Food Control 121 (2021) 107637

Contents lists available at ScienceDirect

Food Control

journal homepage: www.elsevier.com/locate/foodcont

A comparison of analytical test methods in dairy processing

Niamh Burke [a,c,*], Krzysztof Zacharski [a,b], Catherine C. Adley [c], Mark Southern [a,b]

[a] Dairy Processing Technology Centre (DPTC), University of Limerick, Limerick, V94 T9PX, Ireland
[b] Enterprise Research Centre, University of Limerick, Limerick, V94 T9PX3, Ireland
[c] School of Natural Sciences, University of Limerick, Limerick, V94 T9PX, Ireland

ARTICLE INFO

Keywords:
Dairy
Processing
Analytical methods
Sampling
Optimisation

ABSTRACT

Dairy quality strategies start at the beginning of a raw milk supply chain at farm level, but it is the obligation of the manufacturer at a dairy processing plant to ensure quality is upheld from intake to finished product. This is achieved by implementing robust quality systems, measured through sampling plans and analytical test methods. Influences on product quality and composition, and analytical test results within a dairy plant are multi-factorial including: seasonality; the quality of incoming milk and herd health; the level of skilled laboratory technicians; the level of production and the availability of equipment; and finally milk harvesting, transportation and handling. These factors, along with customer and regulatory requirements will determine the level and type of analytical testing required. In the dairy industry, manufacturers oftentimes pay little attention to the need for optimising analytical test strategies or improving laboratory operations, if it is not broken why fix it? The focus of this qualitative research was to differentiate the core current analytical test methods in use at three dairy manufacturing plants for the production of raw milk, skim milk and cream and skim milk powder (SMP). The main objective being to inform and educate each producer on best practice methods. Results displayed similarities across testing categories but demonstrated a range of traditional testing methods in the microbiological analysis compared to advanced instrumentation use in the chemical and compositional analytical category. The dairy industry needs to adapt to a modern, process focused quality system using industry 4.0 analytical processing regimes.

1. Introduction

Modern day dairy processing has seen considerable advancements in equipment and various techniques to develop product in the most efficient and sustainable way possible with a large emphasis placed on reduced energy and waste (Welch & Mitchell, 2000; Sousa et al. 2016; Poyatos-Racionero et al. 2018). In addition to advancements in dairy processing, innovative analytical test techniques and rapid methods of analysis, have paved the way for dairy manufacturers to produce safe, high quality food products. The "farm to fork" concept refers to a system whereby food production, processing, distribution and consumption are integrated to enhance clarity for the consumer (Campanhola and Pandey 2019). To ensure high yield and exceptional quality (Trienekens & Zuurbier, 2008), quality systems in any food industry must be established from raw materials right through to finished product (Aung & Chang, 2014), through optimised quality systems (Santos-Fernández et al. 2014). This is achieved through analytical testing and sampling strategies (Truchaud et al. 1997) which are governed by legislative and regulatory guidelines and standards, and also driven by customer requirements.

An imperative part of any quality system are laboratory quality standards and methods, by both customer and regulatory requirements (Barbé et al. 2017). Standard methods are determined by regulators to guide dairy processors in producing product to meet specific customer and legislative requirements (Burke, Southern, et al., 2018) (Burke, Zacharski, et al., 2018) and analytical tools to investigate foods are continually evolving to allow for more rapid, sensitive analysis (Ramaswamy et al. 2019) (Zacharski et al. 2016).

The use of spectroscopy methods have been traditionally effective in assessing quality attributes in dairy products (Nawrocka & Lamorska, 2013), infrared (IR), near-infrared (NIR), mid-infrared (MIR) and Raman spectroscopy methods for example, have allowed for fast quantitative analysis on the composition of dairy products. Fourier-transform infrared (FTIR) spectroscopy being the most commonly used in dairy processing (Nawrocka & Lamorska, 2013). However, FTIR has seldom if ever been shown to effectively measure milk microbial quality reliably.

* Corresponding author. Dairy Processing Technology Centre (DPTC), University of Limerick, Limerick, V94 T9PX, Ireland.
E-mail address: niamh.burke@ul.ie (N. Burke).

https://doi.org/10.1016/j.foodcont.2020.107637
Received 19 June 2020; Received in revised form 14 September 2020; Accepted 15 September 2020
Available online 17 September 2020

Most standard methods are developed by national standards bodies through ISO (International Standards Organisation) for example, however alternative methods of analysis may be used in place of standard methods, but must be validated by accredited organisations such as AOAC International (Association of Official Agricultural Chemists). Furthermore an alternative method must be validated against a standard method, for example direct microscopic count for Somatic Cell Count (SCC) in raw milk (ISO 2008a) Vs fluoro-opto-electronic methods (ISO 2006) or the application of mid FTIR to liquid milk (ISO 2013). It is the responsibility of the dairy manufacturer to ensure the analytical method chosen to assess food samples meets legal requirements and best practice is followed.

The analysis of a dairy product involves three simple actions, collection of a representative sample, sample preparation and finally analysis using appropriate methods and equipment. Analytical results are never more reliable than the sampling technique and although these activities are independent of each other, they can have significant influences on each other. Analysis is carried out on finished product and processes within a dairy setting. Sampling can be randomised, rotational, by attributes or by variable sampling (Barbosa-Cánovas et al. 2005) and it is essential to clearly define the population that is to be sampled in order to obtain a true analytical result. The production of high quality product is the pinnacle in ensuring how one organisation can differentiate from its competitors.

Quality systems and analytical testing regimes, in particular within the dairy industry, are rarely measured or evaluated to ensure their effectiveness. Additionally, the effects a quality system can have on process, product and environmental optimisation are also rarely assessed. More often than not, when issues arise within these categories, be it product downgrades, contamination or processing issues, management can spend too much time focusing improvement efforts on the symptoms of problems rather than on the causes.

The objective of this study was to compare and contrast the analytical test methods used by three dairy manufacturing plants across a twelve-month period. This study focused on test methods and equipment. A comparison of sampling and calibration methods were not included, but were carried out in accordance with (ISO 707, 2008), and manufacturers requirements respectively. This study evaluated the chemical, compositional and microbiological analytical test methods associated with the processing of raw milk, skim milk and cream, and skim milk powder (SMP). The aim of the study was to identify opportunities for improvements and to inform each company of best practice methods to optimise laboratory procedures.

2. Materials and methods

2.1. Site descriptions

In this study, three large dairy manufacturing plants (A, B and C) were investigated and data were collected, collated and analysed across one production year (2018). All three plants process raw milk from local suppliers in accordance with EU regulations, (EC) 852/2004 on the *hygiene of foodstuffs* (EU 2004), the health rules of milk-based products EC 92/46 (EU 1992), together with a collection of EU food control packages, in particular *the microbiological criteria for foods* (EC) No 2073/2005 (EU 2005) and *on contamination and residue levels*, (EC) 1881/2006 (EU 2006) and (EC) 2377/1990 (EU 1990) (EEC, 2003). Each plant processes in excess of 200 million litres of milk annually and manufacture a variety of dairy products, including butter, cheese and various milk powders.

2.2. Sampling and data analysis

The data were amalgamated from a laboratory information management system (LIMS) and SOP's (Standard Operating Procedures) used by each plant, and combined in Microsoft Office Excel® spreadsheets based on product categories. Analytical test methods for each product were categorised based on chemical and compositional tests and microbiological test with each corresponding reference method applied. Each plant carries out sampling and analysis at various processing points and on finished product, and this can occur hourly throughout a time-dependent production period. Sampling is carried out in accordance with International Standard, Milk and Milk Products: Guidance on Sampling (ISO 707, 2008).

2.3. Equipment validation methods

All analytical test equipment used by each of the three plants is validated and calibrated as per equipment contract requirements and legislative requirements. Equipment validation methods confirm that the analytical technique utilised for a specific test is suitable for its projected use. For the purpose of this research, an analysis of calibration methods have been excluded.

3. Results

3.1. Raw milk

Analysis of raw milk was similar across Plants A & B. The chemical and compositional analysis methods are presented in Table 1. Plant C evaluates raw milk at an off-site laboratory therefore they were not included in the following collation. A process that is not common as yet in the dairy industry due to additional costs. This might change due to demand for independent verification of milk and dairy products quality. The only variation evident in this category was the use of equipment for compositional evaluation (Table 1). Plant A use the Bentley Dairyspec combi© flow cytometry © (FC) equipment, while Plant B use Delta lactoscope© FTIR equipment. An essential aspect of the integrity of the instruments output is the calibration of the equipment. It is fundamental to ensure the instruments reading is close to the value given by the reference method. Different instruments have different calibration systems, therefore no specific procedure is given through the reference method listed in Table 1 (ISO 5764, 2009). Each plant refers to the manufacturers requirements on calibrating the equipment. Microbiological test regimes were similar between Plants A and B on raw milk intake (Table 2). The most significant difference observed in this category was the enumeration of total bacterial counts (TBC) between Plant A and B. Plant A utilise a Bentley bactocount machine to assess TBC's in milk samples, while Plant B use a traditional pour plate method. Furthermore, Plant A assess raw milk samples for thermoduric bacteria while Plant B do not (Table 2).

Table 1
Chemical and compositional tests on raw milk.

Test	Plant A	Plant B
Chemical		
Acidity	Titratable Acidity AOAC validated Resazurin (BS 4285–4 1991)	Titratable Acidity AOAC validated Resazurin (BS 4285–4 1991)
Added Water	Cryoscope (ISO 5764, 2009)	Cryoscope (ISO 5764, 2009)
Compositional:	Bentley Dairyspec ©	Delta Lactoscope FTIR©
Fat	AOAC validated	(ISO 9622, 2013)
Protein	Bentley Dairyspec © AOAC validated	Delta Lactoscope FTIR© (ISO 9622, 2013)
Lactose	Bentley Dairyspec © AOAC validated	Delta Lactoscope FTIR© (ISO 9622, 2013)
Total Solids	Bentley Dairyspec © AOAC validated	Kruss refractometer © AOAC validated

AbbreviationsISO – International Standards Organisation; FTIR - Fourier-transform infrared; AOAC – Association of Official Analytical Chemists.

Table 2
Microbiological tests on raw milk.

Test	Plant A	Plant B
Antibiotics	Charm Rosa Test® Delvo Test	Charm Rosa Test® Delvo Test
SCC	Bentley Dairyspec Combi © AOAC validated	Delta Somascope© (ISO 13366-2, 2006)
TBC	Bentley Bactocount © AOAC validated	Pour plate method (ISO 4833-1, 2013)
Thermoduric Bacteria	Oculer Technology (ISO, 16297)	N/A

Abbreviations: SCC – Somatic Cell Count; AOAC- Association of Official Analytical Chemists; ISO – International Standards Organisation; TBC – Total Bacterial Count; N/A – Not Applicable.

3.2. Cream and skim milk

Analytical tests for chemical and compositional components were identical between plants A and B (Table 3). Plant C only carry out microbiological analysis on cream and skim milk (Table 4), therefore they have also been omitted for this collation. The reasoning for which will be discussed further in the discussion section.

Variation was evident between plants with regards to microbiological analysis on cream and skim milk (Table 4). Plant C carried out a wider range of microbiological analysis than plants A and B. The majority of microbiological analysis was carried out using pour plate methods with the exception of Plant A using a Petrifilm™ for the enumeration of yeast/moulds. Petrifilm™ has been validated as an alternative for aerobic counts by the international American Public Health Association (Nelson et al. 2013).

3.3. Skim milk powder (SMP)

No variation was displayed across the chemical and compositional testing strategies on finished SMP. Differences were visible however, among methods and equipment applied, particularly for protein analysis. Plant A use a Leco machine, a Dumas method, Plant B use a Dickey John GAC III® analyser for a block-digestion method, while Plant C use a Kjedahl machine for a crude protein calculation (Table 5).

Finally, the analytical test matrix for microbiological sampling on SMP was similar across all three plants. Variation was visible between the plants for the enumeration of yeast and moulds and *Staphylococcus aureus*, where plants A and B use a 3M™ petri film in place of a pour plate method, as used by Plant C. Furthermore, Plant C displayed a smaller test matrix for microbiological assessments in comparison to Plants A and B (Table 6). Reasoning for this will be deliberated in the discussion section.

Table 3
Chemical and compositional tests on cream and skim milk.

Test	Plant A	Plant B
Chemical		
Acidity	Titratable Acidity (ISO 6091, 2010)	Titratable Acidity (ISO 6091, 2010)
Nitrates/Nitrites	Merck test strips©	Merck test strips©
pH	pH meter	pH meter
Compositional:	Delta Lactoscope©	Delta Lactoscope©
Fat	(ISO 9622, 2013)	(ISO 9622, 2013)
Protein	Delta Lactoscope© (ISO 9622, 2013)	Delta Lactoscope© (ISO 9622, 2013)
Lactose	Delta Lactoscope© (ISO 9622, 2013)	Delta Lactoscope© (ISO 9622, 2013)
Total Solids	Delta Lactoscope© (ISO 9622, 2013)	Delta Lactoscope© (ISO 9622, 2013)

AbbreviationsISO – International Standards Organisation.

Table 4
Microbiological tests on cream and skim milk.

Test	Plant A	Plant B	Plant C
TBC	Pour plate (ISO 4833-1, 2013)	Pour plate (ISO 4833-1, 2013)	Pour plate (ISO 4833-1, 2013)
Coliforms	Pour plate (ISO 4832, 2015)	Pour plate (ISO 4832, 2015)	Pour plate (ISO 4832, 2015)
Escherichia coli	Pour plate (ISO 4832, 2015)	Pour plate (ISO 4832, 2015)	Pour plate (ISO 4832, 2015)
Yeast/Moulds	Petri film (ISO 6611, 2004)	Pour plate (ISO 6611, 2004)	N/A
Bacillus cereus	N/A	N/A	Pour plate (ISO 7932, 2004)
Streptococcus	N/A	N/A	Pour plate (CCFRA 2.7.1 2007, p.1)
Thermophiles	N/A	N/A	Pour plate (ISO 27265 2009)
Enterobacteriaceae	Pour plate (ISO 21528, 2004)	N/A	Pour plate (ISO 21528, 2004)
Staphylococcus aureus	N/A	N/A	Pour plate (ISO 6888, 2003)
Sulphur Reducing Bacteria	N/A	N/A	Pour plate (ISO 15213, 2003)

Abbreviations: TBC – Total Bacterial Count; ISO – International Standards Organisation; N/A – Not Applicable; CCFRA – Campden & Chorleywood Food Research Association.

Table 5
Chemical and compositional tests on skim milk powder.

Test	Plant A	Plant B	Plant C
Acidity	Titratable Acidity (ISO 6091, 2010)	Titratable Acidity (ISO 6091, 2010)	Titratable Acidity (ISO 6091, 2010)
WPN	Spectrophotometer GEA (2009)	Spectrophotometer GEA (2009)	Spectrophotometer GEA (2009)
Moisture	Oven Method (ISO 5537, 2004)	Dickey John (ISO 5537, 2004)	Oven Method (ISO 5537, 2004)
Protein	Leco (Dumas) (ISO 14891, 2002)	Dickey John (ISO 8968-3, 2007)	Kjedahl (ISO 8968-1, 2014)
Scorched Particles	ADPI Method ADPI (1990)	ADPI Method ADPI (1990)	ADPI Method ADPI (1990)
Solubility	ISO Method (ISO 8156, 2005)	ADPI Method (ISO 8156, 2005)	ADPI Method (ISO 8156, 2005)
Density	Tap Density (ISO 8967, 2005)	Tap Density (ISO 8967, 2005)	Tap Density (ISO 8967, 2005)
Fat	Gravimetric Method (ISO 1736, 2008)	Gravimetric Method (ISO 1736, 2008)	Gravimetric Method (ISO 1736, 2008)

AbbreviationsWPN – Whey Protein Nitrogen; ISO – International Standards Organisation; ADPI – American Dairy Products Institute; GEA – Global Engineering Alliance.

4. Discussion

This study aimed to compare and contrast the analytical test methods, quality standards and equipment in use among three Irish dairy manufacturing plants. Data were collected and categorised for raw milk, skim milk and cream, and skim milk powder (SMP) for one production year (2018). Analytical test matrices were grouped based on core chemical, compositional and microbiological analysis. The overall objective being to identify disparities for optimisation to improve the quality system and laboratory operations.

The key to any quality system is to measure and monitor both product and process in a proficient way as possible, to produce safe,

Table 6
Microbiological tests on skim milk powder.

Test	Plant A	Plant B	Plant C
Antibiotics	Delvotest® (ISO 13969, 2003, p.13,969)	Delvotest® (ISO 13969, 2003)	Delvotest® (ISO 13969, 2003)
TBC	Pour Plate (ISO 4833-1, 2013)	Pour Plate (ISO 4833-1, 2013)	Pour Plate (ISO 4833-1, 2013)
Streptococci spp.	N/A	N/A	Pour plate (CCFRA 2.7.1 2007)
Clostridium	Pour plate (ISO 7937, 2004)	N/A	Pour plate (ISO 7937, 2004)
Coliform	Pour plate (ISO 4832, 2015)	Pour plate (ISO 4832, 2015)	Pour plate (ISO 4832, 2015)
Escherichia coli	Pour plate (ISO 4832, 2015)	Pour plate (ISO 4832, 2015)	Pour plate (ISO 4832, 2015)
CPS	Pour plate (ISO 6888, 2003)	Petri film (ISO 6888, 2003)	Pour plate (ISO 6888, 2003)
Yeast/Moulds	Petri film (ISO 21527–1, 2008)	Pour plate (ISO 6611, 2004)	Pour plate (ISO 6611, 2004)
B. cereus	Pour plate (ISO 7932, 2004)	Pour plate (ISO 7932, 2004)	Pour plate (ISO 7932, 2004)
Thermophiles	Pour plate (ISO 27265, 2009)	N/A	Pour plate (ISO 27265, 2009)
Enterobacteriaceae	Pour plate (ISO 21528, 2004)	Pour plate (ISO 21528, 2004)	Pour plate (ISO 21528, 2004)
Sulphur reducing bacteria	Pour plate (ISO 15213, 2003)	Pour plate (ISO 15213, 2003)	Pour plate (ISO 15213, 2003)

Abbreviations: TBC – Total Bacterial Count; CPS Coagulase positive *Staphylococcus*; N/A – Not applicable; ISO – International Standards Organisation.

high-quality product. This is achieved through robust analytical test matrices determined by both regulatory bodies and customers. Depending on the targeted food system, the processing environment and equipment capabilities, each product will require specific test matrices. Powdered milk, for use in infant formula and medicinal formulations, will require a stringent microbial test matrix (Kent et al. 2015), while exported milk products may require certain tests to be carried out using particular equipment and standards in accordance with a particular country. All three plants manufacture similar dairy ingredients from raw cow milk supplied by local farmers. The analytical test matrix for raw milk was the first parameter categorised and compared. Plant's A and B both intake raw milk on-site. Plant C was omitted from this grouping due to testing milk off-site. Results demonstrated similar analysis across both categories (Tables 1 and 2). The analysis of raw milk upon intake at all dairy plants is heavily legislated. Milk must be free from antibiotic residues and be of high quality. Furthermore, manufacturers pay their suppliers based on high-quality milk, e.g. fat, protein and lactose contents and the level of total bacterial counts (TBC) and somatic cell counts (SCC) will determine milk levies. Therefore little variation across this category was expected. The most significant observation in the raw milk category was the difference in how both plants enumerate for total bacterial counts (TBC's). Plant A use a Bentley Bactocount©, a fully automated flow cytometer that can analyse up to 150 samples per hour. Plant B use a traditional pour plate method (ISO 4833–1 2019), whereby a colony count is obtained after utilising a pour plate technique and incubating at 30 °C under aerobic conditions for 72 h. This is an accepted method for TBC analysis on product downstream from intake. However, it is heavily time-consuming for use on raw milk samples. Laboratory turnaround time (TAT) is an essential key performance indicator (KPI) (Burke, Southern, et al., 2018). Using quicker methods such as flow cytometry (FC) or infrared (IR) techniques will save on TAT, labour, cost of consumables and time to result. Also in this category, it was noted Plant A, test incoming samples for thermoduric bacteria and some milk buyers are introducing thermoduric testing for payment. Thermoduric bacteria counts and indicate poor hygiene practices at farm level. However this test is not widespread as yet.

The test matrices for cream and skim milk again were similar across Plants A and B for chemical and compositional analysis. Both plants use a Delta lactoscope©, an FTIR analyser, (Table 3). Plant C does not carry out in-process chemical and compositional testing due to process control. In the event of loss of process control, evident in final product, corrective action procedures are implemented and testing is applied. More often than not, sampling and analysis within a dairy processing setting is product rather than process-driven. Research suggests, however, optimised process validation can reduce test frequencies and give the manufacturer confidence in the product they are producing. Collation of the microbiological test matrices shows evident variation across the three plants (Table 4). Plants A and B again had a similar test matrix, while Plant C displayed an extended sampling strategy for microbiological analysis. In general, Plant C's quality systems are process-driven rather than product-driven.

No variation was visible across all plants for chemical and compositional analysis on SMP, except for the analysis of protein (Table 5). All three plants utilise different equipment and standard methods, Plant A use standard method (ISO 14891, 2002), while Plant's B and C use two different versions of the standard method (ISO 8963–1/ISO 8963–3). Plant A, use a routine method of combustion according to the Dumas principle. Plant's B and C use similar methods, a crude protein calculation based on the Kjeldahl principle and a block digestion method respectively. The difference between Kjeldahl and Dumas methods have been widely researched (DeVries et al. 2017; Thompson et al. 2002). The Dumas method determines total nitrogen including inorganic matter while Kjeldahl determines only organic nitrogen (Müller, 2017). In terms of analytical reliability and faster time-to-result, the Dumas method would be the more desirable method for milk powder protein analysis.

Finally, for microbiological enumeration on SMP, little variation was evident across the three test matrices. All three plants follow the same standard methods. Plant A uses a rapid Petrifilm™ method for the analysis of yeast/moulds (RYM) while plant B use a Petrifilm™ for the analysis of *Staphylococcus aureus*. There are many Petrifilm™ plate systems e.g. aerobic counts, Coliform counts, *E.coli*/coliform. Using Petrifilm™ in place of a pour plate method would display no significant difference in results. However, Petrifilm™ eliminates the need to prepare media and plate therefore reducing labour time and cost of laboratory consumables (Souza et al. 2015).

This study further highlights the need for rapid microbiological test methods to reduce time and cost in the dairy laboratory setting. It also emphasises the necessity for continuous revision of quality standard methods. Dairy processing has significantly changed in modern times in comparison with historic analytic methods that are still being used. They work but are time consuming, labour and consumable intensive.

5. Conclusion

This study qualitatively assessed and compared the analytical test methods and standards across three dairy manufacturing plants. This research has provided each of the dairy manufacturing plants with the ability to re-assess their test matrices, particularly on in-process analysis across all categories. Finally, this study presents an opportunity for other dairy manufacturing plants to compare their analytical test methods to industry best practice.

CRediT author contribution statement

Catherine C. Adley: Conceptualisation, Writing - review & editing, Methodology, Writing - review & editing. **Mark Southern:** Conceptualisation, Methodology, Data Collection, Data curation, Formal analysis, Writing - original draft, Writing - review & editing, Validation.

Declaration of competing interest

I declare there are no conflicts of interest in this research.

Acknowledgements

This work was supported by the Irish State through funding from the Technology Centre's program - Grant Number TC/2014/0016.

References

ADPI. (1990). Determination of scorched particles and solubility index. In *Standards for grades of dry milks including methods of analysis*. Chicago, USA: American Dry Products Institute.

Aung, M. M., & Chang, Y. S. (2014). Traceability in a food supply chain: Safety and quality perspectives. *Food Control, 39*, 172–184.

Barbé, B., Yansouni, C. P., Affolabi, D., & Jacobs, J. (2017). Implementation of quality management for clinical bacteriology in low-resource settings. *Clinical Microbiology and Infections, 23*(7), 426–433.

Sampling. In Barbosa-Cánovas, G. V., Ortega-Rivas, E., Juliano, P., & Yan, H. (Eds.), *Food powders: Physical properties, processing, and functionality*, (pp. 3–17). (2005) (pp. 3–17). Boston, MA: Springer US.

BS. (1991). *Microbiological examination for dairy purposes - methods for assessment of hygienic conditions* (pp. 4285–4294). London: British Standards Institution.

Burke, N., Southern, M., Ryan, A., & Adley, C. C. (2018). Investigating the current skim milk powder inspection strategies for improvements in process optimisation. *Food Control, 94*, 17–21.

Burke, N., Zacharski, K. A., Southern, M., Hogan, P., Ryan, M. P., & Adley, C. C. (2018). The dairy industry: Process, monitoring, standards, and quality. *Descriptive food science, IntechOpen*.

Agrifood systems. In Campanhola, C., & Pandey, S. (Eds.), *Sustainable food and agriculture*, (pp. 305–330). (2019) (pp. 305–330). Academic Press, Elsevier Inc.

2.7.1 CCFRA. (2007). *Enumeration of presumptive Enterococcus species: Colony count technique*. United Kingdom': Campden & Chorleywood Food Research Association.

DeVries, J. W., Greene, G. W., Payne, A., Zbylut, S., Scholl, P. F., Wehling, P., Evers, J. M., & Moore, J. C. (2017). Non-protein nitrogen determination: A screening tool for nitrogenous compound adulteration of milk powder. *International Dairy Journal, 68*, 46–51.

EU Council Regulation (EEC). (2003). No 2377/90 of 26 june 1990 laying down a community procedure for the establishment of maximum residue limits of veterinary medicinal products in foodstuffs of animal origin'. *Official Journal of the European Union*. L224, last amended in Commission Regulation (EC) No 1029, of 16 June 2003 L 149 15.

No 2073/2005 of 15 November EU Council Regulation (EEC). (2005). On microbiological criteria for foodstuffs. *Official Journal of the European Union*, 22/12/2005, L338/1.

No 1881/2006 of 19 December EU Council Regulation (EEC). (2006). Setting maximum levels for certain contaminants in foodstuffs. *Official Journal of the European Union*. L364/5.

EU Council Regulation (EEC). (No 853/2004). *Of 29 april 2004 laying down specific hygiene rules for food of animal origin'*. Official Journal of the European Union.

GEA. (2009). *GEA niro method No. A 21 Whey protein nitrogen index*. Düsseldorf, Germany: GEA Group.

ISO 13366-1. (2008a). *Milk - enumeration of somatic cells - Part 1: Microscopic method' (reference method)*. Geneva, Switzerland: International Organization for Standardization.

ISO 13366-2. (2006). *Milk - enumeration of somatic cells - part 2: Guidance on the operation of fluoro-opto-electronic counters*. Geneva, Switzerland: International Organization for Standardization.

ISO 13969. (2003). *Milk and milk products - guidelines for a standardized description of microbial inhibitor tests*. Geneva, Switzerland: International Organization for Standardization.

ISO 14891. (2002). *Milk and milk products - determination of nitrogen content - routine method using combustion according to the Dumas principle*. Geneva, Switzerland: International Organization for Standardization.

ISO 15213. (2003). *Microbiology of food and animal feeding stuffs - horizontal method for the enumeration of sulfite-reducing bacteria growing under anaerobic conditions*. Geneva, Switzerland: International Organization for Standardization.

ISO 16297. (2020). *Milk — bacterial count — protocol for the evaluation of alternative methods'*. Geneva, Switzerland: International Organization for Standardization.

ISO 707. (2008). *(IDF 50:2008) 'Milk and Milk products-Guidance on sampling*. Geneva, Switzerland: International Organization for Standardization.

ISO 1736. (2008). *Dried milk and dried milk products – determination of fat content - gravimetric method (reference method)*. Geneva, Switzerland: International Organization for Standardization.

ISO. (2009). *Dried milk - enumeration of specially thermoresistant spores of thermophilic bacteria* (p. 27265). Geneva, Switzerland: International Organization for Standardization.

ISO 21527-1. (2008). *Microbiology of Food and animal feeding stuffs - horizontal method for the enumeration of yeasts and moulds'*. Geneva, Switzerland: International Organization for Standardization.

ISO 21528. (2004). *Microbiology of food chain - horizontal methods for the detection and enumeration of enterobacteriaceae - Part 2: Colony-count method*. Geneva, Switzerland: International Organization for Standardization.

ISO 4832. (2015). *'Microbiology of Food and animal feeding stuffs- horizontal Method for the Enumeration of microorganisms – Part 2: Horizontal Method for the Enumeration of coliforms*. Geneva, Switzerland: International Organization for Standardization.

ISO 4833-1. (2013). *Microbiology of the food chain.- horizontal method for the detection and enumeration of microorganisms – Part 1. Colony count at 30C by the pour plate method'*. Geneva, Switzerland: International Organization for Standardization.

ISO 5537. (2004). *'Dried milk–Determination of moisture content*. Geneva, Switzerland: International Organization for Standardization.

ISO 5764. (2009). *Milk-determination of freezing point - thermistor crysocope method (reference method)*. Geneva, Switzerland: International Organization for Standardization.

ISO 6091. (2010). *Dried milk determination of titratable Acidity (reference method)'*. Geneva, Switzerland: International Organization for Standardization.

ISO 6611. (2004). *Milk and milk products- enumeration of colony forming units of yeasts and moulds- colony count technique at 25 degrees*. Geneva, Switzerland: International Organization for Standardization.

ISO 6888. (2003). *'Microbiology of food and animal feeding stuffs – horizontal method for the enumeration of coagulase-positive staphylococci (Staphylococcus aureus and other species) – Part 3: Detection and MPN technique forlow number'*. Geneva, Switzerland: International Organization for Standardization.

ISO 7932. (2004). *Microbiology of food and animal feeding stuffs- horizontal method for the enumeration of presumptive Bacillus cereus colony count technique at 30 degrees*. Geneva, Switzerland: International Organization for Standardization.

ISO 7937. (2004). *Microbiology of food and animal feeding stuffs- horizontal method for the enumeration of Clostridium perfringens- colony count technique*. Geneva, Switzerland: International Organization for Standardization.

ISO 8156. (2005). *'Dried milk and dried milk products – determination of insolubility index*. Geneva, Switzerland: International Organization for Standardization.

ISO 8967. (2005). *Dried milk and dried milk products- determination of bulk density*. Geneva, Switzerland: International Organization for Standardization.

ISO 8968-1. (2014). *Milk and milk products. Determination of nitrogen content – Part 1: Kjeldahl principle and crude protein calculation'*. Geneva, Switzerland: International Organization for Standardization.

ISO 8968-3. (2007). *Milk-determination of nitrogen content - Part 3: Block digestion method*. Geneva, Switzerland: International Organization for Standardization.

ISO 9622. (2013). *Milk and liquid milk products — Guidelines for the Application of mid-infrared spectrometry'*. Geneva, Switzerland: International Organization for Standardization.

Kent, R. M., Fitzgerald, G. F., Hill, C., Stanton, C., & Ross, R. P. (2015). Novel approaches to improve the intrinsic microbiological safety of powdered infant milk formula. *Nutrients, 7*(2), 1217–1244.

No 92/46/EEC of 16 June EU Council Regulation (EEC). (1992). Laying down the health rules for the production and placing on the market of raw milk, heat-treated milk and milk-based products'. *Official Journal of the European Union*. L 268, 14.9.1992, p 1.

Müller, J. (2017). Dumas or Kjeldahl for reference analysis', comparison and considerations for Nitrogen/protein analysis of food and feed. available https://www.fossanalytics.com. documents ⟩ papers ⟩ laboratories-segment [accessed 12/03/2020].

Nawrocka, A., & Lamorska, J. (2013). Determination of food quality by using spectroscopic methods. In *Advances in agrophysical research* (Vol. 14, pp. 347–367). IntechOpen Publishers.

Nelson, M. T., LaBudde, R. A., Tomasino, S. F., & Pines, R. M. (2013). Comparison of 3M™ Petrifilm™ aerobic count plates to standard plating methodology for use with AOAC antimicrobial efficacy methods 955.14, 955.15, 964.02, and 966.04 as an alternative enumeration procedure: Collaborative study. *Journal of AOAC International, 96*(4), 717–722.

Poyatos-Racionero, E., Ros-Lis, J. V., Vivancos, J.-L., & Martinez-Manez, R. (2018). Recent advances on intelligent packaging as tools to reduce food waste. *Journal of Cleaner Production, 172*, 3398–3409.

Ramaswamy, R., Ahn, J., Balasubramaniam, V., Saona, L. R., & Yousef, A. E. (2019). Food safety engineering. In *Handbook of farm, dairy and food machinery engineering* (pp. 91–113). Elsevier.

Santos-Fernández, E., Govindaraju, K., & Jones, G. (2014). A new variables acceptance sampling plan for food safety. *Food Control, 44*, 249–257.

Sousa, S. G., Delgadillo, I., & Saraiva, J. A. (2016). Human milk composition and preservation: Evaluation of high-pressure processing as a non-thermal pasteurization Technology. *Critical Reviews in Food Science and Nutrition, 56*(6), 1043–1060.

Souza, L. M. J. de, Costa, A. C., Nero, L. A., Couto, E. P., & Ferreira, M. de A. (2015). Evaluation of Petrifilm" system compared with traditional methodology in count of indicators of sanitary-hygienic quality and pathogenic microorganisms in sheep milk. *Food Science and Technology, 35*, 375–379.

Thompson, M., Owen, L., Wilkinson, K., Wood, R., & Damant, A. (2002). A comparison of the Kjeldahl and Dumas methods for the determination of protein in foods, using data from a proficiency testing scheme. *Analyst, 127*(12), 1666–1668.

Trienekens, J., & Zuurbier, P. (2008). Quality and safety standards in the food industry, developments and challenges. *International Journal of Production Economics, 113*(1), 107–122.

Truchaud, A., Le Neel, T., Brochard, H., Malvaux, S., Moyon, M., & Cazaubiel, M. (1997). New tools for laboratory design and management. *Clinical Chemistry, 43*(9), 1709–1715.

Welch, R., & Mitchell, P. (2000). Food processing: A century of change. *British Medical Bulletin, 56*(1), 1–17.

Zacharski, Z. A., Southern, M., & Adley, C. C. (2016). Industry 4.0 vision considerations in a Quality Control sampling regime in a dairy manufacturing plant. In *33rd international Manufacturing Conference (IMC33)*. Ireland: University of Limerick, 31st August – 1st September 2016.