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ARTICLE

DETERMINATION OF THE FAT ACIDITY (LIPOLYSIS) – PRINCIPLE AND CRITICAL POINTS OF THE STANDARDISED METHODS

The determination of the fat acidity (lipolysis) is important in the analysis of the product quality.

Two standardised methods for the determination of this quality criterion of fat exist at the international level:

- **ISO/TS 22113 | IDF/RM 204** for milk, cream and dried milk
- **ISO 1740 | IDF 6** for butter.

The analytical principle is the same for the both methods, that is an extraction of the fat content and an acidity titration of this extracted fat using Tetra n'Butyl Ammonium Hydroxyde in presence of thymol blue as coloured indicator.

In addition to the standardised requirements of the standardised methods, a significant attention must be paid to the following technical points in the implementation of the test:

- **Extraction (or separation for butter)**

  - *For milk, cream and dried milk:*

- The nature (principally type of phosphate) and the pH of the extraction solution (6.6) have a very significant impact on the final result. Because of its unavailability, the product initially expected, namely the sodium tetraphosphate, will be replace by the sodium hexaphosphate in the revision in process of the next standard.
- The « effective » passage to the water bath at 95 °C for a minimum of 15 minutes, because an insufficient passage impacts also the final results.
- The optional use of the centrifugation to complete the separation of the fat content enables the guarantee of a clear separation and the absence of non fat content in the fat phase during the titration, which could distort the results (input of acidity).

  - *For butter*

This operation is not really an extraction but rather a separation. The critical point is to have a « pure » fat content without inclusion of non-fat following a filtration problem.

- **Titration**

For this step, it is necessary to consider the following points:

- First, the titre of the titrant used for the final calculations. The Tetra n'Butyl Ammonium Hydroxyde (TBAH) solution has the tendency to tap $C0_2$ (atmospheric) and to see its titre progressed over time (even if this evolution is less consequent than the other alkaline titrants as soda or alcoholic postash, it is even so significant concerning the results) So, first it should be preserved this titrant of a such evolution (using a soda lime $C0_2$ absorber, for example) and tested a standard reference fat before each analytical set or realised a titration of this titrant in exchange for a standard solution of potassium hydrogen phthalate (which can be realised by the laboratory using pure dried product).
- The realisation of the titration under nitrogen is absolutely necessary so that not to have interferences with the ambient CO_2, which will acidify the contents of the titration vessel and then have the tendency to overestimate the volume of the titrant and the final result.
- The detection reproducibility of the final point. Even if the curve with the Bromo Thymol blue is very visible, this step is crucial to obtain reproducible results. The use of a « final point standard » during an analytical set is not possible for this type of method because of the ambient CO_2 interference, which will generate an evolution of the titration vessel colour. We can also note that automated systems of detection of the final point using an adjusted optical tube with a wavelength between 600 and 620 nm (as recommended in ISO 22113 standard) are totally adapted to ensure the quality and reproducibility of this step.

These technical elements are important control points for this type of methods and can significantly impact the accuracy of the determinations. They should be integrated in the consideration and the implementation of the method.

STANDARDS - REGULATIONS

STANDARDS, DRAFT STANDARDS

Classification in alphabetical order by theme

ISO standards under development

SENSORY ANALYSIS	
ISO/DIS 11136/A1 February 2019	SENSORY ANALYSIS Methodology – General guidance for conducting hedonic tests with consumers in a controlled area – Amendment 1
MICROBIOLOGY OF FOOD AND ANIMAL FEEDING STUFFS	
ISO/DIS 7932/A1 January 2019	MICROBIOLOGY OF FOOD AND ANIMAL FEEDING STUFFS Horizontal method for the enumeration of presumptive Bacillus cereus – Colony count technique at 30 °C – Amendment 1
ISO/DIS 6887-5 March 2019	MICROBIOLOGY OF THE FOOD CHAIN Preparation of test samples, initial suspension and decimal dilutions for microbiological examination – Part 5: Specific rules for the preparation of milk and milk products
QUALITY	
ISO/IEC DIS 17029 December 2018	CONFORMITY ASSESSMENT General principles and requirements for validation and verification bodies
ISO/DIS 2859-2 January 2019	SAMPLING PROCEDURES FOR INSPECTION BY ATTRIBUTES Part 2: Sampling plans indexed by limited quality (LQ) for isolated lot inspection
STATISTICAL INTERPRETATION OF DATA	
ISO/DIS 16269-5 March 2019	STATISTICAL INTERPRETATION OF DATA Part 5: Techniques of estimation and tests relating to means and variances

ISO published standards

METROLOGY	
ISO/TS 28038 December 2018	Determination and use of polynomial calibration functions
MILK, MILK PRODUCTS, INFANT FORMULA AND ADULT NUTRITIONALS	
ISO 15151 (FIL 229) November 2018	MILK, MILK PRODUCTS, INFANT FORMULA AND ADULT NUTRITIONNALS Determination of minerals and trace elements – Inductively coupled plasma atomic emission spectrometry (ICP-AES) method
ISO 15151 (FIL 229) November 2018	MILK, MILK PRODUCTS, INFANT FORMULA AND ADULT NUTRITIONNALS Determination of minerals and trace elements – Inductively coupled plasma mass spectrometry (ICP-MS) method

NEW EU REGULATIONS

Classification is established in alphabetical order of the first keyword

FLAVOURING SUBSTANCES

O.J.E.U. L 275, 6th November 2018 – Commission Regulation (EU) 2018/1649 of 5 November 2018 amending Annex I to Regulation (EC) No 1334/2008 of the European Parliament and of the Council as regards removal from the Union list of certain flavouring substances

http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2018.275.01.0007.01.ENG

FOOD ADDITIVES

O.J.E.U. L 247, 3rd October 2018 – Commission Regulation (EU) 2018/1472 of 28 September 2018 amending Annex II to Regulation (EC) No 1333/2008 of the European Parliament and of the Council and the Annex to Commission Regulation (EU) No 231/2012 as regards “Cochineal, carminic acid, carmines (E 120)”

http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2018.247.01.0001.01.ENG

STANDARDS - REGULATIONS

O.J.E.U. L 251, 5th October 2018 – Commission Regulation (EU) 2018/1481 of 4 October 2018 amending Annexes II and III to Regulation (EC) No 1333/2008 of the European Parliament and of the Council and the Annex to Commission Regulation (EU) No 231/2012 as regards octyl gallate (E 311) and dodecyl gallate (E 312)
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2018.251.01.0013.01.ENG

MATERIALS IN CONTACT WITH FOOD

O.J.E.U. L 009, 11th January 2019 – Commission Regulation (EU) 2019/37 of 10 January 2019 amending and correcting Regulation (EU) No 10/2011 on plastic materials and articles intended to come into contact with food
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2019.009.01.0088.01.ENG

NOVEL FOOD

O.J.E.U. L 272, 31st October 2018 – Commission Implementing Regulation (EU) 2018/1632 of 30 October 2018 authorising the placing on the market of bovine milk basic whey protein isolate as a novel food 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_.2018.272.01.0023.01.ENG

O.J.E.U. L 275, 6th November 2018 – Commission Implementing Regulation (EU) 2018/1648 of 29 October 2018 authorising the placing on the market of xylo-oligosaccharides as a novel food 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_.2018.275.01.0001.01.ENG

O.J.E.U. L 23, 25th January 2019 – Commission Implementing Regulation (EU) 2019/110 of 24 January 2019 authorising an extension of use of *Allanblackia* seed oil as a novel food 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_.2019.023.01.0011.01.ENG

PESTICIDES

O.J.E.U. L 256, 12th October 2018 – Commission Regulation (EU) 2018/1516 of 10 October 2018 amending Annexes II and III to Regulation (EC) No 396/2005 of the European Parliament and of the Council as regards maximum residue levels for penoxsulam, triflumizole and triflumuron in or on certain products
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2018.256.01.0045.01.ENG

O.J.E.U. L 009, 11th January 2019 – Commission Regulation (EU) 2019/38 of 10 January 2019 amending Annexes II and V to Regulation (EC) No 396/2005 of the European Parliament and of the Council as regards maximum residue levels for iprodione in or on certain products
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2019.009.01.0094.01.ENG

O.J.E.U. L 010, 14th January 2019 – Commission Regulation (EU) 2019/50 of 11 January 2019 amending Annexes II, III, IV and V to Regulation (EC) No 396/2005 of the European Parliament and of the Council as regards maximum residue levels for chlorantraniliprole, clomazone, cyclaniliprole, fenazaquin, fenpicoxamid, fluoxastrobin, lambda-cyhalothrin, mepiquat, onion oil, thiacloprid and valifenalate in or on certain products
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2019.010.01.0008.01.ENG

O.J.E.U. L 012, 15th January 2019 – Commission Regulation (EU) 2019/58 of 14 January 2019 amending Annexes II, III and V to Regulation (EC) No 396/2005 of the European Parliament and of the Council as regards maximum residue levels for linuron in or on certain products
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2019.012.01.0001.01.ENG

O.J.E.U. L 022, 22nd January 2019 – Commission Regulation (EU) 2019/89 of 18 January 2019 amending Annexes II, III and V to Regulation (EC) No 396/2005 of the European Parliament and of the Council as regards maximum residue levels for bromadiolone, etofenprox, paclobutrazol and penconazole in or on certain products
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2019.022.01.0013.01.ENG

O.J.E.U. L 022, 22nd January 2019 – Commission Regulation (EU) 2019/90 of 18 January 2019 amending Annexes II, III and V to Regulation (EC) No 396/2005 of the European Parliament and of the Council as regards maximum residue levels for bromuconazole, carboxin, fenbutatin oxide, fenpyrazamine and pyridaben in or on certain products
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2019.022.01.0052.01.ENG

O.J.E.U. L 022, 22nd January 2019 – Commission Regulation (EU) 2019/91 of 18 January 2019 amending Annexes II, III and V to Regulation (EC) No 396/2005 of the European Parliament and of the Council as regards maximum residue levels for buprofezin, diflubenzuron, ethoxysulfuron, ioxynil, molinate, picoxystrobin and tepraloxydim in or on certain products
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2019.022.01.0074.01.ENG

P.D.O.

O.J.E.U. L 302, 28th November 2018 – Commission Implementing Regulation (EU) 2018/1852 of 26 November 2018 approving non-minor amendments to the specification for a name entered in the register of protected designations of origin and protected geographical indications [Mahon-Menorca (PDO) (cheese)]
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2018.302.01.0007.01.ENG

AFNOR VALIDATIONS

During its May and December meetings, the Technical Committee of NF VALIDATION approved by vote:

Commercial name	Date	Certificate	Description
NEW VALIDATIONS			
EXTENSO	Validation date: 25 Oct 2018 **End of validity: 25 Oct 2022**	**EXT-39/01-10/18**	**Detection of antibiotics** Raw commingled cow's milk
SYMPHONY	Validation date: 4 Dec 2018 **End of validity: 4 Dec 2022**	**BKR-23/11-12/18**	**Enumeration of yeasts and molds** All human food products and feed products
SOLUS ONE *SALMONELLA*	Validation date: 4 Dec 2018 **End of validity: 4 Dec 2022**	**SOL-37/04-12/18**	**Detection of *Salmonella*** Ready to eat, ready to reheat (excluding smoked products), heat processed milk and dairy products, egg products
RENEWALS OF VALIDATIONS			
GENEDISC *LISTERIA* SPP.	Validation date: 2 Jul 2010 Renewal: 2 Oct 2014 and 3 Dec 2018 Extension: 26 Oct 2015 **End of validity: 2 Jul 2022**	**GEN-25/07-07/10**	**Detection of *Listeria* spp.** All human food products and industrial production environmental samples
GENEDISC *LISTERIA MONOCYTOGENES.*	Validation date: 2 Jul 2010 Renewal: 2 Oct 2014 and 3 Dec 2018 Extension: 26 Oct 2015 **End of validity: 2 Jul 2022**	**GEN-25/08-07/10**	**Detection of *Listeria monocytogenes*** All human food products and production environmental samples
***LISTERIA* PRECIS™**	Validation date: 15 Sep 2006 Renewal: 2 Jul 2010, 3 Jul 2014 and 3 Dec 2018 Extension: 29 Mar 2007 **End of validity: 15 Sep 2022**	**UNI-03/05-09/06**	**Enumeration of *Listeria monocytegenes*.** All human food products and industrial production environmental samples
ALOA COUNT	Validation date: 15 Sep 2006 Renewal: 2 Jul 2010, 3 Jul 2014 and 4 Oct 2018 Extension: 4 Oct 2012 and 28 Mar 2013 **End of validity: 15 Sep 2022**	**AES-10/05-09/06**	**Enumeration of *Listeria monocytegenes* and *Listeria* spp.** All human food products and production environmental samples
ESIA ONE DAY	Validation date: 27 Nov 2014 Renewal: 4 Oct 2018 Extension: 28 Jan 2016 **End of validity: 27 Nov 2022**	**BIO-12/37-11/14**	**Detection of *Cronobacter* spp.** Milk powders, infant formula and infant cereals with or without probiotics, including ingredients, and production environmental samples

AFNOR VALIDATIONS

BAX SYSTEM PCR ASSAY *SALMONELLA* SPP. (AUTOMATISED)	Validation date: 28.11.2002 Renewal: 23 Oct 2006, 24 Sep 2010, 27 Nov 2014 and 5 Oct 2018 Extension: 30 Jun 2008, 27 Nov 2008, 18 May 2009, 24 Mar 2011, 22 Mar 2012, 28 Jan 2016 and 26 Jan 2018 **End of validity: 28 Nov 2022**	**QUA-18/03-11/02**	**Detection of *Salmonella* spp.** All human food products, feed products and production environmental samples (except primary production environment)
MICROSEQ® *SALMONELLA*	Validation date: 24.09.2010 Renewal: 3 Jul 2014 and 3 Dec 2018 Extension: 11 May 2012 and 4 Jul 2013 **End of validity: 24 Sep 2022**	**ABI-29/02-09/10**	**Detection of *Salmonella* spp.** All human and animal food products, animal faeces and environmental samples from the primary production stage
CHROMID COLI-ID AGAR	Validation date: 19 Jan 1999 Renewal: 5 Feb 2003, 14 Dec 2006, 2 Dec 2010, 27 Nov 2014 and 4 Dec 2018 **End of validity: 19 Jan 2022**	**BIO-12/05-01/99**	**β-glucuronidase positive *E. coli* enumeration at 44 °C** All human food products
CHROMID COLI-ID AGAR	Validation date: 14 Dec 2006 Renewal: 2 Dec 2010, 27 Nov 2014 and 4 Dec 2018 **End of validity: 14 Dec 2022**	**BIO-12/19-12/06**	**β-glucuronidase positive *E. coli* enumeration at 37 °C** All human food products
CHROMID COLI-ID AGAR	Validation date: 14 Dec 2006 Renewal: 2 Dec 2010, 27 Nov 2014 and 4 Dec 2018 **End of validity: 14 Dec 2022**	**BIO-12/20-12/06**	**Coliforms enumeration at 37 °C** All human food products
TEMPO EB	Validation date: 14 Dec 2006 Renewal: 2 Dec 2010, 2 Oct 2014 and 3 Dec 2018 Extension: 3 Feb 2011 **End of validity: 14 Dec 2022**	**BIO-12/21-12/06**	**Enumeration of *Enterobacteriaceae*** All human food products and pet food
EXTENSIONS OF VALIDATIONS			
THERMO SCIENTIFIC SURETECT *LISTERIA* SPP. PCR ASSAY	Validation date: 28 Nov 2013 Renewal: 17 May 2018 Extension: 21 Mar 2014, 3 Jul 2014, 30 Jun 2016 and 5 Oct 2018 **End of validity: 28 Nov 2021**	**UNI-03/09-11/13**	**Detection of *Listeria* spp.** Meat products, milk and dairy products, seafood and fishery products, vegetables and production environmental samples
GENE-UP *LISTERIA* SPP.	Validation date: 29 Sep 2016 Extension: 24 Nov 2016, 3 Jul 2017 and 4 Dec 2018 **End of validity: 29 Sep 2020**	**BIO-12/39-09/16**	**Detection of *Listeria* spp. (except *Listeria Grayi*)** All human food products and production environmental samples

AFNOR VALIDATIONS

THERMO SCIENTIFIC SURETECT *LISTERIA MONOCYTOGENES* PCR ASSAY	Validation date: 4 Nov 2013 Renewal: 22 Nov 2017 Extension: 30 Jan 2014, 21 Mar 2014, 30 Jun 2016 and 5 Oct 2018 **End of validity: 4 Nov 2021**	**UNI-03/08-11/13**	**Detection of *Listeria monocytogenes*** Meat products, milk and dairy products, seafood and fishery products, vegetables and production environmental samples
VIDAS *LISTERIA MONOCYTOGENES* 2 (LMO2) **(includes a 37 °C enrichment step in Fraser broth and a specific protocol in LPT broth at 30 °C)**	Validation date: 12 Mar 2004 Renewal: 17 Jan 2008, 2 Feb 2012 and 30 Jun 2016 Extension: 2 Dec 2004, 14 Dec 2006, 30 Jun 2011, 29 Jan 2016 and 4 Oct 2018 **End of validity: 12 Mar 2020**	**BIO-12/11-03/04**	**Detection of *Listeria monocytogenes*** All human food products and production environmental samples
GENE-UP *LISTERIA MONOCYTOGENES*	Validation date: 24 Oct 2016 Extension: 27 Jan 2017, 3 Jul 2017 and 4 Dec 2018 **End of validity: 24 Nov 2020**	**BIO-12/40-11/16**	**Detection of *Listeria monocytogenes*** All human food products and production environmental samples
THERMO SCIENTIFIC SURETECT *SALMONELLA* SPECIES PCR ASSAY	Validation date: 4 Nov 2013 Renewal: 22 Mar 2018 Extension: 30 Jan 2014, 21 Mar 2014, 30 Jun 2016, 24 Mar 2017 and 3 Dec 2018 **End of validity: 4 Nov 2021**	**UNI-03/07-11/13**	**Detection of *Salmonella* spp.** All human food products, pet food and production environmental samples (except primary production environment)
GENE-UP *SALMONELLA*	Validation date: 30 Jun 2016 Extension: 29 Sep 2016, 24 Mar 2017, 3 Jul 2017, 23 Nov 2017, 26 Jan 2018, 4 Oct 2018 and 3 Dec 2018 **End of validity: 30 Jun 2020**	**BIO-12/38-06/16**	**Detection of *Salmonella* spp.** All human food products, pet food products and production environmental samples (except primary production environment)
GENE-UP *CRONOBACTER* SPP.	Validation date: 23 Mar 2018 Extension: 3 Jul 2018 and 4 Oct 2018 **End of validity: 23 Mar 2022**	**BIO-12/42-03/18**	**Detection of *Cronobacter* spp.** Infant cereals and powdered infant formula with or without probiotics, milk powders, ingredients and industrial environmental samples
THERMO SCIENTIFIC SURETECT *CRONOBACTER* SPECIES PCR ASSAY	Validation date: 3 Dec 2015 Extension: 30 Jun 2016 and 3 Dec 2018 Renewal: 22 Mar 2018 **End of validity: 3 Dec 2023**	**UNI-03/11-12/15**	**Detection of *Cronobacter* spp.** Infant formula and industrial production environmental samples
DATE EXTENSIONS OF END OF VALIDITY			
COMPASS *LISTERIA* AGAR DETECTION	Validation date: 28 Nov 2002 Renewal: 25 May 2007, 24 Sep 2010 & 27 Nov 2014 Extension: 27 Sep 2007, 12 May 2011 & 29 Mar 2013 **End of validity: 28 Nov 2018** **Validity extended till: 31 Jul 2019**	**BKR-23/02-11/02**	**Detection of *Listeria monocytogenes* and *Listeria* spp.** All human food products and production environmental samples

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

IN THE PRESS – ON THE WEB

Classification in alphabetical order of keywords

FOOD ADDITVES

Re-evaluation of propane-1,2-diol esters of fatty acids (E 477) as a food additive

http://www.efsa.europa.eu/en/efsajournal/pub/5497

▸ The EFSA Panel on Food Additives and Flavourings (FAF) provides a scientific opinion re-evaluating the safety of propane-1,2-diol esters of fatty acids (E 477) when used as a food additive. Considering the overall metabolic and toxicity database, the Panel confirmed the previously established ADI for propane-1,2-diol esters of fatty acids (E 477) of 25 mg/kg bw per day expressed as propane 1,2 diol. The Panel concluded that there would not be a safety concern at the reported use levels for E 477. However, the Panel aims to explore the feasibility of establishing a group ADI for those food additives that result in an exposure to propane-1,2-diol, such as E 477, E 1520 and E 405.

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