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THE PREPARATION OF DRIED MILK AND DRIED DAIRY PRODUCTS FOR ANALYSIS OF THEIR COMPOSITION PRINCIPLES AND CRITICAL POINTS

Dried milk and dried dairy products are subject to composition criteria, in particular, moisture and fat contents.

To ensure compliance with these criteria and also monitor the production operations, these products are regularly analysed using chemical or instrumental analytical methods.

For these methods, a preparation of the sample is required. This step is generally described in the standardised analytical methods (AFNOR and/or ISO in particular):

- **ISO 5537: Dried milk – Determination of moisture content**
- **ISO 5543: Caseins and caseinates - Determination of fat content**
- **ISO 21543: Milk products – Guidelines for the application of near infrared spectrometry**

As with all the other dairy products, the steps of sampling and samples preparation of the dried products are essential to achieving results, accurate and representative of the initial sample.
The sub-sampling step is often not necessary for these types of products, because it is not necessary to carry out a mass reduction of the samples received in this context (except for caseins and caseinates in some cases)

You will find below a description of these different steps, their objectives, the operating conditions to set up and critical points to control.

Sampling

As with the most analytical standards, the sampling part is not included in the documents concerning the composition determination of these products. In this case, reference may be made to the ISO 707 | IDF 50 standard which specifies the sampling methods to be applied in a production area.

Once the sample has been taken, it should be packed in an airtight and waterproof container and then stored at room temperature. As these types of products are very hygroscopic, special attention should be paid to the watertight of the container to ensure the stability of the product during storage before analysis: presence of cap with airtight seal in the case of vials, nature of the material and characteristic of its water permeability in the case of bags, etc.

Moreover, the choice of the container (type and volume, and time of storage before analysis) should be carried out taking into account many elements of the preparation step.

The main critical points of this step are:

- **The representativeness of the sample taken in relation to the product to be characterised**
- **Compliance with the choice conditions of the container adapted to the use.**

Preparation

The preparation recommended in the standards seems to be simple (except for caseins and caseinates which will be developed at the end of this chapter), but this step can nevertheless have a significant influence on the later analytical determinations.

Its general principle is to mix the sample by shaking and rotating in a container **with a capacity of about twice the volume of the sample** in order to be able to carry out test samples representative of the sample received.
In fact, during storage, a sedimentation of the grains according to their size and a gas exchange between the external layer of the product and the air in the headspace can be observed, leading to a potential non-homogeneity in the sample received.

From a practical point of view, the initial volume of the container vs the quantity of the sample will therefore determine whether the laboratory should transfer the sample to a container with a larger volume or if shaking is possible in the initial container. In the initial reflection on this point, it will also be necessary to take into account that any transfer involves a risk of moisture absorption.
The choice should therefore be considered beforehand by integrating the analytical methods (criteria, time before analysis, etc.) and the objectives of the test.

A specific method for caseins and caseinates

For caseins and caseinates, after shaking such as that described above, a grinding may be necessary if the particle size of the product is greater than 500 µm (using a specific sieve). This point is specified in the analytical methods and is very important for the accuracy of the results obtained.

Test sample

Once the sample is homogeneous, the laboratory should carry out its test samples for all the determinations in a short time interval so as not to expose the product to ambient air and thus see an evolution of its moisture content.
In the course of these operations, the laboratory has to ensure that the weighing is carried out as quickly as possible and also that the container is closed between each test sample.

If the laboratory wishes to keep the sample for a duplicate analysis, for example, the storage methods must be defined and validated to guarantee that the product does not change over time.

The main critical points of this step are:

- **The choice of the container according to the analysis methods and the objectives**
- **Compliance with the shaking methods as described above, which could cause, if they were not applied, a non-representative and non-homogenous test sample**
- **The shortest possible exposure to the air of the product to be analysed**
- **A quick realisation of the sample tests.**

Conclusion

As you understand, compliance with good practices during these steps is the only solution to ensure the quality of the determinations that are carried out in the laboratories. Indeed, only this respect will ensure the representativeness of the initial sample throughout the analytical process and also the quality of the associated analytical determinations.

Philippe TROSSAT

STANDARDS, DRAFT STANDARDS

Classification in alphabetical order by theme

ISO published standards

MICROBIOLOGY OF THE FOOD CHAIN	
ISO 6579-1/Amd1 March 2020	MICROBIOLOGY OF THE FOOD CHAIN Horizontal method for the detection, enumeration and serotyping of *Salmonella* – Part 1: Detection of *Salmonella* spp. – Amendment 1
MILK-BASED INFANT FORMULA POWDERS	
ISO 23293 (IDF 247) February 2020	MILK-BASED INFANT FORMULA POWDERS Quantification of whey protein content by sodium dodecyl sulfate-capillary gel electrophoresis (SDS-CGE)
QUALITY	
ISO 2859-2 February 2020	Sampling procedures for inspection by attributes – Part 2: Sampling plans indexed by limiting quality (LQ) for isolated lot inspection *Replace ISO 2859-2:1985*
SENSORY ANALYSIS	
ISO 11136/Amd1 January 2020	SENSORY ANALYSIS Methodology – General guidance for conducting hedonic tests with consumers in a controlled area – Amendment 1
STATISTICS	
ISO 5725-4 March 2020	Accuracy (trueness and precision) of measurement methods and results – Part 4: Basic methods for the determination of the trueness of a standard measurement method *Replace ISO 5725-4:1994*

ISO standards under development

CHEESE AND PROCESSSED CHEESE PRODUCTS, CASEINS AND CASEINATES	
ISO/DIS 23319 June 2020	CHEESE AND PROCESSED CHEESE, CASEINS AND CASEINATES Determination of fat content – Gravimetric method
MILK	
ISO/DIS 8196-3 June 2020	MILK Definition and evaluation of the overall accuracy of alternative methods of milk analysis – Part 3: Protocol for the evaluation and validation of alternative quantitative methods of milk analysis
MILK, MILKPRODUCTS AND INFANT FORMULAE	
ISO/DIS 23970 April 2020	MILK, MILK PRODUCTS AND INFANT FORMULAE Determination of melamine and cyanuric acid by liquid chromatography and tandem mass spectrometry (LC-MS/MS)
SENSORY ANALYSIS	
ISO/DIS 11132 April 2020	SENSORY ANALYSIS Methodology – Guidelines for monitoring the performance of a quantitative sensory panel
ISO/DIS 4120 June 2020	SENSORY ANALYSIS Methodology – Triangle test

NEW EU REGULATIONS

Classification is established in alphabetical order of the first keyword

ADDITIVES

O.J.E.U. L 59, 28th February 2020 – Commission Regulation (EU) 2020/279 of 27 February 2020 amending Annex II to Regulation (EC) No 1333/2008 of the European Parliament and of the Council as regards the use of soybean hemicellulose (E 426)
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2020.059.01.0006.01.ENG

NOVEL FOOD

O.J.E.U. L 109, 7th April 2020 – Commission Implementing Regulation (EU) 2020/500 of 6 April 2020 authorising the placing on the market of partially defatted chia seed (Salvia hispanica) powders as novel foods under Regulation (EU) 2015/2283 of the European Parliament and of the Council and amending Commission Implementing Regulation (EU) 2017/2470
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2020.109.01.0002.01.ENG

P.D.O. / P.G.I.

O.J.E.U. C 46, 11th February 2020 – Publication of an application for approval of non-minor amendments to a product specification pursuant to Article 50(2)(a) of Regulation (EU) No 1151/2012 of the European Parliament and of the Council on quality schemes for agricultural products and foodstuffs [Cantal/Fourme de Cantal/Cantalet (cheese) (PDO)]
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.C_.2020.046.01.0014.01.ENG

O.J.E.U. L 48, 21st February 2020 – Commission Implementing Regulation (EU) 2020/237 of 14 February 2020 entering a name in the register of protected designations of origin and protected geographical indications [Bjelovaeski kvargl (cheese) (PGI)]
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2020.048.01.0002.01.ENG

O.J.E.U. C 60, 24th February 2020 – Publication of the amended single document following the approval of a minor amendment pursuant to the second subparagraph of Article 53(2) of Regulation (EU) No 1151/2012 [Edam Holland (cheese) (PGI)]
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.C_.2020.060.01.0007.01.ENG

O.J.E.U. L 51, 25th February 2020 – Commission Implementing Regulation (EU) 2020/247 of 18 February 2020 entering a name in the register of protected designations of origin and protected geographical indications [Queso Castellano (cheese) (PGI)]
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2020.051.01.0003.01.ENG

O.J.E.U. C 64, 27th February 2020 – Publication of an application for approval of non-minor amendments to a product specification pursuant to Article 50(2)(a) of Regulation (EU) No 1151/2012 of the European Parliament and of the Council on quality schemes for agricultural products and foodstuffs [Brie de Meaux (cheese) (PDO)]
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.C_.2020.064.01.0041.01.ENG

O.J.E.U. C 83, 13th March 2020 – Publication of an application for approval of non-minor amendments to a product specification pursuant to Article 50(2)(a) of Regulation (EU) No 1151/2012 of the European Parliament and of the Council on quality schemes for agricultural products and foodstuffs [Brie de Melun (cheese) (PDO)]
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.C_.2020.083.01.0077.01.ENG

O.J.E.U. L 81, 18th March 2020 – Commission Implementing Regulation (EU) 2020/410 of 12 March 2020 approving non-minor amendments to the specification for a name entered in the register of protected designations of origin and protected geographical indications [Queso de Valdeon (cheese) (PGI)]
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2020.081.01.0081.01.ENG

O.J.E.U. C 115, 7th April 2020 – Publication of an application for approval of an amendment, which is not minor, to a product specification pursuant to Article 50(2)(a) of Regulation (EU) No 1151/2012 of the European Parliament and of the Council on quality schemes for agricultural products and foodstuffs [Piave (cheese) (PDO)]
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.C_.2020.115.01.0021.01.ENG

O.J.E.U. L 121, 20th April 2020 – Commission Implementing Regulation (EU) 2020/541 of 7 April 2020 entering a name in the register of protected designations of origin and protected geographical indications [Gyor-Moson-Sopron megyei Csemege sajt (cheese) (PGI)]
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2020.121.01.0003.01.ENG

PESTICIDES

O.J.E.U. L 135, 29th April 2020 – Commission Implementing Regulation (EU) 2020/585 of 27 April 2020 concerning a coordinated multiannual control programme of the Union for 2021, 2022 and 2023 to ensure compliance with maximum residue levels of pesticides and to assess the consumer exposure to pesticides residues in and on food of plant and animal origin
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2020.135.01.0001.01.ENG

AFNOR VALIDATIONS

During its January meeting, the Technical Committee of NF VALIDATION approved by vote:

Commercial name	Date	Certificate	Description
RENEWALS OF VALIDATIONS			
MICROSEQ® *LISTERIA MONOCYTOGENES*	Validation date: 1 Dec 2011 Renewal: 6 Jul 2015 and 30 Jan 2020 **End of validity: 1 Dec 2023**	**ABI-29/05-12/11**	**Detection of *Listeria monocytogenes*** All human food and production environmental samples
COMPASS *LISTERIA* AGAR DENOMBREMENT	Validation date: 4 Dec 2007 Extension: 4 Oct 2013 Renewal: 10 May 2012, 6 Jul 2015 and 30 Jan 2020 **End of validity: 4 Dec 2023**	**BKR-23/05-12/07**	**Enumeration of *Listeria monocytogenes*** All human food products and production environmental samples
VIDAS® *LISTERIA MONOCYTOGENES* II (LMO2)	Validation date: 12 Mar 2004 Extension: 2 Dec 2004, 14 Dec.2006, 30 Jun 2011, 29 Jan 2016 and 4 Oct 2018 Renewal: 17 Jan 2008, 2 Feb 2012, 30 Jun 2016 and 30 Jan 2020 **End of validity: 12 Mar 2024**	**BIO-12/11-03/04**	**Detection of *Listeria monocytogenes*** All human food products and production environmental samples
MICROSEQ® *LISTERIA* SPP.	Validation date: 1 Dec 2011 Renewal: 6 Jul 2015 and 30 Jan 2020 **End of validity: 1 Dec 2023**	**ABI-29/04-12/11**	**Detection of *Listeria* spp.** All human food and production environmental samples
TRANSIA PLATE *LISTERIA*	Validation date: 11 Nov 1995 Renewal: 11 Feb 2000, 11 Dec 2003, 4 Dec 2007, 6 Oct 2011, 6 Jul 2015 and 30 Jan 2020 **End of validity: 21 Nov 2023**	**TRA-02/06-11/95**	**Detection of *Listeria*** All human food products and production environmental samples
VIDAS UP *LISTERIA* (LPT)	Validation date: 10 May 2012 Renewal: 18 Mar 2016 and 2 Apr 2020 **End of validity: 10 May 2024**	**BIO-12/33-05/12**	**Detection of *Listeria* spp.** All human food products and industrial production environmental samples
RAPID' *E. COLI* O157:H7	Validation date: 27 Sep 2007 Renewal: 6 Oct 2011, 14 Oct 2015 and 30 Jan 2020 **End of validity: 27 Oct 2023**	**BRD-07/14-09/07**	**Detection of *E. coli* O157** All human food products and production environmental samples
***SALMONELLA* PRECIS™**	Validation date: 4 Dec 2007 Renewal: 6 Oct 2011, 6 Jul 2015 and 30 Jan 2020 **End of validity: 4 Dec 2023**	**UNI-03/06-12/07**	**Detection of *Salmonella* spp.** All human and animal food products and production environmental samples (except primary production environment)

AFNOR VALIDATIONS

SESAME *SALMONELLA* TEST	Validation date: 4 Dec 2007 Extension: 3 Jul 2009 Renewal: 7 Oct 2011, 6 Jul 2015 and 2 Apr 2020 **End of validity: 4 Dec 2023**	**BKR-23/04-12/07**	**Detection of motile *Salmonella*** All human and animal food products and production environmental samples (except primary production environment)
RAPID'*SAKAZAKII*	Validation date: 10 May 2012 Renewal: 17 Mar 2016 and 2 Apr 2020 **End of validity: 10 May 2024**	**BRD-07/22-05/12**	**Detection of *Cronobacter* spp.** Powdered infant formula and cereals with or without probiotics
IQ-CHECK *CRONOBACTER* SPP.	Validation date: 31 Jan 2013 Extension: 3 Oct 2013 Renewal: 27 Jan 2017 and 02.04.2020 **End of validity: 31 Jan 2025**	**BRD-07/23-01/13**	**Detection of *Cronobacter* spp.** Powdered infant formula and cereals with or without probiotics and production environmental samples
EXTENSIONS OF VALIDATIONS			
BACGENE *LISTERIA* SPP.	Validation date: 26 Jan 2017 Extension: 21 Mar 2019 and 30 Jan 2020 **End of validity: 26 Jan 2021**	**EGS-38/02-01/17**	**Detection of *Listeria* spp.** All human food products and production environmental samples
BACGENE *LISTERIA* MULTIPLEX	Validation date:14 Mar 2017 Extension: 21 Mar 2019 and 30 Jan 2020 **End of validity: 14 Mar 2021**	**EGS-38/05-03/17**	**Detection of *Listeria* spp. and *Listeria monocytogenes*** All human food products and production environmental samples
BACGENE *LISTERIA MONOCYTOGENES*	Validation date: 26 Jan 2017 Extension: 21 Mar 2019 and 30 Jan 2020 **End of validity: 26 Jan 2021**	**EGS-38/03-01/17**	**Detection of *Listeria monocytogenes*** All human food products and production environmental samples
COMPASS *BACILLUS CEREUS* AGAR	Validation date: 5 Feb 2010 Extension: 30 Jan 2020 Renewal: 28 Nov 2013 and 17 May 2018 **End of validity: 5 Feb 2022**	**BKR-23/06-02/10**	**Enumeration of presomptive *Bacillus cereus*** All human and animal food products
SALMA ONE DAY	Validation date: 23 Mar 2017 Extension: 18 May 2017 and 2 Apr 2020 **End of validity: 23 Mar 2021**	**BIO-12/41-03/17**	**Detection of *Salmonella* spp.** All human food products and feed products and industrial production environmental samples from surface area (except primary production environment)
VIDAS EASY *SALMONELLA*	Validation date: 20 Sep 2005 Extension: 30 Jun 2011, 30 Jan 2014, 14 Oct 2015 and 2 Apr 2020 Renewal: 2 Jul 2009, 4 Jul 2013 and 23 Nov 2017 **End of validity: 20 Sep 2021**	**BIO-12/16-09/05**	**Detection of *Salmonella* spp.** All human food products, feed products and production environmental samples (except primary production environment)

AFNOR VALIDATIONS

VIDAS UP *SALMONELLA (SPT)*	Validation date: 6 Oct 2011 Extension: 2 Feb 2012, 6 Jul 2012, 31 Jan 2013, 30 Jan 2014, 15 May 2014, 14 Oct 2015 and 2 Apr 2020 Renewal: 14 Oct 2015, and 28 Nov 2019 **End of validity: 6 Oct 2023**	**BIO-12/32-10/11**	**Detection of *Salmonella* spp.** All human food products, animal feeding stuffs and production environmental samples (including animal faeces and environmental samples from the primary production stage)
EXTENSIONS OF VALIDATIONS VALIDITY			
TRANSIA PLATE *LISTERIA MONOCYTOGENES*	Validation date: 27 Mar 2008 Renewal: 1 Dec 2011 and 18 Mar 2016 **End of validity: 27 Mar 2020** **Validity extended till: 15 Oct 2020**	**TRA-02/11-03/08**	**Detection of *Listeria monocytogenes*** All human food products and production environmental samples
DELVOTEST®T	Validation date: 3 Feb 2012 Renewal: 28 Jan .2016 **End of validity: 3 Feb 2020** **Validity extended till: 15 Oct 2020**	**DSM-28/02-02/12**	**Detection of antibiotics** Cow, goat and sheep milk (with or without azidiol)
SESAME *SALMONELLA* TEST	Validation date: 4 Dec 2007 Renewal: 7 Oct 2011 and 6 Jul 2015 Extension: 3 Jul 2009 **End of validity: 4 Dec 2019** **Validity extended till: 31 May 2020**	**BKR-23/04-12/07**	**Detection of motile *Salmonella*** All human and animal food products and production environmental samples (except primary production environment)
REVEAL 2.0 *SALMONELLA*	Validation date: 7 Oct 2011 Renewal: 6 Jul 2015 **End of validity: 7 Oct 2019** **Validity extended till: 15 Jul 2020**	**NEO-35/01-10/11**	**Detection of *Salmonella* from group A (except *Salmonella Paratyphi* A) through group E** Meat products, dairy products (except milk powders), egg products, seafood and vegetables (except dehydrated products)

The validation certificates and the recapitulative list are available at the following website address:
http://www.afnor-validation.com/afnor-validation-validated-methods/validated-methods.html

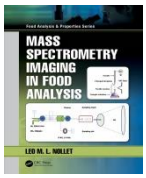
BOOKSHOP: LATEST PUBLICATIONS

The classification in alphabetic order of the first keyword allows you to consult the references according to your interests.
The web site allows you to know more, or to order the book.

MASS SPECTROMETRY

NOLLET Leo M.L. – **Mass spectrometry imaging in food analysis** – CRCPress – May 2020 – ISBN: 9781138370692 – 198 pages

https://www.routledge.com/Mass-Spectrometry-Imaging-in-Food-Analysis/Nollet/p/book/9781138370692



This book explains the novel use of matrix-assisted laser desorption/ionization mass spectrometry imaging in food analysis. It describes how MALDI-MSI will be a useful technique for optical quality assurance. It shows also how MALDI-MSI detects food contaminants and residues, and covers the historical development of the technology.

IN THE PRESS – ON THE WEB

Classification in alphabetical order of keywords

ADDITIFS

Re-evaluation of L(+)-tartaric acid (E 334), sodium tartrates (E 335), potassium tartrates (E 336), potassium sodium tartrate (E 337) and calcium tartrate (E 354) as food additives

https://efsa.onlinelibrary.wiley.com/doi/10.2903/j.efsa.2020.6030

▸ The EFSA Panel on Food Additives and Flavourings (FAF) provides a scientific opinion on tartatic acid-tartrates (E 334-337, 354) when used as food additives in food, particularly in fermented dairy products. The exposure estimates for the different population groups did not exceed the group ADI. However, the Panel emited some recommendations. Les estimations de l'exposition pour les différents groupes de population n'ont pas dépassé la DJA du groupe. Cependant, le groupe scientifique a émis une série e recommandations.

Re-evaluation of acetic acid, lactic acid, citric acid, tartaric acid, mono- and diacetyltartaric acid, mixed acetic and tartaric acid esters of mono- and diglycerides of fatty acids (E 472a-f) as food additives

https://efsa.onlinelibrary.wiley.com/doi/10.2903/j.efsa.2020.6032

▸ The panel on Food Additives and Flavourings (FAF) provided a scientific opinion re-evaluating the safety of acetic acid, lactic acid, citric acid, tartaric acid, mono- and diacetyltartaric acids, mixed acetic and tartaric acid esters of mono- and diglycerides of fatty acids (E 472a-f) when used as food additives in food, particularly in cow milk and fermented dairy products. Considering the exposure estimates, there is no safety concern at their reported uses and use levels. The Panel also proposed a number of recommendations.

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