ABSTRACT

LLOYD, MICHELLE ANN. Flavor and Stability of Whole Milk Powder. (Under the direction of Dr. MaryAnne Drake.)

Flavor variability and stability of US-produced whole milk powder (WMP) are important parameters for maximizing quality and global competitiveness of this commodity. The US industry standard for shelf-life of whole milk powder (WMP) is 6-9 mo, although previous research has demonstrated flavor changes by 3 mo at ambient storage. Two studies were conducted using sensory and instrumental techniques to (1) characterize flavor and flavor stability of domestic WMP and (2) evaluate the influence of packaging atmosphere, storage temperature, and storage time on WMP shelf-life. Descriptive analysis was conducted using a 10-member trained panel. Selected volatiles were quantified using solid phase microextraction (SPME) with gas chromatography/mass-spectrometry: dimethyl sulfide, 2-methylbutanal, 3-methylbutanal, hexanal, 2-heptanone, heptanal, 1-octen-3-ol, octanal, 3-octen-2-one, and nonanal. For the first study, freshly produced (<1 mo) WMP was collected from 4 US production facilities 5 times over a 1 y period. Each sample was analyzed initially and every 2 mo for sensory profile, volatiles, color, water activity, and moisture through 12 mo storage. Multiple linear regression with backwards elimination was applied to generate equations to predict grassy and painty flavors based on selected volatiles. All WMP were between 2-3% moisture and 0.11-0.25 water activity initially. WMP varied in initial flavor profiles with varying levels of cooked, milkfat, and sweet aromatic flavors. During storage, grassy and painty flavors developed, while sweet aromatic flavor intensities decreased. WMP developed painty flavor between 4 and 6 mo. Painty and grassy flavors were confirmed by increased levels ($p<0.05$) of lipid oxidation products such as hexanal, heptanal, and octanal. Hexanal, 2-heptanone, 1-octen-3-ol and nonanal concentrations were the best predictors of grassy flavor ($r=0.62$, $R^2=0.38$, $p<0.0001$) while hexanal, 2-methylbutanal, 3-methylbutanal, octanal, and 3-octen-2-one concentrations were the best predictors of painty flavor ($r=0.78$, $R^2=0.61$, $p<0.0001$). For the second study, two batches of WMP were repackaged in plastic laminate pouches with air or nitrogen and stored at 2 ˚C or 23 ˚C for one y. In addition to sensory and volatile compound analyses, consumer acceptance testing (n=75) was conducted every 3 mo with reconstituted WMP and white and milk chocolate made from each treatment. Data were analyzed using analysis of variance with Fisher's LSD, Pearson's correlation analysis, and principal component analysis, with significance at $p<0.05$. Air stored WMP had higher peroxide values, lipid oxidation volatiles and grassy and painty flavors than nitrogen flushed WMP. Storage temperature did not impact levels of straight chain lipid oxidation volatiles; 23 ˚C storage resulted in higher cooked and milkfat flavors and lower levels of grassy flavor compared with 2 ˚C storage. Consumer acceptance was negatively correlated with lipid oxidation volatiles and painty flavor. Nitrogen flushing prevented the development of painty flavor in WMP stored up to 1 y at either temperature, resulting in chocolate with high consumer acceptance. Nitrogen flushing can be applied to extend the shelf life of WMP; refrigerated storage also plays a role, but to a lesser extent. These results provide baseline information to determine specific factors that can be controlled to optimize US WMP flavor and flavor stability.

Flavor and Stability of Whole Milk Powder

by
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DEDICATION

This book is dedicated to my family and friends who encouraged and supported me in all of my educational pursuits.

BIOGRAPHY

Michelle Lloyd was born at Ellsworth Air Force Base (AFB), South Dakota. She was the first of 10 children born to Bruce and Esther Lloyd. Her family moved a lot while she was growing up, so she had the opportunity to live in the following places: Orem, UT; Grand Forks AFB, ND; Bennion, UT; Hanscom AFB, MA; Kearns, UT and Layton, UT, where her family currently resides.

She discovered the major of food science when filling out her college application and has enjoyed it ever since. She received a BS and MS degree in food science from Brigham Young University (BYU) in Provo, UT. As an undergraduate student, she worked part-time in the BYU Food Quality Assurance Lab, testing the quality and safety of canned food produced by LDS Welfare Services. Upon graduation, she became the supervisor of the lab and worked full-time in this position while earning her master's degree. Her thesis was on the sensory and nutritional quality of nonfat dry milk in long term storage. After receiving her MS degree, she spent a year and a half at BYU mentoring undergraduate students in food storage research before beginning a PhD at NC State under the direction of Dr. MaryAnne Drake. During her time at NC State, she has been an active member of the Food Science Club, serving in the following positions: secretary, historian, publicity committee, outreach committee, website co-chair, and college bowl team member (with a 2nd place win at the Annual Meeting of the Institute of Food Technologists in New Orleans in 2008).

In her spare time, she enjoys preserving memories through taking pictures, scrapbooking, editing home movies, and compiling information about her ancestors. She also enjoys vegetable gardening, cooking, and sharing her food with others. Anyone she associates with will quickly learn that she is passionate about food science and loves to conduct experiments and taste tests, even in her own kitchen. She hopes to use her talents and skills to make the world a better place to live.

Her future plans include returning to Brigham Young University as a faculty member in the Department of Nutrition, Dietetics and Food Science where she will teach food chemistry and food analysis classes and conduct research related to food flavor, stability, and shelf-life.

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    - WMP suppliers
    - California Dairies, Inc. (for their assistance with proximate analyses)
    - The Hershey Company

Finally, thank you to King Benjamin, an exemplary leader who lived over 2100 years ago (and those who preserved his words so that we can have them today). His inspiring words have been my motto throughout my PhD program: "And see that all these things are done in wisdom and order; for it is not requisite that a man should run faster than he has strength. And again, it is expedient that he should be diligent, that thereby he might win the prize; therefore, all things must be done in order" (Book of Mormon, Mosiah 4:27).

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

Chapter 1. Whole Milk Powder Literature Review

Chapter 2. Flavor Variability and Stability of Whole Milk Powder

Chapter 3. Effect of Nitrogen Flushing and Storage Temperature on Flavor and Stability of Whole Milk Powder

Whole Milk Powder Literature Review

Production and Processing

Production Statistics

Production of dried milk products can be traced back to ancient times when milk was dried in the sun (Caric and Kalab, 1987). The development of industrial production equipment and techniques for dried milk began in the early 1800's and became commercially available in the late 1800's (Caric and Kalab, 1987). Since that time, the milk powder production industry has grown significantly, with production of both whole milk powder (WMP) and skim milk powder (SMP). Worldwide production of WMP in 2006 was approximately 3.5 million metric tons, with the European Union, China, New Zealand and Brazil and Argentina ranked as the top producing countries, and the United States ranked 10th (USDA-FAS, 2008). In 2006, the United States produced approximately 14 thousand metric tons of WMP and 656 thousand metric tons of SMP, accounting for nearly 10% of worldwide milk powder production (USDA-FAS, 2008).

Standard of Identity

WMP, which is also known as full cream milk powder, is properly labeled "dry whole milk" by US standard of identity, and can be made from removing the water from fluid whole milk, or by making a powder which reconstitutes to the same specifications as fluid whole milk, such as mixing skim milk powder with anhydrous milkfat or spray drying with a stream of cream along with a stream of skim milk (CFR, 2008; Kelly, 2006). The final product must have a fat content between 26% and 40% by weight, and a moisture content less than 5% on a milk solids not fat basis (CFR, 2008). Vitamins A and/or D may be added, along with any necessary carriers, as long as they reconstitute to 2000 IU/qt and 400 IU/qt, respectively (CFR, 2008). Other optional ingredients include emulsifiers, stabilizers, anticaking agents, antioxidants, and flavorings (CFR, 2008). Most WMP in the US does not contain any optional ingredients, but lecithin is sometimes used to make agglomerated, or instantized WMP, which is often used for infant formulas (Dan Meyer, American Dairy Products Institute, personal communication, 2008). According to Codex Alimentarius, which sets recommended standards for food on an international basis, WMP must meet the following criteria: 26-42% fat, <5% moisture, and 34% protein on a solids not fat basis (CODEX, 1999). Other standards include approved additives, proper hygiene, limits on heavy metals and pesticide residues, and labeling requirements (CODEX, 1999).

Composition

Typically, WMP has a composition of 24.5-27% protein, 36-38.5% lactose, 26-28.5% fat, 5.5-6.5% ash and 2.0-4.5% moisture, as shown in **Figure 1** (Chandan, 1997; USDEC, 2005). In contrast, SMP contains higher proportions of protein (34-37%), lactose (49.5-52%), and ash (8.2-8.6%), due to a much lower fat content (0.6-

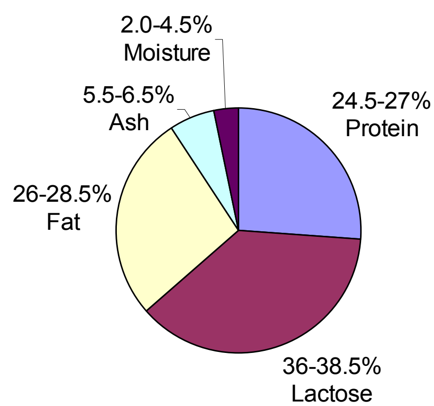

Figure 1. Proximate composition of whole milk powder.

1.25%), but moisture content is similar to WMP (USDEC, 2005). In WMP, approximately 66% of the milkfat is saturated fat, with 31% monounsaturated fat and nearly 3% polyunsaturated fat (**Table 1**) (USDA-ARS, 2007). The composition of milkfat can vary depending on the feed of the cows, especially with regard to the amount and type of fat in the diet (Palmquist et al., 1993). Diets higher in unsaturated fat result in milkfat with higher levels of C18 fatty acids, which decrease the stability of the milkfat (Palmquist et al., 1993). Kim et al. (2002) determined that the surface composition of a 29% fat WMP was 98% fat and 2% lactose, with negligible protein.

Table 1. Lipid composition of whole milk powder[a]

Fatty acids	g per 100 g
Total saturated	16.7
4:0	0.87
6:0	0.24
8:0	0.27
10:0	0.60
12:0	0.61
14:0	2.82
16:0	7.52
18:0	2.85
Total monounsaturated	7.92
16:1	1.20
18:1	6.19
Total polyunsaturated	0.67
18:2	0.46
18:3	0.20

[a]From USDA-ARS, 2007

Quality Parameters

Grading. In the United States, WMP may be graded on a voluntary basis to facilitate marketing (USDA-AMS, 2001). There are two grades for WMP: US Extra Grade and US Standard Grade. Grades are based on the following criteria: flavor, physical appearance, microbial counts, milkfat, moisture, scorched particles, solubility index, and titratable acidity (USDA-AMS, 2001). A summary of the grading criteria is found in **Table 2**. The details for many of these test procedures are described elsewhere (ADPI, 2002; Wehr and Frank, 2004). Other tests used to evaluate WMP quality include free fat (FF), peroxide value (PV), thiobarbituric acid (TBA), hydroxymethylfurfural (HMF), sulfhydryl group concentration, whey protein nitrogen index (WPN), bulk density, dispersibility, and mineral content.

International Standards. Several recommended quality parameters on an international basis include a titratable acidity of no more than 0.18% lactic acid on a solids not fat basis, scorched particles no higher than disk B, solubility index not more than 1.0 ml (CODEX, 1999).

Table 2. Summary[a] of criteria for grading whole milk powder in the United States

	US Extra Grade	US Standard Grade
Flavor	Definite cooked and a slight feed flavor	Definite cooked and feed flavors as well as slight bitter, oxidized, scorched, stale, and storage flavors
Physical appearance	Typical white to light cream color; may have lumps that break with slight pressure; mostly free of dark particles; free of graininess when reconstituted	May have slightly unnatural color; lumps that break with moderate pressure; "reasonably" free of dark particles; "reasonably" free of graininess when reconstituted
Standard plate count	≤10,000 per gram	≤50,000 per gram
Coliform count	≤10 per gram	≤10 per gram
Milkfat	≥26.0 and <40.0%	≥26.0 and <40.0%
Moisture	≤4.5%	<5.0%
Scorched particles	≤15 mg for spray dried; ≤22.5 mg for roller dried	≤22.5 mg for spray dried; ≤32.5 mg for roller dried
Solubility index	Up to 1.0 ml sediment	Up to 1.5 ml sediment
Titratable acidity	Up to 0.15% lactic acid	Up to 0.17% lactic acid

[a]Summarized from USDA-AMS, 2001

Flavor. The flavor of WMP should be "sweet, pleasing, and desirable" (USDA-AMS, 2001). To be considered U.S. Extra Grade, the WMP may have a definite cooked and a slight feed flavor, with no other off-flavors present; U.S. Standard Grade may have definite cooked and feed flavors as well as slight bitter, oxidized, scorched, stale, and storage flavors (USDA-AMS, 2001). These definitions are vague, and attempts have been made to develop more clear standards for flavor using descriptive analysis (Hough et al., 1992; Drake et al., 2003). Hough et al. (1992) developed a methodology to create specific flavor defects in milk and determined threshold concentrations for these defects. Drake et al. (2003) developed a dried dairy ingredient lexicon for use with descriptive analysis of dairy products, including WMP, which will be discussed later.

Appearance. The appearance of WMP should be a typical light cream color, with minimal lumps (**Figure 2**), dark particles, or graininess (**Figure 3**) (USDA-AMS, 2001). Hough et al. (2002) studied WMP with increasing levels of caramel color, along with off flavors, and determined that flavor was more important to consumer acceptance than appearance. The color of WMP can be measured with a colorimeter or spectrophotometer (Clydesdale, 1978). Color values of WMP can vary based on the season and/or diet of the cows (Buma et al., 1977; Grigioni et al., 2007). Changes also occur during storage through Maillard browning, although minimal changes occur at lower (<0.2) water activities (Lim et al., 1994a; Nielsen et al., 1997a; Stapelfeldt et al., 1997a) and <5% moisture (Renner 1988).

Figure 2. Lumps in whole milk powder.

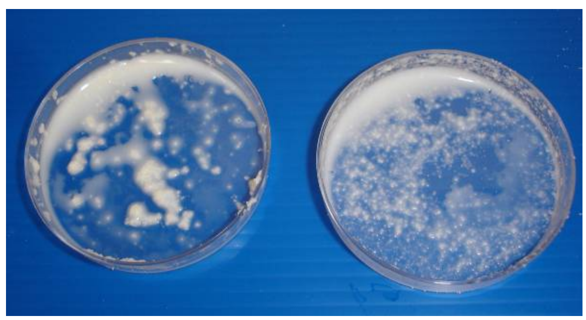

Figure 3. Graininess in whole milk powder after reconstitution.

Bacterial quality. For bacterial quality of WMP, the standard plate count should be <10,000 CFU/g for extra grade and <50,000 CFU/g for standard grade, with coliforms less than 10 CFU/g for either grade (USDA-AMS, 2001). Other researchers have observed ranges of 1,100-14,000 CFU/g for standard plate counts and <10 CFU/g for coliforms (Steen, 1977; Van Mil and Jans, 1991). Standard plate counts typically decrease during storage (Celestino et al., 1997; Steen, 1977). Ronimus et al (2006) observed bacillus at in milk powder that had been stored for over 90 years in a hut in Antarctica.

Titratable acidity. Titratable acidity is determined by titrating the reconstituted WMP with sodium hydroxide to determine the amount of lactic acid present (USDEC, 2005). Titratable acidity should not be more than 0.15% for extra grade and 0.17% for standard grade (USDA-AMS, 2001). Significant positive correlation has been found between psychrotroph counts of raw milk and titratable acidity of WMP (de Oliveira et al., 2000).

Milkfat. Milkfat is typically measured by the Roese-Gottlieb Method, where the fat is extracted with diethyl and petroleum ether, and the fat content is measured gravimetrically after evaporation of the solvent (ADPI, 2002). For both grades of WMP, the milkfat must be between 26.0 and 40.0%, as stated by the US standard of identity (CFR, 2008), but may be up to 42% milkfat according to international standards (CODEX, 1999).

Free fat. Free fat is the fat that can be easily extracted with organic solvent, typically pentane (Walstra et al., 1999; Ziegler and Langiotti, 2003). Kim et al. (2003)

used petroleum ether, following the method of (Buma, 1971a). Free fat includes the fat that is on the surface of the WMP or within cracks (Walstra et al., 1999). The free fat in spray dried WMP is typically around 2% to 3%, while roller-dried WMP has free fat in the range of 60% to 90% (Liang and Hartel, 2004). High levels of free fat in WMP have been associated with greater susceptibility to oxidation (Koc et al., 2003; Vignolles et al., 2007), but other research contradicts this conclusion. Buma (1971a, b, c, d, e, f, g, h, i, j) performed extensive research on free fat in spray dried WMP and did not find any correlation between the development of oxidized flavor and free fat of 1.1%-79% in WMP stored at 30 ºC for 6 mo, although the flavor of the larger particles (containing less free fat) was slightly better than that of smaller particles (containing a larger proportion of free fat). Higher amounts of free fat in WMP decrease its wettability (Kim et al., 2002), which could be an important factor to consider in an industrial setting. Free fat is important when WMP is used in the production of chocolate, because it reduces the amount of cocoa butter, which is a more expensive ingredient, that must be added to produce the desired rheological properties of the chocolate (Chandan, 1997).

Moisture. Moisture is often measured by vacuum or convection oven, but may also be measured by titration (USDEC, 2005). The moisture of WMP must not be more then 4.5% for extra grade and 5% for standard grade, on a milk solids not fat basis (USDA-AMS, 2001), which corresponds to a water activity of around 0.3 (Vuataz, 2002). WMP is typically manufactured to around 2-3% moisture (Van Mil and Jans 1991; Steen 1997), which corresponds to a water activity around 0.2 (Jouppila and Roos, 1994, Thomsen et al., 2005b). Above a water activity of 0.4, lactose is predominately found in the crystallized form and deterioration reactions are more prevalent, including Maillard browning (Jouppila and Roos, 1994; Thomsen et al., 2005b). Van Mil and Jans (1991) concluded that WMP should have a moisture content near 3% to provide optimal stability during storage with regard to flavor and peroxide value.

Scorched particles. Scorched particles are undesirable in WMP; they develop when WMP particles get held up somewhere in the drying process and receive extra heating, causing them to turn a golden brown color (Celestino et al., 1997). For spray dried WMP, the scorched particle content is measured by filtering reconstituted WMP (32.5 g in 250 ml of water) through a cotton disc, allowing it to dry, and comparing it to a photograph of standard discs corresponding to the following amounts of scorched particles: Disc A, 7.5 mg; Disc B, 15.0 mg; Disc C, 22.5 mg; Disc D, 32.5 mg (ADPI, 2002; USDA-AMS, 2001). For extra grade, the scorched particle content should not be more than Disc B if spray dried and Disc C if roller dried. For standard grade, the scorched particle content should not be more than Disc C if spray dried and Disc D if roller dried. A score of B or better is recommended for WMP that will be used for direct reconstitution (Sanderson, 1979 as cited by Celestino et al., 1997). Celestino et al. (1997) observed that WMP processed in the spring and summer were comparable to Disc A for scorched particles, while WMP processed in the fall had a Disc B rating. They gave several possibilities for this difference, including variation in spray dryer operation parameters, higher protein content, and higher calcium content. However, Steen (1977) also measured scorched particles in WMP produced across all seasons at 2 facilities, and did not observe any seasonal effect; 23 out of 24 samples possessed Disc A ratings, with

1 sample out of 6 produced in the spring receiving a B rating. Thus, scorched particle content may be more related to operation of the drier than season.

Solubility index. The solubility index, which is also know as an insolubility index by some methods, is another quality parameter of WMP. It involves reconstituting the WMP under specified conditions, centrifuging in a graduated conical test tube, and measuring the amount of sediment at the bottom. Spray dried WMP should not have more than 1.0 ml of sediment for extra grade and not more than 1.5 ml of sediment for standard grade; roller dried WMP should not have more than 15 ml sediment (USDA-AMS, 2001). A typical solubility index for WMP ranges from 0.14 ml to 1.3 ml (Baldwin and Ackland, 1991; Van Mil and Jans, 1991). Solubility index is affected by processing conditions as well as storage time. Baldwin and Ackland (1991) found that the solubility index of WMP increased as the preheat holding time increased, but did not see any effect due to preheat temperature. They noted an increase in solubility index during storage up to 1 year at 30 ºC. Increases in solubility index during storage have also been observed by other researchers (Celestino et al., 1997; Van Mil and Jans, 1991). Van Mil and Jans (1991) found that the solubility index general increased more during storage in samples of 2.4% and 2.7% moisture than those of 3% moisture. Lloyd et al. (2004) did not observe any definitive increase in sediment for SMP stored up to 29 y in sealed No 10

Figure 4. Solubility index measurement.

cans. Decreases in solubility during storage are related to the Maillard reaction and oxidation (Thomas et al., 2004).

Measures of lipid oxidation. The peroxide value (PV) is a commonly used test to measure primary lipid oxidation products in high fat foods. Nielsen et al. (2003) compared 5 wet-chemistry methods for PV of various foods, including tuna oil, a milk drink (with added oil), rapeseed oil, fish oil, and mayonnaise. All of the methods used were based on the principle that peroxides oxidize iron or iodide, which is further reacted to produce a colored complex that can be measured by titration or spectrophotometer. They selected the method of the International Dairy Federation (IDF) as the most optimal. The IDF method (IDF, 1991) is a standard method for PV of anhydrous milkfat. It is performed by dissolving the milkfat in a chloroform and methanol mixture, followed by addition of ferrous chloride and ammonium thiocyanate to form the red ferric isothiocyanate complex which is measured spectrophotometrically. This method has been applied to WMP following fat extraction, which is typically done with a mixture of chloroform and methanol (Baldwin et al., 1991; Celestino et al., 1997; Liang, 2000; McCluskey et al., 1997b; Newstead and Headifen, 1981; Van Mil and Jans, 1991). The PV procedure should be conducted in the absence of light to prevent false high values (Newstead and Headifen, 1981). PV is expressed as milli-equivalents of oxygen per kg fat. At 2 meq/kg fat, off-flavors may become detectable in WMP (Van Mil and Jans, 1991). Since primary lipid oxidation products break down over time, the peroxide value is not highly correlated with oxidized flavor perception (Baldwin et al., 1991; Pyenson and Tracy, 1946).

The thiobarbituric acid reactive substances (TBARS) assay measures the amount of malondialdehyde, a secondary lipid oxidation product, present in a sample. Some researchers consider TBARS a better measure of oxidation than PV (Chan et al., 1993). TBARS has been found to correlate well with sensory data (Nielsen et al., 1997b; Stapelfeldt et al., 1997b), but not always (Tuohy and Kelly, 1989). Stapelfeldt et al. (1997b) found that TBARS was more highly correlated with sensory data when measured spectrophotometrically at 450 nm rather than the standard 532 nm.

Heat treatment. Upon heating, lactose and free amino groups in milk react in the Maillard reaction (Ellis, 1959). This reaction has been extensively studied in WMP (Hall and Lingnert, 1984; Morales and Jimenez-Perez, 1998; Muir, 1996; Pizzoferrato et al., 1998; Thomas et al., 2004; Thomsen et al., 2005a, b). Maillard reaction products have been shown to possess antioxidant properties (Elias et al., 2008; Hall and Lingnert, 1984). Several tests can be used to indicate the level of heat treatment of WMP: hydroxymethylfurfural (HMF), sulfhydryl group concentration (SH), and whey protein nitrogen index (WPN). Measurement of HMF, a Maillard reaction intermediate, was proposed by Baldwin and Ackland (1991) as an indicator of poor storage conditions because it increases during processing and storage. Sulfhydryl groups formed during processing have been shown to possess antioxidant activity and decrease during storage (Baldwin and Ackland, 1991). WPN measures the amount of undenatured whey protein in a milk powder sample, which varies according to the heat treatment the milk received before drying. It is expressed as mg undenatured whey protein nitrogen per g powder. Lower values indicate a more extreme heat treatment of the fluid milk before spray drying than higher numbers. WPN is typically measured to classify skim milk powder as low, medium or high heat. **Table 3** lists the parameters for each SMP category (Sørensen et al., 1978; USDEC, 2005).

Table 3. Criteria for classification of milk powders

Powder Classification	**WPN (mg undenatured whey protein /g powder)**	**Typical Heat Treatment**
Low heat	≥ 6	72-75°C for 15-20 s
Medium heat	1.51-5.99	85-105°C for 1-2 min
High heat	≤ 1.5	125-135°C for 2-6 min

From (Sørensen et al., 1978; USDEC, 2005)

WPN has also been measured in WMP (Baldwin and Ackland, 1991; Hols and Van Mil, 1991; Ipsen and Hansen, 1988). An equation has been developed to estimate WPN in skim milk powder based on temperature and holding time (Baucke and Newstead, 1972) which can also be applied to WMP (Baldwin and Ackland, 1991). When using WPN with WMP, a correction needs to be made for fat content (Hols and Van Mil, 1991). This can be done by reconstituting the WMP to the same non-fat-solids as the skim milk powder and expressing the results as mg undentured whey protein nitrogen per g skim milk powder equivalent (Sørensen et al., 1978). WPN is a good indicator of the original heat treatment used in the manufacture of WMP because it does not change much during storage (Baldwin and Ackland, 1991).

The original method for WPN was developed by Harland and Ashworth (1947). A modification of their method was published by Kuramoto et al. (1959) which has become the standard procedure recommended by Sørensen et al. (1978) and ADPI (2002). This method involves reconstituting the milk powder and precipitating the casein and the denatured whey protein with sodium chloride, followed by filtration. The filtrate contains all of the undenatured whey protein, which is denatured with hydrochloric acid, forming a turbid solution. The turbidity is measured with a spectrophotometer as a measure of the amount of protein present. An alternative method suggested by Sanderson (1970) uses amido black dye to bind the protein rather than acid precipitation and was shown to be more reliable than the turbidity method. Other potential methods to measure heat treatment of milk powders have also been compared with WPN, including fast protein liquid chromatography (Manji and Kakuda, 1987) and reactive sulfhydryl groups (Anema and Lloyd, 1999).

Bulk density. Bulk density refers to the volume of a specific mass of milk powder and is often measured by placing a sample of milk powder in a graduated cylinder and tapping it by hand or with an instrument until the volume no longer changes (Walstra et al., 1999). Nijdam and Langrish (2005) did not find this technique very useful for WMP because due to its high fat content, the powder was sticky and formed clumps in the tapping process. Often a high bulk density and a small particle size is desired for shipping because it conserves space. In contrast, a lower bulk density with larger particle size is often desired for beverage application and direct reconstitution, as in the case of agglomerated powders. Agglomerated powders take up more space and may not transport as readily because stacking large bags may result in destruction of the agglomerated particles, but these products have the benefit of dissolving more easily in water. The bulk density may be expressed as g per 100 ml; its inverse is bulk volume, would be expressed as ml per 100 g (Westergaard, 2005).

The particle size can affect flowability and cohesion of WMP, which relate to how the dry powder moves through equipment, such as a hopper (Fitzpatrick et al., 2004). Fitzpatrick et al. (2004) found that WMP with a finer particle size was more cohesive, but increasing free fat had no effect. Flowability may be measured by the time it takes for a powder to flow through a funnel, although more sophisticated techniques have been developed (Westergaard, 2005).

Dispersibility. Dispersibility measures how well instantized (agglomerated) WMP goes into solution (USDEC, 2004). The International Dairy Federation (IDF) has a standard method to determine dispersibility and wettability of instantized milk powders (IDF, 1979). The method involves dissolving milk powder in water and filtering it through a sieve. The dispersibility is calculated based on the solids content of the filtrate, the mass of the powder sample, and the water content of the powder. A similar property is wetting time, which is the amount of time for instantized WMP to become wetted when placed on the surface of a beaker of water (IDF, 1979). Kim et al. (2005) suggested that the wettability of WMP was related to the melting point of the fat on the surface of the powder and that a water temperature above this melting point drastically reduced the wetting time. Wettability may also be improved by coating the powder surface with a surfactant such as lecithin (Kim et al., 2005).

Mineral content. The mineral content of WMP can be measured using inductively coupled plasma-optical emission spectroscopy (ICP-OES). Transitional metals such as copper have been analyzed by a number of researchers due to concerns that they promote lipid oxidation (Havemose et al., 2006). Both iron and copper are the main minerals that have been associated with oxidized flavors (Greenbank 1948). Steen (1977) observed that copper content of WMP was higher during the winter (around 0.7 mg/kg) months than the summer (0.4 mg/g), with no seasonal variation observed for iron or zinc.

Processing Methods

Overview. WMP is made from raw whole milk through the following processing steps: (1) standardization (2) heat treatment (3) evaporation (4) homogenization (5) and drying, as described by USDEC (2005) and Walstra et al. (1999). Since raw whole milk has a variable fat content (Palmquist et al., 1993), the milk is first separated into cream and skim milk and then recombined in specific proportions to reach a standard fat content. Second, the milk is heat treated to pasteurize it, to inactivate enzymes such as lipase, and to increase the abundance of sulfhydryl groups, which stabilize the WMP against oxidation (Baldwin et al., 1991; Contarini et al., 1997; Van Mil and Jans, 1991). Third, the milk is evaporated to 40-50% solids to make the drying process much more efficient. Fourth, the milk is homogenized. This step is optional, depending on the desired product. It is useful because is breaks up the fat into small globules that are more stable against oxidation. If the milk is not homogenized, the resulting powder will have a greater free fat content. The final step is drying, which can be accomplished by spray drying or roller drying.

Spray drying. Spray drying involves spraying the concentrated milk into tiny droplets in a heated chamber. The small mist of droplets is generated most commonly by either a pressure atomizer or a centrifugal atomizer (USDEC, 2005). The water evaporates from the small droplets and the milk powder is created. The fine powder formed has a small particle size, which varies with the operating parameters of the spray dryer (Birchal et al., 2005; Caric and Kalab, 1987). If it is a one stage process, the WMP achieves its final moisture content inside the spray dryer (Westergaard, 2005). Most WMP is produced with a two stage process, where the WMP moves from the spray drier to a vibrating fluidized bed and is exposed to additional heated air to complete the drying process (USDEC, 2005; Westergaard, 2005).

Agglomeration. Sometimes the WMP is agglomerated, or instantized, to make it easier to disperse in water. The process makes the particles less dense by incorporation of air. Typically, a small amount (0.1-0.3%) of lecithin is also added during the final stage of spray drying as a wetting agent to facilitate reconstitution (USDEC, 2005; Westergaard, 2005). There are two main agglomeration techniques: "straight through" or "single pass" and "re-wet" agglomeration (USDEC, 2005). The single pass process allows the WMP to be agglomerated during the initial drying process, while the re-wet process uses previously dried WMP that is then re-wetted to around 5-10% moisture, agglomerated, and dried (USDEC, 2005). Agglomerated WMP is typically used in infant formula (Dan Meyer, American Dairy Products Institute, personal communication, 2008).

Roller drying. Roller drying is done by bringing milk into contact with heated cylindrical drums and scraping the dried milk from the drum once in has dried (USDEC, 2005; Westergaard, 2005). Roller drying has become an antiquated process for WMP (Kelly, 2006), with only a couple of plants in the US that are capable of making this product (Dan Meyer, American Dairy Products Institute, personal communication, 2008).

Most, if not all powdered milk is currently spray dried because roller drying is not as consistent and produces milk powder that has more scorched particles and is less soluble (USDEC, 2005; Walstra et al., 1999). Roller dried milk powder has traditionally been preferred in making chocolate, but spray drying has become the preferred drying method in the dairy industry for economic reasons (Keogh et al., 2004).

Processing variables. During processing, there are also a host of factors that influence the quality of WMP produced, including, milk preheat temperature, adherence to good manufacturing practices, and spray dryer operating variables. The preheat temperature of the milk has a major impact on the quality of the resulting WMP (Baldwin and Ackland, 1991; Baldwin et al., 1991; Christensen et al., 1951; Decker et al., 1951; Deruyck, 1991; Oldfield et al., 2000; Stapelfeldt et al., 1997a). The widely used heat treatment for milk being made into WMP is 85-95°C for several minutes (Varnam and Sutherland, 1994), which would result in an undenatured whey protein nitrogen (WPN) value of less than 4.0 mg /g powder (Baldwin and Ackland, 1991). Nanua et al. (2000) used heat treatments of 74°C for 20 s for low heat WMP and 88°C for 15 min for high heat WMP. Ulberth and Roubicek (1995) used 90°C for 60 s in their WMP research. Baldwin and Ackland (1991) recommended a high (125°C) preheat temperature and a short holding time (20 s) for good WMP storage stability. McCluskey et al. (1997b) found good oxidative stability in WMP preheated to 120°C for 20 min, although higher amounts of cholesterol oxidation products were formed in the process. Baldwin et al. (1991) concluded that a heat treatment of 95°C for 20 s would give WMP a shelf life of 9 months at 30 ºC. Recently, Birchal et al. (2005) determined optimal settings for several spray drying variables (emulsion feed flow rate of 1.4 kg/h, atomization flow rate of 50,000 rpm, and inlet air temperature or 160 ºC) to produce agglomerated WMP with the best reconstitution quality. This research was conducted on a pilot scale spray dryer, so the results may not directly apply to an industrial setting with full scale equipment. A spray-drying process has been developed that can produce WMP with around 90% free fat (Koc et al., 2003). Faldt and Sjoholm (1996) found that WMP produced in a pilot-scale spray dryer had a higher free fat content then WMP from large scale equipment. They attributed this to the smaller particle size produced by the pilot-scale dryer, resulting in a greater surface area of fat available for extraction.

Projected Shelf Life

Overview. Some factors that influence shelf-life of WMP include initial milk quality, processing variables, moisture content, packaging, oxygen exposure, exposure to light, and storage temperature (Hough et al., 2002; Lloyd et al., 2004; Nielsen et al., 1997b; Ruckold et al., 2003). The shelf-life of WMP is closely tied to the development of off-flavors, especially those attributed to lipid oxidation. US WMP is reported to have a shelf-life of 6-9 months when stored at temperatures less than 27°C and relative humidity less than 65% (USDEC, 2005), while international WMP claims a shelf life of 12 mo (S. Hess, Hershey Foods, personal communication, 2008). Values in the literature for shelf life of WMP range from 3 months (Carunchia Whetstine and Drake, 2007) to 3 years (Van Mil and Jans, 1991) (**Table 4**). Baldwin et al. (1991) detected oxidized flavor in WMP stored in air at 35°C after 9-12 months. Van Mil and Jans (1991) reported that

WMP processed and stored under optimal conditions could be kept for 3 years without quality loss. Optimal conditions included: (1) moderate heat treatment, (2) moisture content at least 2.8%, (3) storage temperature of 20°C or less, and (4) air and moisture proof oxygen reduced packaging. Since WMP has a high fat content, it is very susceptible to lipid oxidation. The shelf life can be extended by oxygen free packaging, cool and dry storage conditions, or the use of antioxidants.

Table 4. Published values for shelf-life of whole milk powder.

Shelf-Life	Conditions	Source
3 months	21°C and 50% relative humidity	Carunchia Whetstine and Drake, 2007
6-9 months	<27°C and <65% relative humidity	USDEC, 2005
9-12 months	35°C in air, moderate heat treat (>95°C, 20 s)	Baldwin et al., 1991
3 years	Optimal conditions: (1) moderate heat treatment (>95°C, 2 min) (2) moisture content at least 2.8% (3) storage temperature of 20°C or less (4) air and moisture proof reduced O2 packaging	Van Mil and Jans, 1991

Packaging. Several studies have focused on packaging of milk powders (Chan et al., 1993; Lim et al., 1994a, b; Min et al., 1989; Tuohy, 1984a, b; Tuohy et al., 1981). Rehman et al. (2003) compared plastic bags to paper bags for storage of SMP at 37°C and 90% relative humidity for up to 135 days. In both packages, moisture and water activity increased, while dispersibility and pH decreased, and titratable acidity and bulk density remained mostly unchanged. Flowability decreased with both packages, but more so in the paper packages. Overall, they concluded that one type of package was not better than the other.

Lim et al. (1994a) studied WMP stored at ambient temperature, 20°C and 40 °C for 4 months in the following types of packaging: Kraft paper/polyethylene, Kraft paper/ nylon-polyethylene laminate (nitrogen flushed or vacuum packed), and aluminum-polyethylene laminate (nitrogen flushed or vacuum packed). At ambient temperature and 20°C, there were no significant differences between the packaging treatments, but at 40°C the aluminum-polyethylene laminate performed better than the others to limit changes in PV, TBARS, solubility index and L* a* b* values (with changes occurring in a* values). The Kraft paper/ nylon-polyethylene laminate also performed well, while the Kraft paper/polyethylene packaging did not; it showed the most physicochemical changes. Lim et al. (1994b) published a second study estimating the shelf life of the WMP in the various packaging treatments based on sensory and hydroxymethylfurfural (a Maillard reaction intermediate) data. The results, shown in **Table 5**, ranged from 3.5 months to over 2 years, depending on the packaging and temperature of storage.

Table 5. Estimated shelf-life of WMP in various packaging and temperatures[a].

Packaging	Additional Treatment	Storage Temperature (°C)	Projected shelf-life (months)
Kraft paper/polyethylene	None	20	27.1
		30	10.8
		40	4.3
Kraft paper/ nylon-polyethylene laminate	Nitrogen flush	20	39.8
		30	19.1
		40	9.2
Kraft paper/ nylon-polyethylene laminate	Vacuum packaged	20	17.7
		30	10.5
		40	6.3
Aluminum-polyethylene laminate	Nitrogen flush	20	26.2
		30	13.0
		40	6.5
Aluminum-polyethylene laminate	Vacuum packaged	20	20.6
		30	8.5
		40	3.5

[a] From Lim et al., 1994b

Numerous storage studies have shown that WMP and cream powder flavor is better preserved under low oxygen conditions (Andersson and Lingnert, 1998; Coulter, 1947; Hall and Andersson, 1985; Steen, 1977; Van Mil and Jans, 1991). Van Mil and Jans (1991) concluded that WMP should be made with a moderate heat treatment and a moisture content of 2.8-5%, then stored at less than 20°C with lowered oxygen levels. Some research has shown slight benefits from added antioxidants in WMP (Abbot and Waite, 1962, 1965; Decker and Ashworth, 1951; Findlay and Smith, 1945; Hall and Andersson, 1985), but this is not a common commercial practice (Dan Meyer, American Dairy Products Institute, personal communication, 2008).

Ingredient Applications

WMP is a versatile food ingredient that provides calcium and high quality protein (USDEC, 2005). In the US, over 75% of the WMP produced goes into confectionary products such as chocolate, with the remainder used in baked goods, dry sauce and soup mixes, dairy beverages, ice cream, recombined milk, or other uses (Chandan, 1997; USDEC, 2005). If the WMP used in these applications has off flavors, these flavors may carry through into the final product and result in consumer complaints. Off-flavor carry-through was reported by Caudle et al. (2005) for SMP used in yogurt, hot cocoa, and white chocolate. Wright et al. (2008) also reported that off flavors noted in dried whey protein could be detected in protein beverages, resulting in decreased consumer acceptance.

Bolenz et al. (2003) made 16 different milk chocolate samples using a variety of milk ingredients of various free-fat contents, including roller or spray dried WMP, skim milk powder with anhydrous milk fat, or cream powder. All samples were produced to meet legal requirements for milk chocolate. Significant differences were observed between chocolate samples made from different milk ingredients in particle size, viscosity and descriptive sensory attributes. In consumer testing, the parameters that were important to consumer liking included small particle size, more intense milky flavor, and the lack of malty and off-note flavors. Chocolates made with higher free milkfat had a smaller particle size and were better liked by consumers. Liang and Hartel (2004) focused on rheology of chocolate made with various milk powders and observed that the free fat content of the milk powder was the most important property. As free fat increased, the final chocolate product generally had reduced hardness and better bloom inhibition. However, the observed instrumental differences did not affect consumer acceptability, as none of the samples scored significantly different in overall acceptability by a consumer panel. Currently, most chocolate manufacturers use anhydrous milkfat and skim milk powder for economic reasons (Steve Hess, Hershey Foods, personal communication, 2008).

Flavor and Flavor Stability

Flavor

Dairy products with consistent, high quality flavor are essential to maintain consumer loyalty to a brand (Drake, 2004). Milk powders available on the market exhibit wide variation in flavor quality (Drake et al., 2003; Kamath et al., 1999; Lloyd et al., 2004). Drake et al. (2003) evaluated several samples of whole milk powder during the development of a dried dairy ingredients lexicon (standardized descriptive language). Attributes noted in the samples at various intensities included 9 flavors (cooked, caramelized, sweet aromatic, milkfat, cereal, fried/fatty/painty, sweet and salty) and a feeling factor (astringency). During development of the lexicon, one WMP sample also had a fishy flavor that was not detected in any of the other samples. A review of dairy flavors, including WMP, was provided by Nursten (1997). Kinsella et al. (1967) reviewed the flavor of milk fat, and compiled a list of volatile compounds associated with oxidized, cardboard, oily, painty, fishy, grassy, metallic, and other flavors associated with oxidized milkfat, along with threshold values for these compounds in 3.8% milk.

Before the milk arrives at the processing plant, many variables can affect its flavor quality, including the diet of the herd, season of the year, and microbiological quality. The diet that a cow eats will affect the flavor of the milk it produces (Al-Mabruk et al., 2004; Bendall, 2001; Coulon and Priolo, 2002; Croissant et al., 2007; Fearon et al., 1998; Mounchili et al., 2004; Palmquist et al., 1993; Randby et al., 1999; Toso et al., 2002; Urbach, 1990). Several researchers have found that the season of the year affects WMP quality and stability (Baldwin and Ackland, 1991; Baldwin et al., 1991; Biolatto et al., 2007; Negri et al., 2004; Steen, 1977; Van Mil and Jans, 1991). Steen (1977) collected WMP from 2 manufacturing facilities in Denmark over a year period to observe the effect of season on a number of quality attributes, including physical properties, flavor, and storage stability up to 24 mo. The thiobarbituric acid (TBA) value, a measure of lipid oxidation, was higher in the summer, which was consistent with Biolatto and others (2007) who found that straight chain aldehydes were higher in WMP produced during the spring and summer compared to fall and winter. They explained that the variation was likely due to the feed of the cows, which is consistent with Toso and others (2002), who reported higher levels of straight chain aldehydes in raw milk from cows fed grass silage, compared to hay or maize silage. Van Mil and Jans (1991) also found that WMP produced in the spring was less stable and decreased in flavor score more rapidly than WMP produced in other seasons. The microbiological profile of raw milk can also affect the quality of WMP made from it (Celestino et al., 1997; de Oliveira et al., 2000). Ipsen and Hansen (1988) found that high coliform counts in raw milk were detrimental to the flavor of WMP stored for a year.

Storage conditions also have a major impact on flavor of WMP. The two main reactions that generate off-flavors in WMP during storage are lipid oxidation and Maillard browning. Lipid oxidation occurs when oxygen reacts with lipids to form hydroperoxides, which react further to produce aldehydes and ketones, contributing off flavors to the WMP. Lipid oxidation produces tallowy, grassy, metallic, cardboard, fatty/fryer oil, and painty flavors in WMP (Carunchia Whetstine and Drake, 2007; Hall and Lingnert, 1984; Walstra et al., 1999). Control of lipid oxidation is of concern from a quality as well as a safety standpoint, as McCluskey et al. (1997a) found a positive correlation ($p<0.001$) between lipid and cholesterol oxidation products, which have been associated with adverse health effects (Chan et al., 1993).

Hall and Andersson (1985) studied the formation kinetics of volatile compounds in WMP containing various antioxidants and stored in an atmosphere of air or nitrogen for 84 weeks. Many reactions for formation of straight chain aldehydes were zero order (linear); a couple were first order (exponential), with the remainder as mixed models. Formation of other compounds (2-ketones, Strecker aldehydes, alkyl furans, sulfur compounds, and hydrocarbons) best fit a zero order model. The storage atmosphere affected the formation of Strecker aldehydes and alkyl furans, with higher formation rates in air. In general, dimethyl sulfide increased more rapidly in the nitrogen atmosphere. Formation of butane and butene was significantly affected by the storage atmosphere and the addition of antioxidants, but not pentane.

Maillard browning in WMP occurs when a free amino group from the milk protein reacts with lactose. At ambient temperature, Maillard browning occurs in powdered dairy products if the moisture content is greater than or equal to 5% (Renner, 1988). A small amount of Maillard browning may be desirable because some Maillard browning products have antioxidant properties (Benjakul et al., 2005; Calligaris et al., 2004; Yilmaz and Toledo, 2005) and produce desirable aromas and flavor if at low concentrations (Renner, 1988). A large amount of Maillard browning will adversely affect the protein quality as lysine becomes biologically unavailable (Renner, 1988). Van Mil and Jans (1991) observed minimal loss of lysine (<2%) in 2.4-3.0% moisture WMP stored for 3 years at 20 ºC and 10-20% loss for the same WMP stored at 35 ºC.

The Maillard reaction can also adversely affect appearance and flavor. The Maillard reaction produces gluey flavors in WMP (Walstra et al., 1999). Some research suggests that the Maillard reaction is also influenced by lipid oxidation (Hidalgo and Zamora, 2008; Hall et al., 1985; Andersson and Lingnert, 1998). A review on dairy powder stability related to lactose crystallization was compiled by Roos (2002). Lactose in WMP is typically found in the amorphous or glassy state and remains in that state as long as the lactose remains below the temperature of glass transition (T_g); above $T_{g,}$, lactose crystallizes. Amorphous lactose is very hygroscopic and will begin to crystallize at increased temperature and relative humidity (Thomas et al., 2004). For amorphous lactose with a moisture content of 6.8% (corresponding to a relative humidity of 37% and water activity of 0.27), T_g is around room temperature (Roos, 2002). A more detailed discussion of T_g has been given by Thomas et al. (2004). The transition from amorphous to crystalline lactose causes a release of water, resulting in powder caking and Maillard browning. Also, there is increased oxidation because the milkfat is no longer encapsulated by the lactose. Thomsen et al. (2005b) reviewed the effects of Maillard browning on WMP and found that Maillard browning, lactose crystallization and formation of radicals were closely connected. Stapelfeldt et al. (1997a) stored WMP at 3 different water activities (0.1, 0.2, 0.3) and determined that the quality was best maintained between a water activity of 0.1 and 0.2. Samples stored at 45°C and 0.3 water activity showed the most Maillard browning. Both 3-methylbutanal and 2-methylbutanal are flavor compounds produced by the Maillard reaction in milk and WMP, resulting from Strecker degradation of isoleucine and leucine (Bendall, 2001; Carunchia Whetstine and Drake, 2007).

Sensory Analysis

Sensory analysis involves using the human senses to evaluate food properties. Sensory techniques include consumer testing and analytical sensory techniques (Drake, 2007). Consumer testing is used to find out how well a particular product is liked or accepted by the average consumer. Consumer panels typically require large (>50) numbers of people to get statistically valid data (Man and Jones, 2000; Drake, 2007). Descriptive analysis is used to document a complete picture of the sensory properties of a food product from an analytical perspective. Descriptive panels require extensive training of a small group (generally 6-10) of people (Drake, 2007). Trained panels are more frequently used in storage studies and flavor research.

Hough et al. (2002) correlated consumer panel data with trained panel scores for WMP and found that changes in the flavor affected consumer acceptability more than changes in appearance. Baldwin et al. (1991) produced 16 WMP samples with all combinations of 4 preheat temperatures and 4 holding times. This experiment was replicated on 3 occasions to account for variations in season. Samples were stored at 30°C in an air atmosphere and were evaluated by descriptive analysis every 3 months over an 18-month period. Aroma descriptors included cooked, feedy/cabbagey, and oxidized. Flavor descriptors included cooked, creamy, oxidized, and sweet. Texture descriptors included astringency and body. Samples with higher heat treatments and holding times were more intense in cooked aroma and flavor, and feedy/cabbagey aroma. Oxidized aroma and flavor were minimized by high preheat treatments (125 ºC for 10-240 s). Several attributes increased during the dairying season, including sweetness, creaminess, astringency, and body.

Hough et al. (1992) determined the detection and recognition thresholds for the following flavors in reconstituted WMP: cooked, chalky, feed, flat burnt, bitter, oxidized, lipolysis, salty, acid, and chemical. Andersson and Lingnert (1998) used the following descriptors for odor and flavor of cream powder: total odor, total flavor, boiled, buttery, chemical, cardboard. Drake et al. (2003) developed a lexicon for sensory profiling of dried dairy ingredients, including WMP (**Table 6).**

Table 6. Reference materials for descriptive sensory analysis of dried dairy ingredients

Descriptor	**Reference**	**Preparation**
Cooked/sulfurous	Heated milk	Heat pasteurized skim milk to 85 ºC for 45 min
Caramelized/ butterscotch	Autoclaved milk Caramel syrup	Autoclave whole milk at 121 ºC for 30 min Dilute a tablespoon of caramel syrup in 400mL ski milk
Sweet aromatic/ cake mix	Pillsbury White cake mix Vanillin	Dilute 5mg of vanillin in skim milk
Cereal/grass-like	Breakfast cereals (corn flakes, oat and wheaties)	Soak one cup cereal into three cups milk for 30 min and filter to remove cereals
Barny	*p*-cresol	20 ppm in skim milk
Brothy/potato-like	Kroger canned white potato slices Methional	Remove the sliced potatoes from the broth Several drops of 20 ppm methional in methanol in sniffing jars
Animal/gelatin-like/wet dog	Knox-unflavored gelatin	Dissolve one bag of gelatin (28g) in two cups of distilled water
Milkfat/lactone	Heavy cream Delta dodecalactone	40 ppm on filter paper
Fried fatty/painty	(E,E)-2,4-decadienal	2 ppb in skim milk
Fishy	Fresh fish with skin Canned tuna juice	
Mushroom/metallic	Fresh mushroom	Slice fresh mushroom in skim milk for 30 min and filter to remove mushroom slices
Papery/cardboard	Cardboard paper	Soak pieces of cardboard paper in skim milk overnight
Burnt/charcoal	Over toasted bread slice	
Vitamin/rubber	Enfamil liquid Polyvisol vitamins	
Diacetyl	Diacetyl	Diacetyl, 20 ppm on filter paper
Earthy/musty	Potting soil, odor reminiscent of damp basement	
Sweet taste	Sucrose	5% sucrose solution
Salty	NaCl	2% NaCl solution
Sour	Citric acid	1% citric acid solution
Bitter	Caffeine	0.5% caffeine solution
Umami	Monosodium glutamate	1% MSG in water
Astringent	Tea	Soak 6 tea bags in water for 10 min

From Drake et al. (2003)

Instrumental Analysis

Overview. Instrumental analysis techniques are used to characterize the volatile compounds in a food that affect its flavor. These compounds can be separated, identified, and quantified using gas chromatography (GC). GC analysis became available in the 1950's and became more prevalent in flavor research in the 1960's, which has expanded and improved since that time (Harper, 2007). A number of extraction and concentration techniques have been developed to prepare samples for GC, including direct solvent extraction (DSE) with solvent assisted flavor extraction (SAFE), static and dynamic headspace, supercritical fluid extraction (SFE), and solid-phase microextraction (SPME). Further detail about these and other techniques may be obtained from Mariaca and Bosset (1997) who reviewed instrumental analysis techniques for volatile flavor components in milk and dairy products.

It is important to note that no single extraction technique will obtain all of the volatile compounds in a food. It is wise to use a combination of techniques if possible, or to use a technique that will target a compound of interest. Each extraction technique has advantages and disadvantages. For example, solvent extraction works well for semi-volatile compounds, but is very time consuming and highly volatile compounds may be lost or poorly recovered. SPME is a more rapid extraction technique and can extract highly volatile compounds, but may not extract higher molecular weight volatile compounds. Kataoka et al. (2000) reviewed the use of solid-phase microextraction (SPME) in food analysis. Another instrument to analyze volatile compounds is an electronic nose, which has been reviewed by Ampuero and Bosset (2003) in relation to dairy products.

Once volatile compounds have been extracted from a food, they are injected into a gas chromatograph to separate the component compounds. Once the compounds have been separated, they pass through a detector which gives a response corresponding ideally to the amount of a compound that is present. A commonly used detector for flavor research is a flame ionization detector (FID) because it is widely available, reliable, and inexpensive, with good sensitivity for most organic compounds (10-100 pg), along with a wide linear response range (Reineccius, 2003; Sides 2000). The human nose is also a valuable detector, particularly in flavor chemistry where aroma-active compounds are the compounds of interest. When the human nose is used as a detector, the technique is referred to as GC-olfactometry (Figure 5). A trained and experienced sniffer can identify and determine intensities of aroma active compounds as they elute from the GC column (Friedrich and Acree 1998). Mass spectrometers (MS) are also very useful detectors when combined with gas chromatography (GC-MS), as they can be used both to detect and identify unknown volatiles (Smith and Thakur 2003). An MS functions by creating charged fragments from the volatiles that elute off of the GC column, which are separated by their mass to charge ratio using a mass analyzer such as a quadrupole or ion trap (Smith and Thakur 2003). A quadrupole mass analyzer consist of 4 rods with alternating

Figure 5. Gas chromatography-olfactometry.

currents that allow stable ions to be detected, while unstable ions are pumped away (Smith and Thakur 2003). With an ion trap mass analyzer, all ions are held within the mass analyzer by a series of electrodes and the unstable ions are released to the detector according to their mass to charge ratios (Smith and Thakur 2003).

Three methods to identify volatile compounds include mass spectra, odor properties, and retention indices. Mass spectrometry results in "fingerprint" spectra of ion fragments that can be compared with a database of known spectra to identify the compounds in question. Mass spectral matching of an unknown compound with that of a known standard is considered the gold standard for compound identification. The aroma of a compound is also a potentially unique and identifying attribute. Gas chromatography-olfactometry (GC-O) may be used to characterize and identify aroma-active compounds and then compare the aromas and retention indices (RIs) of those compounds with known standards. The third criterion for compound identification is comparison of RIs with those of known standards. In the absence of mass spectra, identical aroma properties and RI values on more than one column that match with a known standard may be considered robust identification. The reason that RI on more than one column is required (without mass spectra) for compound identification is simply because multiple compounds may co-elute and have the same RI on a single column, but this probability is drastically diminished if RI on more than one column is evaluated. The RI (van Den Dool and Kratz, 1963) adjusts the retention time of a compound relative to the retention times of a standard alkane series. This standardized value allows for better comparison of research conducted in different labs, as retention times will vary depending on the GC conditions used, the columns length, and other factors. RI values for various aroma-active volatiles are available through the NIST library (National Institute of Standards and Technology, Gaithersburg, MD) as well as an online database compiled by Terry Acree & Heinrich Arn: http://www.flavornet.org/flavornet.html.

Relating sensory to instrumental analysis. Some research has performed both sensory and instrumental volatile analyses on WMP (Carunchia Whetstine and Drake, 2007; Hall and Andersson, 1985), which gives a more complete understanding of WMP flavor. Hall and Andersson (1985) performed multiple regression analysis to determine which volatile compounds in reconstituted WMP best contributed to various flavor attributes. A 10-member trained panel rated the following flavor attributes: total intensity, like cooked milk, sweet, creamy, like whey cheese, like casein, like cardboard, musty, sourish, green, like mushroom, and flavor acceptance. Twenty-five volatile compounds were measured quantitatively including straight-chain aldehydes (ethanal, propanal plus 2-propanone, n-butanal, n-pentanal, n-hexanal, n-heptanal, n-nonanal), ketones (2-butanone, 2-pentanone, 2-heptanone, 2-nonanone, 2,3-butandione), Strecker aldehydes (2-methyl propanal, 3-methyl butanal, 2-methyl butanal), furans (2-methyl furan, 2-ethyl furan), sulfur compounds (methyl mercapatan, carbon disulfide, dimethyl sulfide, dimethyl disulfide), and hydrocarbons (n-butane, n-pentane, n-heptane). Of these, the following were selected as having an odor threshold high enough to contribute to the flavor of reconstituted WMP: ethanal, propanal plus 2-propanone, n-butanal, n-pentanal, n-hexanal, n-heptanal, n-nonanal, 2,3-butandione, 2-methyl propanal, 3-methyl butanal, 2-methyl butanal, methyl mercaptan, carbon disulfide, dimethyl sulfide, and dimethyl disulfide.

Carunchia Whetstine and Drake (2007) studied flavor volatiles and sensory characteristics of WMP stored for 24 months using a combination of descriptive sensory analysis and GC-O. They found 65 aroma active volatile compounds in WMP. Many of the volatiles were lipid oxidation products with fatty aromas, which increased during storage. This corresponded well with their descriptive analysis results, which showed an increase in fatty/fryer oil flavor during storage.

Conclusions

Although much research has been conducted on the flavor and stability of WMP, the food industry continues to have quality and flavor problems with WMP. These problems are major issues for food companies who use WMP as a food ingredient. Additional research may hold the answers to producing a more consistent quality WMP with good flavor stability for use in the food industry. The objectives of this dissertation are to (1) survey flavor variability and flavor stability of US WMP stored up to 1 y and to (2) evaluate the influence of nitrogen flushing and storage temperature on flavor and shelf-life of WMP.

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Flavor Variability and Flavor Stability of US-Produced Whole Milk Powder

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Flavor of Whole Milk Powder…

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ABSTRACT:

Flavor variability and stability of US-produced whole milk powder (WMP) are important parameters for maximizing quality and global competitiveness of this commodity. This study characterized flavor and flavor stability of domestic WMP. Freshly produced (<1 mo) WMP was collected from 4 US production facilities 5 times over a 1 y period. Each sample was analyzed initially and every 2 mo for sensory profile, volatiles, color, water activity, and moisture through 12 mo storage. The following volatiles were quantified using solid phase microextraction (SPME) with gas chromatography/mass-spectrometry: dimethyl sulfide, 2-methylbutanal, 3-methylbutanal, hexanal, 2-heptanone, heptanal, 1-octen-3-ol, octanal, 3-octen-2-one, and nonanal. Multiple linear regression with backwards elimination was applied to generate equations to predict grassy and painty flavors based on selected volatiles. All WMP were between 2-3% moisture and 0.11-0.25 water activity initially. WMP varied in initial flavor profiles with varying levels of cooked, milkfat, and sweet aromatic flavors. During storage, grassy and painty flavors developed, while sweet aromatic flavor intensities decreased. At 12 mo storage, all WMP displayed grassy and painty flavors. Painty and grassy flavors were confirmed by increased levels ($p<0.05$) of lipid oxidation products such as hexanal, heptanal, and octanal. Hexanal, 2-heptanone, 1-octen-3-ol and nonanal concentrations were best predictors of grassy flavor ($r=0.62$, $R^2=0.38$, $p<0.0001$) while hexanal, 2-methylbutanal, 3-methylbutanal, octanal, and 3-octen-2-one concentrations were best predictors of painty flavor ($r=0.78$, $R^2=0.61$, $p<0.0001$). These results provide

baseline information to determine specific factors that can be controlled to optimize US WMP flavor and flavor stability.

Key words: dairy, flavor, storage, volatiles, dry whole milk

Introduction

In recent years, demand for dairy products has increased on the world market (Wiley 2007; USDEC 2008). In 2007, over 9.5% of US dairy production (on a total solids basis) was exported, which is a significant increase from the 5% exported in 2002 (USDEC 2007). The US is the top producer of skim milk powder (SMP) worldwide, with 656,000 metric tons produced in 2006, representing approximately 21% of world production; whole milk powder production was 14,000 metric tons in 2006, accounting for less than 1% of world production (USDA-FAS 2008). As of 2006, close to 44% of US-produced SMP and 7% of WMP were exported; current forecasts predict increased production and export of both of these commodities (USDA-FAS 2008). Most (78.1%) WMP in the US goes into confectionary products such as chocolate, with the remainder used in dairy products (8%), dry sauce and soup mixes (6.5%), baked goods (4.8%), retail/home (0.7%), institutions (0.2%), meats (0.2%), soups (0.4%) and animal feed (1.1%) (USDEC 2005).

Milk and WMP flavor and stability can be influenced by animal feed (Croissant and others 2007; Urbach 1990; Toso and others 2002; Havemose and others 2006; Shipe and others 1978), season (Baldwin and others 1991; Biolatto and others 2007; Steen 1977; van Mil and Jans 1991) microbiological quality (de Oliveira and others 2000; Shipe and others 1978), storage time before drying (Celestino and others 1997), heat treatment and other processing conditions (Baldwin and Ackland 1991; Stapelfeldt and others 1997; van Mil and Jans 1991; Schuck 2002; Birchal and others 2005), air pollution (Kurtz and others 1969), and storage conditions, including water activity, moisture, packaging, light and temperature (Stapelfeldt and others 1997; Hall and others 1985; Hall and Andersson 1985; Van Mil and Jans 1991; Baldwin and Ackland 1991). Lipid oxidation in WMP is of concern from a flavor as well as a safety standpoint since lipid oxidation products have been positively correlated with cholesterol oxidation products, which have been linked with negative health effects (McCluskey and others 1997; Chan and others 1993).

The shelf-life of whole milk powder has been reported as anywhere from 3 mo (Carunchia Whetstine and Drake 2007) to 3 y (Van Mil and Jans 1991). According to the US Dairy Export Council (2005), the shelf-life of WMP at <27°C and <65% relative humidity is 6-9 mo. A shelf life of 12 mo or longer has been purported with internationally-produced WMP (Van Mil and Jans 1991; Steve Hess, Hershey Foods, personal communication, 2008). The two main deterioration reactions in WMP include the Maillard reaction and lipid oxidation, but lipid oxidation is the main factor limiting the shelf-life of WMP (Farkye 2006; Renner 1988), as US WMP has a fat content between 26-40% (ADPI 2002) with unsaturated fatty acids representing close to 34% of the fat in WMP (31% monounsaturated and nearly 3 % polyunsaturated fat) (USDA-ARS 2007).

Lipid oxidation produces volatile compounds that can be measured by sensory and instrumental techniques to gain a better understanding of the flavor (Drake 2004, 2007). A trained descriptive panel has been used by a number of researchers to evaluate WMP flavor (Carunchia Whetstine and Drake 2007; Drake and others 2003; Hall and Lingnert 1984; Hall and Andersson 1985; Hough and others 2002). Instrumental techniques used to measure lipid oxidation in WMP include peroxide value (PV) (Baldwin and others 1991; Doka and others 2000; van Mil and Jans 1991; Celestino and others 1997; McCluskey and others 1997; Newstead and Headifen 1981; Ulberth and Roubicek 1995; Tuohy and Kelly 1989), thiobarbituric acid reactive substances (TBARS) (McCluskey and others 1997; Ulberth and Roubicek 1995; Tuohy and Kelly 1989), and volatile analysis by gas chromatography (Ulberth and Roubicek 1995; Carunchia Whetstine and Drake 2007; Hall and others 1985; Hall and Andersson 1985).

Comprehensive storage studies on flavor and storage stability of international WMP have been conducted (Steen 1977; van Mil and Jans 1991; Baldwin and others 1991; Baldwin and Ackland 1991), but quantitative information is lacking on flavor and storage stability of US WMP. Some work was conducted by Carunchia Whetstine and Drake (2007), who tracked the flavor of WMP with a trained panel and volatile changes with gas chromatography-olfactometry of two samples of WMP milk powder from a single U.S. facility over 24 mo storage. Their results indicated that the shelf stability of US WMP was 3 mo or less, significantly less than what other studies reported with internationally produced WMP. Flavor variability and stability of US-produced WMP warrants further research so that manufacturers can identify process methods to maximize WMP quality. The objectives of this study were to characterize flavor variability and stability of US WMP. WMP was collected from four US facilities at five different occasions and stored for 12 mo. Both sensory and instrumental analyses were applied to characterize flavor and flavor variability.

Materials and Methods

Samples

Freshly produced (<1 mo) spray dried WMP was obtained from four US production facilities (2 east coast and 2 west coast) in 5 shipments over a year period (Jan, May, Aug, Nov, and the following Jan) (**Figure 1**). These facilities were selected based on their location and total production capacity (continuous rather than seasonal production). Collectively, these facilities represented more than 50% of the 2005 US WMP supply. At each shipment, two 23-kg bags from different production runs were collected from each facility and shipped by expedited carrier to North Carolina State University. One facility was unable to send samples for one shipment due to lack of production at that time, so the total samples received was 38. Upon receipt, each sample was repackaged into a 19-liter bucket with a tight-fitting lid (M&M Industries, Chattanooga, TN, USA) lined with a Teflon® liner (Welch Fluorocarbon, Dover, NH, USA). Initial analyses included moisture, free fat, fat, undenatured whey protein nitrogen (WPN), mineral content, water activity, color, volatile analysis, and descriptive analysis. Samples were stored at 21 ºC and 45% relative humidity and evaluated at two mo intervals for moisture, water activity, color, volatiles, and flavor. At each timepoint, WMP was stirred, a composite sample (1000 g) was removed, and then the bag was twisted closed and the bucket re-sealed. Where indicated, WMP was reconstituted to 10% solids not fat (SNF) before analysis with odor-free deionized water according to the equation in International Dairy Federation Method 99C (IDF 1997): g whole milk powder in 90 g water = 1000/(100–% fat). WMP were protected from light exposure during storage as well as during preparation for volatile and sensory analysis.

Physical, Chemical and Microbiological Properties

Samples were evaluated in duplicate by an independent lab (Tulare, CA) for moisture, free fat, fat, and WPN. Moisture was measured by the vacuum oven method from DA Instruction 918-RL (USDA-AMS 2001). Free fat was evaluated based on the method of Sørensen and others (1978). WPN, fat, standard plate counts, and coliforms were measured according to standard methods described by Wehr and Frank (2004).

Iron, copper, manganese, and zinc were measured in duplicate by the NCSU Analytical Services Laboratory (Raleigh, NC) using a standard dry ash method with inductively coupled plasma optical emission spectroscopy (ICP-OES). Briefly, 2.5g samples were (1) ashed overnight at 500 ºC in a muffle furnace, (2) cooled, (3) mixed with approximately 1.5 ml distilled water and 4 ml 6N HCl (VWR, West Chester, PA, USA) and heated to dryness on a steam plate to remove silicates, (4) transferred to a 50 ml volumetric flask with approximately 4 ml of 6N HCl, and (5) brought to volume with distilled water for ICP-OES analysis (Lisa Lentz, Analytical Services Lab, North Carolina State University, Personal Communication, 2008).

Water activity was measured in duplicate using an Aqualab series 3 TE water activity meter (Decagon Devices, Pullman, WA, USA). CIE L*a*b* values were obtained in triplicate with a Minolta CR-300 Chroma Meter using light source “C” (Konica Minolta Sensing Americas, Inc., Ramsey, NJ, USA) and a 60 x 15 mm Petri dish

(Becton Dickinson Labware, Franklin Lakes, NJ, USA). Dry as well as reconstituted powders (10% SNF) and liquid were filled to the brim (ca 9mm depth) of the larger diameter Petri dish top; the dry powder was compressed with the smaller diameter bottom portion of the dish. Samples were measured at approximately 22 ºC.

Volatile Analysis

Volatile compounds were analyzed in triplicate using solid phase microextraction (SPME) similar to techniques used by Wright and others (2008) and Vazquez-Landaverde (2005) with a Varian CP-3380 gas chromatograph (GC) equipped with a Saturn 2000 ion trap mass selective detector (MSD) from Varian, Inc. (Palo Alto, CA., USA). Chromatography was performed using a 30 m length x 0.25 mm i.d. x .25 µm df Rtx®-5 ms column (Restek, Bellefonte, PA, USA) with helium carrier gas at a flow rate of 1 ml/min. Oven temperature was ramped from 40°C to 250°C at a rate of 8°C/min with 5 min initial and final hold times. MSD conditions were as follows: 80°C manifold, 120°C transfer line 150°C ion trap. Chemical standards and alkanes were obtained from Sigma Aldrich (St. Louis, MO, USA); methanol was obtained from VWR (West Chester, PA, USA).

WMP was reconstituted to 10% SNF using 90 ml odor-free deionized water. Five µl of internal standard (5 µl E-2-hexenal in 5 ml methanol) were added to reconstituted milk to make a final concentration of 45 (µg/kg). Twenty g of the reconstituted sample with internal standard were added to a 40 ml amber vial with 1.0 g sodium chloride (Fisher Scientific, Pittsburgh, PA, USA) and an 8 mm x 13 mm stir bar (VWR, West Chester, PA, USA). A poly(tetrafluoroethyene) coated septum (Supelco, Bellefont, PA, USA) was placed facing the liquid and screwed into place. A water blank was made by adding internal standard to 90 ml deodorized water and then adding 20 g of this solution to a 40 ml vial as described above. An alkane series was prepared by adding 1 µl of alkane solution (approximately 40 mg/kg C7-C24 in hexane) into an empty 40 ml vial and sealed as described above and used to calculate retention indices (RI) of volatile compounds.

Sample vials were placed in a heating block at 40 ºC for 30 min to equilibrate. A 2.0 cm DVB/Carboxen^{TM}/PDMS Stablefex^{TM} (Supelco, Bellefont, PA, USA) was inserted and exposed at a depth of 2.0 cm in the heating block for an additional 30 min, followed by injection into the GC for 5 min at a depth of 3.0 cm. All fibers were conditioned before use according to manufacturer's directions. Volatile compounds were identified using the NIST05 mass spectral library (National Institute of Standards and Technology, Gaithersburg, MD), retention indices (RI) (van Den Dool and Kratz 1963), and by comparison with authentic standard compounds injected under the same conditions. Compounds were selected for quantification based on previous literature (Hall and others 1985; Hall and Andersson 1985; Carunchia Whetstine and Drake 2007; Vazquez-Landaverde and others 2005) and comparison of chromatogram peaks over storage. Multiple point internal standard curves were generated in odor-free water using the same protocol described above to cover five concentration levels in the range found in the samples for each of the following compounds: dimethyl sulfide, 3-methylbutanal, 2-methylbutanal, 2-heptanone, 1-octen-3-ol, 3-octen-2-one, hexanal, heptanal, octanal, and nonanal. Stock solutions were made by dissolving compounds in methanol, with final dilution into 90 ml odor free water with internal standard and evaluating each concentration in duplicate. Data were plotted with the response ratio (peak area of standard/peak area of internal standard) on the y-axis and concentration ratio (concentration of standard/concentration of internal standard) on the x-axis. Regression equations were generated based on the best linear fit of the data.

Sensory Analysis

Approval for use of human subjects was obtained from the Institutional Review Board (IRB) at North Carolina State University. WMP was reconstituted to 10% SNF with odor-free deionized water for descriptive analysis and freshly purchased spring water for consumer testing. All samples were served with 3-digit codes. Evaluations were conducted in individual sensory booths using Compusense® five (Compusense, Guelph, Canada) software for data collection. Sample order was determined using a randomized balanced Williams design (MacFie and others 1989).

Descriptive Analysis. A 10-member trained panel scored WMP flavor attributes of aroma intensity, cooked, milkfat, sweet aromatic, grassy, painty, musty/earthy, and cardboard flavors and sweet taste and astringency using the Spectrum™ method and the dried dairy ingredient lexicon developed by Drake and others (2003) and previously applied to WMP by Carunchia Whetstine and Drake (2007). About 25 ml of reconstituted product was poured into 60 ml plastic soufflé cups with lids (Solo Cup Company, Highland Park, IL, USA) and served at about 20 ºC. Samples were evaluated in duplicate by each panelist every 2 mo.

Panelists were trained using fresh and aged WMP samples. Panelists received a set of eight samples to evaluate in each session, with a half hour break between sessions. Deionized water was provided for panelists to refresh their palates between samples. Empty cups were also provided for sample expectoration. At each session, separate fresh (free of grassy or painty flavors), grassy and painty flavored reconstituted WMP (previously identified and discussed by the panel) and a fluid whole milk sample were provided to help calibrate panelists.

Consumer Acceptance. Two representative WMP samples were selected for consumer testing: one was 3 mo old and characterized by grassy flavor (intensity 2); the other was 6 mo old and characterized by grassy (intensity 1.5) and painty flavors (intensity 3). WMP was reconstituted in 11.4-liter buckets (M&M Industries, Chattanooga, TN, USA) using wire whisks. Consumer testing (n=75) compared overall acceptance of these samples to a fresh fluid control and two freshly produced WMP (<1 mo, free of grassy or painty flavors, confirmed by descriptive sensory analysis) using a 9-point hedonic scale where 1 = dislike extremely and 9 = like extremely. Approximately 90 ml of reconstituted WMP was served in 177 ml foam cups with lids (Dart Container Corporation, Mason, MI, USA) and straws (Solo Cup Company, Highland Park, IL, USA). Samples were prepared a day in advance and stored at 5 ºC until served.

Evaluations were conducted in individual sensory booths using Compusense® five v 4.6 (Compusense, Guelph, Canada) software for data collection. Sample order was determined using a Williams design (MacFie and others 1989). Room temperature filtered water was provided for panelists to cleanse their palates between samples. Panelists were compensated with food treats and grocery store gift cards.

Data Analysis

XLSTAT version 2007.5 software (Addinsoft USA, New York, New York, USA) was used for data analysis, including analysis of variance with Fisher's LSD, Pearson's correlation analysis, and principal component analysis (PCA). P-values <0.05 were considered significant. Multiple linear regression using a mixed model with backward elimination was performed using SAS v 9.1 (SAS Institute, Cary, NC, USA) to determine the best prediction equations for grassy and painty flavors based on the following volatiles: dimethyl sulfide, 3-methylbutanal, 2-methylbutanal, hexanal, 2-heptanone, heptanal, 1-octen-3-ol, octanal, 3-octen-2-one, nonanal. Multiple linear regression was also performed to determine if physicochemical attributes (water activity, moisture, fat, free fat, WPN, mineral content) could predict grassy or painty flavor or hexanal content.

Results and Discussion

Physical, Chemical and Microbiological Properties

In general, US WMP from the 4 facilities was similar to international WMP in moisture, free fat, fat, and other properties (Steen 1977; van Mil and Jans 1991) and values were within recommended domestic WMP guidelines (ADPI 2002), although some differences were noted (**Table 1**). Water activity values ranged from 0.11-0.24, with the mean values from each facility close to 0.2, which has been recommended as optimal to preserve WMP quality during 25 ºC storage (Stapelfeldt and others, 1997; Schuck and others 2007). The moisture content ranged 2.0-3.3%, which was similar to previous reports of international WMP (Steen 1977; van Mil and Jans 1991). WMP from Facility B was higher in moisture and water activity than WMP from other facilities ($p<0.05$). Total fat content was similar between facilities and within domestic (26-40% fat) and international standards (26-42% fat) (CFR 2005, CODEX 1999). Free fat ranged from 1.1-7.7, which was comparable to international WMP (Steen 1977; van Mil and Jans 1991). WMP from Facility C was significantly higher in free fat than the other facilities, with a mean of 4.2%, compared with 3.2%, 2.0%, and 1.6% for Facilities B, A, and D, respectively ($p<0.05$). WMP with high free fat content have been associated with greater susceptibility to oxidation (Vignolles and others 2007). However, Buma (1971) did not find any correlation between the development of oxidized flavor and free fat of 1.1-79% in WMP stored at 30 ºC for 6 mo, although the flavor of the larger particles (containing less free fat) was slightly better than that of smaller particles (containing a larger proportion of free fat). There were no differences for fat or free fat with relation to season ($p>0.05$), but higher values for water activity and moisture were observed with WMP produced in the spring and summer from both west and east coast facilities ($p<0.05$).

US WMP were all classified as medium heat powders, as indicated by a WPN between 1.51-5.99 mg undenatured whey protein/g WMP (USDEC 2005). WMP were higher ($p<0.05$) in WPN (3.7-5.9 mg/g) than international WMP (1.2-4.2 mg/g), which indicates the heat treatment before drying was generally less severe than international

WMP (USDEC 2005; Steen 1977; van Mil and Jans 1991). Research on New Zealand WMP with a WPN from 0.6-6.0 mg/g determined that a lower WPN resulted in higher cooked flavor and delayed the development of oxidized flavor (Baldwin and others 1991; Baldwin and Ackland 1991). Stapelfeldt and others (1997) found medium (WPN 4.5) and high heat (WPN 1.3) WMP to be more stable than low heat (WPN 7.0) WMP. Facility C WMP was lower in WPN than WMP from the other facilities ($p<0.05$), which indicated that fluid milk from this facility received a more severe heat treatment before spray drying (USDEC 2005). There were no differences in WPN related to season ($p>0.05$).

Both iron and copper were below the recommended limits of 10 mg/kg and 1.5 mg/kg, respectively, for WMP (ADPI 2002). These limits have been set because both iron and copper have been associated with oxidized flavor in milk, while other minerals such as manganese and zinc have not been associated with any off flavors (Greenbank 1948). Copper, zinc and manganese were close to previously reported values, while iron was lower (USDA-ARS 2007, Steen 1977). The observed difference in iron content could be due to efforts to keep the iron content of milk as low as possible. Some differences were observed in mineral content between facilities, with Facility A significantly higher ($p<0.05$) in manganese and lower in zinc ($p<0.05$). These observed differences might be due to differences in feed. No clear seasonal variation was observed for any of the minerals ($p<0.05$). Steen (1977) reported that copper content of WMP was higher during the winter (around 0.7 mg/kg) months than during the summer (0.4 mg/kg), with no seasonal variation observed for iron or zinc.

For microbiological quality, 1 of 38 WMP exceeded the recommended standard plate count limit <10,000 CFU/g for extra grade WMP, although it was still within the limit of <50,000 for standard grade WMP; 10 of 38 WMP exceeded the coliform limit of <10 CFU/g with coliform levels of 15 to 256 CFU/g (ADPI 2002). No seasonal variation was observed for standard plate counts or coliforms ($p>0.05$).

There were some color differences between facilities in dry WMP and reconstituted (wet) WMP (**Figure 2**). Compared to the other facilities, Facility A exhibited higher dry L* scores and wet b* scores, which were correlated with WPN ($r = 0.43$ and 0.50, respectively, $p<0.01$). The higher yellow color (wet b*) from Facility A could be due to milk from pasture fed cows used at this facility, because wet b* values were also correlated with grassy flavor ($r = 0.53$, $p=0.001$). Croissant and others (2007) observed higher Hunter b-values in milk from pasture fed cows compared to milk from cows fed a total mixed ration. Some seasonal variation was observed, with initial dry L* values (lightness) lowest in the fall (data not shown, $p<0.05$). Grigioni and others (2007) observed the opposite, with higher L* values in WMP produced in the fall and winter, concluding that this was due to feed differences throughout the year, which could explain our results as well. Buma and others (1977) attributed the seasonal color variation in milk to β-carotene content, which resulted in lower % reflectance (milk was darker in color) during summer and early fall when cows were fed a pasture based diet, with higher levels of beta-carotene. This agrees with our dry b* values, which were higher (more yellow) during the spring, summer, and early fall ($p<0.05$). Comparison of 12 mo color values to initial values revealed an increase for dry L* ($p<0.0001$) and a decrease for dry b*($p<0.0001$), and wet a* ($p<0.001$) values during storage. There were no significant changes for dry a*, wet L*, or wet b* values ($p>0.05$). Stapelfeldt and others (1997) noted only minor changes in color values for WMP stored for 2 mo at 25 ºC and water activity of 0.1-0.2, which is in agreement with our research. Lim and others (1994) also observed an increase in L* value and decrease b* for dry WMP at 20 ºC during storage, but unlike our research, they also observed an increase in a* values.

Volatile Analysis

Volatiles detected in WMP by SPME included straight chain aldehydes (hexanal, heptanal, octanal, and nonanal), Strecker aldehydes (2- and 3-methylbutanal), 2-heptanone, dimethyl sulfide, 1-octen-3-ol, and 3-octen-2-one. These compounds have been previously documented in WMP and were associated with grassy and painty flavors (Carunchia Whetstine and Drake 2007; Hall and others 1985; Hall and Andersson 1985; Biolatto and others 2007). Straight chain aldehydes have been reported to be the most important volatiles from lipid oxidation (Hall and others 1985). Hexanal, 1-octen-3-ol, and 3-octen-2-one have been identified as important flavor compounds resulting from oxidation of linoleic acid (Ulrich and Grosch 1987), which is the main polyunsaturated fatty acid in WMP (USDA-ARS 2007). The Strecker aldehydes 2- and 3-methylbutanal result from isoleucine and leucine degradation in the Maillard reaction (Bendall 2001), but previous research suggests that their formation is also influenced by lipid oxidation (Hidalgo and Zamora 2008; Hall and others 1985; Andersson and Lingnert 1998).

Wide variation was observed in initial concentrations of WMP volatiles between different facilities and shipments (**Figure 3)**. Some WMP were characterized by a lack of volatiles, while some WMP were differentiated from each other by the presence of certain volatiles. WMP from shipments 2 (May) and 3 (Aug) contained higher ($p<0.0001$) initial levels of hexanal, heptanal, octanal and nonanal compared with the other shipments (Nov and Jan). This is consistent with Biolatto and others (2007) who found that straight chain aldehydes were higher in WMP produced during the spring and summer compared to fall and winter, which they related back to the feed of the cows. Toso and others (2002) also reported higher levels of hexanal, heptanal, and nonanal in raw milk from cows fed grass silage, compared to hay or maize silage. Initial dimethyl sulfide levels were highest in WMP from shipment 3 (Aug), which is also in agreement with Biolatto and others (2007). Reddy and others (1967) also observed higher levels of dimethyl sulfide (31 μg/kg against 11 μg/kg) in milk from cows fed freshly cut alfalfa compared to milk from cows fed bromegrass or rye. Higher concentrations of dimethyl sulfide in our WMP may be associated with feed flavors. Patton and others (1956) reported that when dimethyl sulfide was added to water at levels above sensory threshold (12 μg/kg), a cowy or malty flavor was observed. Initial concentrations of 2- and 3-methylbutanal were significantly higher ($p<0.05$) in WMP from Facilities C and D, which also had the lowest WPN (most severe heat treatment). This suggests that the more severe heat treatment resulted in more Maillard reaction products which underwent Strecker degradation to form Strecker aldehydes. Higher levels of 2- and 3-methylbutanal have also been noted in raw milk from cows fed hay, compared to diets of maize silage or grass silage (Toso and others 2002), which could also explain some of the variation we observed in initial concentrations of these volatiles. Facilities C and D also possessed higher levels of 2-heptanone, which has been observed at higher concentrations in fluid milk with more severe heat treatments (Contarini and others 1997; 2002). Contarini and others (2002) suggested that 2-heptanone could be used as a marker for heat treatment of milk. Our research showed that initial 2-heptanone and WPN values were correlated ($r=-0.40$, $p=0.01$), suggesting that initial 2-heptanone formation increases with more severe heat treatments (lower WPN).

Lipid oxidation volatiles (1-octen-3-ol, 3-octen-2-one, hexanal, heptanal, octanal, and nonanal) as well as Strecker aldehydes (2- and 3-methylbutanal), 2-heptanone and dimethyl sulfide increased during storage ($p<0.05$) (**Table 2**). Initial levels of hexanal ranged from 0.8 μg/kg to 50 μg/kg, with a mean of 13μg/g. Ulberth and Roubicek (1995) reported that fresh WMP contained less than 10 μg/kg of hexanal, which was the case for 22 of our 38 samples. During storage, concentrations of dimethyl sulfide, 3-methylbutanal, hexanal, and heptanal were observed at levels above previously reported sensory threshold concentrations (van Gemert 2003). Carunchia Whetstine and Drake (2007) also observed hexanal, octanal, nonanal, and 2- and 3-methylbutanal to be important odor active compounds in stored WMP by gas chromatography-olfactometry.

During storage, some samples developed lipid oxidation volatiles faster than others ($p<0.05$). Combined data across shipments from each facility during storage are presented in **Figure 4.** Facilities A, C, and D were highly characterized by lipid oxidation volatiles at 12 mo storage, while Facility B at 12 mo was more closely related to the other facilities at 10 mo. Facility D was characterized by higher concentrations of dimethyl sulfide, as well as 2- and 3- methylbutanal during storage compared to other facilities. Hexanal did not increase as rapidly in WMP from Facility B compared to other facilities **(Figure 5**), and WMP from facility B was lower in hexanal than WMP from other facilities after 12 mo storage ($p<0.05$).

It is possible that the slower development of hexanal in facility B WMP was related to its higher moisture content (close to 3%) which has been shown by some researchers to improve storage stability of WMP (van Mil and Jans 1991), or to other factors that were not measured. Hexanal content of WMP at 12 mo varied widely, ranging from 490 to 4400 μg/kg (**Figure 6**), with differences between and within facilities ($p<0.05$). Facilities A and D exhibited the widest variation in hexanal content, while the least variation was observed in facility B. Overall there were no significant correlations between WPN and any of the volatiles ($p>0.05$), which may be explained by the relatively small range of WPN values (3.7-5.9 mg/kg) observed in this study compared to the wide range (0.6-6.0 mg/kg) evaluated by Baldwin and others (1991) and Baldwin and Ackland (1991), especially considering that they found the best WMP stability with WPN below 2.7 mg/kg.

Sensory Analysis

Descriptive Analysis. Descriptive analysis of WMP at time zero revealed differences in initial flavor profiles between facilities as well as within each facility (**Figure 7**)**.** Variations were noted between facilities for cooked and milkfat flavor, with more contrasting differences in sweet aromatic/caramelized and additional flavor notes. WMP from Facility C was higher than the others in sweet aromatic/caramelized flavor intensity ($p<0.05$), which could be related to its heat treatment, although WPN was not significantly correlated with sweet aromatic/ caramelized flavor ($r=-0.09$, $p>0.05$). WMP from Facility A was higher than the others in initial grassy flavor ($p<0.05$). Two samples from Facility B exhibited an initial fatty flavor; these samples were also characterized by higher levels of octanal and nonanal ($p<0.05$). Several samples, including 2 from facility D and 1 from Facility C, had a sweet/feed flavor. This same flavor was observed in milk from cows fed a total mixed ration rather than grass (Croissant and others 2007) and has been associated with the Strecker aldehydes 2- and 3-methyl butanal, previously documented at higher concentrations in WMP from these facilities compared to other facilities. Other researchers have reported that a feed flavor was associated with poor air quality in the cows' barn, feeding of baled silage, and feeding <2 h before milking (Mounchili and others 2004). Facilities A and B each had a sample with an initial earthy/musty flavor that remained throughout storage. This flavor, reminiscent of trichloroanisole, has been reported in other foods and is probably a taint acquired from the food packaging during shipment (Gomez-Ariza and others 2006; Martendal and others 2007; Gunschera and others 2004; Whitfield and others 1997; Nerin and Asensio 2007).

Cooked, sweet aromatic, and milkfat flavors decreased in the WMP during storage (**Table 3**); grassy and painty flavors increased, along with a slight increase in astringency, which is consistent with Carunchia Whetstine and Drake (2007). **Figure 8** summarizes WMP flavor from each facility during storage. Initial flavor profiles of WMP were characterized by sweet aromatic, milkfat, and cooked/caramelized flavors.

Between 2-4 mo, WMP were characterized by sweet and grassy flavors. Beyond 4 mo, WMP were highly characterized by painty and grassy flavors.

WMP from Facilities B, C, and D developed a detectable (mean score of ≥ 1) grassy flavor by 3-4 mo (**Figure 9**), which corresponded with a hexanal concentration of 200-300 μg/kg (**Figure 5**). WMP from facility A was significantly higher than the other facilities in grassy flavor initially and throughout 12 mo storage. The milk used by this facility to manufacture WMP came from cows that were primarily pasture fed rather than receiving a total mixed ration. The initial grassy flavor of WMP from this facility was probably due to milk from pasture fed cows rather than storage-associated lipid oxidation; this flavor was correlated with 2-methylbutanal (r=0.69), hexanal (r=0.65), 2-heptanone (r=0.73), and 2-octene-2-one (r=0.76) ($p<0.05$).

WMP from Facilities A, C and D developed a detectable (score of ≥ 1) painty flavor around 6 mo, while the onset of this flavor was delayed by a couple of mo in WMP from facility B (**Figure 10**), corresponding with a hexanal concentration around 300 μg/kg (**Figure 5**). Facility B WMP was less painty than WMP from the other facilities at 12 mo (**Figure 10 and 11**). This could be related to the higher moisture content in WMP from Facility B as well as other processing parameters and properties that were not measured. Van Mil and Jans (1991) noted that WMP stored at 3% moisture had a higher flavor score and lower peroxide value than WMP stored at 2.7% or 2.4 % moisture for up to 36 mo, which supports our observation. There was wide variation in the intensity of painty flavor from each shipment and facility at 12 mo (**Figure 11**). These differences occurred across different facilities as well as within each facility. Nielsen and others

(1997) observed differences in stability based on trained panel perception of oxidized flavor between 15 batches of medium heat (WPN 4.5) WMP processed in the same plant, which is in agreement with our data. With regard to season, 12 mo WMP received the highest painty scores in the winter and spring, and lowest in the summer and fall. Steen (1977) observed lower flavor quality scores for WMP produced in the spring from 2 different facilities. Van Mil and Jans (1991) observed lowest flavor stability in WMP produced in the spring. This could be related to higher levels of mono- and poly-unsaturated fatty acids in the milk that are susceptible to oxidation, resulting from the feed of the cows (Ambruk and others 2004; Havemose and others 2006; Croissant and others 2007).

Consumer Acceptance. Differences were noted in consumer acceptance of fluid, fresh, grassy, and painty flavored WMP (**Figure 12**). One of the fresh WMP samples was not significantly different in acceptance from the fresh fluid milk, indicating that it is possible for fresh WMP to be well accepted by consumers. The decreased consumer acceptance between the 2 fresh WMP can be explained by higher levels of lipid oxidation volatiles in Fresh WMP 2, including a hexanal content five times higher than Fresh WMP 1 and lower intensities of fresh flavors (cooked, milkfat and sweet aromatic/caramelized) than Fresh WMP 1 (data not shown). Both the 3 mo grassy WMP and the 6 mo painty WMP scored significantly ($p<0.05$) lower than the fresh fluid and fresh reconstituted WMP, indicating that these flavors were not liked by consumers and may be considered off-flavors. Interestingly, there was no difference in acceptance between the grassy and painty WMP. This could be due to the fact that neither flavor attribute was typical of milk that the consumers were accustomed to, making them equally unacceptable. These results suggest that off-flavors detected in WMP by trained panelists can also be recognized by consumers. This is consistent with the results of Hough and others (2002) who determined that increased levels of oxidized flavor (produced by adding copper to reconstituted WMP) in WMP documented by a trained panel resulted in decreased consumer acceptance. Caudle and others (2005) noted lower consumer acceptance scores for SMP with trained panel documented fatty or animal off-flavors, when compared to two SMP lacking those flavor attributes. They also performed consumer acceptance on vanilla ice cream, strawberry yogurt, hot cocoa and white chocolate with each SMP incorporated and noted that some applications masked these off-flavors better than others; this could be an area for further research with WMP.

Key Relationships

Grassy flavor in WMP at 12 mo was positively correlated with WPN ($r=0.49$, $p=0.002$), indicating that the heat treatment of the WMP can have an effect on stability, which is in agreement with research by Stapelfeldt and others (1997). However, grassy flavor at 12 mo was not significantly correlated with water activity, moisture, fat, free fat, or mineral content ($p<0.05$). Painty flavor at 12 mo was not significantly correlated with any of the aforementioned attributes ($p<0.05$). This indicates that these variables may not be directly related to stability in the ranges we observed or that the attributes measured did not have enough variation to draw conclusions related to stability. Still, there is evidence that initial moisture/water activity can play a role on stability, as WMP from the facility with the highest moisture/water activity also exhibited lower painty flavor intensity at 12 mo, which is in accordance with research of Van Mil and Jans (1991). Our research did not show any differences in stability related to free fat content at the levels found in our WMP samples, which is in agreement with Buma (1971).

Correlations were observed between volatiles and flavor attributes across 12 mo storage (**Table 4**). Aroma intensity, grassy flavor, fatty/painty flavor, and astringency were positively correlated with each other as well as Strecker aldehydes, lipid oxidation products, and 2-heptanone; these flavor attributes and volatiles were negatively correlated with cooked, milkfat, sweet aromatic, and sweetness. Hexanal was strongly correlated with 2-methylbutanal (r=0.55), 2-heptanone (r=0.74), heptanal (r=0.96), 1-octen-3-ol (r=0.88), octanal (r=0.92), 3-octen-2-one (r=0.89), and nonanal (r=0.90) ($p<0.0001$). This indicates that as hexanal content increased in WMP, these other volatiles also increased. Strong correlations were observed between painty flavor and lipid oxidation volatiles, including hexanal (r=0.70), heptanal (r=0.65), 1-octen-3-ol (r=0.58), octanal (r=0.60), 3-octen-2-one (r=0.55) and nonanal (r=0.58) ($p<0.0001$). The painty flavor in WMP from Facility C was consistently described as "like old rice" by one of the trained panelists. This is reasonable because raw milled rice contains volatiles resulting from oleic, linoleic, and linolenic acids, including hexanal, heptanal, octanal, 2-nonenal, 3-penten-2-one, and 2-pentylfuran (Lam and Proctor 2003).

Regression equations were generated to predict grassy (r=0.62, R^2=0.38) and painty (r=0.78, R^2=0.61) flavors based on volatiles in WMP (**Table 5**). The correlation was lower for grassy flavor, which is likely due to other volatiles that predict grassy flavor that were not measured. Grassy flavor is not solely a storage flavor so it is possible that other non lipid oxidation compounds may contribute to this flavor. Hexanal was a key compound in our predictive equations ($p<0.0001$) for both grassy and painty flavors. The other volatile compounds ranked as follows for grassy flavor: nonanal, $p=0.002$, 2-heptanone, $p=0.012$; 1-octen-3-ol, $p=0.047$. For painty flavor, the terms were ranked as follows: 3-octen-2-one, $p=0.0025$; octanal, $p=0.0098$; 3-methylbutanal, $p=0.010$; 2-methylbutanal, $p=0.021$. Regression on individual volatiles revealed that hexanal explained the most variation in grassy (31%) and painty (59%) flavor intensities. Other volatiles explained 18-28% of the variation in grassy flavor ($p<0.0001$). Nonanal also explained 59% of the variation of painty flavor. Other volatiles individually explained 26-52% of the variation in painty flavor ($p<0.0001$), excluding, dimethyl sulfide and 3-methylbutanal, which were not significant predictors of this flavor ($p>0.05$). Hall and Andersson (1985) generated regression equations using 25 volatile compounds, including 7 of the 10 quantified in the current study (dimethyl sulfide, 3-methylbutanal, 2-methylbutanal, hexanal, 2-heptanone, heptanal, and nonanal) to predict a variety of flavor attributes including cooked milk, creamy, whey cheese, casein, and cardboard, with r values of 0.85-0.95. Cardboard was the main flavor attribute they associated with off-flavor development in WMP during storage. They determined that hexanal alone could account for 79% of the variation in cardboard off-flavor, while their best predictive equation accounted for 86% of the variation in this flavor.

Conclusions

In general, flavor changes occurred in US WMP by 3-4 mo, indicating that the optimal shelf life of WMP was not as long as the 6-9 mo purported or the desired shelf life of 12 mo claimed by international WMP, which is often subjected to higher heat treatments (lower WPN) and flushed with nitrogen to extend shelf-life. Wide variation existed in flavor and flavor stability of US WMP, even within an individual facility. Some of the sources of variability included water activity, moisture, WPN, and feed source. Of the volatiles studied, hexanal was the best predictor of grassy flavor and either hexanal or nonanal could be used as predictors of painty flavor. For optimum quality, hexanal content of WMP should be less than 50 μg/kg. If hexanal or nonanal concentrations are greater than 600 μg/kg or 4 μg/kg, respectively, painty flavors are evident. This study has established the current status of US WMP and provides a baseline for future studies. Future research should determine what feasible measures can be taken by US manufacturing facilities to produce WMP with more consistent flavor and flavor stability and should directly compare flavor and flavor stability of US WMP with international WMP.

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Tables

Table 1. Mean initial analyses[a] of whole milk powder (WMP) from four US facilities across 5 shipments, with minimum and maximum values and comparison with previous research.

	Facility A	Facility B	Facility C	Facility D	US WMP[b]	International WMP[c]	Recommended values[d]
a_w	0.16b (0.004)	0.18a (0.008)	0.15b (0.007)	0.15b (0.007)	0.11-0.24	---	---
Moisture (%)	2.5b (0.05)	2.9a (0.06)	2.4b (0.07)	2.4b (0.07)	2.0-3.3	2.2-3.0	<4.5
WPN (mg/g)	5.4a (0.06)	4.7b (0.05)	4.2c (0.07)	4.5b (0.06)	3.7-5.9	1.2-4.2	---
Fat (%)	29.3a (0.18)	29.0a (0.08)	28.7a (0.26)	29.5a (0.32)	26.2-32.0	26.1-27.6	26-40
Free fat (%)	2.0c (0.07)	3.2b (0.09)	4.2a (0.44)	1.6c (0.12)	1.1-7.7	2.8-6.7	---
Iron (mg/kg)	1.2a (0.13)	1.0a (0.09)	1.5a (0.25)	1.0a (0.13)	0.2-3.1	4.7-7.3	<10
Copper (mg/kg)	0.24b (0.03)	0.35ab (0.03)	0.47a (0.07)	0.27b (0.05)	0.1-0.9	0.4-0.9	<1.5
Mn (mg/kg)	0.66a (0.14)	0.14b (0.01)	0.18b (0.03)	0.13b (0.01)	0.1-2.1	---	---
Zn (mg/kg)	32.3c (0.16)	34.2a (0.13)	34.4a (0.22)	32.9b (0.30)	30.7-35.7	37.0-40.7	---
Standard plate count (CFU/g)	215a (38.4)	3904a (2341.7)	495a (126.9)	273a (81.1)	84-34,000	1100-14,000	<10,000
Coliforms (CFU/g)	13a (6.9)	7a (3.1)	33a (17.6)	24a (8.7)	<10-256	---	<10

[a] Different letters within a row indicate significant differences ($p<0.05$). Values in parenthesis are standard errors of the means.
[b] Ranges from this study. [c]Ranges compiled from Steen 1977; Van Mil and Jans 1991. [d] From ADPI 2002.

Table 2. Mean volatile compounds (μg/kg) [a] in US WMP through 12 mo storage.

Storage time (mo)	0	2	4	6	8	10	12	Flavor threshold[b] (μg/kg)	Characteristic odor/flavor[c]
Dimethyl sulfide	5.6c	7.3bc	9.9a	10.0a	8.3ab	8.6ab	8.9ab	0.03-12	Malty/cowy[d]
R^2=0.98	(0.50)	(0.4)	(0.7)	(1.7)	(0.8)	(0.6)	(0.7)	water	
3-methylbutanal	1.5d	2.8c	3.8ab	3.3bc	3.3bc	4.1a	4.3a	2-200	Malty/chocolate
R^2=0.97	(0.1)	(0.2)	(0.3)	(0.3)	(0.2)	(0.3)	(0.3)	water	
2-methylbutanal	1.8e	2.8d	4.1c	4.1c	4.7c	6.9b	7.8a	130	Malty/chocolate
R^2=0.98	(0.20	(0.3)	(0.3)	(0.3)	(0.3)	(0.3)	(0.5)	skim milk	
Hexanal	13.0f	109.0f	301.9e	533.5d	842.9c	1152.1b	1691.7a	49-50	Green grass
R^2=0.99	(1.1)	(13.4)	(30.2)	(49.8)	(65.9)	(68.6)	(128.8)	3.8% fat milk	
2-heptanone	4.7f	6.7e	7.7de	8.7d	10.7c	15.0b	21.8a	250-500	Soapy/spicy[e]
R^2=1.00	(0.3)	(0.5)	(0.4)	(0.4)	(0.4)	(0.6)	(1.2)	milk	
Heptanal	2.3e	13.3e	26.0e	52.0d	84.6c	123.7b	200.2a	120	Fatty[g]
R^2=0.97	(0.2)	(1.6)	(2.3)	(4.3)	(7.5)	(9.7)	(19.9)	3.8% fat milk	
1-octen-3-ol	0.2e	0.4de	1.0de	1.4d	2.6c	3.8b	6.9a	10	Mushroom
R^2=1.00	(0.04)	(0.09)	(0.12)	(0.15)	(0.24)	(0.36)	(0.79)	skim milk	
Octanal	0.2e	0.8e	3.3de	9.5d	18.1c	28.1b	50.5a	460	Citrus/green
R^2=0.99	(0.04)	(0.12)	(0.52)	(0.90)	(1.47)	(2.15)	(5.12)	3.8% milk	
3-octen-2-one	0.2e	0.7de	2.4de	5.8d	12.1c	21.5b	40.7a	140	Spicy/fatty/mushroom[f]
R^2=1.00	(0.03)	(0.07)	(0.26)	(0.51)	(1.07)	(1.96)	(4.73)	refined vegetable oil	
Nonanal	0.8e	1.1de	1.4de	2.9cd	4.7c	7.3b	13.8a	220	Fatty/citrus
R^2=1.00	(0.1)	(0.1)	(0.1)	(0.2)	(0.4)	(0.7)	(1.5)	3.8% milk	

[a]Values represent pooled means from triplicate measurements from five shipments. Different letters within a row indicate significant differences ($p<0.05$). Values in parenthesis are standard errors of the means. R^2 based on linear regression of standard curve data. [b]From van Gemert 2003, matrix threshold was determined in is provided below reported threshold or threshold range. [c]From Carunchia Whetstine and Drake 2007, unless otherwise specified. [d]From Patton and others 1956, [e]From Maarse 1991. [f]From Ullrich and Grosch 1987. [g]From Burdock and Fenaroli 2004.

Table 3. Mean flavor attributes[a] in US WMP through 12 mo storage.

Storage Time (mo)	0	2	4	6	8	10	12
Aroma intensity	2.04d	1.98e	2.07cd	2.09c	2.12b	2.13b	2.19a
	(0.02)	(0.01)	(0.01)	(0.01)	(0.01)	(0.01)	(0.01)
Cooked/sulfurous	2.53a	2.34b	2.22c	2.11d	1.95e	1.99f	1.95f
	(0.03)	(0.02)	(0.02)	(0.02)	(0.02)	(0.02)	(0.02)
Milkfat	2.62a	2.41b	2.21c	2.09d	1.93e	1.95e	1.93e
	(0.02)	(0.02)	(0.02)	(0.02)	(0.02)	(0.02)	(0.02)
Sweet Aromatic/caramelized	2.45a	2.19b	2.10c	1.95d	1.79e	1.84e	1.80e
	(0.03)	(0.02)	(0.02)	(0.02)	(0.02)	(0.02)	(0.02)
Grassy/hay	ND	1.05e	1.36d	1.55c	1.67b	1.65bc	2.00a
	(0.04)	(0.05)	(0.05)	(0.04)	(0.04)	(0.04)	(0.04)
Fatty/fryer oil/painty	ND	ND	0.51e	0.96d	1.39c	1.60b	1.98a
	(0.02)	(0.03)	(0.04)	(0.06)	(0.06)	(0.06)	(0.06)
Sweetness	2.10b	2.14a	2.06c	1.99d	1.95e	1.88f	1.90f
	(0.02)	(0.01)	(0.01)	(0.01)	(0.01)	(0.01)	(0.01)
Astringency	1.13c	1.08d	1.14c	1.17c	1.32a	1.26b	1.33a
	(0.01)	(0.01)	(0.01)	(0.01)	(0.01)	(0.01)	(0.01)

[a] Values represent pooled means from duplicate panel measurements from five shipments. Different letters within a row indicate significant differences ($p<0.05$). Values in parenthesis are standard errors of the means. ND=not detected. Intensities were scored on a 0 to 15 point universal Spectrum[TM] intensity scale (Meilgaard and others 1999) where 0 = absence of the attribute and 15 = very high intensity of the attribute. Most dried ingredient flavors fall between 0 and 3 on this scale (Drake and others 2003; Caudle and others 2005; Wright and others 2008).

Table 4. Correlation coefficients[a] of flavor attributes and volatiles in whole milk powder collected from 4 facilities in 5 shipments over a 12 mo period and subsequently and stored for 12 mo.

Variables	Aroma intensity	Cooked	Milkfat	Sweet aromatic	Grassy	Fatty/painty	Sweet	Aastringent	Earthy	Cardboard	Dimethyl sulfide	3-methylbutanal	2-methylbutanal	Hexanal	2-Heptanone	Heptanal	1-Octen-3-ol	Octanal	3-Octen-2-one	Nonanal
Aroma intensity	**1.00**	-0.09	**-0.60**	**-0.30**	**0.38**	**0.43**	**-0.44**	**0.28**	0.01	0.11	0.08	**0.15**	**0.34**	**0.50**	**0.41**	**0.48**	**0.43**	**0.45**	**0.42**	**0.42**
Cooked	-0.09	**1.00**	**0.58**	**0.50**	**-0.43**	**-0.55**	**0.19**	**-0.51**	-0.04	0.03	0.06	-0.07	**-0.19**	**-0.41**	**-0.17**	**-0.37**	**-0.35**	**-0.35**	**-0.29**	**-0.31**
Milkfat	**-0.60**	**0.58**	**1.00**	**0.67**	**-0.55**	**-0.74**	**0.36**	**-0.42**	-0.08	-0.12	-0.01	**-0.22**	**-0.37**	**-0.53**	**-0.34**	**-0.45**	**-0.39**	**-0.42**	**-0.36**	**-0.38**
Sweet aromatic	**-0.30**	**0.50**	**0.67**	**1.00**	**-0.40**	**-0.70**	**0.32**	**-0.51**	-0.07	-0.03	0.10	-0.04	**-0.19**	**-0.45**	**-0.20**	**-0.39**	**-0.35**	**-0.36**	**-0.31**	**-0.33**
Grassy	**0.38**	**-0.43**	**-0.55**	**-0.40**	**1.00**	**0.40**	-0.09	**0.27**	0.02	**-0.13**	0.08	**0.12**	**0.25**	**0.39**	**0.23**	**0.35**	**0.31**	**0.33**	**0.27**	**0.27**
Fatty/painty	**0.43**	**-0.55**	**-0.74**	**-0.70**	**0.40**	**1.00**	**-0.33**	**0.55**	-0.11	-0.01	**-0.16**	**0.16**	**0.37**	**0.70**	**0.44**	**0.65**	**0.58**	**0.60**	**0.55**	**0.58**
Sweet	**-0.44**	**0.19**	**0.36**	**0.32**	-0.09	**-0.33**	**1.00**	**-0.43**	-0.08	-0.04	-0.10	**-0.18**	**-0.35**	**-0.35**	**-0.30**	**-0.30**	**-0.22**	**-0.30**	**-0.26**	**-0.25**
Astringent	**0.28**	**-0.51**	**-0.42**	**-0.51**	**0.27**	**0.55**	**-0.43**	**1.00**	0.00	-0.01	-0.04	0.10	**0.27**	**0.48**	**0.30**	**0.44**	**0.42**	**0.44**	**0.40**	**0.40**
Earthy	0.01	-0.04	-0.08	-0.07	0.02	-0.11	-0.08	0.00	**1.00**	-0.03	0.06	-0.01	-0.05	-0.06	-0.04	-0.06	-0.06	-0.06	-0.06	-0.05
Cardboard	0.11	0.03	-0.12	-0.03	**-0.13**	-0.01	-0.04	-0.01	-0.03	**1.00**	0.07	**0.41**	**0.29**	-0.04	**0.22**	-0.05	-0.06	-0.05	-0.04	-0.05
Dimethyl sulfide	0.08	0.06	-0.01	0.10	0.08	**-0.16**	-0.10	-0.04	0.06	0.07	**1.00**	**0.47**	**0.39**	0.01	**0.26**	0.01	0.00	0.02	0.02	0.02
3-methylbutanal	**0.15**	-0.07	**-0.22**	-0.04	**0.12**	**0.16**	**-0.18**	0.10	-0.01	**0.41**	**0.47**	**1.00**	**0.84**	**0.28**	**0.61**	**0.22**	**0.19**	**0.23**	**0.23**	**0.21**
2-methylbutanal	**0.34**	**-0.19**	**-0.37**	**-0.19**	**0.25**	**0.37**	**-0.35**	**0.27**	-0.05	**0.29**	**0.39**	**0.84**	**1.00**	**0.55**	**0.82**	**0.49**	**0.41**	**0.48**	**0.47**	**0.45**
Hexanal	**0.50**	**-0.41**	**-0.53**	**-0.45**	**0.39**	**0.70**	**-0.35**	**0.48**	-0.06	-0.04	0.01	**0.28**	**0.55**	**1.00**	**0.74**	**0.96**	**0.88**	**0.92**	**0.89**	**0.90**
2-Heptanone	**0.41**	**-0.17**	**-0.34**	**-0.20**	**0.23**	**0.44**	**-0.30**	**0.30**	-0.04	**0.22**	**0.26**	**0.61**	**0.82**	**0.74**	**1.00**	**0.73**	**0.68**	**0.74**	**0.75**	**0.74**
Heptanal	**0.48**	**-0.37**	**-0.45**	**-0.39**	**0.35**	**0.65**	**-0.30**	**0.44**	-0.06	-0.05	0.01	**0.22**	**0.49**	**0.96**	**0.73**	**1.00**	**0.94**	**0.97**	**0.94**	**0.97**
1-Octen-3-ol	**0.43**	**-0.35**	**-0.39**	**-0.35**	**0.31**	**0.58**	**-0.22**	**0.42**	-0.06	-0.06	0.00	**0.19**	**0.41**	**0.88**	**0.68**	**0.94**	**1.00**	**0.96**	**0.96**	**0.96**
Octanal	**0.45**	**-0.35**	**-0.42**	**-0.36**	**0.33**	**0.60**	**-0.30**	**0.44**	-0.06	-0.05	0.02	**0.23**	**0.48**	**0.92**	**0.74**	**0.97**	**0.96**	**1.00**	**0.97**	**0.97**
3-Octen-2-one	**0.42**	**-0.29**	**-0.36**	**-0.31**	**0.27**	**0.55**	**-0.26**	**0.40**	-0.06	-0.04	0.02	**0.23**	**0.47**	**0.89**	**0.75**	**0.94**	**0.96**	**0.97**	**1.00**	**0.98**
Nonanal	**0.42**	**-0.31**	**-0.38**	**-0.33**	**0.27**	**0.58**	**-0.25**	**0.40**	-0.05	-0.05	0.02	**0.21**	**0.45**	**0.90**	**0.74**	**0.97**	**0.96**	**0.97**	**0.98**	**1.00**

[a] **Values in bold are significant ($p<0.05$).**

Table 5. Prediction equations[a] for grassy and painty flavor in whole milk powder based on volatile compounds[b].

Flavor	Prediction Equation	Correlation Coefficient
Grassy =	0.92 + (0.00058 × hexanal) + (0.024 × 2-heptanone) + (0.073 ×1-octen-3-ol) – (0.083 × nonanal)	$r = 0.62$, $p<0.0001$
Painty =	0.47 – (0.086 × 3-methylbutanal) + (0.070 × 2-methylbutanal) + (0.00073 × hexanal) + (0.018 × octanal) – (0.022 × 3-octen-2-one)	$r = 0.78$, $p<0.0001$

[a]Generated using multiple linear regression with backwards elimination; variables with p-value >0.10 removed from model. [b]Initial volatile compounds in the model included dimethyl sulfide, 3-methylbutanal, 2-methylbutanal, hexanal,2-heptanone, heptanal, 1-octen-3-ol, octanal, 3-octen-2-one, and nonanal.

Figures

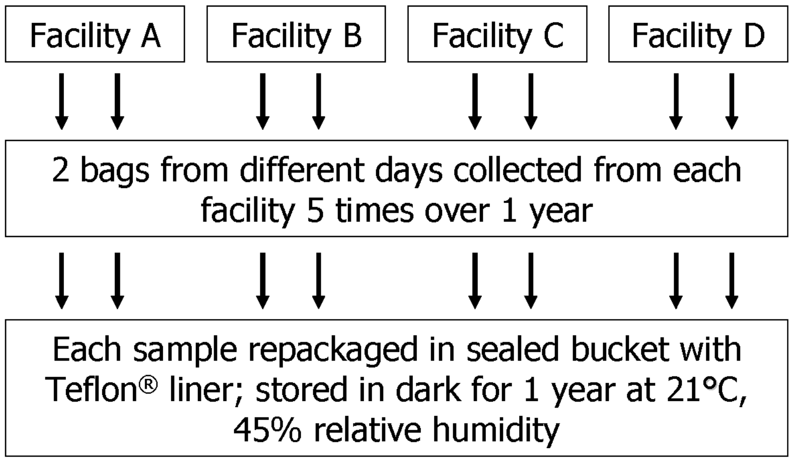

Figure 1. Experimental design.

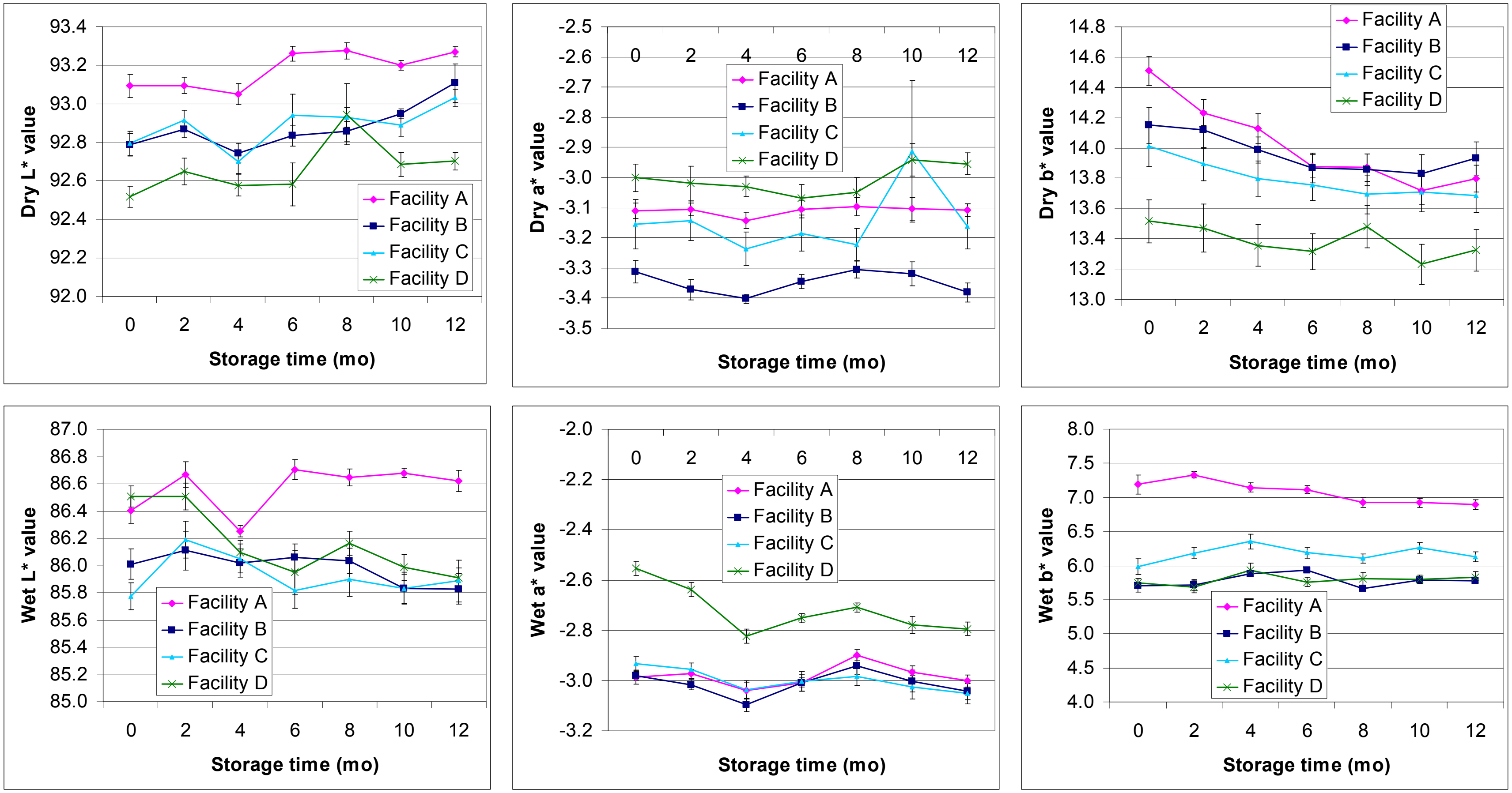

Figure 2. CIE L* a* b* color values for dry and reconstituted (wet) whole milk powder stored up to 12 mo at 21 ºC and 45% relative humidity. Bars represent the standard errors of the means.

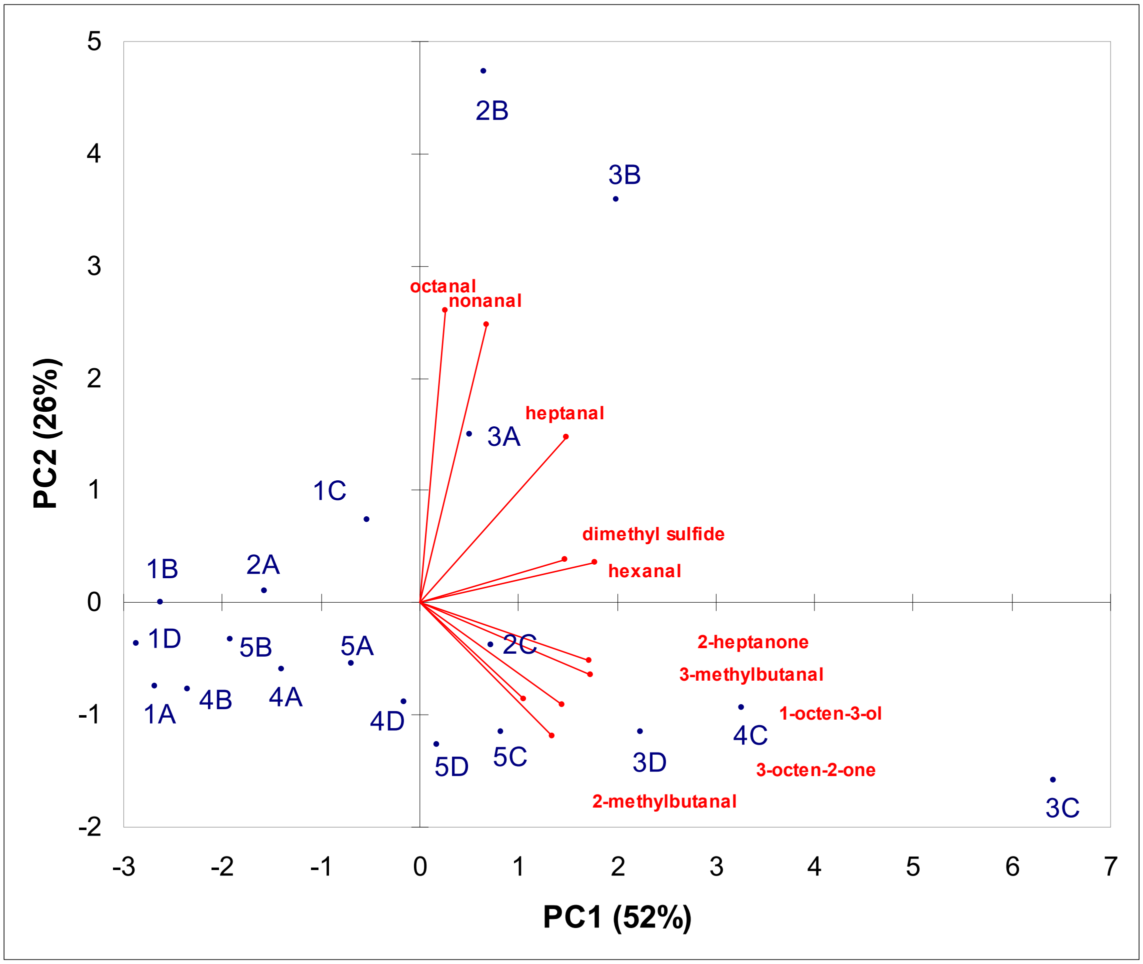

Figure 3. Principal component analysis biplot showing initial volatiles of whole milk powder from four facilities. Numbers represent shipments (1-5) and letters represent facilities (A-D). Each point represents triplicate analyses from 2 bags produced on different days.

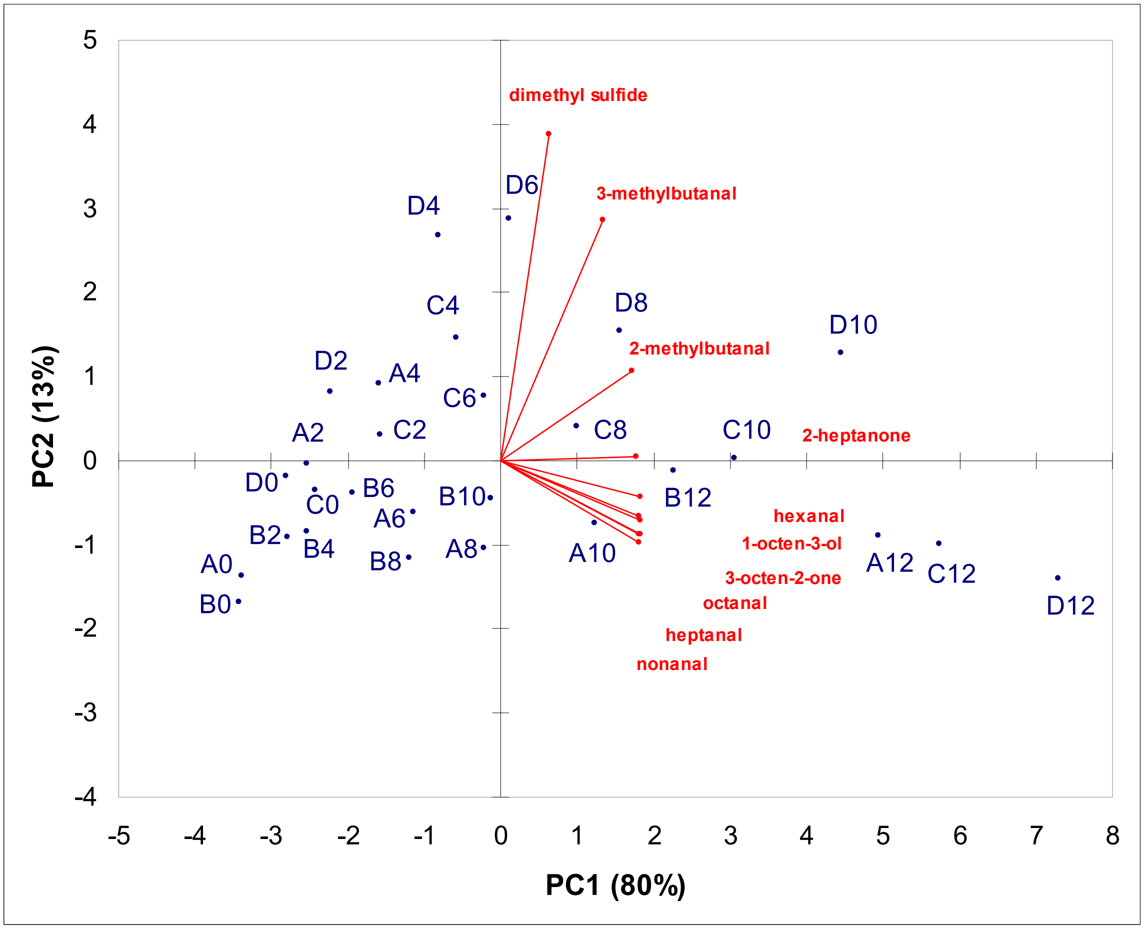

Figure 4. Principal component analysis biplot showing volatiles in whole milk powder from four facilities stored up to 12 mo. Letters represent facilities (A-D) and numbers represent storage time (0-12 mo). Each point represents triplicate analyses from 2 bags produced on different days.

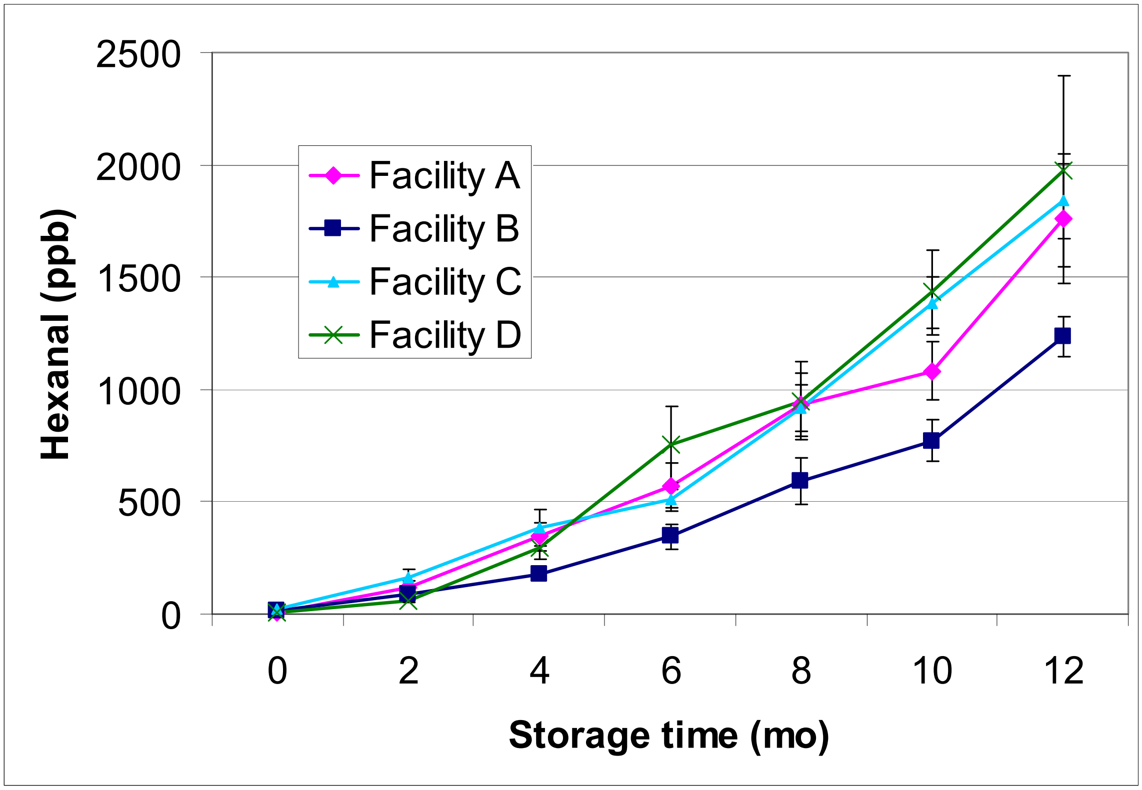

Figure 5. Hexanal development in whole milk powder from four facilities over 12 mo storage. Data are pooled from 5 shipments across a year for each facility (with only 4 shipments from facility D). Bars represent standard errors of the means.

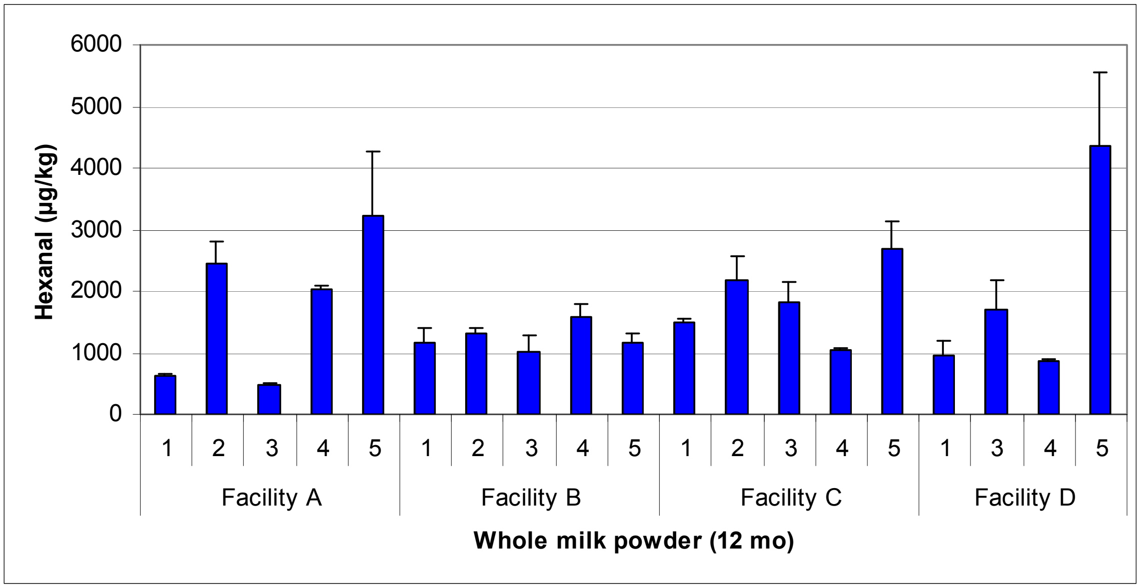

Figure 6. Variation in hexanal concentration in whole milk powder stored for 12 mo at 21 ºC and 45% relative humidity. Data are means of 2 bags received in 5 different shipments across a year for from facility (with no shipment 2 from facility D). Bars represent standard errors of the means.

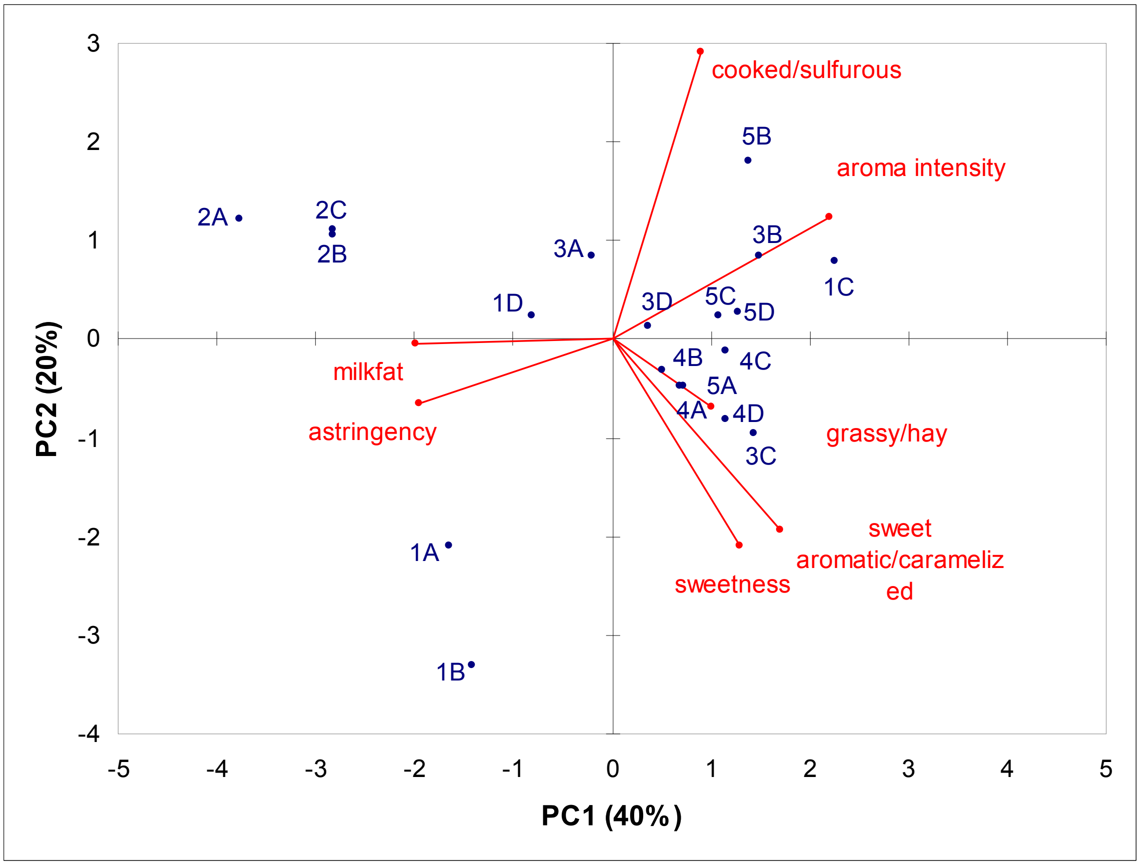

Figure 7. Principal component analysis biplot showing initial descriptive analysis of whole milk powder from four facilities. Numbers represent shipments (1-5) and letters represent facilities (A-D). Each point represents duplicate analyses from 2 bags produced on different days.

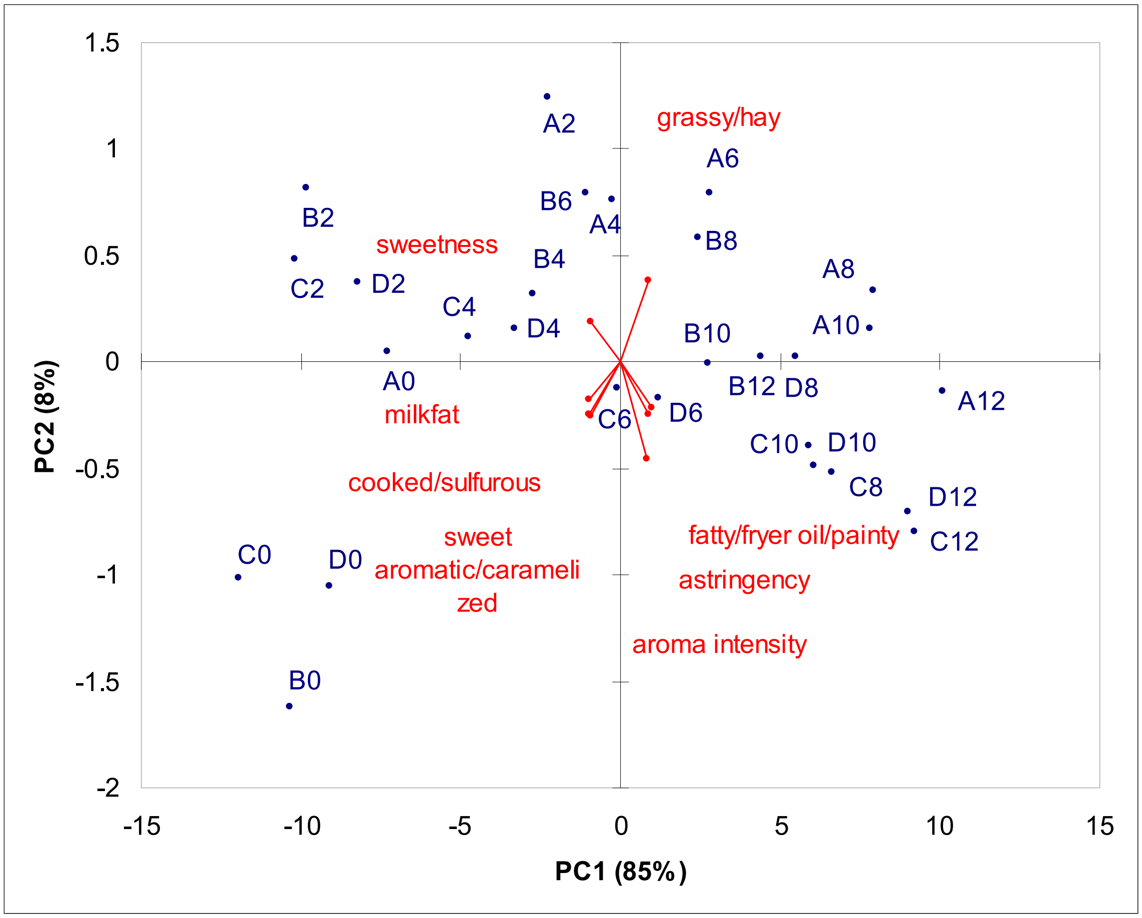

Figure 8. Principal component analysis biplot descriptive analysis of whole milk powder from four facilities stored up to 12 mo. Letters represent facilities (A-D) and numbers represent storage time (0-12 mo). Each point represents duplicate analyses from 2 bags produced on different days.

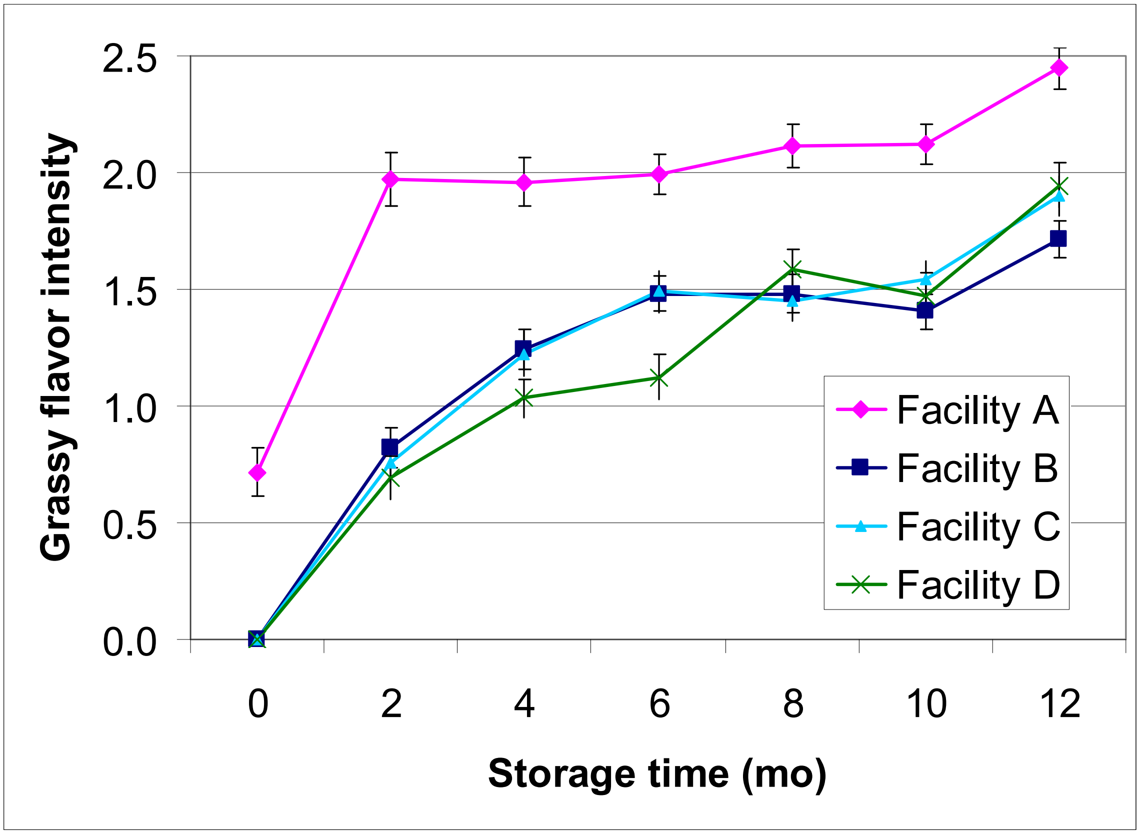

Figure 9. Grassy flavor development in whole milk powder from four facilities over 12 mo storage. Grassy flavor was scored on a 0 to 15 point universal Spectrum^{TM} intensity scale (Meilgaard and others 1999) where 0 = absence of the attribute and 15 = very high intensity of the attribute. Most dried ingredient flavors fall between 0 and 3 on this scale (Drake and others 2003; Caudle and others 2005; Wright and others 2008). Bars represent standard errors of the means.

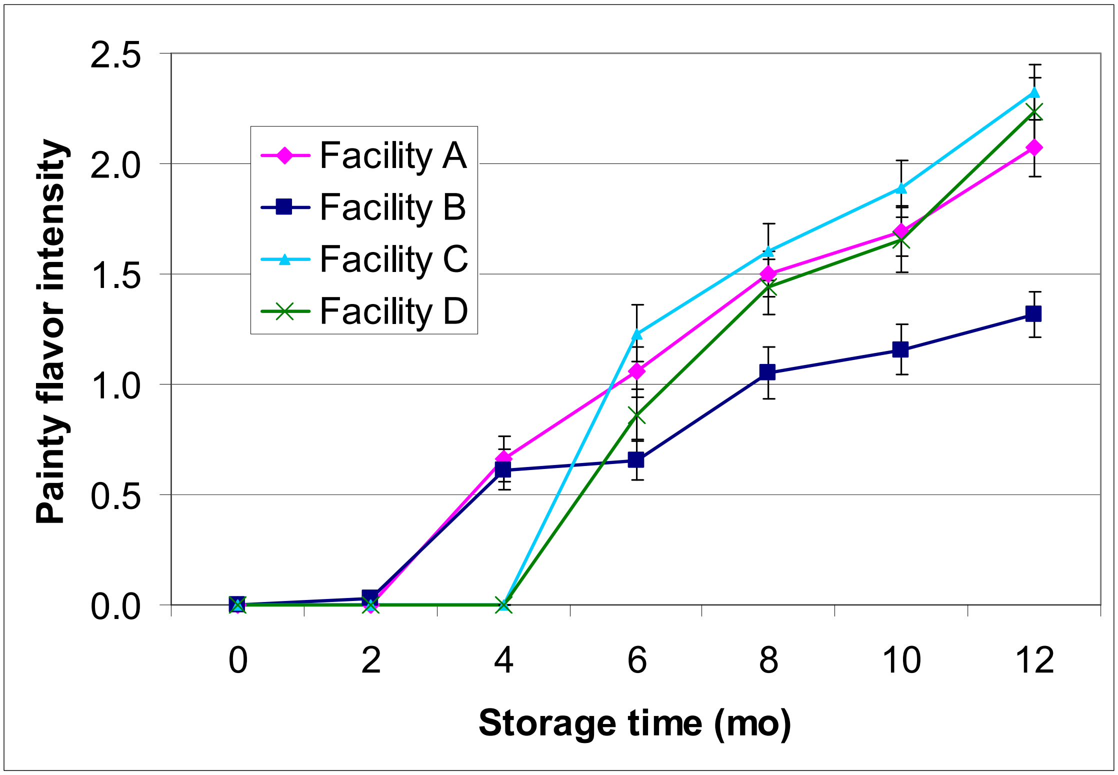

Figure 10. Painty flavor development in whole milk powder from four facilities over 12 mo storage. Painty flavor was scored on a 0 to 15 point universal Spectrum^{TM} intensity scale (Meilgaard and others 1999) where 0 = absence of the attribute and 15 = very high intensity of the attribute. Most dried ingredient flavors fall between 0 and 3 on this scale (Drake and others 2003; Caudle and others 2005; Wright and others 2008). Bars represent standard errors of the means.

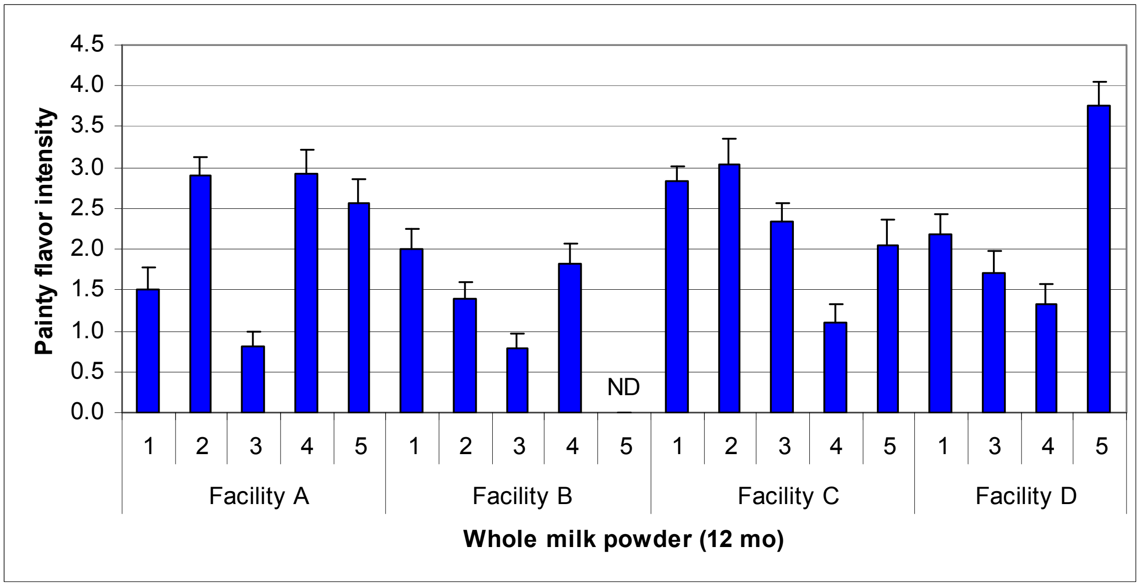

Figure 11. Variation in painty flavor intensity in whole milk powder stored for 12 mo at 21 ºC and 45% relative humidity. Data are means of 2 bags received in 5 different shipments across a year for from facility (with no shipment 2 from facility D). Grassy flavor was scored on a 0 to 15 point universal Spectrum^{TM} intensity scale (Meilgaard and others 1999) where 0 = absence of the attribute and 15 = very high intensity of the attribute. Most dried ingredient flavors fall between 0 and 3 on this scale (Drake and others 2003; Caudle and others 2005; Wright and others 2008). Bars represent standard errors of the means.

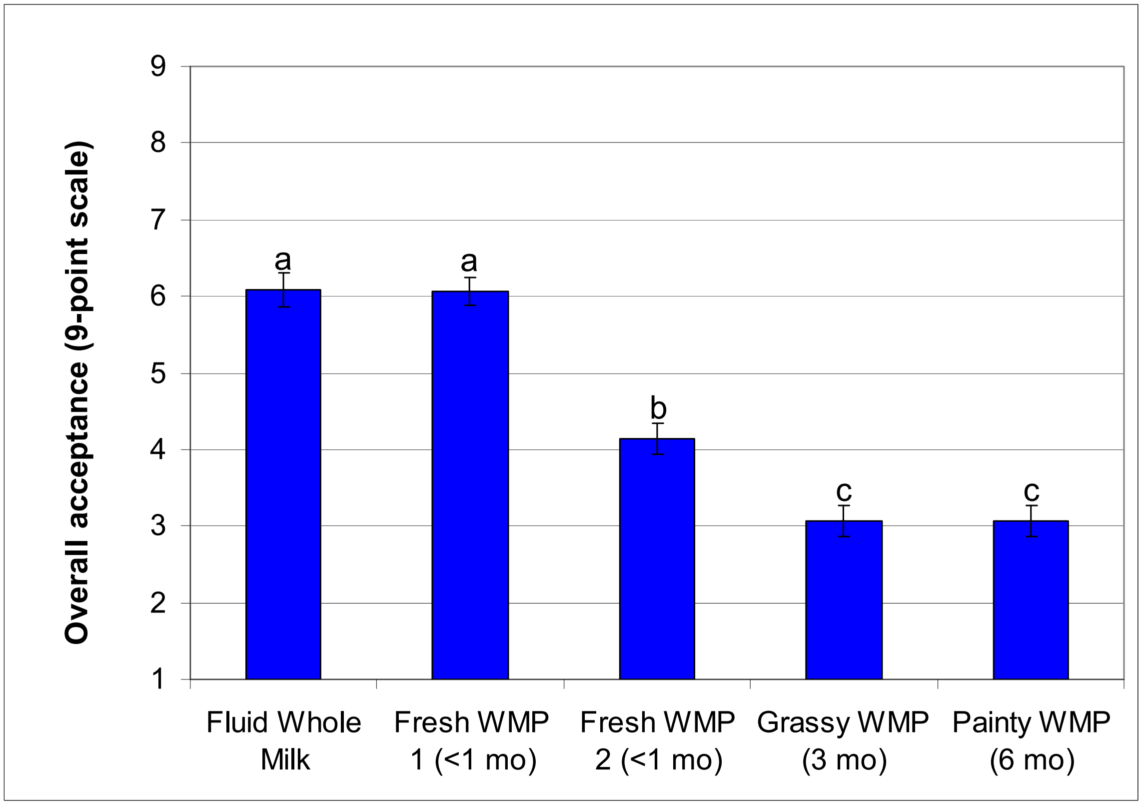

Figure 12. Consumer acceptance of fluid whole milk compared with reconstituted whole milk powder (WMP) of different ages. Acceptance was scored on a 9-point hedonic scale where 1 = dislike extremely and 9 = like extremely. Bars represent standard errors of the means. Different letters indicate significant differences ($p<0.05$).

Effect of Nitrogen Flushing and Storage Temperature on Flavor and Shelf-life of Whole Milk Powder

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Flavor of Whole Milk Powder…

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ABSTRACT:

The US industry standard for shelf-life of whole milk powder (WMP) is 6-9 mo, although previous research has demonstrated flavor changes by 3 mo at ambient storage. This study evaluated the influence of packaging atmosphere, storage temperature, and storage time on WMP shelf-life through sensory and instrumental techniques. Two batches of WMP were repackaged in plastic laminate pouches with air or nitrogen and stored at 2 ºC or 23 ºC for one y. Descriptive analysis was conducted using a 10-member trained panel; volatile analysis was performed using solid-phase microextraction (SPME) with gas chromatography-mass spectrometry (GC-MS). Consumer acceptance (n=75) was conducted every 3 mo with reconstituted WMP and white and milk chocolate made from each treatment. Data were analyzed using analysis of variance with Fisher's LSD, Pearson's correlation analysis, and principal component analysis with significance at $p<0.05$. Air stored WMP had higher peroxide values, lipid oxidation volatiles and grassy and painty flavors than nitrogen flushed WMP. Storage temperature did not impact levels of straight chain lipid oxidation volatiles; 23 ºC storage resulted in higher cooked and milkfat flavors and lower levels of grassy flavor compared with 2 ºC storage. Consumer acceptance was negatively correlated with lipid oxidation volatiles and painty flavor. Nitrogen flushing prevented the development of painty flavor in WMP stored up to 1 y at either temperature, resulting in chocolate with high consumer acceptance. Nitrogen flushing can be applied to extend the shelf life of WMP for use in chocolate; storage temperature also plays a role, but to a lesser extent.

Key words: dairy, flavor, storage, volatiles, whole dry milk

Introduction

Whole milk powder (WMP) contains 26-40% fat, making it very susceptible to oxidation, which can result in off-flavors (USDEC 2005). The US industry standard for shelf-life of WMP is 6-9 mo under storage at <27 ºC and <65% relative humidity (USDEC 2005), although previous research demonstrated flavor changes by 3 mo in ambient temperature storage (Carunchia Whetstine and Drake 2007). Increasing the storage stability of this commodity is of great interest to producers and end users. Some factors that influence WMP flavor and shelf-life include initial milk quality, processing variables, air quality, moisture content, packaging, oxygen exposure, addition of antioxidants, light exposure, and storage temperature (Nielsen and others 1997; Baldwin and Ackland, 1991; Kurtz and others 1969; Christensen and others 1951; Stapelfeldt and others 1997; van Mil and Jans 1991; Hough and others 2002; Heiss and Eichner 1971; Hall and Lingnert 1984; Tamsma and Pallansch 1964).

Van Mil and Jans (1991) reported a 3-year shelf-life for WMP processed and stored under optimal conditions, including: (1) moderate heat treatment of 95 ºC for 2 min (undenatured whey protein nitrogen of 1.7), (2) moisture content at least 2.8%, (3) storage ≤ 20 ºC, and (4) air and moisture proof oxygen reduced packaging. Many researchers have found substantial benefits with reduced oxygen storage for WMP (Chan and others 1993; Tuohy 1984; McCluskey and others 1997; Pyenson and Tracy 1946; van Mil and Jans 1991). Most WMP storage studies have evaluated WMP at ambient or elevated storage temperatures (Stapelfeldt and others 1997; Tamsma and Pallansch 1964; van Mil and Jans 1991), but information is lacking on WMP stored at cooler temperatures. WMP may be classified as low, medium, or high heat based on undenatured whey protein nitrogen (WPN), with values of ≥6, 1.51-5.99, and ≤1.5 mg undenatured whey protein /g powder, respectively (USDEC 2005). Stapelfeldt and others (1997) found WMP to be most stable when the fluid product was heated under medium (80 ºC/20s) or high (88 ºC/20s) heat treatments before spray drying, with water activities between 0.1 and 0.2 (as opposed to 0.3), stored at 25 ºC (rather than 45 ºC). Solubility index has been used by a number of researchers to evaluate WMP quality (Baldwin and Ackland 1991; van Mil and Jans 1991; Nielsen and others 1997; Celestino and others 1997), and peroxide value (PV) is commonly used to evaluate lipid oxidation in WMP (McCluskey and others 1997; Pyenson and Tracy 1946; van Mil and Jans 1991; Decker and others 1951). The development of off-flavors in WMP has been monitored by the development of lipid oxidation volatiles as well as through descriptive sensory analysis (Biolatto and others 2007; Hall and Andersson 1985; Hall and others 1985; Carunchia Whetstine and Drake 2007; Baldwin and others 1991; van Mil and Jans 1991). Off-flavors observed in WMP during storage have been described as fatty, grassy, painty, oxidized (tallowy, waxy, metallic, oily), green, cardboard, casein, and mushroom (Hall and Lingnert 1984; Carunchia Whetstine and Drake 2007; Baldwin and others 1991). Lipid oxidation volatiles detected in WMP included hexanal, heptanal, octanal, nonanal, decanal, Z-4-heptenal, E, E-2,4-heptadienal, E-2-octanal, E-2-decanal, and 3-octen-2-one (Biolatto and others 2007; Carunchia Whetstine and Drake 2007; Hall and others 1985).

Flavor is the main factor influencing consumer acceptance of WMP and other foods (Lee and Morr 1994; Drake 2004; Hough and others 2002; Nursten 1997). Over

75% of the WMP manufactured in the US goes into confectionary products such as chocolate, with the remainder used in baked goods, dry sauce and soup mixes, dairy beverages, ice cream, or other uses (Chandan 1997; USDEC 2001). If the WMP used in these applications has off flavors, these flavors may carry through into the final product and result in consumer complaints. Several researchers have related descriptive analysis to consumer acceptance (Sather and others 1963; Hough and others 2002; Caudle and others 2005; Bolenz and others 2003). Caudle and others (2005) found that off flavors in skim milk powder (SMP) carried though into yogurt, hot cocoa, and white chocolate, resulting in decreased consumer acceptance. Drake (2006) and Wright and others (2008) also reported decreased consumer acceptance of ingredient applications (protein beverages) made from dried whey protein with trained panel documented off-flavors. Bolenz and others (2003) studied milk chocolate made with a variety of dairy ingredients, including WMP, SMP, anhydrous milkfat, and whey protein concentrate, and found that consumer acceptance decreased with the presence of malty or off-notes contributed by dairy ingredients from particular suppliers. However, research is lacking on consumer acceptance of stored WMP and chocolate made from stored WMP.

The objective of this research was to evaluate the influence of WMP packaging atmosphere, storage temperature, and storage time on WMP shelf life, as measured by (1) flavor and stability of WMP and (2) consumer acceptance of WMP and chocolate made with WMP. Both instrumental and sensory tests were conducted to achieve these objectives.

Materials and Methods

WMP

The experimental design for the study was replicated twice (**Figure 1)**. Freshly produced (<1 mo), non-agglomerated, WMP (500 kg) was shipped from a commercial production facility in CA to Hershey, PA where it was repackaged in pouches made up of a 340 gauge nylon-vinyl alcohol-polyethylene laminate with an oxygen transmission rate of 1.5 ml/m^2/day (Weldon, Oshkosh, WI, USA). Approximately 5.4 kg WMP were put into each pouch. Half of the pouches were treated with a vacuum to -0.9 bar and nitrogen to -0.2 bar before sealing. The other half were sealed with ambient air and no vacuum. Each treatment was protected from light and stored at 2 ºC or 23 ºC in temperature controlled rooms at 50% relative humidity for one year and evaluated every 2 mo, unless otherwise specified. Where indicated, WMP was reconstituted to 10% solids not fat (SNF) before analysis with odor-free deionized water according to the equation in International Dairy Federation Method 99C (IDF 1997): g whole milk powder in 90 g water = 1000/(100–% fat). WMP were protected from light exposure during storage as well as during preparation for volatile and sensory analysis.

Physicochemical Tests

Initial Tests. Upon receipt, a subsample of each WMP was sent to an independent lab (Tulare, CA) for duplicate measurements of moisture, undenatured whey protein nitrogen index (WPN), fat, and free fat. Moisture content was measured using the vacuum oven method from DA Instruction 918-RL (USDA-AMS 2001). WPN and fat

(Mojonnier method) were measured as described by Wehr and Frank (2004). Free fat was measured based on the method of Sørensen and others (1978). Copper and iron were measured in duplicate by the NCSU Analytical Services Laboratory (Raleigh, NC) using a standard dry ash method with inductively coupled plasma optical emission spectroscopy (ICP-OES). Briefly, 2.5 g WMP were (1) ashed overnight at 500 ºC in a muffle furnace, (2) cooled, (3) mixed with approximately 1.5 ml distilled water and 4 ml 6N HCl (VWR West Chester, PA, USA) and heated to dryness on a steam plate to remove silicates, (4) transferred to a 50 ml volumetric flask with approximately 4 ml of 6N HCl, and (5) brought to volume with distilled water for ICP-OES analysis (Lisa Lentz, Analytical Services Lab, North Carolina State University, Personal Communication, 2008). Water activity was measured in duplicate using an Aqualab series 3 TE water activity meter (Decagon Devices, Pullman, WA, USA). Both water activity and moisture were also measured at 2 mo intervals through 12 mo storage.

Headspace Oxygen. Headspace oxygen of each WMP package was measured before other analyses at each time point (every 2 mo) using a Mocon HS750 headspace analyzer (Mocon Inc., Minneapolis, MN, USA) calibrated to ambient air (20.9% oxygen). A 1 cm rubber septum with adhesive on one side was attached to the package. Then, 10 ml of headspace gas was drawn out with a syringe and injected into the analyzer to generate a percent oxygen reading. If a nitrogen flushed treatment was above 6.5% oxygen (indicative of a poor package seal), this package was excluded from further study. Extra packages were stored to replace packages that indicated a poor seal by oxygen measurement.

Peroxide Value. Peroxide value (PV) was measured every 2 mo using the Peroxysafe^{TM} STD Kit with the Saftest^{TM} System (MP Biomedicals United States, Solon, OH).

Color. CIE L*a*b* values were obtained in triplicate every 2 mo with a Minolta CR-300 Chroma Meter using light source "C" (Konica Minolta Sensing Americas, Inc., Ramsey, NJ , USA) using a 60 x 15 mm petri dish (Becton Dickinson Labware, Franklin Lakes, NJ, USA). Dry as well as reconstituted powders (10% SNF) and liquid were filled to the brim (ca 9mm depth) of the larger diameter petri dish top; the dry powder was compressed with the smaller diameter bottom portion of the dish. Samples were measured at approximately 22 ºC.

Solubility Index. Every 4 mo, solubility index was determined in duplicate according to the American Dairy Products Institute (ADPI, 2002). WMP was reconstituted to 10% SNF based on the equation described previously. Solubility index was reported as ml sediment in conical centrifuge tubes (Kimble No. 45167-50, Fisher Scientific, Pittsburgh, PA, USA) after centrifugation at 164 x g.

Volatile Analysis

Volatile compounds of WMP were analyzed in triplicate at 0, 2, 3, 4, 6, 8, 10, and 12 mo using solid phase microextraction (SPME) and a Varian CP-3380 gas chromatograph (GC) equipped with a Saturn 2000 ion trap mass selective detector (MSD) from Varian, Inc. (Palo Alto, CA., USA), similar to the method reported by Wright and others (2008). Briefly, chromatography was performed using a 30 m length x 0.25 mm i.d. x .25 µm df Rtx®-5 ms column (Restek, Bellefonte, PA, USA) with helium carrier gas at a flow rate of 1 ml/min. Oven temperature was ramped from 40 ºC to 250 ºC at a rate of 8 ºC/min with 5 min initial and final hold times. MSD conditions were as follows: 80 ºC manifold, 120 ºC transfer line 150 ºC ion trap. Chemical standards and alkanes were obtained from Sigma Aldrich (St. Louis, MO, USA); methanol was obtained from VWR (West Chester, PA, USA).

WMP was reconstituted to 10% SNF using 90 ml odor-free deionized water. Five µl internal standard (5 µl E-2-hexenal in 5 ml methanol) were added to reconstituted milk to make a final concentration of 45 (µg/kg). Twenty g of the reconstituted sample with internal standard were added to a 40 ml amber vial with 1.0 g sodium chloride (Fisher Scientific, Pittsburgh, PA, USA) and an 8 mm x 13 mm stir bar (VWR, West Chester, PA, USA). A poly(tetrafluoroethyene) coated septum (Supelco, Bellefont, PA, USA) was placed facing the liquid and screwed into place. A water blank was made by adding internal standard to 90 ml deodorized water and then adding 20 g of this solution to a 40 ml vial as described above. An alkane series was prepared by adding 1 µl of alkane solution (approximately 40 mg/kg C7-C24 in hexane) into an empty 40 ml vial and sealed as described above and used to calculate retention indices (RI) of volatile compounds (van Den Dool and Kratz 1963).

Sample vials were placed in a heating block at 40 ºC for 30 min to equilibrate. A 2.0 cm DVB/Carboxen^{TM}/PDMS Stablefex^{TM} (Supelco, Bellefont, PA, USA) was inserted and exposed at a depth of 2.0 cm in the heating block for an additional 30 min, followed by injection into the GC for 5 min at a depth of 3.0 cm. All fibers were conditioned before use according to manufacturer's directions. Volatile compounds were identified using the NIST05 mass spectral library (National Institute of Standards and Technology, Gaithersburg, MD), retention indices (RI), and by comparison with authentic standard compounds injected under the same conditions. Compounds were selected for quantification based on previous literature (Hall and others 1985; Hall and Andersson 1985; Carunchia Whetstine and Drake 2007; Vazquez-Landaverde and others 2005) and comparison of chromatogram peaks over storage. Multiple point internal standard curves were generated in odor-free water using the same protocol described above to cover five concentration levels in the range found in the samples for each of the following compounds: dimethyl sulfide, 3-methylbutanal, 2-methylbutanal, 2-heptanone, 1-octen-3-ol, 3-octen-2-one, hexanal, heptanal, octanal, and nonanal. Stock solutions were made by dissolving compounds in methanol, with final dilution into 90 ml odor free water with internal standard. Data were plotted with the response ratio (peak area of standard /peak area of internal standard) on the y-axis and concentration ratio (concentration of standard/concentration of internal standard on the x-axis. Regression equations were generated based on the best linear fit of the data.

Sensory Analysis

Approval for use of human subjects was obtained from the Institutional Review Board (IRB) at North Carolina State University. WMP was reconstituted to 10% SNF with odor-free deionized water for descriptive and consumer testing. All samples were served with 3-digit blinding codes. Samples were presented using a Williams design for a balanced order of presentation (MacFie and others 1989).

Descriptive Analysis. Descriptive analysis profiles for each sample were determined by 10-member trained panel (7 females, 3 males, ages 22 – 47 y, each with at least 100 h of prior experience with descriptive analysis of dairy ingredients) using the Spectrum™ method (Meilgaard and others 1999) and the dried dairy ingredient lexicon developed by Drake and others (2003) and previously applied to WMP by (Carunchia Whetstine and Drake 2007). The following attributes were rated: aroma intensity, cooked, milkfat, sweet aromatic/caramelized, grassy, painty, and cardboard flavors and sweet taste and astringency, About 25 ml were poured into 60 ml plastic soufflé cups with lids (Solo Cup Company, Highland Park, IL, USA) and served at 20 ºC. Samples were evaluated in duplicate by each panelist initially and after 2, 3, 4, 6, 8, 10 and 12 mo storage.

Panelists were trained using fresh and aged WMP samples. Evaluations were conducted in individual sensory booths using Compusense® five version 4.6 (Compusense, Guelph, Ontario, Canada) software for data collection. Panelists received a set of eight samples to evaluate in each session, with a half hour break between sessions. Deionized water was provided for panelists to refresh their palates between samples. Empty cups were also provided for sample expectoration. Warm-up samples of fresh fluid milk, as well as examples of fresh (free of grassy or painty flavors), grassy and painty flavored reconstituted WMP (previously evaluated and discussed by the panel) were provided at each session for panelist calibration.

Consumer Acceptance. Consumer acceptance was conducted every 3 mo with reconstituted WMP and white and milk chocolate made from each treatment, with each product tested on separate days. A freshly produced WMP or chocolate manufactured with freshly produced WMP was also included as a control. A fresh fluid milk control purchased from a local retail outlet was also included as a control in the reconstituted WMP testing. Consumers (n=75) rated overall acceptance (9-point hedonic scale, 1= dislike extremely and 9 = like extremely). For WMP, approximately 90 ml reconstituted WMP was served in 177 ml foam cups with lids (Dart Container Corporation, Mason, MI, USA) and straws (Solo Cup Company, Highland Park, IL, USA). Samples were prepared a day in advance and stored at 5 ºC until served.

Both milk and white chocolate bars were made by an industry partner using a fresh control WMP and each of the treatments (**Table 1)**. Ingredients were mixed with part of the cocoa butter and refined to 25 microns. Then the remainder of the cocoa butter was added and the mixture was conched for 3 h at 70°C. Then, the chocolate was tempered using seed crystal formed from a small amount of the chocolate. The tempering progress was monitored using a Chocolate Temper Meter (Tricore, Elgin, IL, USA). Once a proper temper was reached, samples were molded into bars and sealed in foil laminate packages (Printpack, Inc., Elgin, IL, USA). Chocolate was shipped to North Carolina State University and stored for 2 weeks at 20 ºC before consumer testing.

Two 25 x 25 mm squares of each chocolate treatment were served in 60 ml soufflé cups with lids. Consumers included faculty, staff, and students from North Carolina State University. Evaluations were conducted in individual sensory booths using Compusense® five version 4.6 (Compusense Inc., Guelph, Ontario, Canada) software for data collection. Room temperature filtered water was provided for panelists to cleanse their palates between samples. Panelists were compensated with food treats and grocery store gift cards.

Statistical Analysis

Statistical analysis was performed using XLSTAT version 2007.5 (Addinsoft USA, New York, New York, USA), with $p<0.05$ considered significant. The following tests were performed: analysis of variance with Fisher's least significant difference, Pearson's correlation analysis, and principal component analysis (PCA).

Results and Discussion

Physicochemical Tests

Initial Testing. The initial properties of the WMP were typical compared with previously reported US WMP values (**Table 2**) (USDEC 2005; Farkye 2006; USDA-ARS 2007; Liang and Hartel 2004). Initial values for moisture and water activity were 2.5% ± 0.2 and 0.17 ± 0.03, respectively. By 12 mo, both moisture and water activity increased ($p<0.05$) to levels of 3.3% ± 0.4 and 0.28 ± 0.04, respectively. The 0.8% increase in moisture we observed was reasonable, considering the 0.6% increase in moisture noted by Lim and others (1994) in WMP stored in a paper/nylon/polyethylene laminate for 4 mo. Initial water activity was near the 0.2 recommended by other researchers (Stapelfeldt and others 1997; Schuck and others 2007). The WPN values were within in the range recommended by Baldwin and others (1991) to improve storage stability and to prevent oxidized flavor. Both iron and copper content were well below the recommended limits of 10 mg/kg and 1.5 mg/kg, respectively (ADPI 2002).

Headspace Oxygen. Means of headspace oxygen in packaged WMP across storage were 4.0 ± 1.1% for nitrogen flushed WMP and 18.8 ± 0.7% for air packaged WMP **(Figure 2)**. No significant differences in headspace oxygen were observed with relation to storage time or storage temperature ($p<0.05$). Van Mil and Jans (1991) found that WMP quality was maintained for 36 mo when packaged at 6% or <3% oxygen, with no change in trained panel flavor score and a peroxide value below 0.3 meq/kg fat. In contrast, Andersson and Lingnert (1998) found that sensory changes occurred in cream powder stored at 0.03% oxygen at 30 ºC for 25 weeks.

Peroxide Value. Initial PV's for WMP were less than 0.05 meq/kg fat, which fits with the recommendation of Rossell (1989) that fresh fat should have a PV <1 meq/kg fat. Peroxide formation was significantly higher in the air packaged whole milk powder (**Figure 3**) compared to nitrogen flushed packages. There were no differences due to storage temperature ($p<0.05$). Air packaged treatments reached 1 meq/kg fat level by 6 mo, while nitrogen flushed treatments remained well below 1 meq/kg fat for the duration of 12 mo storage. The air stored, 23 ºC treatment reached 2.7 meq/kg fat at 10 mo, and then decreased by 12 mo. Since peroxides are primary lipid oxidation products, they typically reach a maximum level and then taper off as secondary lipid oxidation products are formed (van Mil and Jans 1991; Liang 2000; McCluskey and others 1997). Baldwin and others (1991) measured PV's from 0.03 to 3 meq/kg fat in New Zealand WMP stored in air for up to 18 mo. van Mil and Jans (1991) measured PV up to 8 meq/kg fat in WMP stored up to 36 mo at 20 ºC.

Color. When compared to the initial value, differences in color values among the treatments were noted after 12 mo storage (**Table 3**). Dry L* values for all treatments were all significantly lighter than the control, with 23 ºC stored samples closer to the control than the 2 ºC samples. None of the treatments were different from the control for dry a*. For dry b*, the 2 ºC stored samples were significantly lower than the 23 ºC samples. For wet L* and wet a*, the 2 ºC samples were not significantly different from the control. All samples were significantly higher than the control for wet b* values. Stapelfeldt and others (1997) found increased lightness (Hunter L-value) and yellowness (Hunter b-value) during storage for dry WMP, with only small changes in Hunter L- and b- values for WMP at low water activity, which was in agreement with our results.

Solubility Index. Solubility index was ≤ 0.1 ml for all treatments initially and did not change significantly during 12 mo of storage (data not shown). These results agree with those of Lloyd and others (2004), who did not find any correlation between age and solubility index of SMP stored up to 29 y. Unlike our results, Celestino and others 1997 noted an increase from 0.1 ml to 0.4 ml in WMP stored up to 8 mo at 25 ºC; similar increases were observed by Baldwin and Ackland (1991) and van Mil and Jans (1991), who observed increases from 0.3 ml to 0.9 ml and <0.1 to 0.6, respectively, for WMP stored up to 12 mo at 30 ºC. Differences in solubility index have been noted with preheat holding time before drying, season, and other factors, but further research is needed to understand the mechanism for these differences, which could be related to denaturation of whey proteins, aggregation of casein micelles, or crystallization of lactose (van Mil and Jans 1991; Celestino and others 1997). These values were all below the 1.0 ml limit set by the USDA for extra grade WMP (USDA-AMS 2001).

Volatile Analysis

The volatile compounds selected for quantification increased during storage (**Table 4**). Initial volatile results were as expected for fresh WMP, with lipid oxidation products such as hexanal, heptanal, nonanal, and octanal at very low levels compared with stored samples (Ulberth and Roubicek 1995; Carunchia Whetstine and Drake 2007; Hall and Andersson 1985). Volatiles increased during storage at different rates, depending on the treatment (**Figure 4)**. Air stored samples were characterized by an increase in lipid oxidation volatiles during storage, while nitrogen flushing slowed the development of lipid oxidation volatiles. The impact of treatment on hexanal formation is shown in **Figure 5**. Hexanal increased most rapidly in the air-packaged, 23 ºC WMP. Hexanal reached a maximum at 10 mo, and then decreased by 12 mo, which could be due to breakdown of hexanal (Neilson and others 2006; Wright and others 2008). Storage at 23 ºC in either air or nitrogen was associated with increased levels of 2-heptanone (**Table 5**). Ulberth and Roubicek (1995) also noted higher levels of 2-heptanone in WMP stored at increased temperature.

Differences were noted between the initial volatile concentrations and the different treatments after 12 mo storage (**Table 5**). The following volatiles were significantly lower in the nitrogen treatments when compared to air (**Figure 4, Table 5**): 2-methylbutanal, hexanal, heptanal, 1-octen-3-ol, octanal, 3-octen-2-one, and nonanal. This is consistent with research of Hall and others (1985) who found that nitrogen flushing inhibited formation of Strecker aldehydes such as 2-methylbutanal, and straight chain aldehydes such as hexanal in WMP stored at 25 ºC for 84 weeks. They noted that a nitrogen storage atmosphere did not impact the formation of 2-ketones, such as 2-heptanone, which is in agreement with our results. They also observed, as we did, that dimethyl sulfide was significantly higher in the nitrogen flushed, room temperature WMP. The following volatiles were significantly higher at 23 ºC compared to 2 ºC: dimethyl sulfide, 3-methylbutanal, 2-methylbutanal, 2-heptanone, heptanal, octanal, 3-octen-2-one, and nonanal; hexanal content was not affected by storage temperature. These results indicate that both the storage temperature and packaging atmosphere can play a role in extending the shelf-life of whole milk powder by inhibiting the formation of lipid oxidation volatiles. However, nitrogen flushing had a much greater effect than storage temperature in preventing the formation of hexanal, which is a key lipid oxidation product that has been associated with cardboard, grassy and painty off-flavors (Andersson and Lingnert 1998; Carunchia Whetstine and Drake 2007; Hall and Andersson 1985).

Sensory Analysis

Descriptive Analysis

Sensory changes were documented across storage (**Figure 6)**. Initial descriptive profiles were typical of fresh WMP, including cooked, sweet aromatic and milkfat flavors and the absence of grassy or painty flavors (Carunchia Whetstine and Drake 2007; Hall and Lingnert 1984; Hall and Andersson 1985); however air stored samples >3 mo were characterized by grassy and painty flavors and a decrease in "fresh" flavors. Nitrogen flushed WMP at both temperatures stored for 12 mo did not have any detectable painty flavors, and were closer in flavor profiles of fresh WMP than air stored WMP after 12 mo (**Table 6**). The 23 ºC air stored WMP after 12 mo was significantly lower than all of the other treatments for milkfat, and sweet aromatic flavors, and significantly higher in painty flavor, indicating that it was the least well preserved. The 2 ºC air stored WMP also exhibited a detectable level of painty flavor. No significant differences in flavor attributes were noted between the 2 ºC and 23 ºC nitrogen flushed WMP, indicating that both treatments were similar from a sensory standpoint.

Overall, the 23 ºC stored WMP had higher intensities of cooked and milkfat flavors, while 2 ºC storage resulted in higher intensities of grassy flavor. Often grassy flavor is associated with hexanal (Carunchia Whetstine and Drake 2007), but there was no significant difference in hexanal concentration between the 2 storage temperatures ($p>0.05$). Many other selected volatile components, however, were distinct between the two storage temperatures (**Table 6**), which suggests that other lipid oxidation components or that a combination of these components with hexanal may contribute to grassy flavor perception. Painty flavor developed in air stored samples by 6 mo (**Figure 7**). Nitrogen flushing and 2 ºC storage inhibited the development of painty flavor in WMP.

Relationships were observed between sensory perception and volatile compounds (**Figure 8)**. Grassy flavor was correlated ($p<0.05$) with 1-octen-3-ol ($r=0.44$), hexanal ($r=0.46$), heptanal ($r=0.48$), and nonanal ($r=0.40$) concentration. Painty flavor was correlated ($p<0.0001$) with 2-methylbutanal ($r=0.66$), 1-octen-3-ol ($r=0.88$), 3-octen-2-one ($r=0.94$), hexanal ($r=0.95$), heptanal ($r=0.95$), octanal ($r=0.87$), and nonanal ($r=0.95$)

concentration. These results are consistent with Hall and Andersson (1985), who found that changes in flavor were mostly due to lipid oxidation products, especially straight chain aldehydes. Carunchia Whetstine and Drake (2007) noted straight chain aldehydes among the odor active compounds that increased in whole milk powder stored up to 2 y. They did not detect any grassy or painty flavors in the initial samples by descriptive analysis, but these flavors developed during storage. Peroxide value was correlated ($p<0.0001$) with hexanal ($r=0.91$) and painty flavor ($r=0.86$). Baldwin and others (1991) found a correlation of 0.73 between PV and trained panel perception of oxidized flavor. Van Mil and Jans (1991) reported that trained judges could detect oxidized off flavors in WMP when PV's reached 2 meq/kg fat, which occurred around 2 y for their WMP. Our research showed distinct flavor changes before the PV reached 1 meq/kg fat, which could be due to use of a different PV method, or a more sensitive trained panel.

Consumer Acceptance

Fluid whole milk scored significantly higher ($p<0.05$) than the WMP control (6.6 versus 6.1) for overall acceptance. When compared to the fresh control WMP, consumer acceptance of reconstituted WMP dropped significantly after 3 mo of storage for all treatments (**Figure 9**). This was likely due to a loss of "fresh" flavors and the development of grassy and painty flavors in the WMP, which were more prevalent in the air stored WMP. Nitrogen flushed WMP scored significantly higher in overall acceptance than air treatments over 12 mo storage, consistent with trained panel profiles and lipid oxidation compounds. Storage temperature of WMP did not influence consumer acceptance of reconstituted WMP ($p>0.05$).

Consumer acceptance was positively correlated with trained panel attributes associated with freshly produced WMP, including cooked/sulfurous, milkfat, and sweetness; acceptance was negatively correlated with astringency, painty, and grassy flavors, as well as the following lipid oxidation products: 1-octen-3-ol, 3-octen-2-one, heptanal, hexanal, nonanal, and octanal (**Table 7**). Hough and others (2002) had good success relating trained panel sensory attributes of WMP to consumer acceptance, finding that WMP with increased levels of oxidized flavor measured by a trained panel resulted in decreased consumer acceptance. Bishov and others (1971) conducted consumer testing on a variety of freeze-dried fruits, vegetables, and meat stored between 0-2% oxygen for 1 yr at 38 ºC, and concluded that lower oxygen levels resulted in better quality (increased consumer acceptance) for all foods studied.

Differences in WMP treatments were more subtle when incorporated into chocolate, probably due to flavor masking by the other ingredients. Air packaged, 23 ºC treatments were less acceptable than other treatments after 3 mo for white chocolate (**Figure 10**), and after 6 mo for milk chocolate (**Figure 11**). Differences noted between white and milk chocolate were also likely due to differing degrees of flavor masking among these two products. Both Caudle and others (2005) and Childs and others (2007) noted differences in flavor carry-through (measured by consumer acceptance testing) of dried ingredients in different ingredient applications. Nitrogen flushed WMP stored up to one y at both temperatures produced both white and milk chocolate that was not significantly different from the fresh control. Unlike consumer acceptance of reconstituted WMP, acceptance of both white and milk chocolate was not significantly correlated ($p>0.05$) with grassy flavor in WMP (**Table 7**), indicating that this off-flavor was well-masked by the other ingredients. Consumer acceptance of both white and milk chocolate was positively correlated with WMP sweet taste and negatively correlated with painty flavor and lipid oxidation volatiles (**Table 7**).

WMP stored at 2 ºC produced chocolate with higher acceptance than WMP stored at 23 ºC (**Figures 10 and 11**). Also, milk chocolate acceptance was negatively correlated ($p<0.05$) with the presence of 2-methylbutanal and 3-methylbutanal in the WMP, which are associated with malty flavor (Avsar and others 2004; Carunchia Whetstine and Drake 2007). Bolenz and others (2003) found that consumer acceptance was negatively impacted by the presence of malty ($r = -0.789$) flavor in chocolate contributed by dry dairy ingredients. As mentioned previously, these compounds were found at higher levels in WMP stored at 23 ºC, regardless of storage atmosphere. This result suggests that a cooler storage temperature of 2 ºC, in combination with nitrogen flushing, could also be beneficial to shelf-life extension of WMP, especially if it is to be used in chocolate.

Conclusions

Optimum shelf-life of WMP from a flavor standpoint was about 3 mo. By 3 mo, WMP exhibited a loss of "fresh" flavors and/or developed a grassy off-flavor, which significantly decreased consumer acceptance of reconstituted WMP. After 6 mo, painty off-flavors in WMP were detected by consumers in chocolate. Nitrogen flushing greatly enhanced storage stability of WMP by preventing the development of painty flavor.

Packaging headspace oxygen levels should be as low as possible (<6.5% oxygen) to prevent lipid oxidation and off flavors. Cooler storage temperatures also enhanced storage stability of WMP, but to a lesser extent than nitrogen flushing. WMP stored in nitrogen flushed packaging for up to a year could be used to manufacture milk chocolate with a high level of consumer acceptance. However, the shelf life of chocolate made from fresh or stored WMP has not been compared. Further research should track changes in consumer acceptance and flavor changes in chocolate bars for up to a year after production (standard shelf life).

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Tables

Table 1. Percentages (w/w) of ingredients in milk and white chocolate formulations.

Ingredient	Milk Chocolate	White Chocolate
Sucrose (Domino Specialty Ingredients, Baltimore, MD, USA)	45.47	50.47
Chocolate liquor (The Hershey Company, Hershey, PA, USA)	11	0
Whole milk powder	25	25
Deodorized cocoa butter (ADM, Decatur, IL, USA)	18	24
Vanillin (Rhodia, Inc., Cranbury, NJ, USA)	0.03	0.03
Lecithin (Degussa Texturant Systems; Atlanta, GA, USA)	0.4	0.4
PGPR[a] (Danisco USA, New Century, KS, USA)	0.1	0.1

[a] = polyglycerol polyricinoleate, an emulsifier

Table 2. Initial analyses of whole milk powder.

	Mean[a]	Typical value
a_w	0.17 (0.01)	0.16-0.18[b]
Moisture (%)	2.46 (0.06)	2.5[d]
Fat (%)	30.05 (0.45	26.1-40[c,e]
WPN (mg/g)	4.40 (0.10)	1.2-4.2[e,f]
Free Fat (%)	1.61 (0.09)	2.8-6.7[e]
Iron (mg/kg)	1.35 (0.06)	4.7[d]
Copper (mg/kg)	0.19 (0.09)	0.8[d]

[a]Means from duplicate analyses of 2 batches of WMP before storage. Values in parentheses are standard errors of the means. [b] Farkye 2006. [c]USDEC 2005. [d]ASDA-ARS 2007. [e]Steen 1977. [f]Van Mil and Jans 1991

Table 3. Mean color values[a] for whole milk powder stored in air or nitrogen at 2 ºC or 23 ºC for 12 mo compared with initial color values.

	Initial Value	12 mo			
		Air		Nitrogen	
		2 ºC	23 ºC	2 ºC	23 ºC
Dry L*	92.64c	93.23a	92.92b	93.24a	92.86b
	(0.07)	(0.02)	(0.03)	(0.07)	(0.05)
Dry a*	-3.11a	-2.93a	-2.99a	-3.31a	-3.10a
	(0.17)	(0.16)	(0.13)	(0.04)	(0.17)
Dry b*	13.51a	12.51b	13.49a	12.72b	13.60a
	(0.14)	(0.10)	(0.03)	(0.02)	(0.12)
Wet L*	87.19a	86.76ab	86.06c	86.72abc	86.20bc
	(0.20)	(0.17)	(0.20)	(0.00)	(0.12)
Wet a*	-2.64a	-2.70ab	-2.76b	-2.63a	-2.76b
	(0.03)	(0.01)	(0.02)	(0.02)	(0.01)
Wet b*	6.20a	5.81c	6.03b	5.94bc	6.04b
	(0.06)	(0.02)	(0.03)	(0.01)	(0.02)

[a]Different letters within a row indicate significant differences ($p<0.05$). Values in parentheses are standard errors of the means.

Table 4. Mean concentrations (μg/kg) of selected volatile compounds[a] in whole milk powder during 1 y of storage

Storage time (mo)	**0**	**2**	**3**	**4**	**6**	**8**	**10**	**12**	**Regression equation[b]**
dimethyl sulfide	22.4bc	12.0d	5.9d	10.5d	14.7cd	23.2bc	33.3a	29.4ab	y = 5.09x
	(6.8)	(1.0)	(0.5)	(1.0)	(0.7)	(2.7)	(6.5)	(6.4)	R^2=0.98
3-methylbutanal	5.2bcd	3.7d	1.8e	4.0d	4.9cd	7.0a	6.3ab	6.0abc	y = 9.35x
	(1.4)	(0.4)	(0.2)	(0.3)	(0.4)	(0.5)	(0.7)	(0.9)	R^2=0.97
2-methylbutanal	3.5de	2.4ef	2.0f	4.0cd	5.0c	7.7b	9.0a	7.6b	y = 8.52x
	(0.9)	(0.3)	(0.2)	(0.3)	(0.5)	(0.4)	(1.0)	(1.0)	R^2=0.98
hexanal	32.8e	57.9e	122.9de	396.0cd	494.1bc	709.7b	1339.9a	784.0b	y = 18.67x
	(2.1)	(9.1)	(37.6)	(52.2)	120.3)	(139.7)	(259.5)	(167.9)	R^2=0.99
2-heptanone	9.8c	7.2c	6.3c	7.4c	9.6c	16.4b	19.6ab	22.8a	y = 50.87x
	(2.3)	(1.0)	(0.4)	(0.8)	(1.1)	(1.9)	(3.4)	(3.9)	R^2=1.00
heptanal	4.3d	7.0d	13.3cd	37.5bcd	45.1bc	59.2b	128.8a	100.1a	y = 33.99x
	(0.3)	(1.0)	(3.3)	(4.3)	(10.5)	(11.0)	(23.8)	(24.0)	R^2=0.97
1-octen-3-ol	0.1d	0.4d	0.7cd	1.1cd	1.5bc	2.5b	4.4a	4.4a	y = 43.02x
	(0.0)	(0.0)	(0.1)	(0.1)	(0.2)	(0.2)	(0.8)	(0.9)	R^2=1.00
octanal	4.2e	8.8de	8.7de	12.8cd	14.3cd	17.7c	38.7a	28.9b	y = 66.74x
	(1.0)	(4.4)	(5.0)	(0.7)	(0.4)	(0.6)	(1.5)	(1.9)	R^2=0.99
3-octen-2-one	0.2c	0.7c	1.0c	3.3bc	4.1bc	6.3b	15.6a	13.8a	y = 39.23x
	(0.0)	(0.1)	(0.3)	(0.4)	(1.2)	(1.4)	(3.2)	(4.1)	R^2=1.00
nonanal	1.0cd	0.9d	1.2cd	2.3bcd	2.9bc	3.7b	7.5a	5.9a	y = 128.75x
	(0.2)	(0.1)	(0.2)	(0.2)	(0.6)	(0.6)	(1.3)	(1.4)	R^2=1.00

[a]Different letters within a row indicate significant differences ($p<0.05$). Values in parentheses are standard errors of the means. [b]Based on standard curves generated using water with E-2-hexenal as an internal standard.

Table 5. Mean concentrations (µg/kg) of selected volatile compounds in whole milk powder stored in air or nitrogen at 2 ºC or 23 ºC for 12 mo compared with initial values[a].

	Initial Value	12 mo storage			
		Air		Nitrogen	
		2 ºC	23 ºC	2 ºC	23 ºC
Dimethyl sulfide	25.8b	16.4b	26.5ab	32.6ab	54.3a
	(6.8)	(5.6)	(9.0)	(3.8)	(17.5)
3-methylbutanal	5.1a	4.0a	8.6a	3.9a	8.0a
	(1.4)	(0.8)	(2.2)	(0.3)	(1.3)
2-methylbutanal	3.5c	4.5c	12.9a	4.9bc	9.2b
	(0.9)	(0.9)	(1.7)	(0.6)	(1.4)
Hexanal	20.7b	1090.3a	1050.5a	100.7b	135.3b
	(2.1)	(347.8)	(344.0)	(2.1)	(37.5)
2-heptanone	10.5b	8.3b	39.1a	9.7b	35.9a
	(2.3)	(1.3)	(5.0)	(0.6)	(6.6)
Heptanal	2.7b	133.1a	144.5a	8.0b	14.9b
	(0.3)	(49.0)	(53.7)	(0.7)	(4.6)
1-Octen-3-ol	0.0c	4.1ab	7.1a	0.3bc	2.8bc
	(0.0)	(1.6)	(2.1)	(0.3)	(0.3)
Octanal	3.7c	29.0ab	47.0a	12.5bc	16.4bc
	(1.0)	(6.7)	(13.2)	(1.2)	(1.4)
3-Octen-2-one	0.2c	13.4ab	27.7a	0.2bc	1.5bc
	(0.0)	(5.2)	(10.9)	(0.2)	(0.5)
Nonanal	0.8b	7.7a	8.9a	0.7b	1.2b
	(0.2)	(2.7)	(3.0)	(0.1)	(0.2)

[a]Different letters within a row indicate significant differences ($p<0.05$). Values in parentheses are standard errors of the means.

Table 6. Mean intensities of flavor attributes in whole milk powder stored in air or nitrogen at 2 ºC or 23 ºC for 12 mo compared with initial values[a].

	Initial value	12 mo storage			
		Air		Nitrogen	
		2 ºC	23 ºC	2 ºC	23 ºC
Aroma Intensity	2.00b	2.12a	2.12a	2.03ab	2.08a
Cooked/sulfurous	2.52a	2.02c	1.97c	2.35b	2.37b
Milkfat	2.72a	2.08c	1.87d	2.49b	2.51b
Sweet Aromatic/Caramelized	2.16b	1.98c	1.76d	2.35ab	2.43a
Grassy	ND	2.13a	1.74a	0.61b	0.76b
Painty/Fatty	ND	1.03b	1.60a	ND	ND
Sweet taste	2.21a	1.88b	1.88b	2.15a	2.10a
Astringency	1.10b	1.24a	1.35a	1.07b	1.07b

[a]Different letters within a row indicate significant differences ($p<0.05$). ND=not detected

Attributes were scored on a 0 to 15 point universal Spectrum™ intensity scale where 0 = absence of the attribute and 15 = extremely high intensity of the attribute (Meilgaard and others 1999). Most reconstituted dried dairy ingredient flavors fall between 0 and 4 on this scale (Drake and others 2003; Drake 2004; Carunchia Whetstine and others 2007; Wright and others 2008).

Table 7. Correlation coefficients and p-values[a] for overall acceptance of reconstituted whole milk powder (WMP), white chocolate, and milk chocolate with trained panel attributes and instrumental measurements from WMP stored for 3, 6 or 12 mo in air or nitrogen at 2 ºC or 23 ºC.

	WMP acceptance		**White chocolate acceptance**		**Milk chocolate acceptance**	
	r	**p-value**	**r**	**p-value**	**r**	**p-value**
Temp	0.34	0.237	-0.22	0.452	-0.35	0.215
Time	**-0.54**	**0.045**	-0.47	0.087	-0.38	0.176
%O2	-0.46	0.100	**-0.58**	**0.031**	-0.49	0.074
aroma intensity	-0.46	0.100	**-0.89**	**< 0.0001**	**-0.80**	**0.001**
astringency	**-0.68**	**0.007**	**-0.75**	**0.002**	**-0.61**	**0.019**
cooked/sulfurous	**0.76**	**0.002**	0.24	0.401	0.02	0.952
painty	**-0.77**	**0.001**	**-0.78**	**0.001**	**-0.66**	**0.009**
grassy	**-0.84**	**0.000**	-0.26	0.367	-0.02	0.954
milkfat	**0.91**	**< 0.0001**	0.49	0.076	0.31	0.287
sweet aromatic	0.47	0.088	0.53	0.050	0.41	0.140
sweetness	**0.83**	**0.000**	**0.72**	**0.004**	**0.54**	**0.048**
1-octen-3-ol	**-0.62**	**0.017**	**-0.62**	**0.018**	**-0.56**	**0.038**
2-heptanone	-0.16	0.592	-0.29	0.312	-0.40	0.162
2-methylbutanal	-0.31	0.282	-0.50	0.067	**-0.58**	**0.030**
3-methylbutanal	-0.02	0.947	-0.45	0.110	**-0.57**	**0.035**
3-octen-2-one	**-0.59**	**0.027**	**-0.67**	**0.009**	**-0.62**	**0.018**
dimethyl sulfide	0.29	0.311	0.01	0.966	-0.11	0.713
Heptanal	**-0.67**	**0.009**	**-0.77**	**0.001**	**-0.67**	**0.009**
Hexanal	**-0.70**	**0.006**	**-0.84**	**0.000**	**-0.75**	**0.002**
Nonanal	**-0.64**	**0.013**	**-0.76**	**0.001**	**-0.71**	**0.005**
Octanal	**-0.67**	**0.009**	**-0.67**	**0.009**	**-0.60**	**0.022**
WMP acceptance	**--**	**--**	**0.57**	**0.035**	0.38	0.177
White chocolate acceptance	**0.57**	**0.035**	**--**	**--**	**0.91**	**< 0.0001**
Milk chocolate acceptance	0.38	0.177	**0.91**	**< 0.0001**	**--**	**--**

[a]Values in bold are significant (p<0.05).

Figures

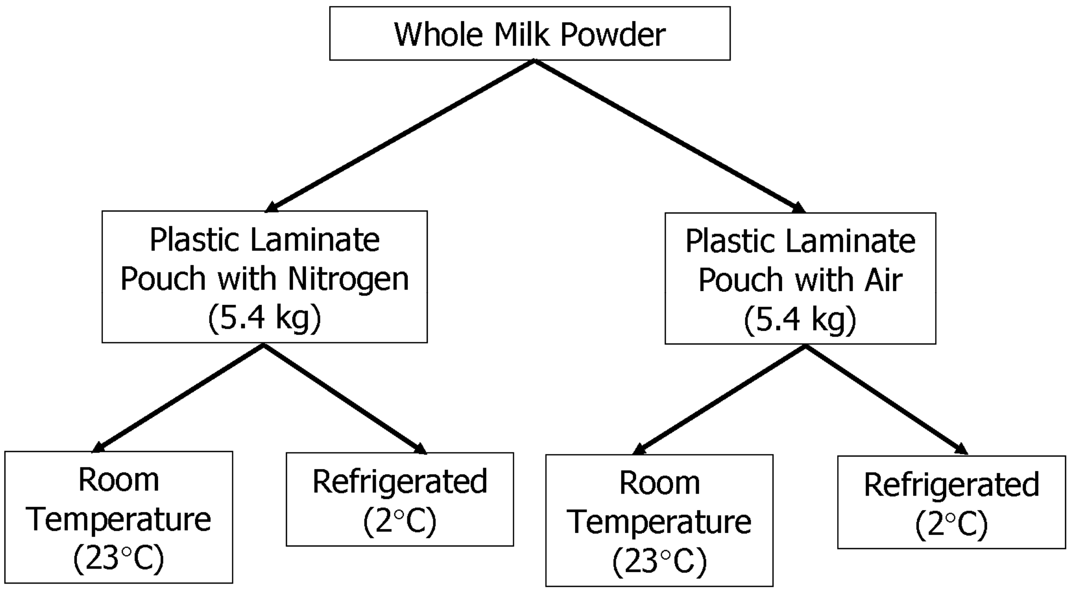


Figure 1. Experimental scheme for whole milk powder packaging study.

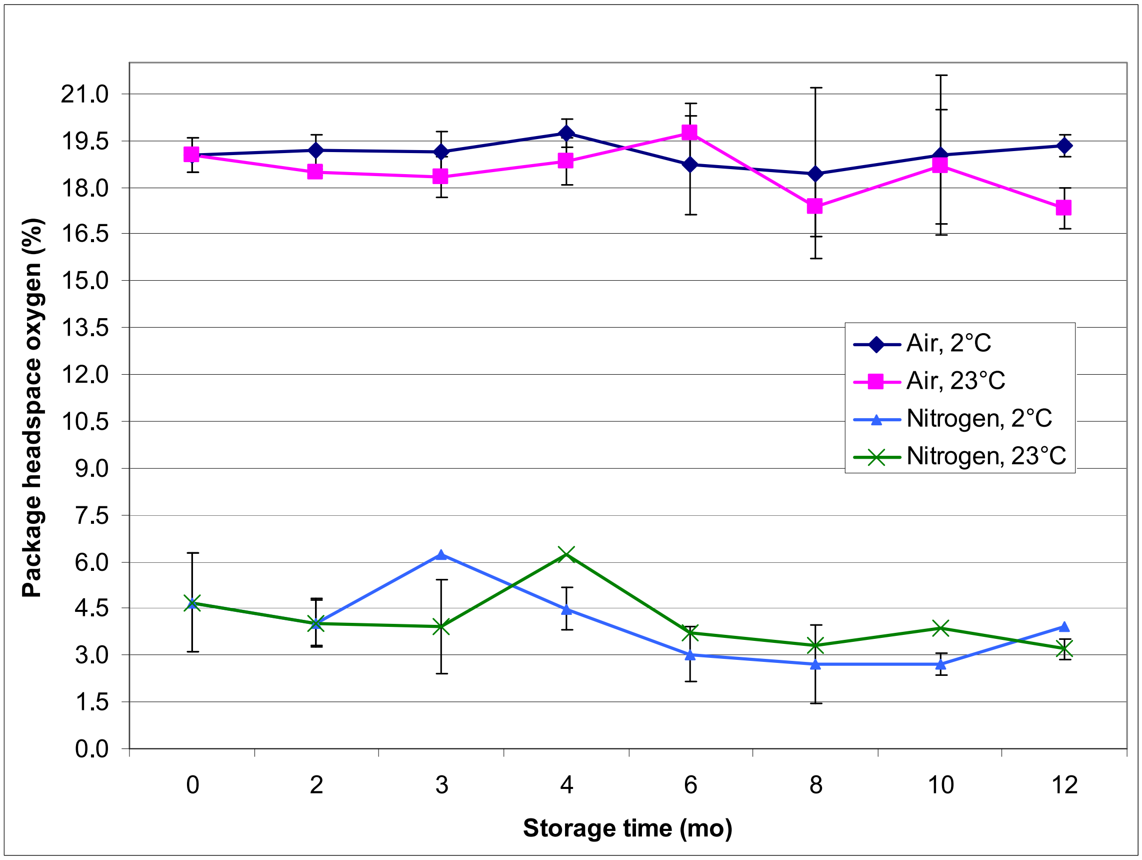


Figure 2. Oxygen content of whole milk powder stored in air or nitrogen at 2 ºC or 23 ºC. Bars represent standard errors of the means.

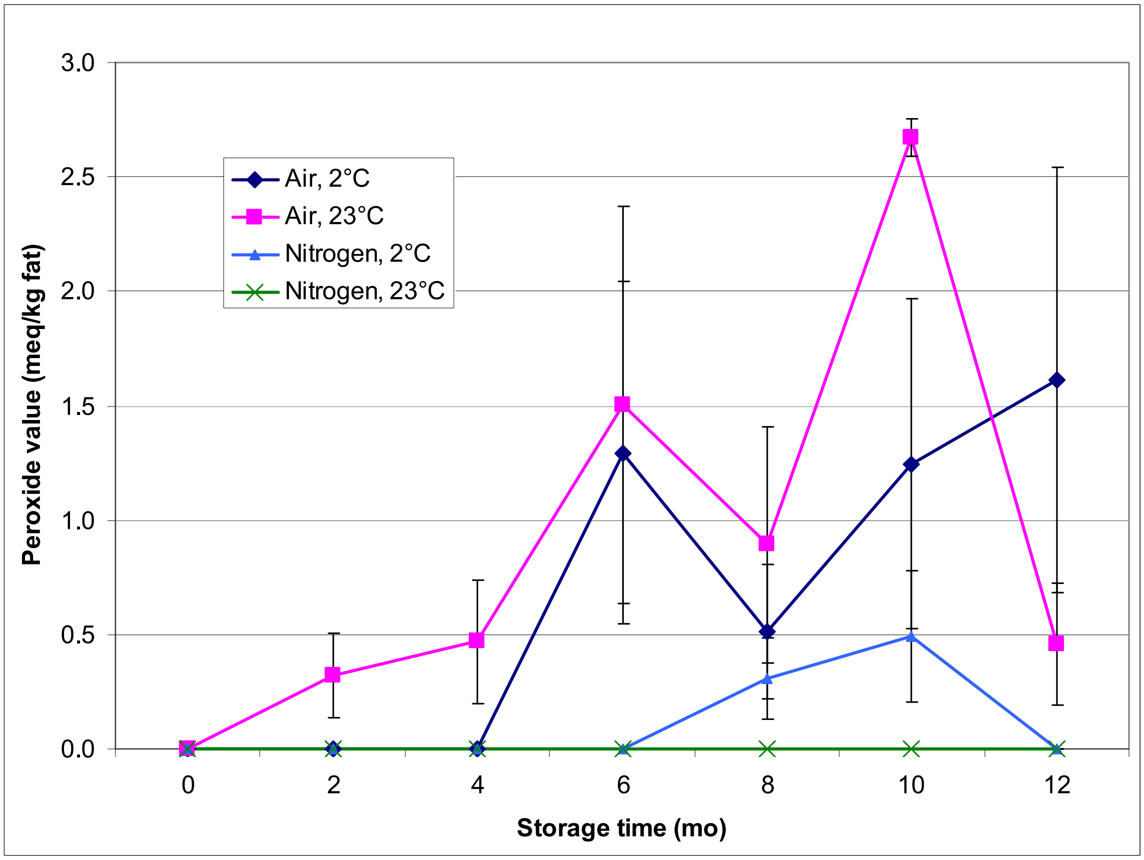


Figure 3. Development of peroxides in whole milk powder stored in air or nitrogen at 2 ºC or 23 ºC. Each point represents the mean from duplicate measurements. Bars represent standard errors of the means.

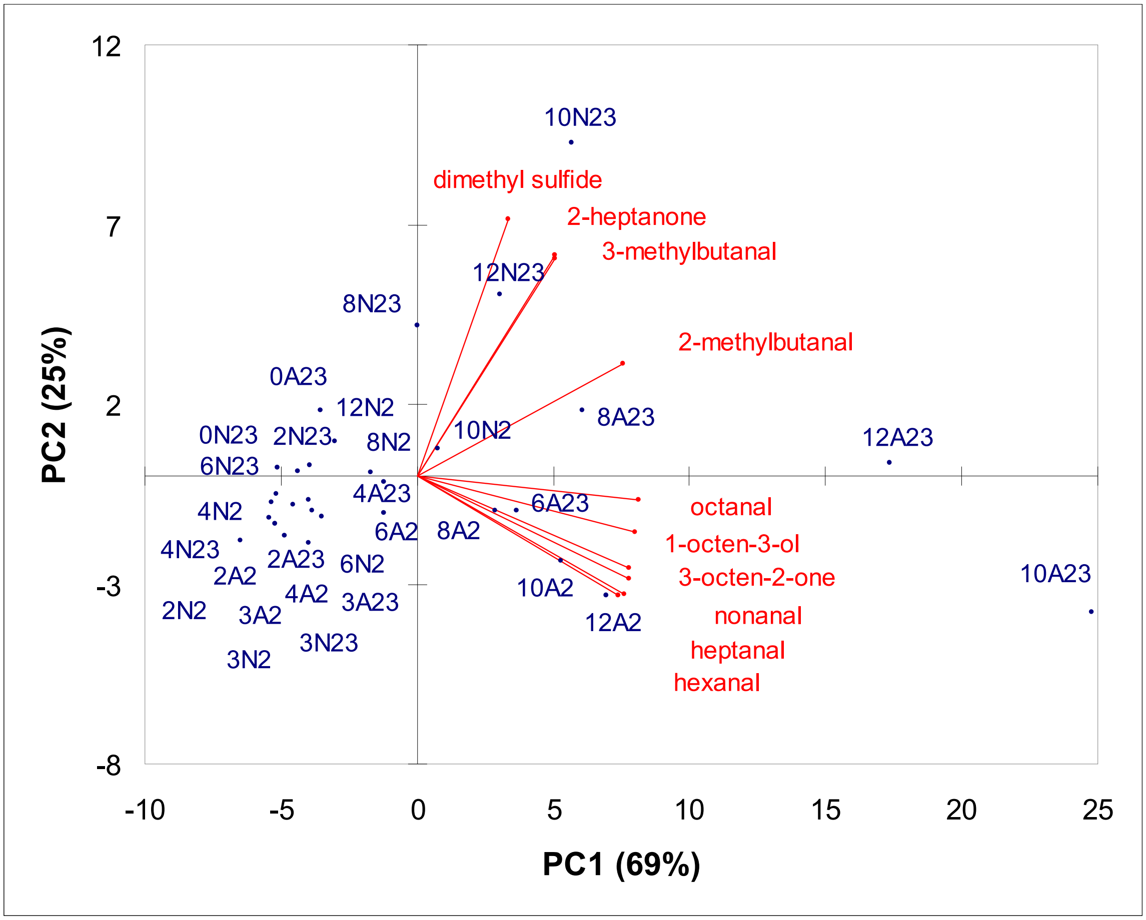


Figure 4. Principal component analysis biplot of whole milk powder volatiles. Codes represent storage time (0-12 mo), packaging (A = air, N = nitrogen), and storage temperature (2 ºC or 23 ºC).

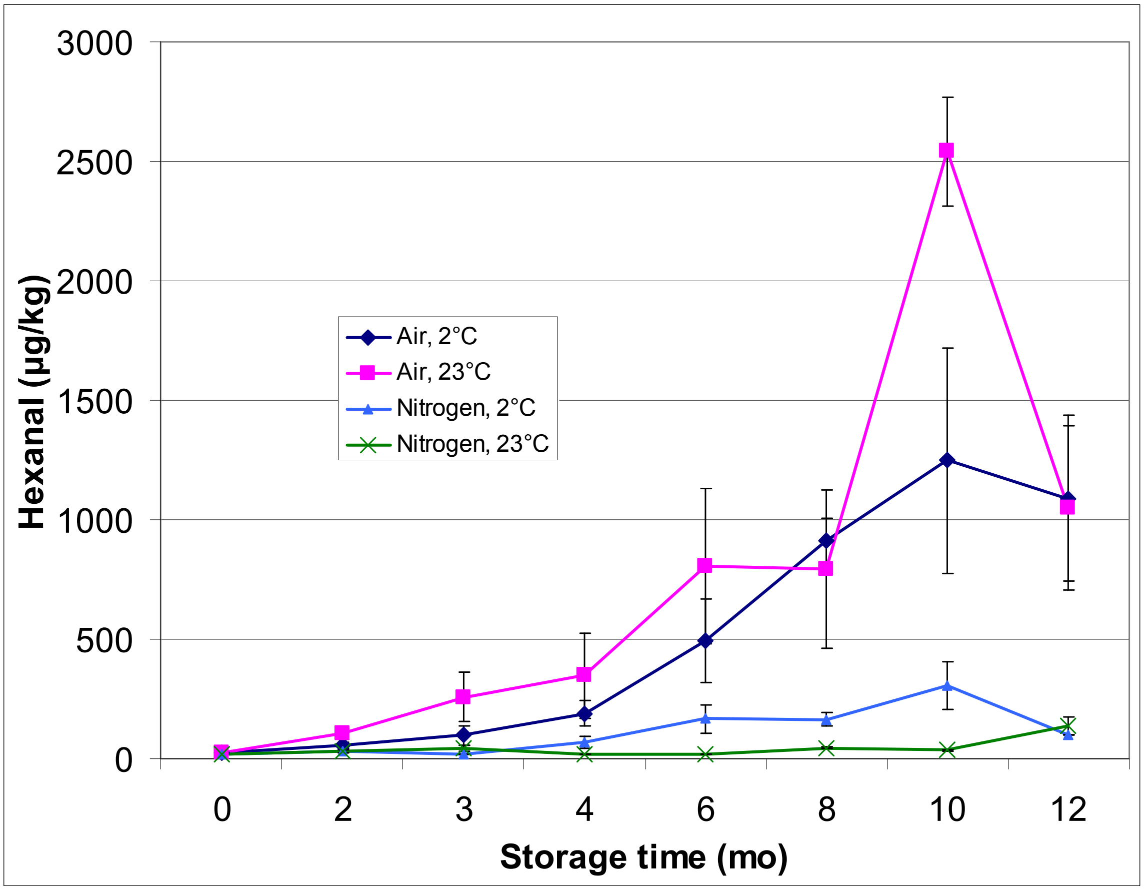


Figure 5. Development of hexanal in whole milk powder stored in air or nitrogen at 2 ºC or 23 ºC. Each point represents the mean from triplicate measurements. Bars represent standard errors of the means.

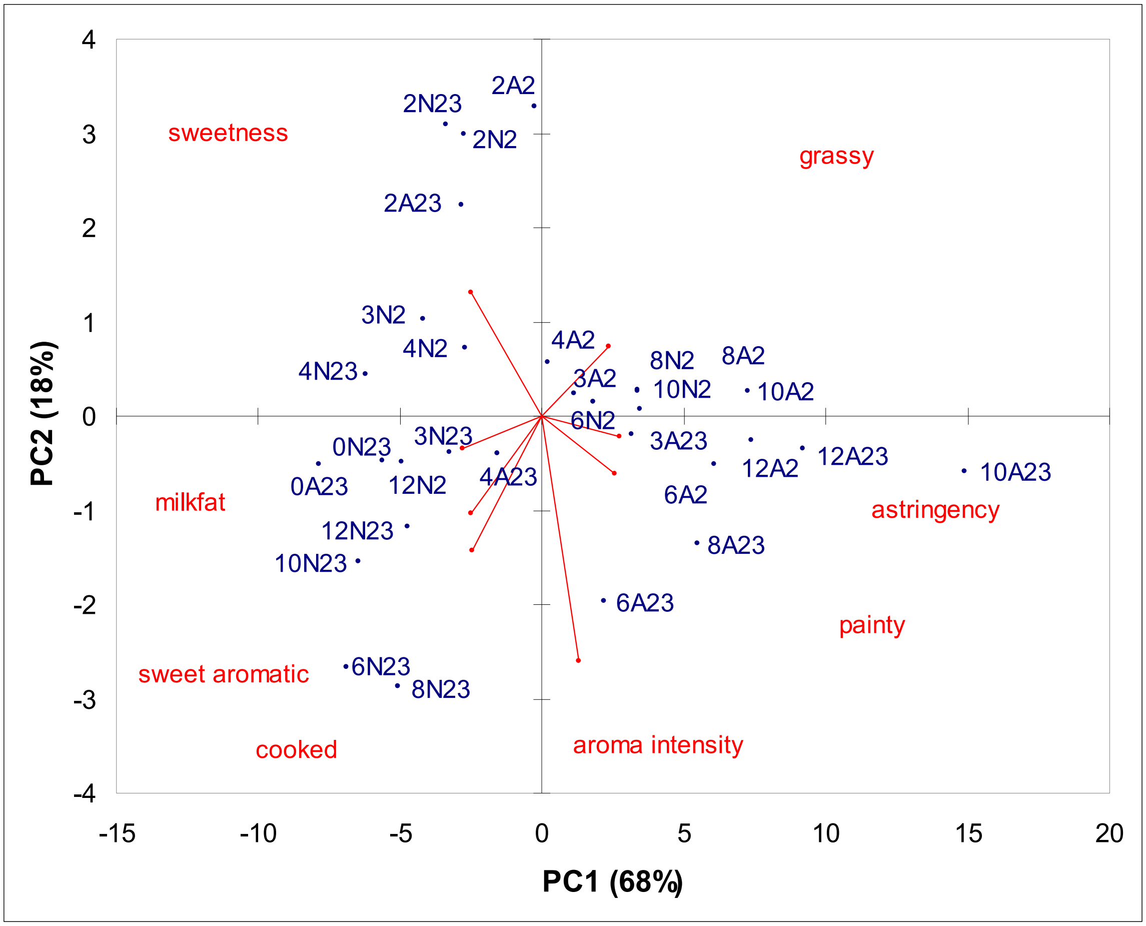


Figure 6. Principal component analysis biplot of whole milk powder descriptive sensory analysis. Codes represent storage time (0-12 mo), packaging (A = air, N = nitrogen), and storage temperature (2 ºC or 23 ºC).

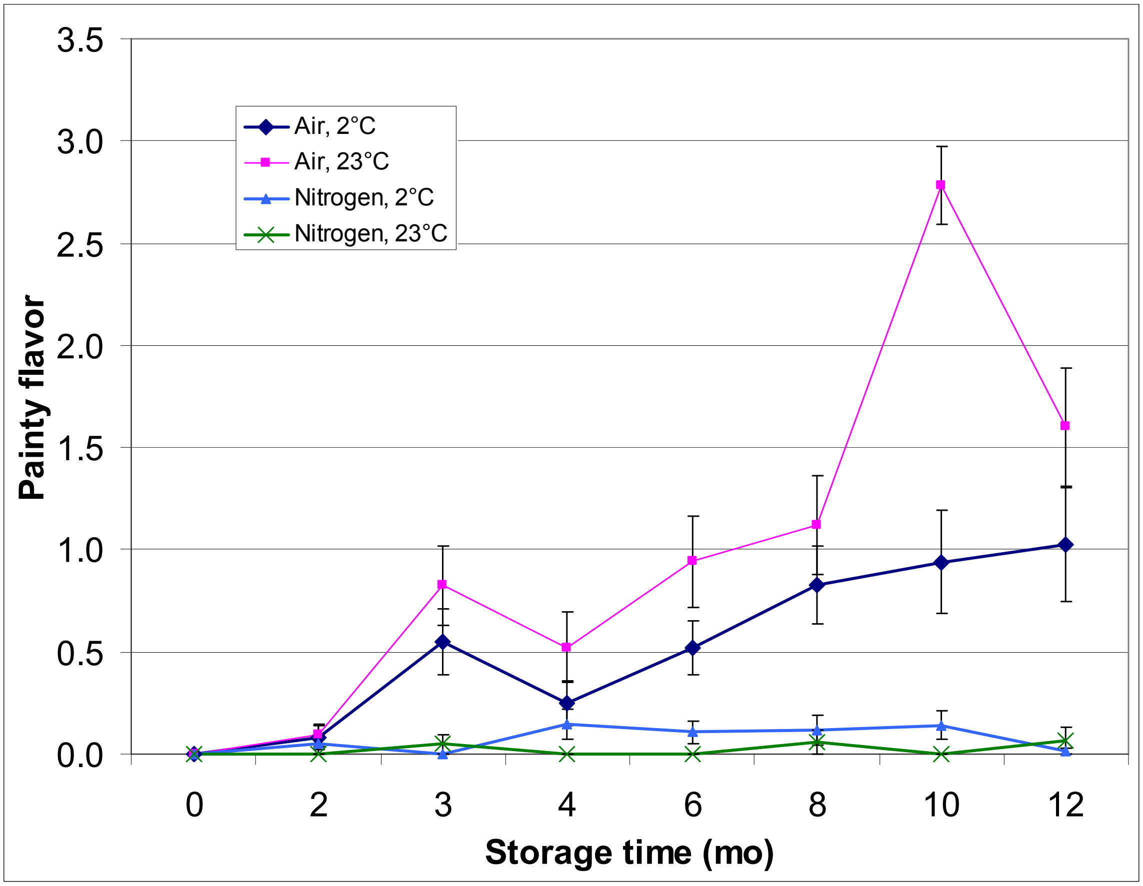


Figure 7. Development of painty flavor in whole milk powder stored in air or nitrogen at 2 ºC or 23 ºC. Each point represents the mean from duplicate measurements by each trained panelist. Bars represent standard errors of the means. Painty flavor intensity was scored on a 0 to 15 point universal Spectrum^{TM} intensity scale where 0 = absence of the attribute and 15 = extremely high intensity of the attribute (Meilgaard and others 1999). Most reconstituted dried dairy ingredient flavors fall between 0 and 4 on this scale (Drake and others 2003; Drake 2004; Carunchia Whetstine and others 2007; Wright and others 2008). Panel mean values below 0.5 are considered not detected (ND).

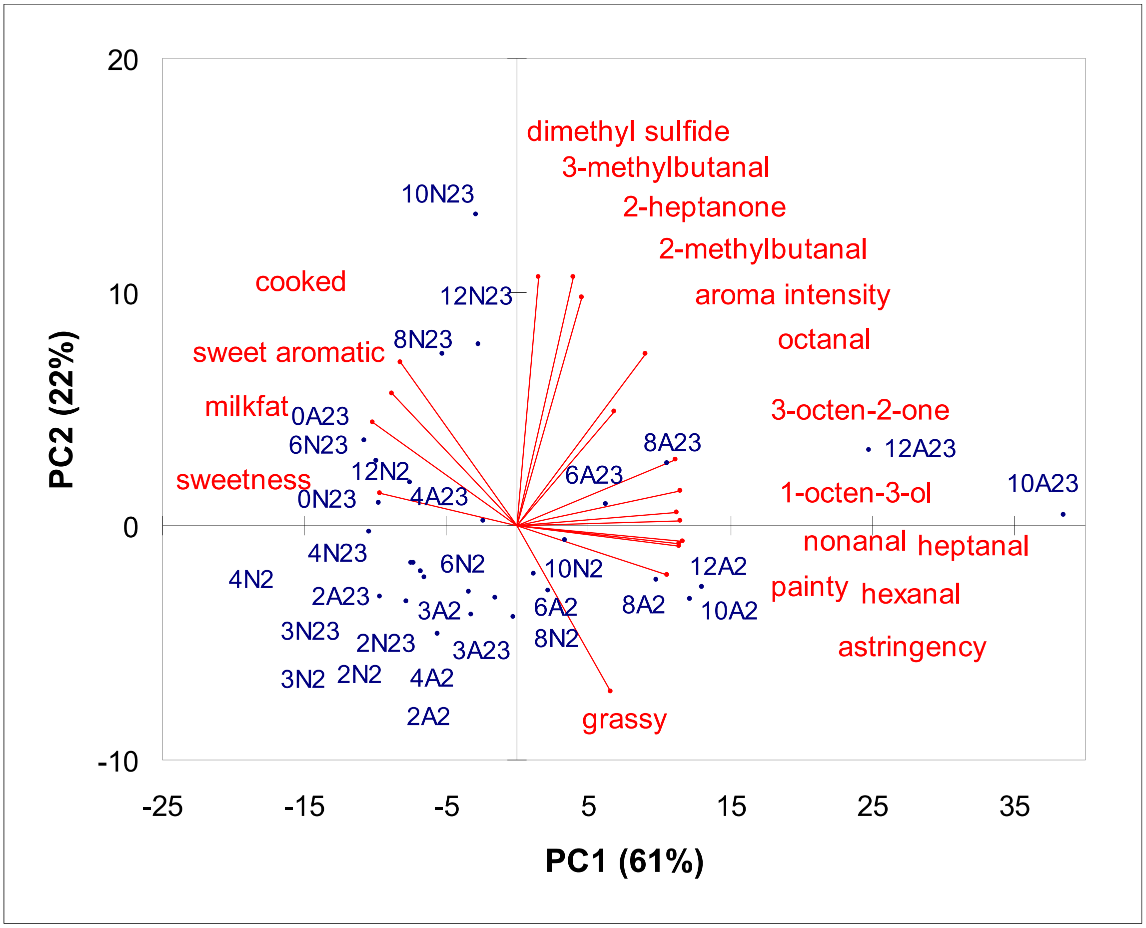


Figure 8. Principal component analysis biplot of whole milk powder volatiles and descriptive sensory analysis profiles. Codes represent storage time (0-12 mo), packaging (air or nitrogen), and storage temperature (2 ºC or 23 ºC).

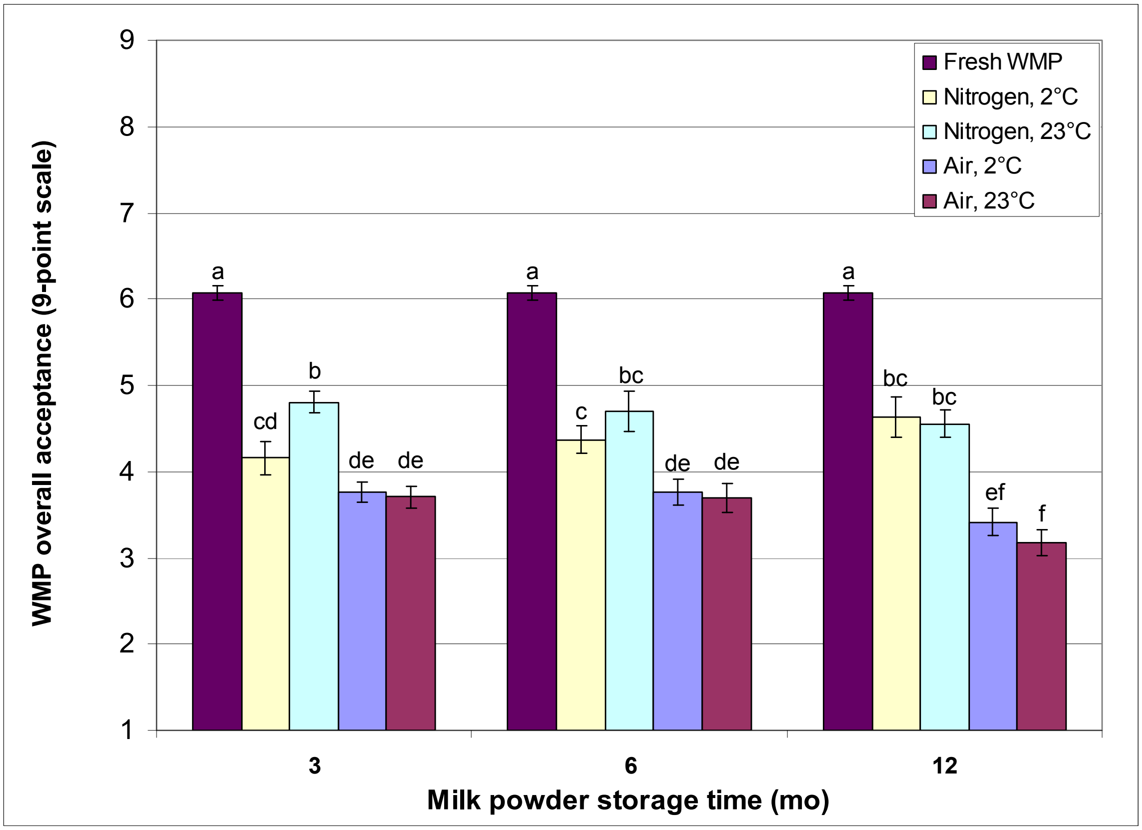


Figure 9. Consumer acceptance of reconstituted whole milk powder (WMP) stored up to one year in air or nitrogen at 2 ºC or 23 ºC, evaluated every 3 mo. Acceptance was scored on a 9-point hedonic scale where 1 = dislike extremely and 9 = like extremely. Different letters indicate significant differences ($p<0.05$).

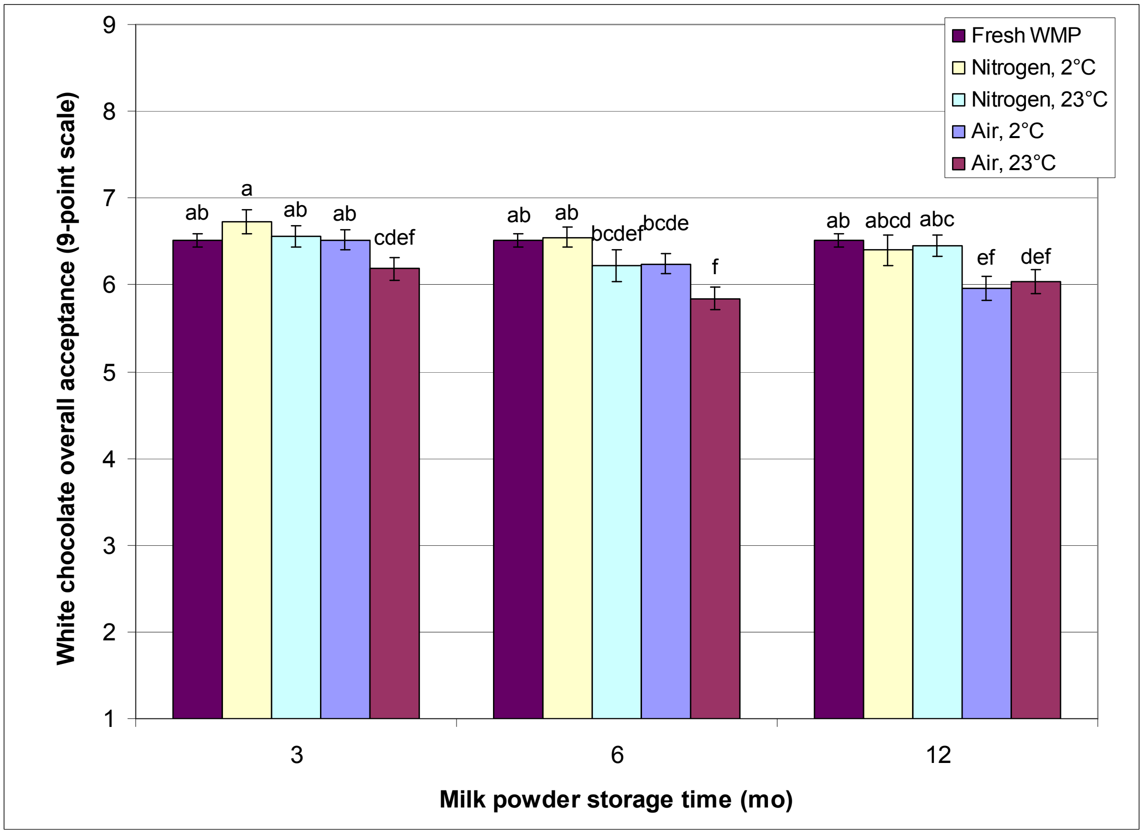


Figure 10. Consumer acceptance of white chocolate made from whole milk powder (WMP) stored up to a year in air or nitrogen at 2 ºC or 23 ºC, evaluated every 3 mo. Acceptance was scored on a 9-point hedonic scale where 1 = dislike extremely and 9 = like extremely. Different letters indicate significant differences ($p<0.05$).

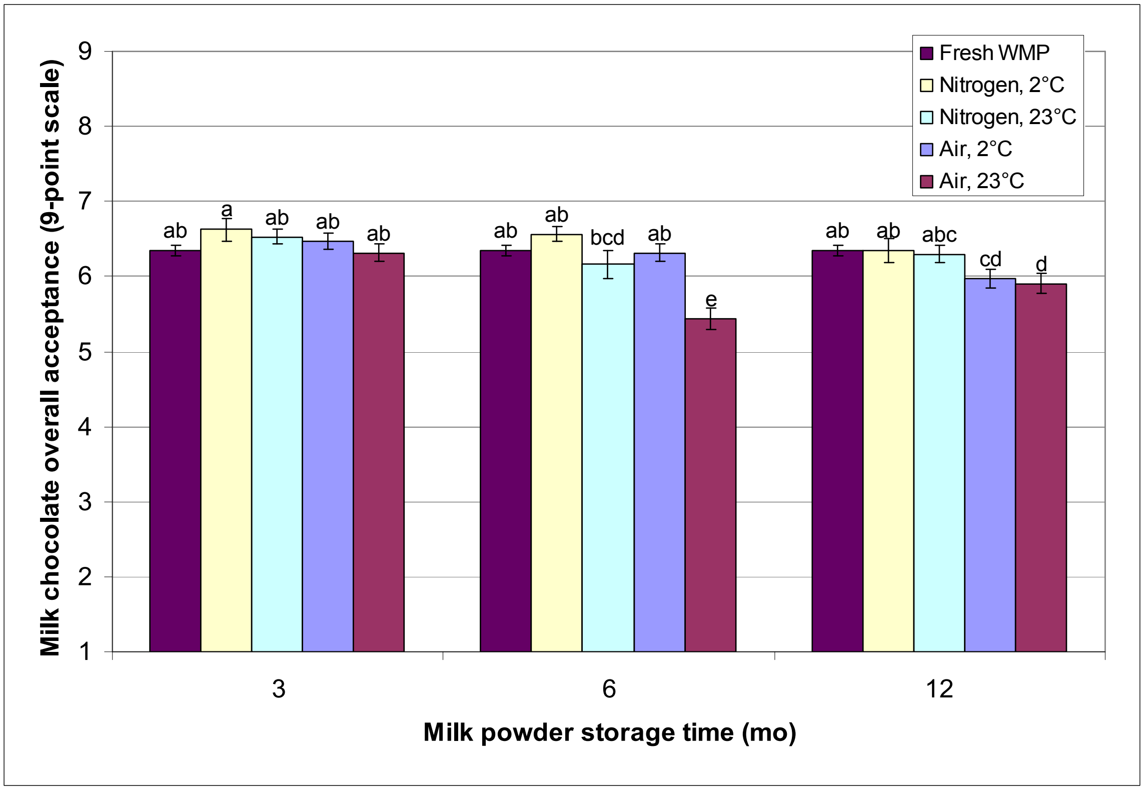


Figure 11. Consumer acceptance of milk chocolate made from whole milk powder (WMP) stored up to a year in air or nitrogen at 2 ºC or 23 ºC, evaluated every 3 mo. Acceptance was scored on a 9-point hedonic scale where 1 = dislike extremely and 9 = like extremely. Different letters indicate significant differences ($p<0.05$).

APPENDICIES

Appendix 1. Guide to reconstitute whole milk powder (WMP) to 10% solids not fat, based on International Dairy Federation Method 99C:1997.

% Fat	g WMP in 90 g water	g WMP in 500 g water(serves 10-12 for descriptive analysis)	g WMP in 2 gallons (7571 g) water (for consumer panel)	% Fat (reconstituted)
26.0	13.5	75.1	1136.8	3.9
26.1	13.5	75.2	1138.4	3.9
26.2	13.6	75.3	1139.9	3.9
26.3	13.6	75.4	1141.4	4.0
26.4	13.6	75.5	1143.0	4.0
26.5	13.6	75.6	1144.6	4.0
26.6	13.6	75.7	1146.1	4.0
26.7	13.6	75.8	1147.7	4.0
26.8	13.7	75.9	1149.2	4.1
26.9	13.7	76.0	1150.8	4.1
27.0	13.7	76.1	1152.4	4.1
27.1	13.7	76.2	1154.0	4.1
27.2	13.7	76.3	1155.6	4.2
27.3	13.8	76.4	1157.1	4.2
27.4	13.8	76.5	1158.7	4.2
27.5	13.8	76.6	1160.3	4.2
27.6	13.8	76.7	1161.9	4.2
27.7	13.8	76.8	1163.5	4.3
27.8	13.9	76.9	1165.2	4.3
27.9	13.9	77.1	1166.8	4.3
28.0	13.9	77.2	1168.4	4.3
28.1	13.9	77.3	1170.0	4.3
28.2	13.9	77.4	1171.6	4.4
28.3	13.9	77.5	1173.3	4.4
28.4	14.0	77.6	1174.9	4.4
28.5	14.0	77.7	1176.6	4.4
28.6	14.0	77.8	1178.2	4.5
28.7	14.0	77.9	1179.9	4.5
28.8	14.0	78.0	1181.5	4.5
28.9	14.1	78.1	1183.2	4.5
29.0	14.1	78.2	1184.9	4.5
29.1	14.1	78.4	1186.5	4.6
29.2	14.1	78.5	1188.2	4.6
29.3	14.1	78.6	1189.9	4.6
29.4	14.2	78.7	1191.6	4.6

Appendix 1 (continued). Guide to reconstitute whole milk powder (WMP) to 10% solids not fat, based on International Dairy Federation Method 99C:1997.

% Fat	g WMP in 90 g water	g WMP in 500 g water(serves 10-12 for descriptive analysis)	g WMP in 2 gallons (7571 g) water (for consumer panel)	% Fat (reconstituted)
29.5	14.2	78.8	1193.3	4.6
29.6	14.2	78.9	1194.9	4.7
29.7	14.2	79.0	1196.6	4.7
29.8	14.2	79.1	1198.4	4.7
29.9	14.3	79.3	1200.1	4.7
30.0	14.3	79.4	1201.8	4.8
30.1	14.3	79.5	1203.5	4.8
30.2	14.3	79.6	1205.2	4.8
30.3	14.3	79.7	1207.0	4.8
30.4	14.4	79.8	1208.7	4.9
30.5	14.4	79.9	1210.4	4.9
30.6	14.4	80.1	1212.2	4.9
30.7	14.4	80.2	1213.9	4.9
30.8	14.5	80.3	1215.7	4.9
30.9	14.5	80.4	1217.4	5.0
31.0	14.5	80.5	1219.2	5.0
31.1	14.5	80.6	1221.0	5.0
31.2	14.5	80.7	1222.7	5.0
31.3	14.6	80.9	1224.5	5.1
31.4	14.6	81.0	1226.3	5.1
31.5	14.6	81.1	1228.1	5.1
31.6	14.6	81.2	1229.9	5.1
31.7	14.6	81.3	1231.7	5.2
31.8	14.7	81.5	1233.5	5.2
31.9	14.7	81.6	1235.3	5.2
32.0	14.7	81.7	1237.1	5.2
32.1	14.7	81.8	1238.9	5.3
32.2	14.7	81.9	1240.8	5.3
32.3	14.8	82.1	1242.6	5.3
32.4	14.8	82.2	1244.4	5.3
32.5	14.8	82.3	1246.3	5.3
32.6	14.8	82.4	1248.1	5.4
32.7	14.9	82.5	1250.0	5.4
32.8	14.9	82.7	1251.9	5.4
32.9	14.9	82.8	1253.7	5.4

Appendix 1 (continued). Guide to reconstitute whole milk powder (WMP) to 10% solids not fat, based on International Dairy Federation Method 99C:1997.

% Fat	g WMP in 90 g water	g WMP in 500 g water(serves 10-12 for descriptive analysis)	g WMP in 2 gallons (7571 g) water (for consumer panel)	% Fat (reconstituted)
33.0	14.9	82.9	1255.6	5.5
33.1	14.9	83.0	1257.5	5.5
33.2	15.0	83.2	1259.3	5.5
33.3	15.0	83.3	1261.2	5.5
33.4	15.0	83.4	1263.1	5.6
33.5	15.0	83.5	1265.0	5.6
33.6	15.1	83.7	1266.9	5.6
33.7	15.1	83.8	1268.8	5.6
33.8	15.1	83.9	1270.8	5.7
33.9	15.1	84.0	1272.7	5.7
34.0	15.2	84.2	1274.6	5.7
34.1	15.2	84.3	1276.5	5.7
34.2	15.2	84.4	1278.5	5.8
34.3	15.2	84.6	1280.4	5.8
34.4	15.2	84.7	1282.4	5.8
34.5	15.3	84.8	1284.3	5.9
34.6	15.3	84.9	1286.3	5.9
34.7	15.3	85.1	1288.3	5.9
34.8	15.3	85.2	1290.3	5.9
34.9	15.4	85.3	1292.2	6.0
35.0	15.4	85.5	1294.2	6.0
35.1	15.4	85.6	1296.2	6.0
35.2	15.4	85.7	1298.2	6.0
35.3	15.5	85.9	1300.2	6.1
35.4	15.5	86.0	1302.2	6.1
35.5	15.5	86.1	1304.3	6.1
35.6	15.5	86.3	1306.3	6.1
35.7	15.6	86.4	1308.3	6.2
35.8	15.6	86.5	1310.3	6.2
35.9	15.6	86.7	1312.4	6.2
36.0	15.6	86.8	1314.4	6.3
36.1	15.6	86.9	1316.5	6.3
36.2	15.7	87.1	1318.6	6.3
36.3	15.7	87.2	1320.6	6.3
36.4	15.7	87.4	1322.7	6.4

Appendix 1 (continued). Guide to reconstitute whole milk powder (WMP) to 10% solids not fat, based on International Dairy Federation Method 99C:1997.

% Fat	g WMP in 90 g water	g WMP in 500 g water(serves 10-12 for descriptive analysis)	g WMP in 2 gallons (7571 g) water (for consumer panel)	% Fat (reconstituted)
36.5	15.7	87.5	1324.8	6.4
36.6	15.8	87.6	1326.9	6.4
36.7	15.8	87.8	1329.0	6.4
36.8	15.8	87.9	1331.1	6.5
36.9	15.8	88.0	1333.2	6.5
37.0	15.9	88.2	1335.3	6.5
37.1	15.9	88.3	1337.4	6.6
37.2	15.9	88.5	1339.6	6.6
37.3	15.9	88.6	1341.7	6.6
37.4	16.0	88.7	1343.8	6.6
37.5	16.0	88.9	1346.0	6.7
37.6	16.0	89.0	1348.1	6.7
37.7	16.1	89.2	1350.3	6.7
37.8	16.1	89.3	1352.5	6.8
37.9	16.1	89.5	1354.7	6.8
38.0	16.1	89.6	1356.8	6.8
38.1	16.2	89.8	1359.0	6.8
38.2	16.2	89.9	1361.2	6.9
38.3	16.2	90.0	1363.4	6.9
38.4	16.2	90.2	1365.7	6.9
38.5	16.3	90.3	1367.9	7.0
38.6	16.3	90.5	1370.1	7.0
38.7	16.3	90.6	1372.3	7.0
38.8	16.3	90.8	1374.6	7.0
38.9	16.4	90.9	1376.8	7.1
39.0	16.4	91.1	1379.1	7.1
39.1	16.4	91.2	1381.4	7.1
39.2	16.4	91.4	1383.6	7.2
39.3	16.5	91.5	1385.9	7.2
39.4	16.5	91.7	1388.2	7.2
39.5	16.5	91.8	1390.5	7.3
39.6	16.6	92.0	1392.8	7.3
39.7	16.6	92.1	1395.1	7.3
39.8	16.6	92.3	1397.4	7.3
39.9	16.6	92.4	1399.7	7.4
40.0	16.7	92.6	1402.1	7.4

Appendix 2. Sample descriptive analysis ballot.

Name____________________________ WMP Flavor Study 28 Nov 2007

	CODE	WU-1	WU-2	WU-3								
Aroma	Aroma Intensity	2	2.1	2.4								
	Aroma Quality	Sweet	Grassy	Grassy; Painty								
Flavor	Cooked/ Sulfurous	2.8	2.1	1.7								
	Milkfat	2.8	2.0	1.6								
	Sweet Aromatic/ Caramelized	2.8	1.9	1.5								
	Grassy/hay	-	2.5	1.5								
	Painty/Fatty/ Fryer Oil	-	-	2.6								
	Other	-	-	-								
Basic	Sweetness	2.2	2	1.7								
	Other	-	-	-								
	Astringency	1	1.2	1.5								

Appendix 3. Consumer panel questionnaire for reconstituted whole milk powder.

Welcome to Milk Panel!

Panelist Code: ________________________

Panelist Name: ____

<u>Review Instructions</u>

Today you will be evaluating
6 samples of milk.

Please take a drink of water between samples to refresh your sense of taste.

There are 4 questions about each sample.

There are no right or wrong answers. Just give us your honest opinion.

Appendix 3 (continued). Consumer panel questionnaire for reconstituted whole milk powder.

Question # 1.

Do you have any allergies to milk? (no relation to lactose intolerance)

- ☐ Yes
- ☐ No

Question # 2.

Review Instructions

SUBJECT CONSENT TO SENSORY EVALUATION

MILK

11/13/2007

I agree to participate in the sensory evaluation of **milk** for the Department of Food Science at North Carolina State University. I understand that participation is voluntary and that I may withdraw my participation at any time. I also understand that information I provide is confidential and that results will not be associated with my name.

If you have any allergies to milk you should NOT participate in this study.

Please enter your name below. By entering your name below, you are providing consent to participate in today's evaluation of **milk**.

Please click on Instructions (below) if you would like to view the consent again.

____

____

____

Appendix 3 (continued). Consumer panel questionnaire for reconstituted whole milk powder.

Question # 3.

Please indicate your gender.

- ☐ Male
- ☐ Female

Question # 4.

Please indicate your age.

- ☐ 18 or younger
- ☐ 19-24
- ☐ 25-29
- ☐ 30-39
- ☐ 40-49
- ☐ 50-59
- ☐ 60 and over

Question # 5.

How often do you typically consume milk?

- ☐ Never
- ☐ Less than once per month (occasionally)
- ☐ At least once per month
- ☐ At least 2-3 times per month
- ☐ At least once per week
- ☐ Several times per week
- ☐ Everyday

Question # 6.

What do you use milk for? Check all that apply.

- ☐ To drink
- ☐ With cereal
- ☐ Milkshake/smoothie
- ☐ Baking
- ☐ Soups/sauces
- ☐ Do not use
- ☐ Other

Appendix 3 (continued). Consumer panel questionnaire for reconstituted whole milk powder.

Question # 7.

Please list other ways that you use milk:

____

____

Question # 8.

What type of milk do you typically consume? Check all that apply:

- ☐ Nonfat (skim) milk
- ☐ 1% milk
- ☐ 2% milk
- ☐ Whole milk
- ☐ Other

Question # 9.

Please list what other types of milk you consume:

____

____

Question # 10 - Sample ______

Please indicate how you feel about the OVERALL ACCEPTANCE of sample _____.

Overall Acceptance

Overall Acceptance

dislike extremely	dislike very much	dislike moderately	dislike slightly	neither like nor dislike	like slightly	like moderately	like very much	like extremely
1	2	3	4	5	6	7	8	9

Question # 11 - Sample ______

Please indicate how you feel about the following attributes in sample _____.

Appendix 3 (continued). Consumer panel questionnaire for reconstituted whole milk powder.

Flavor

Flavor

dislike extremely	dislike very much	dislike moderately	dislike slightly	neither like nor dislike	like slightly	like moderately	like very much	like extremely
1	2	3	4	5	6	7	8	9

Mouthfeel/Texture

Mouthfeel/Texture

dislike extremely	dislike very much	dislike moderately	dislike slightly	neither like nor dislike	like slightly	like moderately	like very much	like extremely
1	2	3	4	5	6	7	8	9

Question # 12 - Sample ______

Please rate the intensity of the following attributes in sample _____.

Freshness Intensity

Freshness Intensity

low				moderate		high		
1	2	3	4	5	6	7	8	9

Question # 13 - Sample ______

Please share any comments you have about sample _____.

____

____

THANK YOU!

Please return your samples
to the booth turntable
and get your treat
from the workers in the kitchen.

Come back tomorrow to taste milk chocolate!

Appendix 4. Consumer panel questionnaire for chocolate.

Welcome to Chocolate Panel!

We are glad to have you participate!

Panelist Code: ______________________

Panelist Name: ____

Question # 1.

Do you have any allergies to milk or soy?

m Yes
m No

Question # 2.

Review Instructions

SUBJECT CONSENT TO SENSORY EVALUATION
Chocolate

2/14/2007
I agree to participate in a consumer panel on **chocolate** for the Department of Food Science at North Carolina State University. I understand that participation is voluntary and that I may withdraw my participation at any time. I also understand that information I provide is confidential and that results will not be associated with my name.

If you have any allergies to MILK or SOY you should NOT participate in this study.

Appendix 4 (continued). Consumer panel questionnaire for chocolate.

Please enter your name below. By entering your name below, you are providing consent to participate in today's evaluation of **chocolate** which contains milk and soy ingredients.
Please click on Instructions (below) if you would like to view the consent again.

____

____

Question # 3.

Please indicate your gender.

- ☐ Male
- ☐ Female

Question # 4.

Please indicate your age.

- ☐ 18 or younger
- ☐ 19-24
- ☐ 25-29
- ☐ 30-39
- ☐ 40-49
- ☐ 50-59
- ☐ 60 and over

Question # 5.

What types of chocolate do you typically consume? (check all that apply)

- ☐ Milk Chocolate
- ☐ Semi-sweet Chocolate
- ☐ Dark Chocolate
- ☐ White Chocolate
- ☐ Other

Appendix 4 (continued). Consumer panel questionnaire for chocolate.

Question # 6.

Please explain what other types of chocolate you consume:

____

____

Question # 7.

How often do you typically consume any kind of chocolate (milk chocolate, semi-sweet, dark, white, etc.)?

- ☐ Never
- ☐ Less than once per month (occasionally)
- ☐ At least once per month
- ☐ At least 2-3 times per month
- ☐ At least once per week
- ☐ Several times per week
- ☐ Everyday

Question # 8 - Sample ______

Please indicate how you feel about the OVERALL ACCEPTANCE of sample _____

Overall Acceptance

Overall Acceptance

dislike extremely	dislike very much	dislike moderately	dislike slightly	neither like nor dislike	like slightly	like moderately	like very much	like extremely
1	2	3	4	5	6	7	8	9

Question # 9 - Sample ______

Please indicate how you feel about the following attributes in sample _____.

Appearance

Appearance

dislike extremely	dislike very much	dislike moderately	dislike slightly	neither like nor dislike	like slightly	like moderately	like very much	like extremely
1	2	3	4	5	6	7	8	9

Appendix 4 (continued). Consumer panel questionnaire for chocolate.

Milk Chocolate Flavor

Milk Chocolate Flavor

dislike extremely	dislike very much	dislike moderately	dislike slightly	neither like nor dislike	like slightly	like moderately	like very much	like extremely
1	2	3	4	5	6	7	8	9

Mouthfeel/Texture

Mouthfeel/Texture

dislike extremely	dislike very much	dislike moderately	dislike slightly	neither like nor dislike	like slightly	like moderately	like very much	like extremely
1	2	3	4	5	6	7	8	9

Question # 10 - Sample ______

Please rate the intensity of the following attributes in sample _____.

Chocolate Flavor Intensity

Chocolate Flavor Intensity

low				moderate		high		
1	2	3	4	5	6	7	8	9

Freshness Intensity

Freshness Intensity

low				moderate		high		
1	2	3	4	5	6	7	8	9

Question # 11 - Sample ______

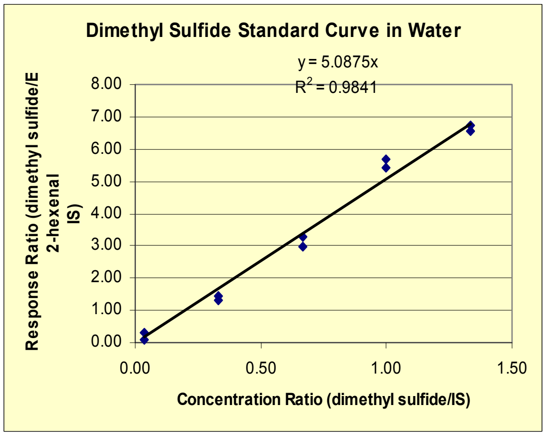
Do you have any comments about sample _____?

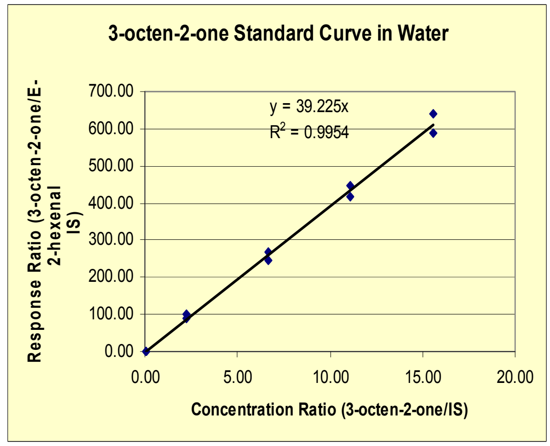
____

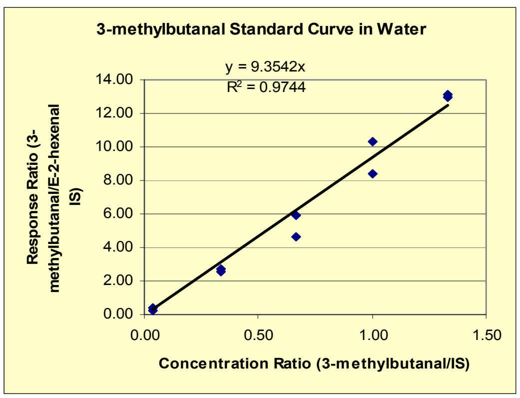
THANK YOU!

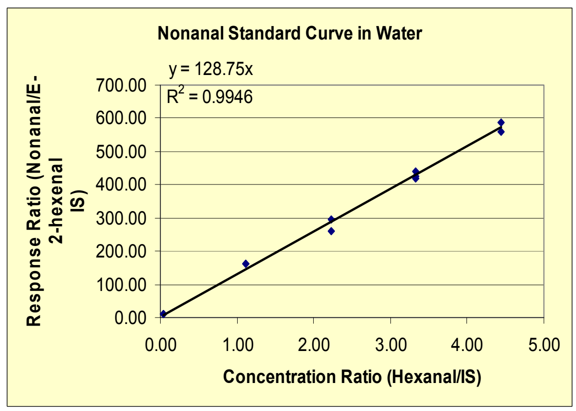
Please return any unwanted samples
to the booth turntable and collect your treat from the workers in the kitchen.
Come back tomorrow to taste white chocolate.

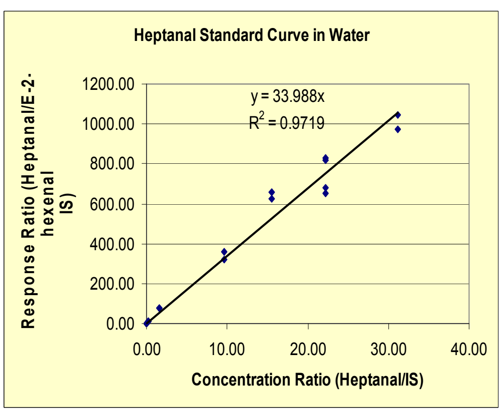
Appendix 5. Standard curve summary for selected volatiles, based on E-2-hexenal internal standard.

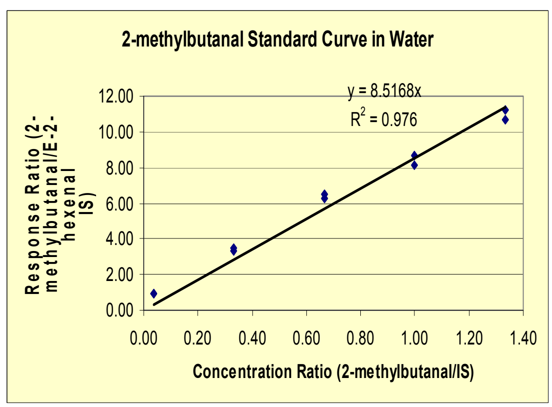




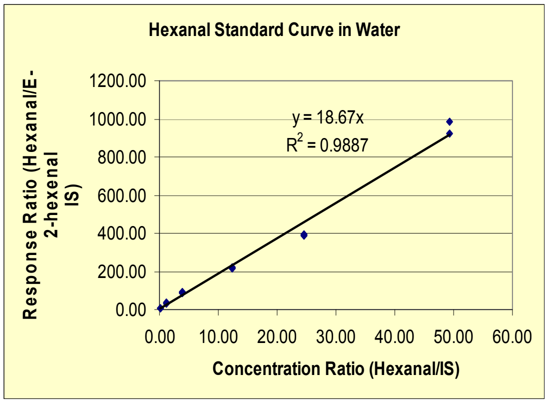


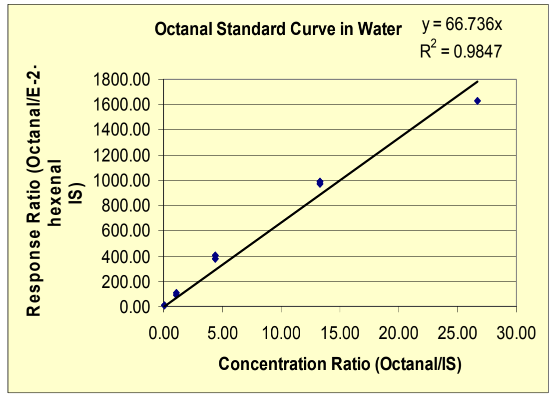


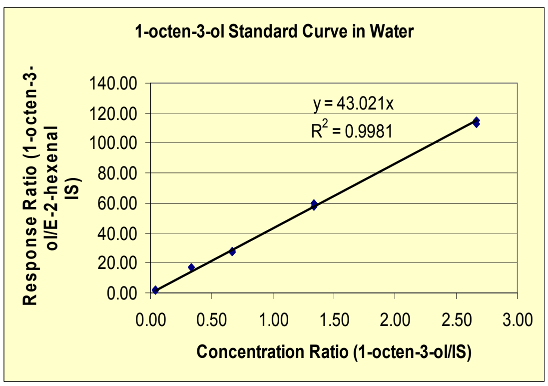


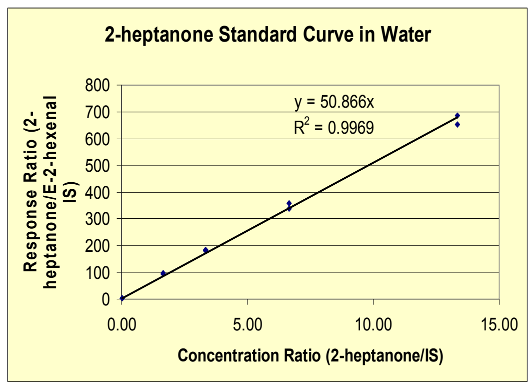


Appendix 5 (continued). Standard curve summary for selected volatiles, based on E-2-hexenal internal standard.









Appendix 6. Selected volatiles in whole milk powder from four facilities stored up to 12 months. Means represent data pooled from five shipments collected over a year period.

Facility	Time	1-octen-3-ol	2-heptanone	2-methyl-butanal	3-methyl-butanal	3-octen-2-one
A	**0**	0.2ij	2.7o	1.0m	0.9hi	0.04kl
	2	0.9hij	3.8mno	2.2klm	2.2gh	0.4kl
	4	1.7fghij	4.9lmno	3.1ijkl	3.0fg	2.4jkl
	6	1.9fghij	6.2jklm	3.3ijk	2.6fg	5.8ijkl
	8	2.4efgh	7.8hijkl	4.0hij	2.6fg	10.5ghijk
	10	3.6def	10.7defgh	5.9defg	3.2efg	18.2efg
	12	7.2b	17.9b	7.3bcd	3.8def	35.6bc
B	**0**	0.01j	2.9no	0.8m	0.7i	0.03l
	2	0.2ij	5.4klmno	1.7lm	2.1gh	0.3kl
	4	0.6hij	6.0jklmn	1.9klm	1.9ghi	1.3kl
	6	0.7hij	6.9ijkl	2.6jkl	2.0ghi	3.3jkl
	8	1.6ghij	8.3ghijk	3.4hijk	2.3g	7.7hijkl
	10	1.9fghi	12.0cdef	4.5ghi	2.8fg	11.9ghij
	12	3.8de	19.2b	6.9cde	3.7def	22.8def
C	**0**	0.4ij	8.0ghijkl	3.3ijk	2.4g	0.4kl
	2	0.3ij	11.0defg	4.6ghi	3.7def	1.5kl
	4	0.9hij	11.4cdef	6.5def	5.2ab	3.8ghijk
	6	1.4ghij	12.6cd	5.7efg	4.4bcde	7.3hijkl
	8	3.0defg	14.5c	5.7efg	3.9cdef	14.4fghi
	10	4.2cd	19.4b	8.8ab	4.7bcd	25.4cde
	12	7.4b	26.3a	8.6ab	5.1abc	46.0b
D	**0**	0.1ij	5.4klmno	2.1klm	2.1ghi	0.1kl
	2	0.3ij	6.3ijklm	2.9jkl	3.2efg	0.6kl
	4	0.7hij	8.8fghij	5.0fgh	5.5ab	2.3jkl
	6	1.6fghij	9.2efghi	4.7ghi	4.4bcde	6.9hijkl
	8	3.6def	12.4cde	6.0defg	4.7abcd	17.0efgh
	10	5.9bc	19.0b	9.0a	6.1a	33.0cd
	12	9.9a	24.0a	8.4abc	4.7abcd	62.2a

Different letters within a column are significantly different ($p<0.05$).

Appendix 6 (continued). Selected volatiles in whole milk powder from four facilities stored up to 12 months. Means represent data pooled from five shipments collected over a year period.

Facility	Time	dimethyl sulfide	heptanal	hexanal	nonanal	octanal
A	**0**	4.9fg	1.6l	7.6l	0.5k	0.2i
	2	8.5cdefg	15.7jkl	121.3kl	0.9jk	0.6i
	4	11.8	33.6ijkl	344.7ijkl	1.5ijk	5.1i
	6	6.7efg	62.2ghij	571.0ghi	3.3fghijk	10.6fghi
	8	6.0efg	94.7defgh	931.2def	4.9defghi	17.4efgh
	10	7.2defg	132.5cde	1083.5cde	6.7def	25.4de
	12	10.1bcde	246.9a	1760.5ab	14.2b	55.2b
B	**0**	4.1g	2.7l	11.4l	1.5ijk	0.5i
	2	4.9fg	10.3kl	85.9kl	1.3jk	0.8i
	4	5.8efg	17.3jkl	179.8jkl	1.4jk	1.5i
	6	8.0cdefg	31.8ijkl	346.7ijkl	2.0hijk	6.3hi
	8	4.9fg	49.1hijkl	594.3fghi	3.3fghijk	11.3fghi
	10	7.5defg	67.6fghi	772.9efg	4.4efghij	17.1efgh
	12	9.1bcdef	114.1def	1235.6cd	8.2cd	27.4cde
C	**0**	6.1efg	3.6kl	23.7l	0.6k	0.2i
	2	6.5efg	19.2jkl	160.2kl	1.3jk	1.3i
	4	8.9bcdef	27.1ijkl	387.0hijk	1.6hijk	3.0i
	6	8.6cdefg	52.0hijk	508.8ghij	3.0ghijk	10.5ghi
	8	9.8bcde	89.2efgh	917.9def	5.1defgh	21.5efg
	10	8.0cdefg	137.6cd	1385.3c	7.5de	33.4cd
	12	7.0efg	198.4ab	1840.1a	14.1b	53.6b
D	**0**	7.6cdefg	1.1l	8.2l	0.3k	0.0i
	2	9.9bcde	6.7kl	58.9kl	0.8jk	0.3i
	4	13.8ab	25.9ijkl	295.0ijkl	1.2jk	3.8i
	6	18.3a	64.9fghij	752.4efgh	3.4fghijk	10.7fghi
	8	13.8ab	110.5defg	949.7def	5.9defg	23.0def
	10	12.7bc	167.1bc	1431.9bc	11.4bc	39.2c
	12	9.4bcdef	248.4a	1971.3a	19.7a	68.9a

Different letters within a column are significantly different ($p<0.05$).

Appendix 7. Flavor intensities of whole milk powder from four facilities stored up to 12 months. Means represent data pooled from five shipments collected over a year period.

Facility	Time	aroma intensity	sweet aromatic/caramelized	milkfat	sweetness	cooked/sulfurous	grassy/hay	fatty/fryer oil/painty	astringency
A	0	1.95j	2.25c	2.54b	2.04defg	2.36de	0.75k	ND	1.21efgh
	2	2.04ghi	2.002fghi	2.21cd	2.11abcd	2.13ghi	1.97bc	ND	1.13hijk
	4	2.08efgh	2.00ghi	2.13def	2.04efg	2.15gh	1.96bc	0.66ij	1.14ghijk
	6	2.10defg	1.86klmn	1.99hijk	1.99ghi	2.03jkl	1.99bc	1.06gh	1.17fghi
	8	2.15bcd	1.69q	1.86lm	1.94ijk	1.88n	2.14bc	1.49de	1.33abc
	10	2.15bcd	1.76opq	1.88lm	1.87l	1.93mn	2.12b	1.65cd	1.28cd
	12	2.21a	1.72pq	1.86m	1.90kl	1.90mn	2.45a	2.08ab	1.34abc
B	0	2.09efgh	2.44b	2.70a	2.08bcde	2.70a	ND	ND	1.14ghijk
	2	1.94j	2.27c	2.47b	2.17a	2.38de	0.82jk	ND	1.05l
	4	2.07fgh	2.08efg	2.21cd	2.08bcde	2.19gh	1.24ghi	0.61ij	1.15ghijk
	6	2.03hi	1.98ghij	2.14de	2.01fgh	2.11hij	1.48f	0.65ij	1.15ghijk
	8	2.06gh	1.86klmn	2.00ghij	2.00gh	1.97klm	1.48f	1.06gh	1.26de
	10	2.09efg	1.92ijkl	2.05efgh	1.90kl	2.04ijk	1.40fgh	1.16fgh	1.21ef
	12	2.12cdef	1.88jklm	2.04fghi	1.94ijk	1.99klm	1.70de	1.33efg	1.28cd
C	0	2.04ghi	2.61a	2.73a	2.13abc	2.60ab	ND	ND	1.07kl
	2	1.95j	2.25c	2.49b	2.14ab	2.54bc	0.75k	ND	1.05l
	4	2.07fgh	2.18cde	2.30c	2.07cdef	2.34de	1.22hi	ND	1.10jkl
	6	2.14bcde	1.94hijk	2.11def	1.99ghi	2.19gh	1.49ef	1.23efg	1.6fghij
	8	2.18ab	1.78nopq	1.91klm	1.93jkl	2.02kl	1.46fgh	1.58d	1.32bc
	10	2.16bc	1.83lmno	1.95ijkl	1.89kl	1.96lmn	1.55ef	1.96bc	1.26de
	12	2.22a	1.79nopq	1.92jklm	1.8kl	1.95lmn	1.90cd	2.32a	1.35ab
D	0	2.10cdefg	2.47b	2.48b	2.15ab	2.44cd	ND	ND	1.11ijkl
	2	1.99ij	2.23cd	2.46b	2.15ab	2.30ef	0.69	ND	1.10jkl
	4	2.06gh	2.13def	2.19d	2.04efg	2.22fg	1.04ij	ND	1.15fghijk
	6	2.08efgh	2.05fgh	2.10defg	1.98ghij	2.14ghi	1.13i	0.86hi	1.21efg
	8	2.10defg	1.81mnop	1.93jklm	1.96hij	1.97klmn	1.61ef	1.44def	1.37ab
	10	2.14bcde	1.86klmn	1.91jklm	1.89kl	1.99klm	1.44fg	1.66cd	1.31bcd
	12	2.19ab	1.80mnop	1.93jklm	1.90kl	1.96klmn	1.94bcd	2.31a	1.39a

Different letters within a column are significantly different ($p<0.05$). ND=not detected

Appendix 8. Color, water activity (aw), and moisture of whole milk powder from four facilities stored up to 12 months. Means represent data pooled from five shipments collected over a year period.

Facility	Time	Dry L*	Dry a*	Dry b*	Wet L*	Wet a*	Wet b*	aw	Moisture (%)
A	0	93.09cde	-3.11bcde	14.51a	86.40bcd	-2.99ghij	7.19ab	0.156defg	2.51hijk
	2	93.09bcde	-3.11cdef	14.23ab	86.67ab	-2.97fghij	7.33a	0.155defg	2.75cdefg
	4	93.05cdef	-3.14bcde	14.13bcd	86.25cde	-3.04ijk	7.15ab	0.152efg	2.68efghi
	6	93.26ab	-3.11bcde	13.88cdef	86.70a	-3.01ghij	7.11bc	0.146fg	2.67fghi
	8	93.28a	-3.10bcde	13.87cdef	86.65ab	-2.90ef	6.93cd	0.147fg	2.77cdef
	10	93.20abc	-3.10bcde	13.72efghi	86.68ab	-2.97fghi	6.92cd	0.147fg	2.64fghi
	12	93.27ab	-3.11bcde	13.80efgh	86.62ab	-3.00ghij	6.90d	0.150efg	2.58ghij
B	0	92.79hijkl	-3.31fghij	14.15bc	86.01efghij	-2.98fghij	5.71kl	0.175ab	2.86bcde
	2	92.87ghij	-3.37ij	14.12bcd	86.11efgh	-3.02ghijk	5.71kl	0.170abcd	3.06a
	4	92.74ijklm	-3.40j	13.99bcdef	86.02efghij	-3.10k	5.88ijk	0.180a	3.01ab
	6	92.83hijk	-3.35hj	13.87cdef	86.06efghi	-3.01ghij	5.93hij	0.173bc	2.90abc
	8	92.86hijk	-3.31fghij	13.86cdef	86.04efghij	-2.94fg	5.67l	0.177ab	3.02ab
	10	92.95defgh	-3.32ghij	13.83defg	85.83hij	-3.00ghij	5.79ijkl	0.179a	2.96ab
	12	93.11abcd	-3.38ij	13.93bcdef	85.83ij	-3.04ijk	5.77jkl	0.174abc	2.88abcd
C	0	92.79hijkl	-3.15cdefg	14.01bcde	85.78j	-2.93fg	5.99ghi	0.152efg	2.40jk
	2	92.91fghi	-3.14cdef	13.89cdef	86.19def	-2.95fgh	6.18efg	0.151efg	2.69defgh
	4	92.70jklm	-3.24efghij	13.80efgh	86.05efghij	-3.04ijk	6.35e	0.151efg	2.58ghi
	6	92.94defgh	-3.18defgh	13.76efghi	85.81ij	-3.00ghij	6.19efg	0.146fg	2.60fghi
	8	92.93efgh	-3.22efghi	13.69fghi	85.90ghij	-2.98fghij	6.11fgh	0.145fg	2.68efghi
	10	92.89fghi	-2.91a	13.71efghi	85.83hij	-3.03hijk	6.26ef	0.148fg	2.66fghi
	12	93.03defg	-3.16cdefg	13.69fghi	85.89ghij	-3.05jk	6.13fgh	0.139g	2.35k
D	0	92.52n	-3.00abc	13.52ghij	86.51abc	-2.55a	5.75jkl	0.148fg	2.37k
	2	92.65lmn	-3.02abcd	13.47ij	86.50abc	-2.64ab	5.69kl	0.153efg	2.74cdefg
	4	92.58mn	-3.03abcd	13.36j	86.10efghi	-2.82de	5.94hij	0.153efg	2.69defghi
	6	92.58mn	-3.07abcde	13.32j	85.95fghij	-2.75cd	5.76jkl	0.158cdef	2.65fghi
	8	92.95defgh	-3.05abcde	13.48hij	86.16defg	-2.71bc	5.81ijkl	0.160bcdef	2.51hijk
	10	92.69klmn	-2.94ab	13.23j	85.99efghij	-2.78cd	5.80ijkl	0.166abcde	2.50ijk
	12	92.70jklmn	-2.96ab	13.33j	85.91fghij	-2.79cd	5.83ijkl	0.157cdef	2.49ijk

Different letters within a column are significantly different ($p<0.05$).

Appendix 9. Two-way analysis of variance to compare the effects of packaging atmosphere (air or nitrogen) and temperature (2 ºC or 23 ºC) on volatiles in whole milk powder during storage up to 12 months.

Time (months)		**A2**	**A23**	**N2**	**N23**	**Atm**	**Temp**	**Interaction (AtmxTemp)**
2	Dimethyl sulfide	9.8	12.4	13.7	12.4	NS	NS	NS
3	Dimethyl sulfide	6.3	5.7	4.6	7.2	NS	NS	NS
4	Dimethyl sulfide	9.0	10.6	12.9	9.8	NS	NS	NS
6	Dimethyl sulfide	15.2	12.5	11.9	13.3	NS	NS	NS
8	Dimethyl sulfide	16.8	25.7	20.9	29.8	0.021	0.002	NS
10	Dimethyl sulfide	19.8	30.8	34.7	86.7	0.003	0.006	NS
12	Dimethyl sulfide	16.4	26.5	20.2	54.3	NS	NS	NS

NS=not significant ($p>0.05$). A=air, N=nitrogen, 2=2 ºC, 23=23 ºC.

Appendix 9 (continued). Two-way analysis of variance to compare the effects of packaging atmosphere (air or nitrogen) and temperature (2 ºC or 23 ºC) on volatiles in whole milk powder during storage up to 12 months.

Time (months)		**A2**	**A23**	**N2**	**N23**	**Atm**	**Temp**	**Interaction (AtmxTemp)**
2	3-methylbutanal	2.7	2.9	3.0	6.2	0.003	0.005	0.013
3	3-methylbutanal	2.1	2.0	1.3	2.0	NS	NS	NS
4	3-methylbutanal	3.2	5.0	3.4	4.4	0.007	0.003	0.019
6	3-methylbutanal	4.5	6.0	3.3	3.7	0.013	NS	NS
8	3-methylbutanal	6.3	9.1	5.1	6.7	NS	0.001	NS
10	3-methylbutanal	4.7	6.6	5.2	12.3	0.003	< 0.0001	0.009
12	3-methylbutanal	4.0	8.6	3.4	8.0	NS	NS	NS

NS=not significant ($p>0.05$). A=air, N=nitrogen, 2=2 ºC, 23=23 ºC.

Appendix 9 (continued). Two-way analysis of variance to compare the effects of packaging atmosphere (air or nitrogen) and temperature (2 ºC or 23 ºC) on volatiles in whole milk powder during storage up to 12 months.

Time (months)		A2	A23	N2	N23	Atm	Temp	Interaction (AtmxTemp)
2	2-methylbutanal	1.7	2.6	2.5	2.8	NS	NS	NS
3	2-methylbutanal	2.0	2.1	1.7	2.2	NS	NS	NS
4	2-methylbutanal	3.1	4.8	3.5	4.5	0.006	NS	0.006
6	2-methylbutanal	4.2	6.6	3.0	2.9	0.008	NS	0.134
8	2-methylbutanal	6.1	10.0	5.1	8.5	< 0.0001	< 0.0001	0.007
10	2-methylbutanal	5.3	13.7	6.3	10.7	NS	0.000	NS
12	2-methylbutanal	4.5	12.9	3.8	9.2	NS	0.000	NS

NS=not significant (p>0.05). A=air, N=nitrogen, 2=2 ºC, 23=23 ºC.

Appendix 9 (continued). Two-way analysis of variance to compare the effects of packaging atmosphere (air or nitrogen) and temperature (2 ºC or 23 ºC) on volatiles in whole milk powder during storage up to 12 months.

Time (months)		A2	A23	N2	N23	Atm	Temp	Interaction (AtmxTemp)
2	Hexanal	58.1	104.5	32.7	32.2	0.003	NS	NS
3	Hexanal	97.4	258.2	86.8	44.9	NS	NS	NS
4	Hexanal	243.4	492.4	99.1	703.4	NS	NS	NS
6	Hexanal	494.0	806.4	166.6	18.2	NS	NS	NS
8	Hexanal	911.0	794.2	164.7	845.6	NS	NS	NS
10	Hexanal	1247.7	2541.8	304.0	37.3	< 0.0001	NS	0.028
12	Hexanal	1090.3	1050.5	859.7	135.3	0.005	NS	NS

NS=not significant ($p>0.05$). A=air, N=nitrogen, 2=2 ºC, 23=23 ºC.

Appendix 9 (continued). Two-way analysis of variance to compare the effects of packaging atmosphere (air or nitrogen) and temperature (2 ºC or 23 ºC) on volatiles in whole milk powder during storage up to 12 months.

Time (months)		A2	A23	N2	N23	Atm	Temp	Interaction (AtmxTemp)
2	2-Heptanone	5.1	10.8	4.5	7.9	NS	NS	NS
3	2-Heptanone	5.4	7.2	4.9	7.5	NS	NS	NS
4	2-Heptanone	5.4	10.2	4.4	9.5	NS	0.000	NS
6	2-Heptanone	5.3	14.2	5.1	14.4	NS	< 0.0001	NS
8	2-Heptanone	8.4	25.0	6.8	22.5	NS	< 0.0001	NS
10	2-Heptanone	7.3	24.1	9.2	50.0	< 0.0001	< 0.0001	0.000
12	2-Heptanone	8.3	39.1	7.8	35.9	NS	< 0.0001	NS

NS=not significant ($p>0.05$). A=air, N=nitrogen, 2=2 ºC, 23=23 ºC.

Appendix 9 (continued). Two-way analysis of variance to compare the effects of packaging atmosphere (air or nitrogen) and temperature (2 ºC or 23 ºC) on volatiles in whole milk powder during storage up to 12 months.

Time (months)		A2	A23	N2	N23	Atm	Temp	Interaction (AtmxTemp)
2	Heptanal	7.1	11.4	4.5	4.6	0.014	NS	NS
3	Heptanal	10.1	23.7	11.7	7.1	NS	NS	NS
4	Heptanal	25.8	43.8	13.4	62.7	NS	NS	NS
6	Heptanal	45.5	70.7	22.7	2.2	NS	NS	NS
8	Heptanal	77.7	63.8	21.7	64.5	NS	NS	NS
10	Heptanal	114.6	241.9	36.2	5.5	< 0.0001	NS	0.015
12	Heptanal	133.1	144.5	107.9	14.9	NS	NS	NS

NS=not significant (p>0.05). A=air, N=nitrogen, 2=2 ºC, 23=23 ºC.

Appendix 9 (continued). Two-way analysis of variance to compare the effects of packaging atmosphere (air or nitrogen) and temperature (2 ºC or 23 ºC) on volatiles in whole milk powder during storage up to 12 months.

Time (months)		A2	A23	N2	N23	Atm	Temp	Interaction (AtmxTemp)
2	1-Octen-3-ol	0.3	0.6	0.3	0.5	NS	0.002	NS
3	1-Octen-3-ol	0.6	0.9	0.6	0.6	NS	NS	NS
4	1-Octen-3-ol	0.7	1.3	0.6	1.8	NS	NS	NS
6	1-Octen-3-ol	1.4	2.1	1.2	0.5	NS	NS	NS
8	1-Octen-3-ol	2.8	2.4	1.8	2.8	NS	NS	NS
10	1-Octen-3-ol	3.1	8.1	2.4	1.1	0.002	NS	0.009
12	1-Octen-3-ol	4.1	7.1	3.5	2.8	NS	NS	NS

NS=not significant ($p>0.05$). A=air, N=nitrogen, 2=2 ºC, 23=23 ºC.

Appendix 9 (continued). Two-way analysis of variance to compare the effects of packaging atmosphere (air or nitrogen) and temperature (2 ºC or 23 ºC) on volatiles in whole milk powder during storage up to 12 months.

Time (months)		A2	A23	N2	N23	Atm	Temp	Interaction (AtmxTemp)
2	Octanal	8.2	11.0	6.2	9.2	NS	NS	NS
3	Octanal	9.2	10.3	7.8	7.4	0.004	NS	NS
4	Octanal	10.6	13.9	9.6	16.5	0.007	NS	NS
6	Octanal	12.3	19.7	10.5	8.0	0.019	NS	NS
8	Octanal	16.1	20.8	11.0	21.5	NS	NS	NS
10	Octanal	25.7	64.6	21.5	28.0	< 0.0001	< 0.0001	0.001
12	Octanal	29.0	47.0	23.2	16.4	NS	NS	NS

NS=not significant (p>0.05). A=air, N=nitrogen, 2=2 ºC, 23=23 ºC.

Appendix 9 (continued). Two-way analysis of variance to compare the effects of packaging atmosphere (air or nitrogen) and temperature (2 ºC or 23 ºC) on volatiles in whole milk powder during storage up to 12 months.

Time (months)		A2	A23	N2	N23	Atm	Temp	Interaction (AtmxTemp)
2	3-Octen-2-one	0.6	1.4	0.3	0.5	< 0.0001	< 0.0001	< 0.0001
3	3-Octen-2-one	0.6	2.5	0.4	0.4	0.017	0.044	NS
4	3-Octen-2-one	1.5	4.6	0.6	6.0	NS	NS	NS
6	3-Octen-2-one	2.5	8.5	0.9	0.2	0.026	NS	NS
8	3-Octen-2-one	5.4	8.4	1.1	9.1	NS	NS	NS
10	3-Octen-2-one	8.7	33.7	1.9	0.3	< 0.0001	0.000	< 0.0001
12	3-Octen-2-one	13.4	27.7	12.4	1.5	0.016	NS	NS

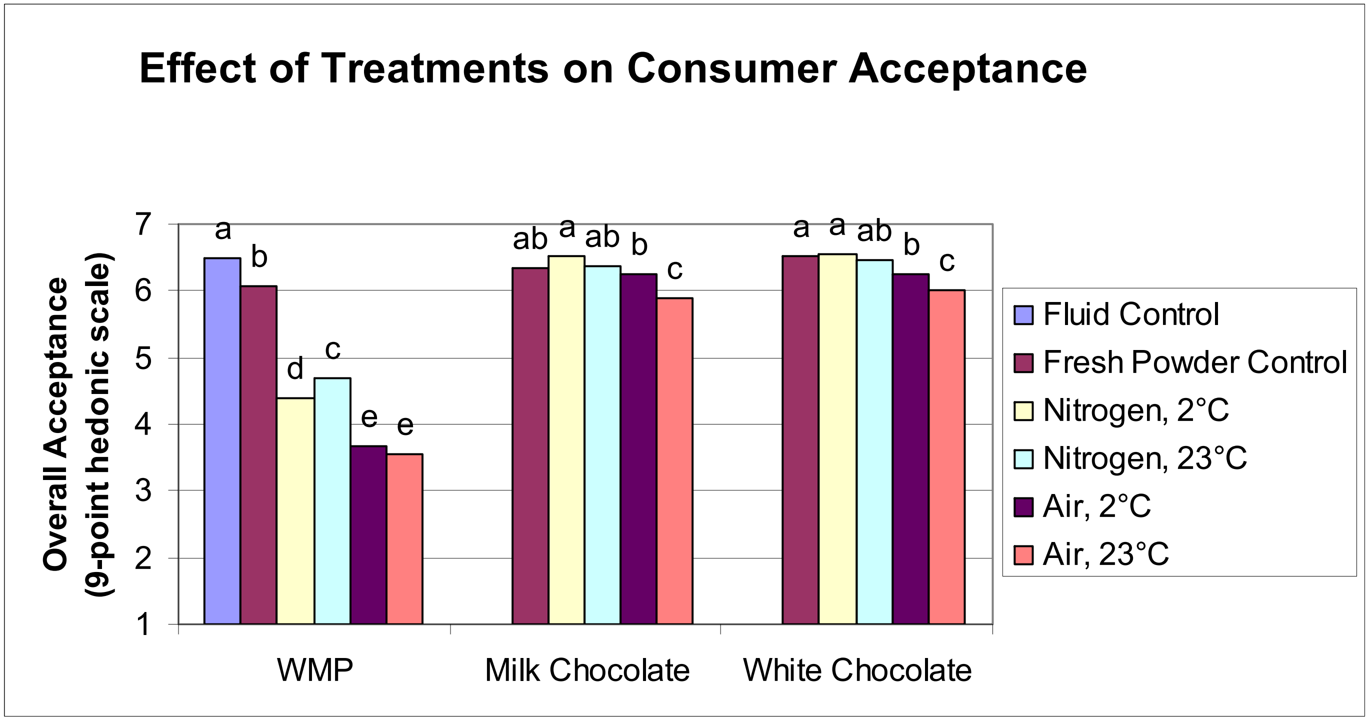
NS=not significant ($p>0.05$). A=air, N=nitrogen, 2=2 ºC, 23=23 ºC.

Appendix 9 (continued). Two-way analysis of variance to compare the effects of packaging atmosphere (air or nitrogen) and temperature (2 ºC or 23 ºC) on volatiles in whole milk powder during storage up to 12 months.

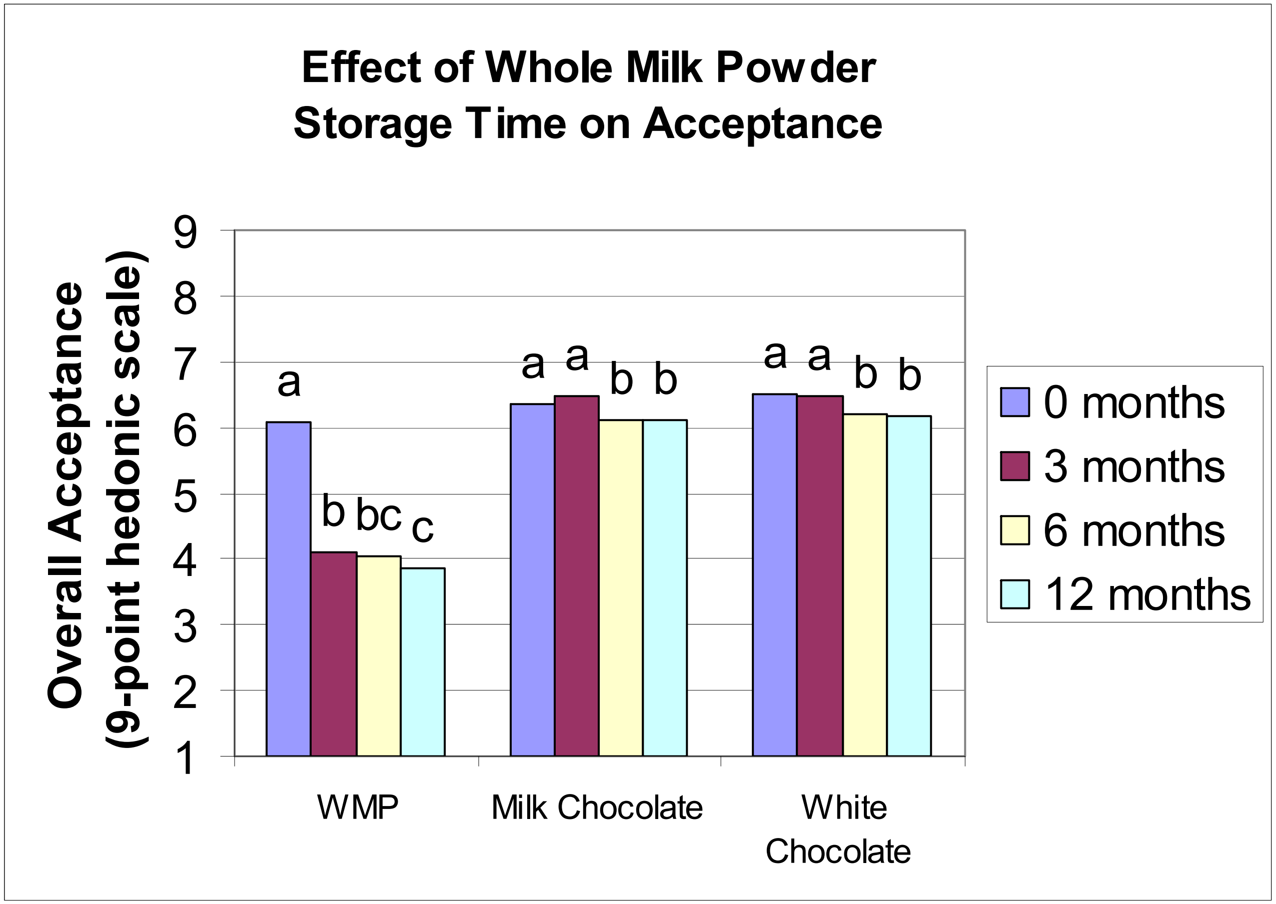
Time (months)		A2	A23	N2	N23	Atm	Temp	Interaction (AtmxTemp)
2	Nonanal	0.7	1.2	0.6	0.8	0.046	0.005	NS
3	Nonanal	1.7	1.4	0.7	1.2	NS	NS	NS
4	Nonanal	1.5	3.0	1.0	3.4	0.018	NS	NS
6	Nonanal	2.4	5.2	1.5	0.7	0.018	NS	NS
8	Nonanal	3.6	4.4	2.0	4.3	NS	NS	NS
10	Nonanal	5.3	14.9	1.8	1.5	< 0.0001	0.000	0.000
12	Nonanal	7.7	8.9	5.7	1.2	NS	NS	NS

NS=not significant ($p>0.05$). A=air, N=nitrogen, 2=2 ºC, 23=23 ºC.

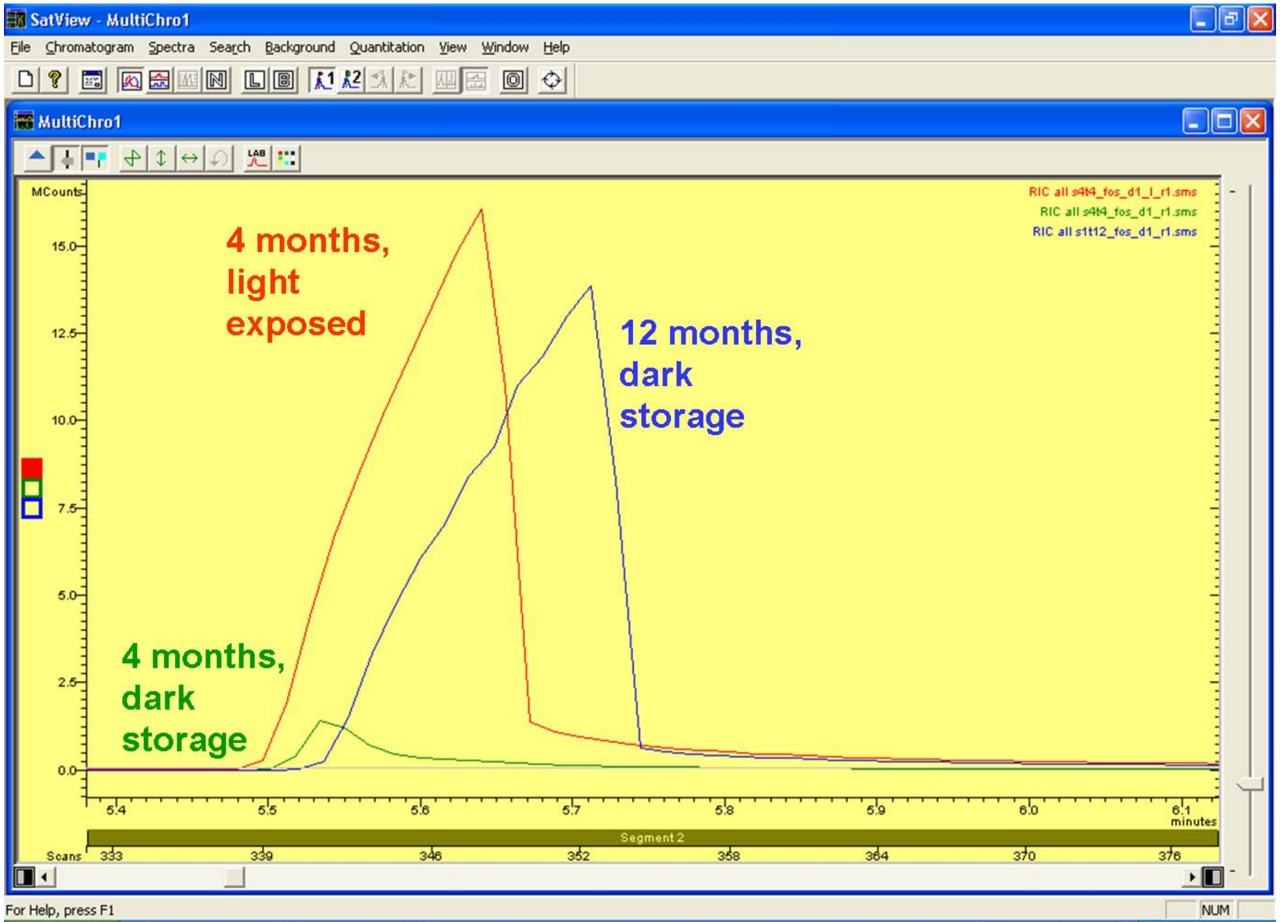
Appendix 10. Effect of packaging atmosphere (nitrogen or air) and storage temperature (2 ºC or 23 ºC) on consumer acceptance of reconstituted whole milk powder, milk chocolate, and, white chocolate. Data pooled across 12 mo storage.



Appendix 11. Effect of storage time on consumer acceptance of reconstituted whole milk powder, milk chocolate, and, white chocolate. Data pooled across treatments of atmosphere (nitrogen or air) and storage temperature (2 ºC or 23 ºC).



Appendix 12. Effect of light exposure on hexanal in WMP. The green and red peaks are the same samples, except that the one in red was exposed to light, and the green one was stored in dark storage. The blue peak is a different sample (from the same facility) at 12 months (to provide a point of reference).



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