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RESULTS OF THE VALIDATION TESTS FOR THE NF V 04-287 A/B METHOD

The results observed between the acido-butyrometric method (NF V 04-287) and the reference method by extraction (ISO 1735) differ for the determination of fat content. So, a "correction" equation was calculated on the basis of proficiency tests organised by ACTALIA Cecalait from 2003 to 2013.

To validate this equation, a validation test was conducted on cheeses, which were not represented in the samples set used for the initial calculation of the correction. These tests were carried out at the end of 2015 and at the beginning of 2016 (processed cheese). A comparative analysis of 30 samples (8 fresh cheese samples, 8 soft cheese samples, 8 hard cheese samples and 6 processed cheese samples) was performed by many laboratories using the butyrometric (NF V 04-287 A or B) and the extraction (ISO 1735) methods.

The results obtained by the butyrometric method were then transformed using the initially calculated equation ($Y = 0.974 X + 0.345$) and compared to the results obtained by the extraction method

These test were performed within the context of the CNIEL analytical commission and AFNOR V04 "milk and dairy products" commission.

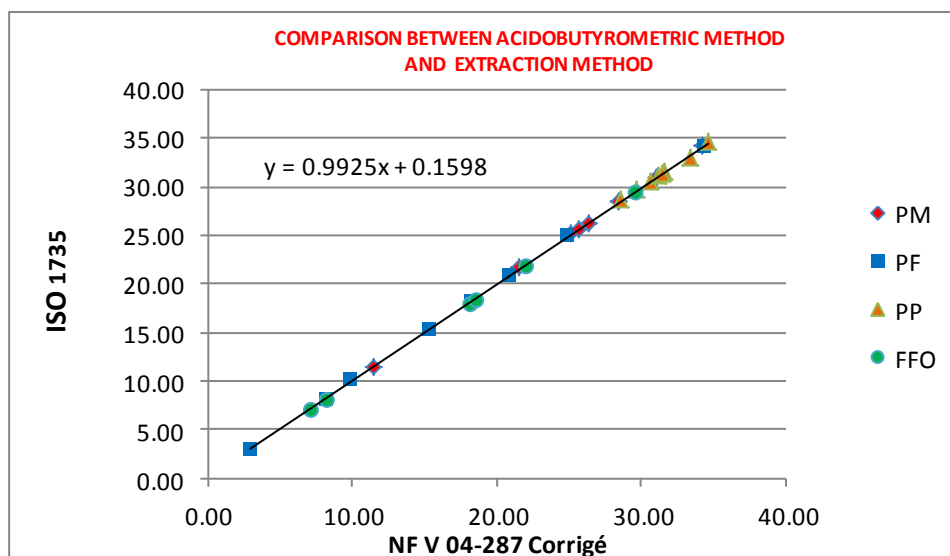
Number of participating laboratories

The table below presents the number of the participating laboratories in the tests according to the method used:

| Type of cheese | Number of laboratories ISO 1735 | Number of laboratories NF V 04-287 |
|----------------------|------------------------------------|---------------------------------------|
| Soft cheese PM | 6 | 8 |
| Fresh cheese PF | 5 | 5 |
| Herd cheese PP | 4 | 9 |
| Processed cheese FFO | 4 | 8 |

Final results

The figure below presents the relation between the acido-butyrometric method after correction and the extraction method on 30 cheese samples:



ARTICLE

The table below presents the results of the comparison between the both methods on 30 cheese samples (before and after correction):

| | ISO 1735 | NF V 04 287 | NF V 04 287 Corr | Ecart |
|----------------|--------------|--------------|------------------|-------------|
| PM 1 | 34,28 | 34,87 | 34,31 | 0,03 |
| PM 2 | 30,97 | 31,49 | 31,01 | 0,05 |
| PM 3 | 25,15 | 25,40 | 25,09 | -0,06 |
| PM 4 | 25,70 | 26,10 | 25,77 | 0,07 |
| PM 5 | 26,23 | 26,72 | 26,37 | 0,14 |
| PM 6 | 28,52 | 28,92 | 28,51 | -0,01 |
| PM 7 | 11,47 | 11,39 | 11,44 | -0,03 |
| PM 8 | 21,64 | 21,81 | 21,59 | -0,05 |
| PF1 | 8,18 | 8,04 | 8,17 | -0,01 |
| PF2 | 3,01 | 2,64 | 2,92 | -0,10 |
| PF3 | 34,26 | 34,92 | 34,36 | 0,10 |
| PF4 | 24,91 | 25,15 | 24,85 | -0,07 |
| PF5 | 20,93 | 21,12 | 20,92 | -0,01 |
| PF6 | 18,31 | 18,37 | 18,24 | -0,07 |
| PF7 | 10,16 | 9,78 | 9,87 | -0,30 |
| PF8 | 15,45 | 15,34 | 15,29 | -0,16 |
| PP1 | 28,75 | 29,05 | 28,64 | -0,10 |
| PP2 | 30,46 | 31,10 | 30,63 | 0,18 |
| PP3 | 33,07 | 33,94 | 33,40 | 0,33 |
| PP4 | 29,67 | 30,15 | 29,72 | 0,04 |
| PP5 | 34,55 | 35,19 | 34,62 | 0,07 |
| PP6 | 31,06 | 31,65 | 31,17 | 0,11 |
| PP7 | 31,58 | 32,18 | 31,69 | 0,11 |
| PP8 | 31,34 | 31,98 | 31,49 | 0,15 |
| FFO1 | 29,58 | 30,05 | 29,62 | 0,04 |
| FFO2 | 18,07 | 18,17 | 18,04 | -0,02 |
| FFO3 | 7,15 | 6,95 | 7,12 | -0,03 |
| FFO4 | 21,98 | 22,20 | 21,97 | -0,01 |
| FFO5 | 8,21 | 8,07 | 8,21 | 0,00 |
| FFO6 | 18,46 | 18,61 | 18,47 | 0,01 |
| Moyenne | 23,10 | 23,38 | 23,12 | 0,01 |
| Différence | | 0,27 | 0,01 | |
| Sy,x | | 0,091 | 0,091 | |
| Min | 3,01 | | | -0,30 |
| Max | 34,55 | | | 0,33 |

Conclusion:

Firstly, these tests confirmed the deviations previously observed between the both methods (mean bias of about 0.27 g/100 g for all cheeses and all contents). Apply the correction equation (calculated on the basis of the proficiency tests results) to the results obtained by the acido-butyrometric method allows to obtain fat contents equivalent to those obtained by the extraction method. Indeed, the study of the simple linear regression between the both sets of data (corrected acido-butyrometric method and extraction method) allows to obtain a mean deviation between the both methods equal to +0.01 g/100 g and a regression slope close to 1.00 (0.9925).

We can then conclude that the equation calculated to adjust the acido-butyrometric method is efficacy for the extraction method in fresh, soft, hard and processed cheeses.

STANDARDS - REGULATIONS

STANDARDS, DRAFT STANDARDS

Classification in alphabetical order by theme

ISO standards under development

| SENSORY ANALYSIS | |
|------------------------------|--|
| ISO/DIS 6658 July 2016 | SENSORY ANALYSIS Methodology - General guidance |
| CREME | |
| ISO/DIS 19660 August 2016 | CRAM Determination of fat content - Acido-butyrometric method |
| MILK | |
| ISO/DIS 19662 August 2016 | MILK Determination of fat content - Acido-butyrometric method (Gerber method) |

ISO published standards

| MILK AND MILK PRODUCTS | |
|--|---|
| ISO 8968-4 (IDF 20-4) April 2016 | MILK AND MILK PRODUCTS Determination of nitrogen content - Part 4: Determination of protein and non-protein nitrogen content and true protein content calculation (reference method) <i>Replace ISO 8968-4:2001 and ISO 8968-5:2001</i> |
| REFERENCE MATERIALS | |
| ISO/TR 16476 June 2016 | REFERENCE MATERIALS Establishing and expressing metrological traceability of quantity values assigned to reference materials |
| MICROBIOLOGY OF FOOD CHAIN | |
| ISO 16140-1 June 2016 | MICROBIOLOGY OF FOOD CHAIN Method validation - Part 1: Vocabulary <i>Replace ISO 16140:2003 and ISO 16140/A1:2011</i> |
| ISO 16140-2 June 2016 | MICROBIOLOGY OF FOOD CHAIN Method validation - Part 2: Protocol for the validation of alternative (proprietary) methods against a reference method <i>Replace ISO 16140:2003 and ISO 16140/A1:2011</i> |
| ISO 17468 June 2016 | MICROBIOLOGY OF FOOD CHAIN Technical requirements and guidance on establishment or revision of a standardized reference method |

NEW EU REGULATIONS

Classification is established in alphabetical order of the first keyword

METAL

O.J.E.U. L 183, 8th July 2016 – Commission Recommendation (EU) 2016/1111 of 6 July 2016 on the monitoring of nickel in food

http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2016.183.01.0070.01.ENG

NOVEL FOOD

O.J.E.U. L 196, 21st July 2016 – Commission Implementing Decision (EU) 2016/1189 of 19 July 2016 authorising the placing on the market of UV-treated milk as a novel food under Regulation (EC) No 258/97 of the European Parliament and of the Council

http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2016.196.01.0050.01.ENG

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

O.J.E.U. C 176, 18th May 2016 – Publication of an amendment application 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 [Maroilles / Marolles (PDO) (cheese)]

http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.C_.2016.176.01.0021.01.ENG

O.J.E.U. C 176, 18th May 2016 – Publication of an amendment application 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 [Maroilles / Marolles (PDO) (cheese)]

http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.C_.2016.176.01.0021.01.ENG

O.J.E.U. C 188, 27th May 2016 – Publication of an amendment application 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 [Gorgonzola (PDO) (cheese)]

http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.C_.2016.188.01.0044.01.ENG

O.J.E.U. L 155, 14th June 2016 – Commission Implementing Regulation (EU) 2016/924 of 1 June 2016 entering a name in the register of protected designations of origin and protected geographical indications [Allgäuer Sennalpkäse (PDO) (cheese)]

http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2016.155.01.0002.01.ENG

O.J.E.U. L 155, 14th June 2016 – Commission Implementing Regulation (EU) 2016/927 of 1 June 2016 entering a name in the register of protected designations of origin and protected geographical indications [Soumaintrain (PGI) (cheese)]

http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2016.155.01.0005.01.ENG

O.J.E.U. L 155, 14th June 2016 – Commission Implementing Regulation (EU) 2016/928 of 1 June 2016 approving non-minor amendments to the specification for a name entered in the register of protected designations of origin and protected geographical indications [Queso Tetilla / Queixo Tetilla (PDO) (cheese)]

http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2016.155.01.0006.01.ENG

O.J.E.U. C 261, 19th July 2016 – Publication of an application 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 [Raclette de Savoie (PGI) (cheese)]

http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.C_.2016.261.01.0016.01.ENG

O.J.E.U. C 263, 20th July 2016 – Publication of an application 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 [Burrata di Andria (PGI) (cheese)]

http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.C_.2016.263.01.0007.01.ENG

PESTICIDES

O.J.E.U. L 167, 24th June 2016 – Commission Regulation (EU) 2016/1002 of 17 June 2016 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 AMTT, diquat, glufosinate and tritosulfuron in or on certain products

http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2016.167.01.0001.01.ENG

O.J.E.U. L 167, 24th June 2016 – Commission Regulation (EU) 2016/1003 of 17 June 2016 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 abamectin, acequinocyl, acetamiprid, benzovindiflupyr, bromoxynil, fludioxonil, fluopicolide, fosetyl, mepiquat, proquinazid, propamocarb, prohexadione and tebuconazole in or on certain products

http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2016.167.01.0046.01.ENG

O.J.E.U. L 172, 29th June 2016 – Commission Regulation (EU) 2016/1015 of 17 June 2016 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 1-naphthylacetamide, 1-naphthylacetic acid, chloridazon, fluazifop-P, fuberidazole, mepiquat and tralkoxydim in or on certain products

http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2016.172.01.0001.01.ENG

O.J.E.U. L 172, 29th June 2016 – Commission Regulation (EU) 2016/1015 of 17 June 2016 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 ethofumesate, etoxazole, fenamidone, fluoxastrobin and flurtamone in or on certain products

http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2016.172.01.0022.01.ENG

PHARMACOLOGICALLY ACTIVE SUBSTANCES

O.J.E.U. L 125, 13th May 2016 – Commission Implementing Regulation (EU) 2016/710 of 12 May 2016 amending Regulation (EU) No 37/2010 as regards the substance "copper carbonate"

http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2016.125.01.0006.01.ENG

AFNOR VALIDATIONS

During their May and June meetings, the Technical Committee of NF VALIDATION approved by vote:

| Commercial name | Date | Certificate | Description |
|---|---|------------------------|---|
| NEW VALIDATION | | | |
| 3M™ MOLECULAR DETECTION ASSAY 2 - LISTERIA | Validation date: 18 May 2016 End of validity: 18 May 2020 | 3M-03/14-05/16 | Detection of <i>Listeria</i> spp. All human food products and production environment samples |
| LESS PLUS FOR LISTERIA + | Validation date: 1 Jul 2016 End of validity: 1 Jul 2020 | NEO-35/05-07/16 | Detection of <i>Listeria</i> spp. All human food products and production environment samples |
| LESS PLUS FOR LISTERIA MONOCYTOGENES + | Validation date: 1 Jul 2016 End of validity: 1 Jul 2020 | NEO-35/06-07/16 | Detection of <i>Listeria monocytogenes</i> All human food products and production environmental samples |
| RENEWAL OF VALIDATION | | | |
| 3M™ PETRIFILM™ RAPID COLIFORM COUNT PLATE | Validation date: 18 Mar 1997 Renewal: 13 Dec 2001, 19 Sep 2005, 28 Nov 2008, 31 Jan 2013 and 1 Jul 2016 End of validity: 18 Mar 2021 | 3M-01/05-03/97A | Enumeration of coliforms - reading after 14 hours All human food products |
| 3M™ PETRIFILM™ RAPID COLIFORM COUNT PLATE | Validation date: 18 Mar 1997 Renewal: 13 Dec 2001, 19 Sep 2005, 28 Nov 2008, 31 Jan 2013 and 1 Jul 2016 End of validity: 18 Mar 2021 | 3M-01/05-03/97B | Enumeration of coliforms - reading after 24 hours All human food products |
| 3M™ PETRIFILM™ RAPID COLIFORM COUNT PLATE | Validation date: 18 Mar 1997 Renewal: 13 Dec 2001, 4 May 2006, 28 Nov 2008, 31 Jan 2013 and 1 Jul 2016 End of validity: 18 Mar 2021 | 3M-01/05-03/97C | Enumeration of gas producing colonies - reading after 24 hours All human food products except processed pork products |
| SIMPLE METHOD FOR SALMONELLA (SMS) | Validation date: 7 May 2004 Renewal: 27 Mar 2008, 22 Mar 2012 and 18 May 2016 Extension: 2 Jul 2007 End of validity: 7 May 2020 | AES-10/04-05/04 | Detection of <i>Salmonella</i> spp. All human and animal food products, and production environment samples (except primary production stage environment) |
| 3M™ MOLECULAR DETECTION ASSAY FOR SALMONELLA | Validation date: 30 Nov 2012 Renewal: 1 Jul 2016 Extension: 31 Jan 2013 and 21 Mar 2014 End of validity: 30 Nov 2020 | 3M-01/11-11/12 | Detection of <i>Salmonella</i> spp All human food products (including spices, aromatic herbs, instant coffees and teas, bouillon cubes/concentrates, milk powders and cocoa powders) and production environment samples (except primary production environment samples) |

AFNOR VALIDATIONS

| | | | |
|---|---|------------------------|--|
| 3M™ PETRIFILM™ SELECT <i>E. COLI</i> COUNT PLATE | Validation date: 14 Jun 2001 Renewal: 7 Apr 2005, 18 May 2009, 23 May 2013 and 1 Jul 2016 Extension: 6 Feb 2015 End of validity: 14 Jun 2021 | 3M-01/08-06/01 | Enumeration of <i>E. coli</i> All human food products, pet food and production environment samples |
| 3M™ MOLECULAR DETECTION ASSAY FOR <i>E. COLI</i> O157 (INCLUDING H7) | Validation date: 29 Mar 2013 Renewal: 1 Jul 2016 End of validity: 29 Mar 2021 | 3M-01/12-03/13 | Detection of <i>E. coli</i> O157 Raw beef meat, raw dairy products, raw fruits and vegetables |
| ALOA ONE DAY | Validation date: 27 Sep 2000 Renewal: 7 Apr 2005, 30 Jun 2008, 6 Jul 2012 and 1 Jul 2016 Extension: 10 Mar 2006, 15 Sep 2006, 1 Apr 2010, 6 Oct 2011 and 28 Mar 2013 End of validity: 27 Sep 2020 | AES-10/03-09/00 | Detection of <i>Listeria monocytogenes</i> and <i>Listeria</i> spp All human food products and production environment samples |
| VIDAS <i>LISTERIA</i> MONOCYTOGENES 2 (LMO2) | Validation date: 12 Mar 2004 Renewal: 17 Jan 2008, 2 Feb 2012, 30 Jun 2016 Extension: 2 Dec 2004, 14 Dec 2006, 30 Jun 2001 and 29 Jan 2016 End of validity: 12 Mar 2020 | BIO-12/11-03/04 | Detection of <i>Listeria monocytogenes</i> All human food products and environ- mental samples |

EXTENSIONS OF VALIDATIONS

| | | | |
|---|--|------------------------|---|
| THERMO SCIENTIFIC SURETECT SALMONELLA SPECIES PCR ASSAY | Validation date: 4 Nov 2013 Extension: 30 Jan 2014, 21 Mar 2014 and 30 Jun 2016 End of validity: 4 Nov 2017 | UNI-03/07-11/13 | Detection of <i>Salmonella</i> spp All human food products and pet food |
| THERMO SCIENTIFIC SURETECT <i>LISTERIA</i> MONOCYTOGENES PCR ASSAY | Validation date: 4 Nov 2013 Extension: 21 Mar 2014, 17 Mar 2016 and 30 Jun 2016 End of validity: 4 Nov 2017 | UNI-03/08-11/13 | Detection of <i>Listeria monocytogenes</i> All human food products and environ- mental samples |
| THERMO SCIENTIFIC SURETECT <i>LISTERIA</i> SPP PCR ASSAY | Validation date: 28 Nov 2013 Extension: 21 Mar 2014, 3 Jul 2014 and 30 Jun 2016 End of validity: 28 Nov 2017 | UNI-03/09-11/13 | Detection of <i>Listeria</i> spp All human food products and environ- mental samples |
| THERMO SCIENTIFIC SURETECT <i>CRONOBACTER</i> SPECIES PCR ASSAY | Validation date: 3 Dec 2015 Extension: 30 Jun 2016 End of validity: 3 Dec 2019 | UNI-03/11-12/15 | Detection of <i>Cronobacter</i> spp Infant formula and production environ- ment 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>

FORTHCOMING EVENTS

Classified in chronological order

MILK AND DAIRY PRODUCTS

16-21 October 2016

Rotterdam, The Netherlands

IDF World Dairy Summit 2016

www.idfwds2016.com

IN THE PRESS – ON THE WEB

Classification in alphabetical order of keywords

STANDARDISATION

Report of the 39th session of the Codex Alimentarius Commission

[http://www.fao.org/fao-who-codexalimentarius/sh-](http://www.fao.org/fao-who-codexalimentarius/sh-proxy/fr/?lnk=1&url=https%253A%252F%252Fworkspace.fao.org%252Fsites%252Fcodex%252FMeetings%252FCX-701-39%252FREPORT%252FREP16_CACe.pdf)

[proxy/fr/?lnk=1&url=https%253A%252F%252Fworkspace.fao.org%252Fsites%252Fcodex%252FMeetings%252FCX-701-39%252FREPORT%252FREP16_CACe.pdf](http://www.fao.org/fao-who-codexalimentarius/sh-proxy/fr/?lnk=1&url=https%253A%252F%252Fworkspace.fao.org%252Fsites%252Fcodex%252FMeetings%252FCX-701-39%252FREPORT%252FREP16_CACe.pdf)

► This report presents the 39th session of the Codex Alimentarius Commission held from 27th June - 1st July 2016 in Rome, Italy. This document presents the list of the amendments, the adopted standards or documents, the draft standards adopted at step 5, the revoked standards and the approved new work.

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