



HAL
open science

Nutritional, sensory and physico-chemical characterization of protein-standardized UHT milk

W Rattray, P Gallmann, P Jelen

► **To cite this version:**

W Rattray, P Gallmann, P Jelen. Nutritional, sensory and physico-chemical characterization of protein-standardized UHT milk. *Le Lait*, 1997, 77 (2), pp.279-296. hal-00929527

HAL Id: hal-00929527

<https://hal.science/hal-00929527>

Submitted on 11 May 2020

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.

Nutritional, sensory and physico-chemical characterization of protein-standardized UHT milk

W Rattray¹, P Gallmann², P Jelen^{1*}

¹ *Department of Agricultural, Food and Nutritional Science,
University of Alberta, Edmonton, Alberta, Canada;*

² *Federal Dairy Research Institute, Liebefeld, Switzerland*

(Received 3 May 1996; accepted 30 September 1996)

Summary — The protein content of 1.7% fat milk was standardized 'downwards' from ~ 3.4% to values in the range ~ 3.2–2.6%, by mixing with ultrafiltration (UF) permeate obtained from skim milk or quarg acid whey. Normal or protein-standardized milk products were subjected to direct (150 °C for 2.7 s) or indirect (138 °C for 1 s) ultra-high temperature (UHT) heat treatments and stored at 4 or 25 °C for up to 12 weeks. Standardization with skim milk permeate caused no change in the milk pH (6.7) and all milk products had excellent stability during direct or indirect UHT heating. Upon storage at 4 or 25 °C for up to 12 weeks no age gelation occurred, and increases in apparent viscosity and sedimentation were minimal. Nutritional quality was slightly reduced by the addition of skim milk permeate, due mainly to minor decreases in the content of protein, calcium, phosphorus and potassium. Sensory quality was similar to normal UHT milk, as shown by periodic triangle sensory tests. The use of acid whey permeate for downward protein standardization led to a better profile of micronutrients, compared to milk with skim milk permeate. In other respects its use was unsatisfactory; although the pH shift upon addition of acid whey permeate to milk was minor (from pH 6.7 to pH 6.50–6.65), neutralization to pH 6.8 was required to avoid protein coagulation during direct or indirect UHT heating. In both direct or indirect UHT milk, high apparent viscosity and excessive sedimentation developed upon storage and off-flavours were unacceptably strong.

protein standardization / UHT milk / skim milk permeate / acid whey permeate

Résumé — **Caractérisation nutritive, sensorielle et physico-chimique de lait UHT standardisé en protéine.** La teneur en protéines du lait partiellement écrémé (1,7 % MG) a été standardisée à la baisse depuis ~ 3,4 % à 3,2–2,6 % par addition de perméat provenant du traitement par ultrafiltration de lait écrémé ou de lactosérum obtenu lors de la fabrication de quarg. Les échantillons lactés ordinaires et standardisés en protéines ont subi un traitement ultra-haute température (UHT) selon une méthode de contact direct (2,7 s à 150 °C) ou indirect (15 s à 138 °C) avant d'être stockés pour une durée de 12 semaines à 4 °C ou 25 °C. La standardisation avec perméat de lait écrémé n'a produit aucun

* Correspondence and reprints

changement dans le pH du lait (6,7) et tous les échantillons ont montré une excellente stabilité face aux deux méthodes de traitement UHT. Au cours du stockage, à 4 ou 25 °C, jusqu'à 12 semaines, aucune gélification n'apparaissait et l'augmentation de la viscosité et la formation de sédiments restaient minimales. La qualité nutritionnelle était légèrement réduite par l'addition de perméat de lait écrémé, principalement à cause de la diminution de la teneur en protéines, calcium, phosphate et potassium. Des tests sensoriels triangulaires n'ont pas montré de différence organoleptique entre le lait UHT ordinaire et le lait UHT standardisé avec perméat de lait écrémé. L'utilisation de perméat de lactosérum de quarg pour la réduction standard en protéines produit une meilleure balance en éléments nutritifs comparé à l'addition de perméat de lait écrémé. Sur les autres points, l'addition de perméat de lactosérum de quarg n'est pas satisfaisante ; bien que la baisse du pH soit faible (de 6,7 à 6,5–6,65), une neutralisation à un pH de 6,8 a été nécessaire, afin d'éviter la coagulation des protéines durant le traitement UHT direct aussi bien qu'indirect. Pour les deux traitements UHT, une viscosité accrue et une sédimentation excessive sont apparues durant le stockage, ainsi que de fortes saveurs indésirables.

standardisation de protéines / lait UHT / perméat de lait écrémé / perméat de lactosérum acide

INTRODUCTION

There is considerable international interest in standardizing the protein content of milk (Marshall, 1995; Rattray and Jelen, 1996a). Standardization of milk protein 'upward' by ultrafiltration (UF) is advantageous in the manufacture of cheese and yoghurt, leading to improvements in product consistency, texture and yield (Maubois, 1991; Puhán, 1992). Upward standardization could also enable many countries to meet protein specifications in condensed milk and milk powder, desired by international markets, which could not otherwise be achieved due to the naturally low protein content of their milk. Standardization of milk protein 'downward' by use of UF permeate would be desirable where milk has a naturally high protein content, allowing more realistic valorization of milk and providing for a convenient method of permeate utilization. Recently, the International Dairy Federation approved the principle of enrichment of condensed milk or milk powder with isolated lactose or skim milk UF permeate for downward standardization of protein (Anonymous, 1995a). The increased production of UF permeate, the technological ease of protein standardization (Rønkilde Poulsen, 1978; Friis, 1986) and the current interest in standardization

of fluid milk increase the possibility that other permeate types will be considered.

Because heating is involved in most dairy processes, the effect of standardization on the heat stability of milk needs to be evaluated. In laboratory scale trials, Rattray and Jelen (1996b) showed that standardization of type A skim milk with skim milk permeate or sweet whey UF permeate caused a general increase in heat stability at 140 °C, in the pH range 6.4–7.1, but the use of acid whey UF permeate caused a general decline in heat stability. Peter et al (1996) standardized 2% fat milk with skim milk permeate, and showed a similar heat stability trend in the presence of homogenized milk-fat. These studies were not extended to a more practical situation, a good example being the manufacture of ultra-high temperature (UHT) milk.

Downward standardization of pasteurized milk with permeate made by UF of skim milk or UF of rennet casein whey has little effect on sensory quality (Rønkilde Poulsen, 1978; Peter et al, 1996; Rattray and Jelen, 1996c). The use of acid whey permeate leads to off-flavours, especially if it is derived from fermented dairy products (Rattray and Jelen, 1996c). The influence of UF permeate addition on the sensory quality of more severely heated milk, such

as UHT milk, is unknown; this could differ due to the presence of heat-generated, sulphur-containing flavour compounds and the possibility that off-flavours produced during microbial fermentation would be volatilized during heating, especially during the cooling stage by vacuum flashing of steam involved in direct UHT heating.

The principal factors affecting the manufacture of an acceptable UHT milk are: (1) thermal stability; milk must tolerate a heat treatment of 135–150 °C for 20–22 s, achieved by direct or indirect heating (Hinrichs and Kessler 1995); (2) physical stability on storage; UHT milk is prone to age gelation, creaming and sedimentation, which typically restrict its shelf life to 3–6 months at ambient temperature (Nieuwenhuijse, 1995); (3) sensory quality; the flavour of freshly made UHT milk has been described as 'cooked', 'sulphurous' or 'cabbagey', becoming 'stale' or 'oxidized' upon storage (Calvo and Holz, 1992; Andersson and Öste, 1995b; Nursten, 1995); (4) nutritional quality; heating leads to the destruction of certain vitamins (Andersson and Öste, 1995a); which, though undesirable, may be tolerable in the western diet.

In the present study, milk products with the same target fat content (1.7%) but protein contents in the range 3.36–2.6% were prepared by blending whole milk and skim milk with industrial permeates obtained by UF of skim milk or quarg acid whey. General objectives of this work were to determine the effect of protein standardization on: (a) the ability of milk to withstand direct or indirect UHT heating; (b) the nutritional and sensory properties; and (c) the storage stability of the UHT milk produced. In a related work (Ratray et al, 1996), the freezing point and furosine content of the UHT milk products were determined.

MATERIALS AND METHODS

A schematic summary of the procedure used to prepare the protein-standardized milk products is shown in figure 1. The procedure was carried out on three separate occasions using three batches of whole milk, skim milk and UF permeates. In general, for every trial each analysis was carried out in duplicate and means and standard deviations were calculated using all data ($n = 6$).

Protein standardization of milk

Skim milk and whole milk were obtained from the Federal Dairy Research Institute, Liebefeld, Switzerland. The UF permeates were kindly provided by Emmi Milch AG (Lucerne, Switzerland). Skim milk permeate was produced by industrial-scale UF of normal skim milk using an APV Pasilac DDS plate and frame UF unit equipped with a polysulphone membrane (GR 61 PP) with a cut-off of ~ 20 000 Da; UF was carried out at 50 °C and an average cross-membrane pressure of 550 kPa. Industrial-scale UF of quarg acid whey yielded acid whey permeate. Quarg manufacture involved fermentation of skim milk at 22 °C for 25 h to pH 4.7, using a commercial culture of lactic acid bacteria. The acid whey produced was ultrafiltered at 47 °C and an average cross-membrane pressure of 400 kPa, using a spiral wound polysulphone membrane capable of retaining molecules of > 5 nm diameter.

Whole milk, skim milk and UF permeates were kept at 4 °C for < 24 h, during which time their fat and protein contents were determined, to enable calculation of the required amounts to be blended to obtain fat- and protein-standardized milk. About 80 kg of each milk was prepared, containing ~ 1.7% (w/w) fat and ~ 3.4, 3.2, 2.9 or 2.6% (w/w) protein. Control milk contained ~ 3.4% (w/w) protein and was prepared by the sole mixing of whole milk and skim milk. Milk containing skim milk permeate was termed S1, S2 or S3 milk in order of increasing level of addition (decreasing protein content); likewise, milk with acid whey permeate was denoted as A1, A2 or A3 milk.

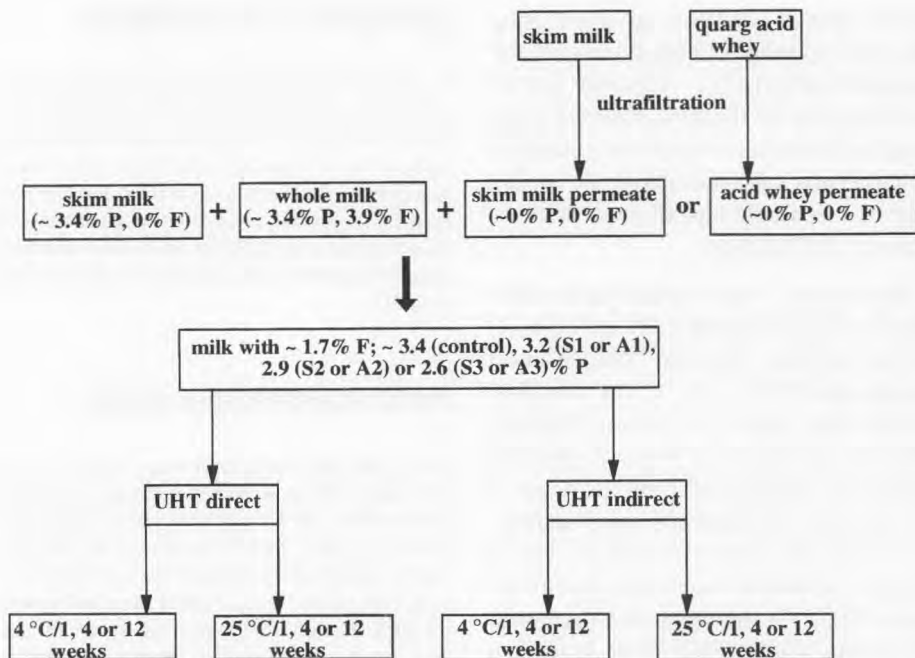


Fig 1. Schematic outline of methods used to prepare fat- and protein-standardized direct or indirect UHT milk products and subsequent storage conditions. F: fat; P: protein; S1, S2 or S3 denote milk with increasing amounts of skim milk permeate; A1, A2 or A3 denote milk with increasing amounts of acid whey permeate.

Diagramme montrant les étapes utilisées pour le traitement UHT par méthode directe et indirecte des échantillons standardisés en matière grasse et protéine et les conditions du stockage consécutif. Abréviations : F, matière grasse ; P, protéine ; S1, S2 ou S3, lait avec une quantité croissante en perméat de lait écrémé ; A1, A2 ou A3, lait avec une quantité croissante en perméat de lactosérum acide.

Ultra-high temperature heating

The UHT milk products were prepared using a pilot scale UHT machine (APV Baker AS, Kolding, Denmark) with direct and indirect heating capabilities and a throughput of ~160 L/h. For direct UHT treatment, milk was pre-heated to 89 °C, heated directly by steam infusion to 150 °C for a 2.7 s holding period, vacuum flash-cooled to 83 °C, further cooled to 65 °C and homogenized at 18.5 MPa and 65 °C under aseptic conditions, using a three-stage reciprocating homogenizer. After homogenization, the milk was cooled to 4 °C and transferred aseptically into glass bottles. Indirect UHT treatment involved heat treatment of milk by the use of plate heat exchangers throughout. Milk was pre-heated to 65 °C, homogenized at 18.5 MPa with

the same homogenizer as above, pre-heated to 90 °C for 2.4 s, heated to 138 °C and held for 1 s, cooled to 4 °C, followed by aseptic filling into glass bottles. In some heating trials, the pH of protein-standardized milk was adjusted to 6.8 prior to heating using 30% NaOH. Milk products were stored at 4 or 25 °C in darkness for up to 12 weeks, the typical maximum storage time allowed for UHT milk.

Compositional analyses

Prior to the preparation of protein-standardized milk, the fat and protein content of skim milk, whole milk and the two permeate types were determined by standard methods (IDF, 1987a,

1993, respectively). After 1 week of storage at 4 °C, the UHT milk products were assayed for total solids (IDF, 1987b), protein (IDF, 1993), fat (IDF, 1987a), lactose (IDF, 1974) and ash (Anonymous, 1987).

The nutritional quality of each protein-standardized UHT milk was further assessed by measuring the content of certain minerals and vitamins after 1 week of storage at 4 °C. Chloride and phosphorus were determined according to IDF standards (1988, 1990, respectively). Sodium, potassium, calcium and magnesium were measured by the following procedure: to a known mass of milk (~5 g), 5 mL of 65% nitric acid was added and the mixture heated at 90 °C for 1 h to completely solubilize all minerals. The sample was then combusted in an air/acetylene flame where the intensity of light emitted at 589 or 769.9 nm was proportional to the content of sodium or potassium respectively, while the absorption of light at 422.7 or 285.2 nm was proportional to the content of calcium or magnesium, respectively. Spectral measurements were carried out using a Video 22E atomic absorption/atomic emission spectrophotometer (Instrumentation Laboratory Inc, Lexington, MA, USA).

The concentration of vitamin B₁ and B₂ was determined after 1 week of storage at 4 °C, according to the methods of Tagliaferri et al (1992a, b, respectively). Vitamins A and E were assayed together by a new method (Anonymous, 1995b) developed at the Swiss Federal Dairy Research Institute.

The control milk and protein-standardized milk products were assayed for non-casein nitrogen (NCN) (IDF, 1964) and non-protein nitrogen (NPN), before, as well as 1, 4, or 12 weeks after UHT heating. The NPN was determined by addition of 12% trichloroacetic acid to milk to precipitate the proteins, filtration of the milk and measurement of the total nitrogen in the filtrate.

Physical stability

The physical stability of UHT milk products was assessed by measuring their apparent viscosities and sedimentation tendencies after 1, 4 or 12 weeks of storage at 4 or 25 °C. The apparent viscosity was determined using a capillary viscometer made by Jena Glaswerk (Schott & Gen, Mainz, Germany) and an Auto Paar, DMA 55 (Auto Paar KG, Graz, Austria) densitometer.

Sedimentation was calculated as g wet sediment per kg milk, after determining the mass of sediment remaining upon inversion and drainage, for 30 min of an exactly known mass of milk in a 1-L capacity bottle. In some UHT milk products, protein aggregation was so extensive that phase separation occurred and the sediment had a custard-like consistency. In these samples, the mass of sediment was estimated after careful decantation of the supernatant. The composition of the sediments (total solids, protein, lactose, fat, ash, calcium and phosphorus) was determined by the same methods as above.

Sensory evaluation

For sensory evaluation a panel combining six untrained individuals and six trained and certified individuals, all from the Federal Dairy Research Institute, Liebefeld, Switzerland was used. The direct or indirect UHT milk products were evaluated on separate days with no more than seven samples per panelist on each occasion. All products were equilibrated to ~15 °C and aliquots (~50 mL) placed in small plastic cups. A hedonic sensory score system was used, in which odour was assigned a maximum (most liked) weighting of 2 (5 × 0.4) points and taste a maximum of 8 (5 × 1.6) points. Panelists were encouraged to give descriptive comments, and were assisted by a list of possible defects.

RESULTS AND DISCUSSION

Heat stability

In preliminary experiments, each milk product was UHT-heated at its native pH; for control milk or milk with skim milk permeate, this was at pH 6.7. Immediately after direct or indirect UHT heating, there were no visible signs of destabilization. The result was expected; normal milk can be heated for up to 20 min at 140 °C without visible protein coagulation, and its heat stability is even greater upon the addition of skim milk permeate (Peter et al, 1996; Rattray and Jelen, 1996b). The improved heat stability of skim milk blended with skim milk permeate has been attributed to a lower concentration

of minerals and possibly reduced collision frequency between casein micelles. In accordance with normal industrial practice, all further UHT heating of control milk or milk with skim milk permeate was carried out at their natural pH (~ 6.7).

Addition of increasing amounts of acid whey permeate to raw milk caused the pH to decline from ~ 6.7 to values in the range ~ 6.50 – 6.65 . This minor pH shift was accompanied by a large decline in heat stability; direct UHT heating of milk with even 3.14% (w/w) protein, corresponding to the lowest level of added permeate, led to visible coagulation in the steam infusion chamber. To avoid damage to the UHT machine, no further preliminary tests of milk products standardized with acid whey permeate at an unadjusted pH of 6.50–6.65 were carried out; destabilization would probably have been even greater due to the higher levels of acid whey permeate and the greater heat load applied during indirect UHT heating.

Rattray and Jelen (1996b) observed that the addition of acid whey permeate to skim milk caused a general decline in heat stability over the pH range 6.4–7.1; this was especially evident at the unaltered pH of milk, where some samples heat-coagulated at 90 °C. Reduced heat stability was attributed to the high calcium content of the permeate, which shifted the heat stability-pH curve of type A milk to more alkaline pH values and led to a faster rate of heat-induced pH decline. An exception was an increased heat stability of milk with acid whey permeate at pH 6.8; extra calcium may have inhibited the formation of soluble complexes between β -lactoglobulin and κ -casein and hence heat-labile κ -casein-depleted micelles. In view of this observation, further UHT heating trials of milk with acid whey permeate were carried out after adjusting the pH to 6.8. It was found that direct or indirect UHT heating was possible without visible coagulation of milk; henceforth, all milk

products with acid whey permeate were neutralized to pH 6.8 before UHT heating.

The pH of all milk products with acid whey permeate declined from the neutralized values (6.8) to ~ 6.7 upon direct or indirect UHT heating, but the pH of milk containing skim milk permeate was unchanged (~ 6.7). Rattray and Jelen (1996b) found that skim milk with acid whey permeate experienced a rapid pH decline when heated at 140 °C, possibly due to its relatively high calcium content which increased the sensitivity of soluble calcium salts to thermal precipitation with accompanying release of H^+ ions.

Composition

Compositional data confirmed the expected differences between the two permeate types especially with respect to lactose and mineral content (table I). Much of the minerals in milk are associated with casein and other proteins, and thus would be retained during UF (Green et al, 1984; Premaratne and Cousin, 1991), accounting for the relatively low mineral content of the skim milk permeate. The concentration of total salt and that of individual salts except phosphorus in the acid whey permeate was very similar to that in normal milk, indicating that acidification caused dissolution of salts from casein micelles, allowing the entire mineral component to permeate the UF membrane. Thus milk with acid whey permeate had a concentration of total minerals that was similar to normal milk, but a greater concentration of soluble minerals; this appeared to have a major influence on its physical stability after UHT heating (see below). Control milk contained ~ 22 mg/100 g more phosphorus than the acid whey permeate, similar to a value reported by Walstra and Jenness (1984) for organic phosphorus in milk (21.6 mg/100 g). Organic phosphorus is esterified to casein and thus would not dissociate upon acidification of milk.

Table 1. Mean composition¹ (*n* = 6) of skim milk ultrafiltration permeate, acid whey ultrafiltration permeate, or UHT direct-treated milk products measured after 1 week of storage at 4 °C.

Composition moyenne (n = 6) du perméat de lait écrémé, perméat de lactosérum acide ou des échantillons de lait traités par la méthode UHT directe après 1 semaine de stockage à 4 °C.

Product	Total solids	Protein	Fat % (w/w)	Lactose	Ash	Calcium	Phosphorus (mg/100 g)	Potassium
Skim milk permeate	5.38 (0.18)	0.19 (0.04)	0.0 (0.0)	4.97 (0.17)	0.46 (0.01)	27.9 (0.2)	6.8 (0.8)	24.0 (1.4)
Acid whey permeate	5.77 (0.14)	0.22 (0.06)	0.0 (0.0)	4.32 (0.06)	0.73 (0.01)	122.4 (8.3)	67.0 (1.3)	161.0 (6.0)
Control	10.75 (0.37)	3.36 (0.03)	1.69 (0.08)	4.98 (0.24)	0.74 (0.01)	118.5 (5.4)	89.0 (4.2)	168.0 (9.9)
S1	10.54 (0.17)	3.18 (0.05)	1.71 (0.13)	4.99 (0.25)	0.73 (0.02)	110.5 (0.7)	86.0 (1.4)	165.0 (11.3)
S2	10.07 (0.24)	2.90 (0.06)	1.63 (0.09)	5.02 (0.23)	0.71 (0.02)	106.5 (4.2)	81.0 (1.4)	164.5 (10.6)
S3	9.58 (0.22)	2.55 (0.05)	1.57 (0.23)	4.99 (0.22)	0.68 (0.02)	95.5 (5.3)	73.5 (0.7)	150.5 (4.9)
A1	10.42 (0.40)	3.14 (0.09)	1.7 (0.20)	4.96 (0.28)	0.72 (0.04)	117.5 (8.1)	86.0 (0.1)	153.5 (0.7)
A2	10.08 (0.36)	2.91 (0.03)	1.63 (0.12)	4.86 (0.29)	0.75 (0.02)	118.0 (8.6)	84.0 (4.2)	154.5 (6.3)
A3	9.68 (0.16)	2.58 (0.05)	1.58 (0.25)	4.78 (0.33)	0.79 (0.03)	121.0 (9.9)	82.5 (0.7)	157.5 (4.9)

¹ Protein standardization with skim milk permeate or acid whey permeate had no effect on the concentration of magnesium [9.3 (0.3) mg/100 g] or chloride [100.4 (2.3) mg/100 g]. S1, S2, S3 indicate milk with increasing amounts of skim milk permeate; A2, A2, A3 indicate milk with increasing amounts of acid whey permeate. Numbers in brackets show the standard deviations.

¹ *La standardisation en protéine par perméat de lait écrémé ou perméat de lactosérum acide n'a aucun effet sur la teneur en magnésium [9,3 (0,3) mg/100 g] ou la teneur en chlore [100,4 (2,3) mg/100 g]. S1, S2, S3, lait avec une quantité croissante en perméat de lait écrémé ; A1, A2, A3, lait avec une quantité croissante en perméat de lactosérum acide. Les chiffres entre parenthèses représentent la déviation standard.*

The lower lactose content of the acid whey permeate as compared to skim milk permeate can be explained by the fact that the former originated from quarg; the manufacture of quarg involves fermentation of skim milk, whereby some of the lactose is converted into lactic acid. The 'protein' in the UF permeates was probably NPN; low molecular weight peptides, amino acids, urea and other nitrogenous compounds are permeable through UF membranes.

Analyses of the UHT milk products gave ~ 3.4, 3.2, 2.9 or 2.6% (w/w) protein and ~ 1.7% (w/w) fat, indicating that the raw materials had been combined appropriately. Control milk and milk with skim milk permeate had a similar concentration of lactose, which was reduced in milk with acid

whey permeate. Standardization of milk with skim milk permeate caused a slight reduction in the total ash content, more specifically in calcium, phosphorus and potassium, while the concentration of magnesium and chloride was unchanged. Standardization of milk with acid whey permeate had a very little effect on the content of total ash, calcium, phosphorus, magnesium or chloride. The mineral composition of bovine milk exhibits a significant degree of natural variability; the range for calcium is 110–130 mg/L and potassium varies from 110–170 mg/L (Flynn and Power, 1985). This natural variability should be considered when evaluating the importance of the changes in mineral composition caused by protein standardization.

Enrichment of milk with either permeate type did not alter the concentration of vitamin B₁ (thiamine), as shown in table II. Most (83–95%) of the thiamine in milk is not bound to proteins and should be freely permeable through UF membranes (Premaratne and Cousin, 1991), and thus the concentration of thiamine in the permeates and hence in protein-standardized milk should be similar to that in normal milk. The level of thiamine in the direct or indirect UHT milk samples was almost identical, as reported previously by Lembke et al (1968), indicating that the greater heat load applied to indirect UHT milk did not lead to extra destruction of the vitamin. Considering that information on the kinetics of the thermal destruction of thiamine in milk is rather equivocal (Andersson and Öste, 1995a), this result cannot be explained readily.

Premaratne and Cousin (1991) observed that a five-fold concentration of milk by UF led to an ~ 1.5-fold increase (from 178 to 251 µg/100 g) in the concentration of vitamin B₂ (riboflavin), due to its association with milk proteins; this is consistent with the present results, which revealed that the concentrations of riboflavin in the skim milk permeate or acid whey permeate were lower than in normal skim milk. Thus, addition of either permeate to milk should have slightly reduced the concentration of riboflavin. However, no consistent effect of standardization on the level of riboflavin was noticed, possibly because the levels of permeate used were relatively low. Furthermore, the concentration of riboflavin in the skim or whole milk used to prepare the control or protein-standardized milk products was not determined, and this may have con-

Table II. Mean ($n = 2$) concentration of vitamin A, E, B₁ and B₂ in skim milk ultrafiltration permeate acid whey ultrafiltration permeate and direct or indirect UHT milk measured after 1 week of storage at 4 °C.

Composition moyenne (n = 2) en vitamines A, E, B₁, B₂ du perméat de lait écrémé, perméat de lactosérum acide ou des échantillons de lait traités par méthode UHT directe et indirecte après 1 semaine de stockage à 4 °C.

Product	Vitamin A		Vitamin E		Vitamin B ₁ (µg/100 g)		Vitamin B ₂	
	Direct	Indirect	Direct	Indirect	Direct	Indirect	Direct	Indirect
Skim milk permeate	0		0		13		128	
Acid whey permeate	0		0		16		135	
Control	31	22	81	59	21	20	148	153
S1	23	18	62	55	21	20	156	139
S2	21	19	56	52	21	21	156	148
S3	16	19	39	50	20	22	143	146
A1	27	20	71	58	19	21	144	153
A2	23	21	60	58	20	20	152	150
A3	17	21	45	55	19	20	149	140

S1, S2, S3 indicate milk with increasing amounts of skim milk permeate; A1, A2, A3 indicate milk with increasing amounts of acid whey permeate.

S1, S2, S3, lait avec une quantité croissante en perméat de lait écrémé ; A1, A2, A3, lait avec une quantité croissante en perméat de lactosérum acide.

tributed to the fluctuations in the riboflavin content of the UHT milk products. The concentration of riboflavin was not influenced by the type of UHT treatment used, which was expected in view of its reported high heat stability (Oamen et al, 1989). The concentrations of thiamine in the UHT milk samples were about half that of the value reported (Fink and Kessler, 1985) for raw milk (35–45 mg/L), concurring with the reported heat sensitivity of this vitamin (Ford et al, 1969; Burton et al, 1970).

Standardization of milk with either permeate caused the concentration of vitamin A (retinol) and vitamin E (mostly α -tocopherol) to fluctuate slightly, but without a clear trend. As retinol and α -tocopherol are fat-soluble vitamins, this would account for their absence in the fat-free UF permeates. Likewise, the concentration of these vitamins in skim milk should have been very low. Because control and protein-standardized milk products had approximately the same fat content, the concentration of fat-soluble vitamins was expected to be unchanged, as indeed seemed to be the case; variations in vitamin concentration were probably caused by fluctuations in the concentration of fat. The type of UHT heating had no effect on the concentration of retinol or α -tocopherol in the milk products, indicating the thermal stability of these vitamins, as documented earlier (Ford et al, 1969; Burton et al, 1970; Le Maguer and Jackson, 1983).

Concentration of non-casein and non-protein nitrogen

Direct or especially indirect UHT heating caused the concentration of NCN to decline (table III), indicating that heat denaturation of serum proteins occurred, resulting in their insolubilization at pH 4.6. Whey protein denaturation would take place to a greater extent in the more severely heated indirect UHT milk products. The loss of solubility of

whey proteins after heating is the basis of several methods used to classify milk, evaporated milk and milk powders, according to the heat load received (Pellegrino et al, 1995).

Protein standardization caused a small reduction in the NCN content of milk, evident before or after UHT heating and throughout storage, and clearly related to the low concentration of NCN in the permeates, particularly the skim milk permeate. The low NCN content in the UF permeates was expected, due to the impermeability of UF membranes to whey proteins. The greater content of NCN in the acid whey permeate was probably due to the catabolism of protein during the fermentation of milk, involved in quarg manufacture; utilization of casein by lactic acid bacteria requires its hydrolysis into small peptides and free amino acids, capable of being transported across the cell membrane (Pritchard and Coolbear, 1993).

Storage of milk at 4 or 25 °C caused the NCN content of all milk samples to increase slightly. This indicates that hydrolysis of protein occurred during storage to liberate polypeptides, small peptides and free amino acids, soluble at pH 4.6. In unconcentrated UHT milk, hydrolysis of casein is probably caused by plasmin and possibly by bacterial proteinases (Kokak and Zadow, 1985; Manji et al, 1986; Harwalkar 1992; Alkanhal et al, 1994; Nieuwenhuijse, 1995). The products of hydrolysis include γ - and *para*- κ -casein, both of which are soluble at pH 4.6, and thus would contribute to an increased concentration of NCN. The increase in NCN was slightly greater at 25 than at 4 °C, probably because of greater residual enzymatic activity at the higher temperature. Additionally, plasminogen, the inactive precursor of plasmin, is converted more rapidly to plasmin at a higher storage temperature (Manji et al, 1986). Protein hydrolysis occurred at a slightly slower rate in the indirect UHT milk products, possi-

Table III. Influence of protein standardization and UHT heating on the mean ($n = 6$) non-casein nitrogen or non-protein nitrogen content of milk.

Influence de la standardisation en protéines et du traitement UHT sur la teneur moyenne ($n = 6$) en azote non caséique et en azote non protéique des échantillons de lait.

Product	Non-casein nitrogen (mmol/kg)			Non-protein nitrogen (mmol/kg)		
	Before heating	After direct UHT ^{1,2}	After indirect UHT ^{1,2}	Before heating	After direct UHT ^{1,3}	After indirect UHT ^{1,3}
Skim milk permeate		17.0 (1.6)			18.3 (1.2)	
Acid whey permeate		27.0 (2.5)			31.7 (2.3)	
Control	85.0 (2.8)	43.7 (0.6)	37.0 (2.8)	20.0 (1.4)	21.7 (1.5)	22.0 (0.0)
S1	79.5 (2.1)	40.3 (1.2)	36.5 (3.5)	20.0 (1.4)	21.7 (1.5)	22.0 (0.0)
S2	74.0 (2.8)	38.7 (0.6)	35.5 (2.1)	20.0 (1.4)	21.0 (1.0)	21.5 (0.7)
S3	69.5 (3.5)	36.7 (1.5)	33.0 (1.4)	20.0 (1.4)	21.0 (1.7)	21.5 (0.70)
A1	84.5 (3.5)	41.0 (1.7)	36.0 (1.4)	21.0 (1.4)	22.0 (1.7)	22.5 (0.7)
A2	79.5 (3.5)	42.0 (1.4)	36.5 (2.1)	21.5 (0.7)	23.5 (0.7)	23.0 (1.4)
A3	74.5 (7.2)	41.5 (3.5)	37.5 (2.1)	22.0 (1.4)	24.5 (0.7)	24.5 (0.7)

¹ Non-casein nitrogen or NPN were measured after 1 week of storage of UHT milk at 4 °C; ² storage of UHT milk products caused the NCN content to increase gradually. After 12 weeks storage at 4 °C, the mean increase in NCN for direct UHT milk products was 9.9 (1.8) mmol/kg or at 25 °C was 11.8 (2.1) mmol/kg. After 12 weeks storage at 4 °C, the mean increase in NCN for indirect UHT milk products was 7.7 (1.1) mmol/kg or at 25 °C was 10.2 (2.5) mmol/kg. Although protein standardization did affect the initial level of NCN, this had no effect on the subsequent magnitude of the increase in NCN upon storage; ³ storage of direct or indirect UHT milk products for 12 weeks at 4 or 25 °C caused negligible (< 1 mmol/kg milk) changes in NPN content, S1, S2, S3 indicate milk with increasing amounts of skim milk permeate; A1, A2, A3 indicate milk with increasing amounts of acid whey permeate. Numbers in brackets show the standard deviations.

¹ Azote non caséique et azote non protéique mesuré après 1 semaine de stockage du lait UHT à 4 °C; ² le stockage des échantillons de lait UHT a produit une augmentation graduelle de l'azote non caséique. Après 12 semaines, l'accroissement moyen en azote non caséique pour le traitement UHT indirect était de 7,7 (1,1) mmol/kg pour un stockage à 4 °C et de 10,2 (2,5) mmol/kg pour un stockage à 25 °C. Bien que la standardisation en protéine affecte la teneur initiale en azote non caséique, cela n'a aucune influence sur le degré d'accroissement consécutif en azote non caséique observé lors du stockage; ³ le stockage des échantillons de lait traités par méthode UHT directe et indirecte pour une période de 12 semaines à 4 °C ou 25 °C a produit des changements négligeables de teneur en azote non protéique (< 1 mmol/kg lait). S1, S2, S3, lait avec une quantité croissante en perméat de lait écrémé; A1, A2, A3, lait avec une quantité croissante en perméat de lactosérum acide. Les chiffres entre parenthèses représentent la déviation standard.

bly because the more severe heat treatment led to a greater inactivation of plasmin and any bacterial proteinases that may have been present.

After direct or especially indirect UHT heating, the NPN content of milk increased slightly, indicating that a small amount of protein was ultimately hydrolyzed into small peptides or possibly free amino acids. Stan-

dardization with skim milk permeate had no effect on the NPN content of milk, but when acid whey permeate was used the concentration of NPN increased slightly, evident before or after UHT heating; this was due to the similar NPN content of control milk (21 mmol/kg) and skim milk permeate (18 mmol/kg), while acid whey permeate had a higher NPN content (32 mmol/kg).

The similarity in the NPN content of normal milk and skim milk permeate indicates that most low molecular weight nitrogenous compounds present in the milk permeated the UF membranes. The higher NPN content of the acid whey permeate implies the hydrolysis of protein, originating from activity of bacteria during quarg manufacture; as described above this may have also contributed to a higher NCN content.

Storage at 4 or 25 °C had a negligible effect on the concentration of NPN in any milk sample. This suggests that the main effect of protein hydrolysis was to increase the content of relatively large polypeptides (NCN), and that smaller-sized peptides and free amino acids were not liberated for up to 12 weeks of storage at 25 °C. Thus, the concentration of NCN is a more sensitive index of proteolysis, at least for relatively short storage periods of UHT milk, in agreement with the observations of Nieuwenhuijse (1995).

Apparent viscosity

The apparent viscosities of UHT milk products (table IV) showed two distinct trends: (1) standardization of milk with increasing amounts of skim milk permeate caused a gradual decline in viscosity; (2) the A1 milk had a lower viscosity than control milk, but A2 or especially A3 products developed very high viscosities upon storage.

Due to the absence of protein and fat, the viscosities of the UF permeates were low (1.08 mPa·s for skim milk permeate; 1.13 mPa·s for acid whey permeate) and their addition to milk was expected to cause the viscosity to decrease, as indeed was the case for S1, S2, S3 or A1 milk, which showed consistently lower viscosities than control milk. Age gelation occurs normally after 6–24 months of storage, whereupon the apparent viscosity increases suddenly to > 10 mPa·s (Kokak and Zadow, 1985;

Table IV. Mean ($n = 6$) apparent viscosity of skim milk ultrafiltration permeate, acid whey ultrafiltration permeate and of UHT-heated milk.

Valeur moyenne ($n = 6$) de la viscosité apparente du perméat de lait écrémé, perméat de lactosérum acide ou des échantillons de lait traités par méthode UHT.

Product	Apparent viscosity (mPa·s)	
	After direct UHT ^{1,2}	After indirect UHT ^{1,2}
Skim milk permeate	1.08 (0.017)	
Acid whey permeate	1.13 (0.018)	
Control	1.74 (0.03)	1.69 (0.01)
S1	1.72 (0.02)	1.63 (0.01)
S2	1.64 (0.03)	1.59 (0.04)
S3	1.54 (0.03)	1.73 (0.15)
A1	1.65 (0.05)	1.62 (0.07)
A2	1.81 (0.21)	1.70 (0.06)
A3	NM ³	NM ³

¹ Apparent viscosity values were measured after 1 week of storage of UHT milk at 4 °C; ² storage of direct or indirect UHT S1, S2, S3 or A1 milk, for up to 12 weeks at 4 or 25 °C caused small increases (< 0.12 mPa·s) in the apparent viscosity. The A2 milk developed a very high viscosity upon storage for 4 or 12 weeks at 4 or 25 °C; viscosity could not be measured, because the excessive sedimentation impeded the flow of sample through the capillary viscometer; ³ NM: not measured; the viscosity of A3 milk could not be measured upon storage for 1, 4 or 12 weeks at 4 or 25 °C due to excessive sedimentation. S1, S2, S3 indicate milk with increasing amounts of skim milk permeate; A1, A2, A3 indicate milk with increasing amounts of acid whey permeate. Numbers in brackets show the standard deviations.

¹ Les valeurs de viscosité apparente données sont celles obtenues après 1 semaine de stockage à 4 °C du lait traité en UHT; ² le stockage des échantillons de lait S1, S2, S3 ou A1 traités par méthode UHT directe et indirecte pour une période de 12 semaines à 4 ou 25 °C a produit un léger accroissement de la viscosité apparente (< 0,12 mPa·s); ³ les échantillons de lait A2 ou A3 ont développé une viscosité très importante lors du stockage, les mesures de viscosité n'ont pu être effectuées à cause de la formation excessive de sédiment bloquant l'écoulement des échantillons dans le tube capillaire du viscosimètre; pour la même raison, la viscosité des échantillons de lait A3 n'a pu être mesurée immédiatement après le traitement UHT. S1, S2, S3, lait avec une quantité croissante en perméat de lait écrémé; A1, A2, A3, lait avec une quantité croissante en perméat de lactosérum acide. Les chiffres entre parenthèses représentent la déviation standard.

Kondal Reddy et al, 1991); by this definition, none of the milk products with skim milk permeate or the lowest level of acid whey permeate underwent age gelation.

A2 or A3 milk developed very high viscosities, indicating a destabilization effect caused by the presence of acid whey permeate and consistent with the presence of very large amounts of sediment in these milk samples, as discussed below.

Sedimentation

Sedimentation tendencies are shown in table V. Although the matrix of data is rather complex, the following are clear trends: (1) standardization of milk with skim milk permeate caused small but consistent reductions in sedimentation, evident for both direct or indirect UHT milk products; (2) the amount of sediment obtained from con-

Table V. Influence of protein standardization, UHT heating and temperature of storage on the mean ($n = 6$) mass of sediment obtained from 1 kg of milk¹.

Influence de la standardisation en protéine, du traitement UHT et de la température de stockage sur la masse moyenne ($n = 6$) de sédiment obtenue à partir d'1 kg de lait.

Heat treatment	Storage conditions (weeks)	g Sediment/kg milk		
		A1	A2	A3
Direct UHT (4 °C)	1	5.2 (1.0)	7.9 (2.7)	12.1 (2.1)
	4	6.1 (0.3)	11.6 (3.9)	16.4 (0.7)
	12	9.7 (0.3)	32.2 (7.4)	<i>129.5 (29.3)²</i>
Indirect UHT (4 °C)	1	1.9 (0.3)	1.3 (0.6)	13.5 (2.9)
	4	1.6 (0.7)	1.3 (0.5)	<i>303.5 (31.8)²</i>
	12	2.1 (0.9)	2.7 (0.6)	<i>320.2 (12.2)²</i>
Direct UHT (25 °C)	4	6.5 (0.4)	22.3 (2.2)	70.3 (6.2)
	12	6.5 (0.3)	50.5 (9.5)	<i>190.3 (9.6)²</i>
Indirect UHT (25 °C)	4	3.7 (0.6)	3.1 (0.5)	<i>198.6 (11.8)²</i>
	12	5.2 (1.9)	5.4 (1.3)	<i>210.8 (21.9)²</i>

¹ Sedimentation values for control milk or milk with skim milk ultrafiltration permeate were in the range ~1.5–5.1 g sediment/kg milk; standardization with skim milk permeate slightly reduced the rate of sedimentation; ² values in italics indicate extreme sedimentation, leading to sediments with weak custard-like consistencies, submerged beneath a translucent layer of liquid. In these cases, the mass of sediment was estimated after careful decantation of the supernatant. A1, A2 or A3 indicate milk with increasing amounts of acid whey ultrafiltration permeate. Numbers in brackets show the standard deviations.

¹ La quantité de sédiment pour l'échantillon contrôlé de lait ou le lait standardisé avec perméat de lait écrémé se trouvait dans la fourchette 1,5–5,1 g sédiment/kg lait, la standardisation avec perméat de lait écrémé réduisant légèrement la sédimentation; ² les valeurs en italique indiquent une sédimentation extrême, produisant un sédiment à consistance crémeuse submergé dans une couche liquide translucide. Dans ces circonstances, la masse de sédiment a été évaluée après décantation de la couche surnageante. A1, A2 ou A3, lait avec une quantité croissante en perméat de lactosérum acide. Les chiffres entre parenthèses représentent la déviation standard.

trol milk or milk with skim milk permeate increased gradually upon storage, the temperature of storage having little effect on the trend; (3) in general, milk with acid whey permeate displayed very high sedimentation upon storage.

The sedimentation of UHT milk should be affected by viscosity and the number, size and density of the sedimenting particles; factors leading to increased intermolecular protein interactions, including heat treatment, protein concentration and an increased concentration of salts should increase the rate of sedimentation. Dalgleish (1992) argued that sedimentation in UHT milk was due essentially to the action of gravity on casein micelles; by calculation this author showed that native micelles could sediment and their limited aggregation would cause a large increase in the rate of sedimentation. For milk with skim milk permeate, the reduced concentration of casein micelles may explain the reduced rate of sedimentation observed; this effect may have more than offset the tendency of lower viscosity (table IV) to increase the rate of sedimentation.

In UHT milk with acid whey permeate, sediments had viscous, semi-solid, custard-like consistencies and occupied from about one-third to two-thirds of the milk volume. In the most unstable milk products, phase separation occurred; the sediment was submerged beneath a greenish, translucent, whey-like layer of liquid. The composition of sediment obtained from 12-week old A3 milk is shown in table VI. The high concentration of protein, ash, calcium and phosphorus in the sediment suggests that the low colloidal stability of the protein was caused by an increased concentration of minerals. In the products with phase separation, it appeared that the entire casein component destabilized; the translucent nature of the supernatant indicated the absence of light-scattering casein micelles.

Two basic possibilities or possibly their combination ensue regarding the adverse effect of extra salts on the colloidal stability of milk proteins in UHT milk: (1) although neutralization to pH 6.8 allowed milk with acid whey permeate to be UHT-heated without visible coagulation, the presence of extra minerals may still have promoted protein aggregation on a microscopic scale during heating: such aggregation may have manifested itself as an increased rate of sedimentation during storage; (2) extra minerals could have promoted interactions between casein micelles during storage, leading to increased aggregation and hence a faster rate of sedimentation.

It seems likely that the presence of extra soluble minerals in the acid whey permeate milk products led to limited protein aggregation during heating at pH 6.8; protein coagulation was rapid when milk with the lowest level of acid whey permeate was directly UHT-heated at its unaltered pH

Table VI. Mean ($n = 2$) composition of sediment obtained from protein-standardized, direct or indirect UHT A3 milk determined after 12 weeks storage at 25 °C.

Valeur moyenne (n = 2) de la composition du sédiment contenu dans les échantillons de lait standardisés en protéine A3 traités par méthode UHT directe et indirecte après 12 semaines de stockage à 25 °C.

Component		A3 direct UHT	A3 indirect UHT
Total solids	(g/100 g)	15.94(0.47)	14.44(0.43)
Protein	(g/100 g)	5.00 (0.10)	8.93 (0.09)
Fat	(g/100 g)	1.15 (0.09)	3.88 (0.34)
Lactose	(g/100 g)	4.64 (0.20)	4.52 (0.19)
Ash	(g/100 g)	1.52 (0.03)	1.00 (0.02)
Calcium	(mg/100 g)	365 (15)	194 (7)
Phosphorus	(mg/100 g)	226 (1.6)	127 (0.7)

Numbers in brackets show the standard deviations.

Les chiffres entre parenthèses représentent la déviation standard.

(6.65), which was quite close to the neutralized milk pH of 6.8. Interactions between casein micelles might also occur upon storage and the presence of extra minerals could have a similar effect on these interactions, though the rate of collision would be reduced to 4 or 25 °C, compared to the temperature of UHT heating.

It is generally accepted that age gelation of UHT milk involves aggregation of casein micelles into a three-dimensional network (Harwalkar, 1992). It is thought that the micelles develop an increased propensity to interact, due to alterations of micelle structure at the surface; controversy exists as to whether these changes are related to non-enzymatic, physico-chemical changes or are caused by the action of plasmin or bacterial proteinases (Harwalkar, 1992). The casein in UHT milk becomes more sensitive to precipitation by calcium upon storage (Nakai et al, 1964; Samel et al, 1971); possibly, enzymatic or physico-chemical changes at the surface of the casein micelle might reduce the ability of κ -casein to stabilize the α_s - and β -caseins to calcium. In the present study, the increased ratio of soluble calcium to protein in the acid whey permeate milk products could have increased the rate of aggregation of casein micelles. However, age gelation is characterized by the development of a continuous network of casein micelles to form a thixotropic gel, without phase separation. Furthermore, age gelation is characterized by a sudden and large increase in apparent viscosity, in contrast to the more gradual process observed in the present study. Therefore, the destabilization of UHT milk with acid whey permeate was probably related to a greater rate of sedimentation rather than age gelation.

Sensory quality

The results of the sensory evaluations of the UHT milk products are shown in table VII, from which the following trends can be

noted: (1) direct UHT milk products had higher sensory scores than indirect UHT products; (2) storage of milk at 25 °C caused a gradual decline in sensory score, while at 4 °C the sensory score was almost unchanged; (3) milk at all three levels of added skim milk permeate had a sensory score similar to control milk upon storage at 4 or 25 °C for up to 12 weeks; (4) standardization of UHT milk with acid whey permeate reduced the sensory score, especially in the case of indirect UHT milk products.

The flavour of UHT milk is often described as being initially 'cooked', becoming 'stale' or 'oxidized' upon storage (Andersson and Öste, 1995b; Nursten, 1995). The cooked flavour is due to the heat-induced activation of sulphhydryl groups of proteins, especially β -lactoglobulin and bovine serum albumin; activation of sulphhydryl groups may lead to the formation of volatile sulphur-containing compounds, including hydrogen sulphide, methanethiol, dimethyl sulphide and dimethyldisulphide. These compounds have been associated frequently with a cooked flavour (Steely, 1994), though the influence of numerous other heat-generated compounds on flavour is relatively unknown (Badings and Neeter, 1980; Andersson and Öste, 1995b; Nursten, 1995).

The flavour of control milk or milk standardized with skim milk permeate after 1 week of storage at 4 °C was described as 'cooked', flavour intensity being greater in the indirect UHT milk products. Andersson and Öste (1992) demonstrated that the higher heat load used during the manufacture of the indirect UHT milk leads to a more intense cooked flavour, due to greater denaturation of whey proteins.

Although the overall sensory scores of control milk or milk containing skim milk permeate were similar, a number of panelists commented on a less intense cooked flavour in the standardized milk samples;

Table VII. Influence of protein standardization, UHT heat treatment and storage on the mean ($n = 12$) sensory score of milk.

Influence de la standardisation en protéine du traitement UHT et du stockage sur la moyenne ($n = 12$) du score d'évaluation sensorielle des échantillons de lait.

Heat treatment	Storage conditions (weeks)	Control	Overall sensory score (maximum: 10 points)					
			S1	S2	S3	A1	A2	A3
Direct UHT	(4 °C)							
	1	8.4 (1.9)	8.3 (1.7)	8.5 (1.7)	6.5 (2.3)	7.4 (1.9)	7.9 (1.6)	5.7 (1.8)
	4	9.1 (2.0)	8.6 (1.0)	8.6 (1.1)	8.3 (1.4)	7.9 (1.2)	6.9 (2.0)	5.2 (1.0)
Indirect UHT	(4 °C)							
	1	8.6 (1.3)	7.8 (1.7)	7.2 (1.1)	7.4 (1.8)	7.5 (1.9)	6.0 (1.5)	3.1 (1.2)
	4	6.8 (1.1)	7.2 (2.1)	7.2 (0.8)	7.2 (1.4)	7.2 (2.1)	6.0 (1.5)	3.0 (1.0) ¹
Direct UHT	(25 °C)							
	4	6.8 (1.7)	6.7 (1.2)	6.5 (1.1)	6.4 (1.1)	4.8 (1.9)	4.6 (1.2) ¹	4.1 (1.3) ¹
	12	6.6 (2.2)	6.4 (2.0)	6.0 (2.1)	6.1 (1.1)	4.6 (1.0)	4.4 (0.5) ¹	3.6 (1.2) ¹
Indirect UHT	(25 °C)							
	4	7.2 (1.4)	5.6 (3.2)	7.1 (2.3)	6.6 (1.1)	6.9 (1.9)	5.6 (1.0)	3.4 (0.6) ¹
	12	7.0 (1.4)	7.5 (1.6)	7.1 (1.7)	7.0 (1.0)	6.9 (1.6)	5.4 (0.8)	3.0 (0.5) ¹

¹ Samples were shaken thoroughly to disperse the large amounts of sediment prior to sensory analysis. S1, S2 or S3 indicate milk with increasing amounts of skim milk ultrafiltration permeate; A1, A2 or A3 indicate milk with increasing amounts of acid whey ultrafiltration permeate. Numbers in brackets show the standard deviations.

¹ *Les échantillons ont été agités vigoureusement avant l'analyse sensorielle, afin de disperser la quantité importante de sédiment. S1, S2 ou S3, lait avec une quantité croissante en perméat de lait écrémé; A1, A2 ou A3, lait avec une quantité croissante en perméat de lactosérum acide. Les chiffres entre parenthèses représentent la déviation standard.*

this effect was most pronounced after 1 week of storage, when the cooked flavour of all milk products was most intense. In addition to the trivial effect of a reduced concentration of whey proteins, another explanation for this observation could be that the presence of skim milk permeate increased the dissolved oxygen content of the milk. The dissolved oxygen content of the permeate was not measured, but may have been relatively high if conditions during UF were turbulent. The presence of oxygen leads to a disappearance of the cooked flavour of UHT milk, which has been correlated with the oxidation of free sulphhydryl groups (Thomas et al, 1975; Fink and Kessler, 1986; Andersson and Öste, 1992).

Storage of control milk and milk products containing skim milk permeate led to a disappearance of the cooked flavour; milk products became blander and had stale or oxidized flavours, which reduced the hedonic sensory score. The development of oxidized flavour in UHT milk upon storage has been attributed to the auto-oxidation of lipids during storage and Maillard browning to generate a variety of aldehydes and ketones (Jeon et al, 1978; Andersson and Öste, 1995b). Probably the reactions responsible for the development of stale flavour might proceed at a faster rate at 25 than at 4 °C, leading to the lower sensory quality of milk products stored at 25 °C.

Milk with acid whey permeate initially also had a cooked flavour, which became oxidized upon storage. Of greater importance was the presence of strong off-flavours; flavour was described by panelists as being 'unclean', 'unnatural', 'fermented' or 'acidic'. Rattray and Jelen (1996c) reported that the sensory quality of pasteurized milk was reduced drastically upon standardization with a whey permeate obtained from a fermented dairy product. The reduced sensory quality was attributed to low molecular weight compounds produced by lactococcal fermentation of lactose and the relatively high lactic acid and salt content of the acid whey permeate. During direct UHT heating, vacuum flash cooling of the milk may have led to volatilization and loss of some of the off-flavours present in the acid whey permeate; this may explain the higher sensory score of direct UHT milk as compared to indirect UHT milk. Despite the modest improvement in sensory quality, direct UHT milk products with acid whey permeate were still considered unacceptable for consumption.

As described above, some of the UHT milk products with acid whey permeate showed extremely high sedimentation with phase separation upon storage. Prior to sensory analysis, these samples were shaken to disperse the gel-like material to obtain milk with a uniform appearance and texture. It should be noted that the sediment did not have the character of acid-destabilized milk and that shaking was sufficient to produce milk samples with a smooth, homogenous texture. Had the milk samples not been shaken, panelists would have been presented with a gel-like material submerged beneath a greenish liquid — without doubt an extremely unappealing product, and completely inconsistent with the normal visual attributes of UHT milk. Thus, the negative sensory impression of some UHT milk products with acid whey permeate would have been much greater if their true appearance had been considered.

CONCLUSIONS

Milk with protein content down-standardized by the addition of skim milk permeate was equivalent to normal milk during direct or indirect UHT processing and with respect to heat stability, storage stability and sensory quality; but nutritional quality was reduced slightly, due to minor decreases in the content of protein, calcium, phosphorus and potassium. Changes in other micronutrients such as magnesium, potassium and vitamins A, B₁, B₆ and E were negligible. Downward standardization of milk with acid whey permeate improved the complement of micronutrients, compared to the use of skim milk permeate, as changes in calcium and phosphorus were almost negligible. In other respects, the use of acid whey permeate was unsatisfactory; neutralization to pH 6.8 was required to avoid coagulation during UHT heating, products underwent excessive sedimentation, developed high viscosities during storage and possessed unacceptably strong off-flavours.

ACKNOWLEDGMENTS

This study would have been impossible without the help of R Baderscher and the staff of Sektion Chemie at EFAM, who carried out the extremely tedious compositional assays; our sincerest gratitude to them. We thank JO Bosset and D Fuchs for the determination of vitamins. The expertise of W Strahm was essential for the collection of raw material and preparation and heat treatment of the milk products. Thanks are due also to Emmi Milch, Lucerne, for the provision of UF permeates. We thank P Eberhard and H Eyer for useful discussions in the course of the preparation of the UHT milk products. Gratitude is conveyed to U Bühler-Moor for helping to organize the sensory assays and to those who participated in the sensory work. Thanks are also extended to our colleague J Bourgois at the University of Alberta who provided the French translations. W Rattray extends sincere thanks to EFAM for the invitation to participate in a collaborative study, and for the hospitality received during a visit there.

REFERENCES

- Alkanhal HA, Abu-Lehia IH, Al-Saleh AA (1994) Changes in the quality of fresh and recombined ultra-high temperature milk during storage. *Int Dairy J* 4, 221-236
- Andersson I, Öste R (1992) Sensory development and free SH-groups in UHT milk. *Milchwissenschaft* 47, 438-441
- Andersson I, Öste R (1995a) Nutritional quality of heat processed liquid milk. In: *Heat-induced Changes in Milk* (Fox PF, ed) IDF spec issue 9501, International Dairy Federation, Brussels, Belgium, 279-307
- Andersson I, Öste R (1995b) Sensory quality of UHT milk. In: *Heat-induced Changes in Milk* (Fox PF, ed) IDF spec issue 9501, International Dairy Federation, Brussels, Belgium, 318-330
- Anonymous (1987) Bestimmung der Asche. Gravimetrisch. *Schweizerisches Lebensmittelbuch*, sect 1.5.3
- Anonymous (1995a) Report of meeting of commission D, memento 1996. In: *Report, 79th Annual Sessions of the IDF*, Vienna, Austria. International Dairy Federation, Brussels, Belgium, 27-29
- Anonymous (1995b) Bestimmung von Vitamin A und Vitamin E. Gehaltsbestimmung in Milch, Milchprodukten und Käse mit HPLC. *Doc No ME008010.612*. Federal Dairy Research Institute, Liebefeld, Switzerland
- Badings HT, Neeter R (1980) Recent advances in the study of aroma compounds of milk and dairy products. *Neth Milk Dairy J* 34, 9-30
- Burton H, Ford JE, Perkin AG et al (1970) Comparison of milks processed by the direct and indirect methods of ultra-high temperature sterilization. *J Dairy Res* 37, 529-533
- Calvo MM, Holz L (1992) Flavour of heated milks. A review. *Int Dairy J* 2, 69-81
- Dalgleish DG (1992) Sedimentation of casein micelles during the storage of ultra-high milk products - a calculation. *J Dairy Sci* 75, 371-379
- Fink R, Kessler HG (1985) Reaction kinetics study of thiamine losses in stored UHT milk. *Milchwissenschaft* 40, 709-712
- Fink R, Kessler HG (1986) Reaction kinetics evaluation of the oxidative changes in stored UHT milk. *Milchwissenschaft* 41, 90-94
- Flynn A, Power P (1985) Nutritional aspects of minerals in bovine and human milks. In: *Developments in Dairy Chemistry. 3. Lactose and Minor Constituents* (Fox PF, ed) Elsevier, London, 183-215
- Ford JE, Porter JWG, Thompson SY, Toothill J, Edwards-Webb J (1969) Effects of ultra-high temperature (UHT) processing and of subsequent storage on the vitamin content of milk. *Milchwissenschaft* 40, 713-716
- Friis MT (1986) La standardisation automatique de demain de la matière grasse et des protéines réalisée par Pasilac. *Tech Lait Market* 1007, 25-26
- Green ML, Scott J, Anderson M, Griffin MA, Glover, FA (1984) Chemical characterization of milk concentrated by ultrafiltration. *J Dairy Res* 51, 267-273
- Harwalkar VR (1992) Age gelation of sterilized milks. In: *Advanced Dairy Chemistry. 1. Proteins* (Fox PF, ed) Elsevier, London, 691-734
- Hinrichs J, Kessler HG (1995) Thermal processing of milk - processes and equipment. In: *Heat-induced Changes in Milk* (Fox PF, ed) IDF spec issue 9501, International Dairy Federation, Brussels, Belgium, 9-21
- IDF (1964) Determination of the casein nitrogen content of milk. *Standard 29*. International Dairy Federation, Brussels, Belgium
- IDF (1974) Determination of the lactose content of milk. *Standard 28A*. International Dairy Federation, Brussels, Belgium
- IDF (1987a) Milk. Determination of fat content. Gravimetric method. *Standard 1C*. International Dairy Federation, Brussels, Belgium
- IDF (1987b) Milk, cream and non-sweetened concentrated milk. Determination of total solids (reference method). *Standard 21B*. International Dairy Federation, Brussels, Belgium
- IDF (1988) Cheese and processed cheese products. Determination of chloride content. Potentiometric titration method. *Standard 88A*. International Dairy Federation, Brussels, Belgium
- IDF (1990) Milk. Determination of total phosphorus content. Spectrometric method. *Standard 42B*. International Dairy Federation, Brussels, Belgium
- IDF (1993) Milk. Determination of nitrogen content. *Standard 20B*. International Dairy Federation, Brussels, Belgium
- Jeon IJ, Thomas EL, Reineccius GA (1978) Production of volatile flavour compounds in ultra-high temperature processed milk during aseptic storage. *J Agric Food Chem* 26, 1183-1188
- Kokak HR, Zadow JG (1985) Age gelation of UHT milk as influenced by storage temperature. *Aust J Dairy Technol* 40, 14-21
- Kondal Reddy K, Nguyen MH, Kailasapathy K, Zadow JG (1991) The effects of some treatments and storage temperatures on UHT whole milk. *Aust J Dairy Technol* 46, 57-63
- Le Maguer I, Jackson H (1983). Stability of vitamin A in pasteurized and ultra-high temperature processed milks. *J Dairy Sci* 66, 2451-2458
- Lemke A, Frahm H, Wegner HH (1968) Ernährungsphysiologische Untersuchungen zur Ultraheizerhitzung der Milch. *Kiel Milchwirtsch Forschungsber* 20, 331-342
- Manji B, Kakuda Y, Arnott DR (1986) Effect of storage temperature on age gelation of ultra-high tem-

- perature milk processed by direct and indirect heating systems. *J Dairy Sci* 69, 2944-3001
- Marshall KR (1995) Protein standardization of milk products. In: *Milk Protein Definition and Standardization*. IDF special issue 9502, International Dairy Federation, Brussels, Belgium, 49-54
- Maubois JL (1991) New applications of membrane technology in the dairy industry. *Aust J Dairy Technol* 46, 91-95
- Nakai S, Wilson HK, Herreid EO (1964) Measurement of protein stability in sterile concentrated milk. *J Dairy Sci* 47, 1056-1061
- Nieuwenhuijse JA (1995) Changes in heat-treated milk products during storage. In: *Heat-induced Changes in Milk* (Fox PF, ed) IDF special issue 9501, International Dairy Federation, Brussels, Belgium, 231-255
- Nursten HE (1995) Heat-induced changes in the flavour of milk. In: *Heat-induced Changes in Milk* (Fox PF, ed) IDF special issue 9501, International Dairy Federation, Brussels, Belgium, 308-317
- Oamen EE, Hansen AP, Swartzel KR (1989) Effect of ultra-high temperature steam injection processing and aseptic storage of labile water-soluble vitamins in milk. *J Dairy Sci* 72, 614-619
- Pellegrino L, Resmini P, Luf W (1995), Assessment (indices) of heat treatment of milk. In: *Heat-induced Changes in Milk* (Fox PF, ed) IDF spec issue 9501, International Dairy Federation, Brussels, Belgium, 409-453
- Peter S, Rattray W, Jelen P (1996) Heat stability and sensory quality of fat- and protein-standardized milk. *Milchwissenschaft* 51, 611-616
- Premaratne RJ, Cousin MA (1991) Changes in the chemical composition during ultrafiltration of skim milk. *J Dairy Sci* 74, 788-795
- Pritchard GG, Coolbear T (1993) The physiology and biochemistry of the proteolytic system of the lactic acid bacteria. *FEMS Microbiol Rev* 12, 179-206
- Puhan Z (1992) Standardization of milk protein by membrane processes for product manufacture. In: *New Applications of Membrane Processes*. IDF special issue 9201, International Dairy Federation, Brussels, Belgium, 23-32
- Rattray W, Jelen P (1996a) Protein standardization of milk and dairy products. *Trends Food Sci Technol* 7, 227-234
- Rattray W, Jelen P (1996b) Thermal stability of skim milk with protein content standardized by the addition of ultrafiltration permeates. *Int Dairy J* 6, 157-170
- Rattray W, Jelen P (1996c) Freezing point and sensory quality of skim milk as affected by addition of ultrafiltration permeates for protein standardization. *Int Dairy J* 6, 569-579
- Rattray W, Gallmann P, Jelen P (1997) Influence of protein standardization and UHT heating on the furosine value and freezing point of milk. *Lait* 77, 297-305
- Rønkilde Poulsen P (1978) Feasibility of ultrafiltration for standardizing protein in milk. *J Dairy Sci* 61, 807-814
- Samel R, Weaver RWV, Gammack DB (1971) Changes on storage in milk processed by ultra-high temperature sterilization. *J Dairy Res* 38, 323-332
- Steely JS (1994) Chemiluminescence detection of sulfur compounds in cooked milk. *ACS Symp Ser* 564, 22-35
- Tagliaferri E, Bosset J, Eberhard P, Bütikofer U, Sieber R (1992a) Untersuchung einiger Kriterien zum Nachweis von Veränderungen der Vollmilch nach thermischen und mechanischen Behandlungen sowie nach verschiedenen langen Belichtungszeiten. II. Bestimmung des Vitamins B₁ mit Hilfe einer neuentwickelten RP-HPLC Methode. *Mitt Gebiete Lebensm Hyg* 83, 435-452
- Tagliaferri E, Sieber R, Bütikofer U, Eberhard P, Bosset J (1992b) Untersuchung einiger Kriterien zum Nachweis von Veränderungen der Vollmilch nach thermischen und mechanischen Behandlungen sowie nach verschiedenen langen Belichtungszeiten. II. Bestimmung des Vitamins B₂ mit Hilfe einer neuentwickelten RP-HPLC Methode. *Mitt Gebiete Lebensm Hyg* 83, 467-491
- Thomas EL, Burton H, Ford JE, Perkin AG (1975) The effect of oxygen content on flavour and chemical changes during aseptic storage of whole milk after ultra-high temperature processing. *J Dairy Res* 42, 285-295
- Walstra P, Jenness R (1984) *Dairy Chemistry and Physics*. John Wiley and Sons, New York, 42-57