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## EVALUATION OF THE MPA™ INFRARED ANALYSER ON RAW MILK

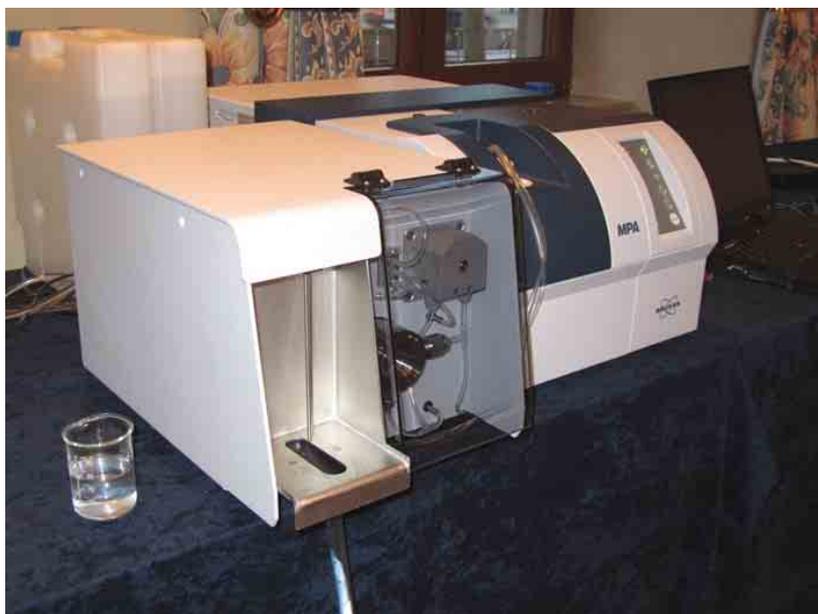
The MPA™ is a near infrared TF spectrophotometer manufactured by Bruker Optik (Germany, Bruker Group Corporation) and commercialised in France by Bruker Optics. It is used for the determination of the principal components in milk and in liquid (retentate, serum, cream, milk-based drinks...) and solid (powder, cheese, butter, yoghurt) dairy products.

This apparatus allows to analyse:

- liquid samples in a sample compartment with an InGaAs detector cooled thermo-electrically (range 12800 – 4000 cm<sup>-1</sup>) and,
- solid samples, by transmission module with a Si detector at room temperature (range 15500 – 9000 cm<sup>-1</sup>), or by reflection module with a PbS detector (range 12800-3600 cm<sup>-1</sup>). Fiber optic probes can also be installed. The heart of the instrument is a permanent alignment interferometer with gold mirrors.

The apparatus is computer controlled with Opus software, which ensures the signal treatment and the PLS calibrations.

The instrument was already evaluated on homogenised milk, so a complementary module adapted to the analysis of raw milk was installed. It includes a pipetting system, a heater, a HP pump and a homogeniser.



The evaluation tests were performed in Actilait-Cecalait® physico-chemistry laboratory (reference and instrumental analyses) from March to September 2012. After preliminary tests of contamination between samples for fat and crude protein, the repeatability and accuracy on raw milk for fat, dry matter and crude protein were evaluated.

The prediction models used have been developed by the supplier thanks the Opus software.

The calculation parameters are in relation with the ISO 21543/IDF 201 standard.

### A – PRELIMINARY TESTS

The objectives of these tests were to evaluate the contamination between samples. Ten sets of whole raw milk were then analysed according the Lait 1 – Lait 2 – Lo1 – Lo2 sequence on 2 levels of different rates. Fat and crude protein parameters were noted. Lo samples (about 15 g/l in fat and crude protein) were used in place of water samples in order to work at constant gain.

The following table summarises the results obtained:

	MILK A		MILK B	
	Fat	Crude protein	Fat	Crude protein
M (g/l)	37.88	31.13	58.6	39.70
Tc (%)	0.25	0.86	0.44	0.68

**Table 1:** MPA contamination criteria for fat and crude protein on raw milk samples

*M: results mean, Tc: contamination rates*

*The contamination rate was calculated according to the following formula:*

$$Tc(\%) = 100 * [\text{sum}(Lo1) - \text{sum}(Lo2)] / [\text{sum}(Lo2) - \text{sum}(Lo2)]$$

With no standard criteria, it can be noted that the relative contamination rates obtained vary between 0.25 and 0.86%. The values obtained are in accordance with the ISO 9622/IDF 141 standard, which fixes the maximal limit at 1%.

## B – EVALUATION OF THE REPEATABILITY AND THE ACCURACY

### B.1 – Samples

The tests were performed on 30 samples of tank milk from Rhône-Alpes region. Bronopol was added to the samples to give a final concentration of 0.02%.

### B.2 – Procedure

The repeatability and the accuracy of the instrument for fat, dry matter and crude protein were evaluated using all the milk samples. The infrared quantitative analysis of each sample was carried out in consecutive duplicate. The instrumental values were carried out by a calibration and adjustment of the manufacturer, optimised by the integration of 10 specific samples.

The following reference methods were used:

- Dry matter: drying method according to ISO 6731/IDF 21 (single tests);
- Fat: acido-butyrometric method according to NF V 04-210 (single tests) ;
- Crude protein: Kjeldahl method according to 8968/IDF 20 (single tests), with conversion crude protein = AT x 6.38

### B.3 – Results

The tables and figures below summarise the results obtained:

	n	min	max	M	Sx	Sr	Sr (%)	r
<b>Dry matter (g/100g)</b>	30	12.36	12.96	12.776	0.127	0.013	0.11	0.04
<b>Fat (g/l)</b>	30	37.60	44.95	42.872	1.568	0.094	0.22	0.26
<b>Crude protein (g/kg)</b>	30	32.68	34.91	33.834	0.475	0.064	0.19	0.18

**Table 2:** MPA repeatability criteria for dry matter, fat and crude protein on raw milk samples

*n: number of results; min and max: minimum and maximum value; M and Sx: mean and standard deviation of the results; Sr and Sr%: absolute and relative standard deviation of repeatability; r: maximal deviation of repeatability (95% of cases)*

	n	min	max	Y	Sy	d	Sd	Sy,x	Sy,x%	b	a
<b>Dry matter (g/100g)</b>	30	12.30	13.00	12.79	0.16	-0.02	0.07	0.066	0.51	1.168	-2.12
<b>Fat (g/l)</b>	30	35.50	42.60	40.78	1.57	2.09	0.29	0.298	0.69	0.985	-1.47
<b>Crude protein (g/kg)</b>	30	32.39	34.77	33.71	0.55	0.12	0.23	0.230	0.68	1.059	-2.12

**Table 3:** MPA accuracy criteria for dry matter, fat and crude protein on raw milk samples

*n, min, max: number of results, minimum and maximum value; Y,X: mean results using reference and instrumental methods; Sy: standard deviation of the results from the reference method; d, Sd: mean and standard deviation of deviations; Sy,x and Sy,x%: absolute and relative standard deviation; b, a: slope and intercept of the linear regression*

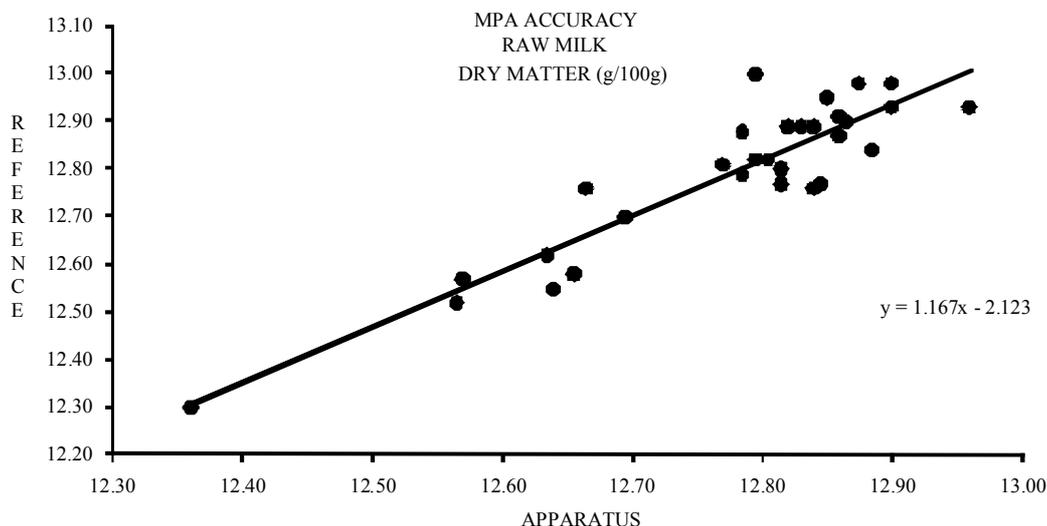


Figure 1: Relation between the MPA and the reference results for dry matter on raw milk samples

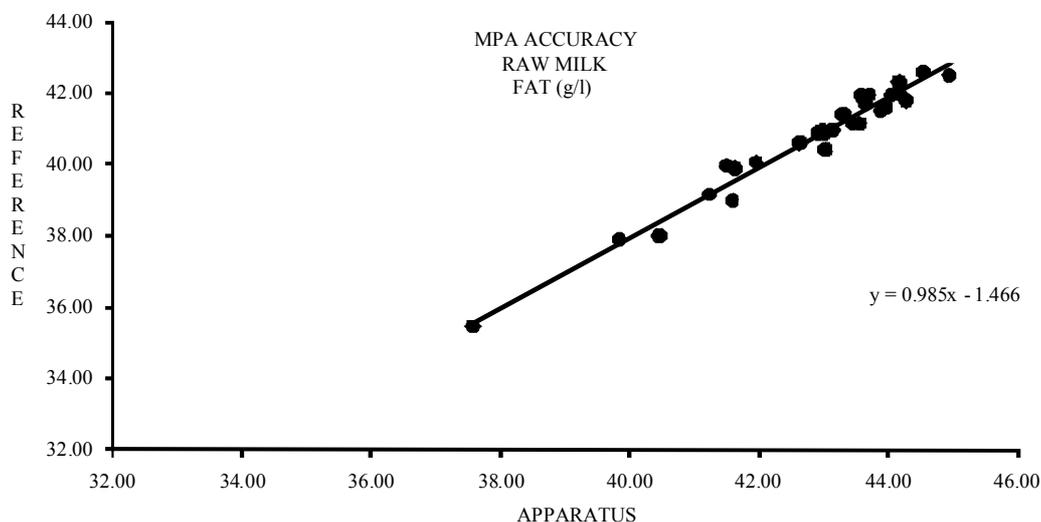


Figure 2: Relation between the MPA and the reference results for fat on raw milk samples

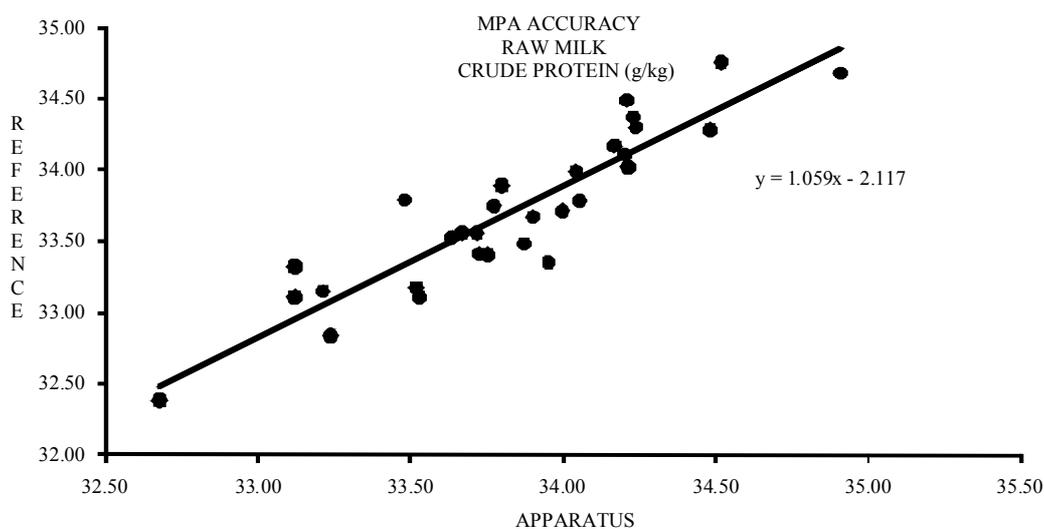


Figure 3: Relation between the MPA and the reference results for crude protein on raw milk samples

It can be noted that:

⇒ For dry matter, the residual standard deviation is equal to 0.066 g/100 g. The slope is equal to 1.167 and is not significantly different from 1.00 (P = 5%),

⇒ For fat, the residual standard deviation is equal to 0.298 g/l. The slope is equal to 0.985 and is not significantly different from 1.00 (P = 5%),

⇒ For crude protein, the residual standard deviation is equal to 0.230 g/kg. The slope is equal to 1.059 and is not significantly different from 1.00 (P = 5%).

#### **B.4 - Conclusion**

Concerning the performance of repeatability, the standard deviations of repeatability are respectively equal to 0.013 g/100 g, 0.064 g/l and 0.094 g/kg for dry matter, fat and crude protein. Despite the absence of standard criteria for the analysis of milk by near infrared, it can be noted that the results obtained are in accordance with the recommendations of the ISO 9622/IDF 141 standard dedicated to the mid infrared analysers on raw milk, which fixes the maximal limit to 0.14 g/l for the standard deviations of repeatability of fat and protein.

Concerning the performance of accuracy, the residual standard deviations of regression observed enable accuracy of estimation ( $2.S_{y,x}$  at 5% risk) equal to 0.132 g/100 g for dry matter, 0.596 g/l for fat and 0.460 g/kg for crude protein. Despite the absence of standard criteria for the analysis of milk by near infrared, it can be noted that the values obtained for fat are in accordance with the maximal limit of the standard deviation of deviations (0.7 g/l) of the ISO 9622/IDF 141 standard.

#### **CONCLUSION**

Because of the absence of standard criteria specific to near infrared method, it is difficult to interpret the MPA repeatability and accuracy performances. However, it can be noted that the performances obtained for fat are in accordance with the standardised limits (ISO 9622/IDF 141) and close to the performances published. Moreover, as the evaluation was performed with general manufacturer's calibration, the repeatability and accuracy values observed are most probably maximums and can be improved by enrichment of the model with appropriate samples.

*According to the evaluation report of the MPA™ infrared analyser (raw milk) – X. QUERVEL and Ph. TROSSAT – December 2012*

**STANDARDS, DRAFT STANDARDS**

Classification in alphabetical order by theme

**ISO standards under development**

<b>MICROBIOLOGY OF FOOD AND ANIMAL FEED</b>	
ISO/DIS 16140-1 June 2013	MICROBIOLOGY OF FOOD AND ANIMAL FEED Method validation – Part 1: Vocabulary
ISO/DIS 16140-2 June 2013	MICROBIOLOGY OF FOOD AND ANIMAL FEED Method validation – Part 2: Protocol for the validation of alternative (proprietary) methods against a reference method
ISO/DIS 17468 June 2013	MICROBIOLOGY OF FOOD AND ANIMAL FEED Technical requirements and guidance on establishment or revision of standard methods
<b>MILK AND MILK PRODUCTS</b>	
ISO/DIS 11816-1 February 2013	MILK AND MILK PRODUCTS Determination of alkaline phosphatase activity – Part 1: Fluorimetric method for milk and milk-based drinks
<b>SENSORY ANALYSIS</b>	
ISO/DIS 8589/A1 June 2013	SENSORY ANALYSIS General guidance for the design of test rooms – Amendment 1
<b>STATISTICS</b>	
ISO/DIS 13528 April 2013	Statistical methods for use in proficiency testing by interlaboratory comparisons

**ISO published standards**

<b>FOODSTUFFS</b>	
FD CEN/TR 16338 January 2013	FOODSTUFFS Detection of food allergens – Template for supplying information about immunological methods and molecular biological methods
ISO 6887-6 March 2013	MICROBIOLOGY OF FOOD AND ANIMAL FEED Preparation of test samples, initial suspension and decimal dilutions for microbiological examination Part 6: Specific rules for the preparation of samples taken at the primary production stage
ISO 13307 March 2013	MICROBIOLOGY OF FOOD AND ANIMAL FEED Primary production stage – Sampling techniques

**NEW EU REGULATIONS**

Classification is established in alphabetical order of the first keyword

**ADDITIVES**

**O.J.E.U. L 77, 20<sup>th</sup> March 2013** – Commission Regulation (EU) No 244/2013 of 19 March 2013 amending Annex III to Regulation (EC) No 1333/2008 of the European Parliament and of the Council as regards the use of Tricalcium phosphate [E 341 (iii)] in nutrient preparations intended for use in foods for infants and young children  
<http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2013:077:0003:0004:EN:PDF>

**O.J.E.U. L 79, 21<sup>st</sup> March 2013** – Commission Regulation (EU) No 256/2013 of 20 March 2013 amending Annex III to Regulation (EC) No 1333/2008 of the European Parliament and of the Council as regards the use of Sodium ascorbate (E 301) in vitamin D preparations intended for use in foods for infants and young children  
<http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2013:079:0024:0026:EN:PDF>

**ANIMAL AND PUBLIC HEALTH RULES**

**O.J.E.U. L 90, 28<sup>th</sup> March 2013** – Commission Implementing Regulation (EU) No 300/2013 of 27 March 2013 amending Regulation (EU) No 605/2010 laying down animal and public health and veterinary certifications conditions for the introduction into the European Union of raw milk and dairy products intended for human consumption  
<http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2013:090:0071:0077:EN:PDF>

**HEALTH CLAIMS**

**O.J.E.U. L 22, 25<sup>th</sup> January 2013** – Commission Implementing Decision of 24 January 2013 adopting guidelines for the implementation of specific conditions for health claims laid down in Article 10 of Regulation (EC) No 1924/2006 of the European Parliament and of the Council  
<http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2013:022:0025:0028:EN:PDF>

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

**O.J.E.U. C 33, 5<sup>th</sup> February 2013** – 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 [Jihoceska Niva (cheese) (PGI)]  
<http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:C:2013:033:0005:0009:EN:PDF>

**O.J.E.U. L 36, 7<sup>th</sup> February 2013** – Commission Implementing Regulation (UE) n° 110/2013 of 6 February 2013 entering a name in the register of protected designations of origin and protected geographical indications [Gruyère (cheese) (PGI)]  
<http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2013:036:0001:0002:EN:PDF>

**O.J.E.U. C 57, 27<sup>th</sup> February 2013** – 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 [Lietuviskas Varskes Suris (cheese) (PGI)]  
<http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:C:2013:057:0024:0027:EN:PDF>

**O.J.E.U. C 57, 27<sup>th</sup> February 2013** – 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 [Pecorino di Picinisco (cheese) (PDO)]  
<http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:C:2013:057:0028:0032:EN:PDF>

**O.J.E.U. L 55, 27<sup>th</sup> February 2013** – Corrigendum to Regulation (EU) No 1151/2012 of the European Parliament and of the Council of 21 November 2012 on quality schemes for agricultural products and foodstuffs  
<http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2013:055:0027:0027:EN:PDF>

**O.J.E.U. C 60, 1<sup>st</sup> March 2013** – 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 [Queso Los Beyos (cheese) (PGI)]  
<http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:C:2013:060:0011:0014:EN:PDF>

**O.J.E.U. L 61, 5<sup>th</sup> March 2013** – Notice concerning the entry into force of the Agreement between the European Union and the Republic of Moldova on the protection of geographical indications of agricultural products and foodstuffs  
<http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2013:061:0001:0001:EN:PDF>

**O.J.E.U. L 61, 5<sup>th</sup> March 2013** – Notice concerning the entry into force of the Agreement between the European Union and the Republic of Moldova on the protection of geographical indications of agricultural products and foodstuffs

<http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2013:061:0001:0001:EN:PDF>

**O.J.E.U. C 77, 15<sup>th</sup> March 2013** – 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 [Stelvio / Stilsfer (cheese) (PDO)]

<http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:C:2013:077:0029:0034:EN:PDF>

**O.J.E.U. C 77, 15<sup>th</sup> March 2013** – 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 [Puzzone di Moena / Spretz Tzaori (cheese) (PDO)]

<http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:C:2013:077:0021:0024:EN:PDF>

**O.J.E.U. L 82, 22<sup>th</sup> March 2013** – Commission Implementing Regulation (EU) No 269/2013 of 18 March 2013 approving non-minor amendments to the specification for a name entered in the register of protected designations of origin and protected geographical indications [Danablu (cheese) (PGI)]

<http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2013:082:0045:0046:EN:PDF>

### **PESTICIDES**

**O.J.E.U. L 25, 26<sup>th</sup> January 2013** – Commission Regulation (EU) No 34/2013 of 16 January 2013 amending Annexes II, III and IV to Regulation (EC) No 396/2005 of the European Parliament and of the Council as regards maximum residue levels for 2-phenylphenol, ametoctradin, *Aureobasidium pullulans* strains DSM 14940 and DSM 14941, cyproconazole, difenoconazole, dithiocarbamates, folpet, propamocarb, spinosad, spiroadiclofen, tebufenpyrad and tetraconazole in or on certain products

<http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2013:025:0001:0048:EN:PDF>

**O.J.E.U. L 88, 27<sup>th</sup> March 2013** – Commission Regulation (EU) No 251/2013 of 22 March 2013 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 aminopyralid, bifentazate, captan, fluazinam, fluopicolide, folpet, kresoxim-methyl, penthiopyrad, proquinazid, pyridate and tembotrione in or on certain products

<http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2013:088:0001:0044:EN:PDF>

### **PHARMACOLOGICALLY ACTIVE SUBSTANCES**

**O.J.E.U. L 38, 9<sup>th</sup> February 2013** – Commission Implementing Regulation (EU) No 116/2013 of 8 February 2013 amending the Annex to Regulation (EU) No 37/2010 on pharmacologically active substances and their classification regarding maximum residue limits in foodstuffs of animal origin, as regards the substance eprinomectin

<http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2013:038:0014:0016:EN:PDF>

**AFNOR VALIDATIONS**

During its 31 January-1<sup>st</sup> February 2013 meeting, the Technical Committee of NF VALIDATION approved by vote:

Commercial name	Date	Certificate	Description
<b>NEW VALIDATIONS</b>			
<b>IQ-CHECK CRONOBACTER SPP.</b>	Validation date: 31 Jan 2013 <b>End of validity: 31 Jan 2017</b>	<b>BRD-07/23-01/13</b>	<b>Detection of <i>Cronobacter</i> spp.</b> Powdered infant formula and environmental samples
<b>QIAGEN MERICON SALMONELLA SPP.</b>	Validation date: 1 Feb 2013 <b>End of validity: 1 Feb 2017</b>	<b>QIA-36/01-02/13</b>	<b>Detection of <i>Salmonella</i> spp.</b> All human food and animal feeding stuffs and environmental samples (except primary production stage environment)
<b>RENEWALS OF VALIDATIONS</b>			
<b>3M™ PETRIFILM™ RAPID COLIFORM COUNT PLATE</b>	Validation date: 18 Mar 1997 Renewal: 13 Dec 2001, 19 Sep 2005, 28 Nov 2008 and 31 Jan 2013 <b>End of validity: 18 Mar 2017</b>	<b>3M-01/05-03/97A</b>	<b>Enumeration of coliforms, reading after 14 hours</b> All human food products
<b>3M™ PETRIFILM™ RAPID COLIFORM COUNT PLATE</b>	Validation date: 18 Mar 1997 Renewal: 13 Dec 2001, 19 Sep 2005, 28 Nov 2008 and 31 Jan 2013 <b>End of validity: 18 Mar 2017</b>	<b>3M-01/05-03/97B</b>	<b>Enumeration of total coliforms, reading after 24 hours (gas and non gas producing colonies)</b> All human food products
<b>3M™ PETRIFILM™ RAPID COLIFORM COUNT PLATE</b>	Validation date: 18 Mar 1997 Renewal: 13 Dec 2001, 4 May 2006, 28 Nov 2008 and 31 Jan 2013 <b>End of validity: 18 Mar 2017</b>	<b>3M-01/05-03/97C</b>	<b>Enumeration of gas producing colonies, reading after 24 hours</b> All human food products except processed pork products
<b>LISTERIA PRECIS™</b>	Validation date: 8 Apr 2005 Extension: 15 Sep 2006 and 29 Mar 2007 Renewal: 24 Sep 2009 and 31 Jan 2013 <b>End of validity: 8 Apr 2017</b>	<b>UNI-03/04-04/05</b>	<b>Detection of <i>Listeria monocytogenes</i></b> All human food products and environmental samples
<b>RAPID' STAPH</b>	Validation date: 4 Feb 2005 Renewal: 27 Jan 2009 and 31 Jan 2013 <b>End of validity: 4 Feb 2017</b>	<b>BRD-07/09-02/05</b>	<b>Coagulase-positive <i>Staphylococcus</i> enumeration</b> All human food products and environmental samples

**EXTENSIONS OF VALIDATIONS**

<p><b>3M™ MOLECULAR DETECTION ASSAY SALMONELLA</b></p>	<p>Validation date: 30 Nov 2012 Extension: 31 Jan 2013 <b>End of validity: 30 Nov 2016</b></p>	<p><b>3M-01/11-11/12</b></p>	<p><b>Detection of <i>Salmonella</i></b> All human food products (except spices, aromatic herbs, instant coffees and teas, bouillon cubes/concentrates, milk powders and cocoa powders) and environmental samples (except primary production stage environment)</p>
<p><b>VIDAS UP SALMONELLA</b></p>	<p>Validation date: 6 Oct 2011 Extension: 2 Feb 2012, 6 Jul 2012 and 31 Jan 2013 <b>End of validity: 6 Oct 2015</b></p>	<p><b>BIO-12/32-10/11</b></p>	<p><b>Detection of <i>Salmonella</i></b> All human food products, animal feeding stuffs and production environment samples (including animal faeces and environmental samples from the primary production stage)</p>

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**

29-31 May 2013  
Aarhus, Denmark

ICAR Technical workshop 2013 and  
health data conference

<http://www.icar2013.dk>

3-7 June 2013  
Rotterdam, The Netherlands

IDF/ISO analytical week 2013

<http://www.idf-iso-analytical-week.org/ColumnsPage.php?siteID=1716&ID=1717>

## IN THE PRESS – ON THE WEB

Classification in alphabetical order of keywords

### **METHODS OF ANALYSIS AND SAMPLING**

#### **Report of the thirty-fourth session of the Codex Committee on methods of analysis and sampling**

<http://www.codexalimentarius.org/meetings-reports/en/>

► This report presents the 34<sup>th</sup> session of the Codex Committee on methods of analysis and sampling held from 4-8 March 2013 in Budapest, Hungary. The conclusions will be submit for adoption / examination by the 36<sup>th</sup> session of the Codex Alimentarius.

### **SALMONELLA**

#### **DuPont real-time *Salmonella* test certified**

<http://www.foodproductiondaily.com/Quality-Safety/DuPont-real-time-salmonella-test-certified>

► DuPont developed a new Bax system for detecting *Salmonella*. This tool is based on the polymerase chain reaction and was identified as effective in raw ground beef, cream cheese, bagged lettuce and dry pet food.

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