CECALAIT-S NEWSLETTER

ACTALIA
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ACTALIA Cecalait

Rue de Versailles – B.P. 70129
39801 POLIGNY CEDEX
FRANCE
www.cecalait.fr
www.actilia.eu

ARTICLE

EVALUATION OF THE FT-NIR DAIRYQUANT B4 ANALYSER

The DairyQuant B4 is a near infrared spectrometer (range 3700-14885 cm^{-1}) manufactured by Q-Interline (Denmark) and commercialised in France by Inforlab-Chimie. It is used for the determination of composition components in solid dairy products (butter, cheese…).

This instrument uses a high resolution industrial infrared spectrometer based on Fourier transform (IRTF) incorporating a patented double pivot Michelson interferometer with mirrors. This construction ensures the stability of the optical alignment. A resolution of 32 cm^{-1} is used for the butter application.

Various parameters (fat, protein, dry matter or moisture, chloride…) can be determined with a PLS calibration.
The apparatus is computer controlled with InfraQuant software, which ensures the signal treatment for the routine analyses. A second software (Horizon QI) for the development of calibrations can be supplied.

The tests:

The evaluation tests, realised in reusable Teflon caps, were performed in ACTALIA Cecalait physico-chemistry laboratory (reference and infrared analyses) in September and October 2013. The repeatability and the accuracy were evaluated for moisture and salt in unsalted and salted butter.

The calculation were performed according to ISO 21543/IDF 201 : 2006 standard.

1- EVALUATION OF THE REPEATABILITY

1.1- Samples

The tests were performed on 15 samples of unsalted butter and 30 samples of salted butter from supermarket. Then, butter come from many different sites of production.

1.2- Procedure

The repeatability of the instrument is evaluated using 15 samples of unsalted and salted butter for moisture and 30 samples of salted butter for salt (NaCl). The quantitative analyses were performed in two sets (unsalted butter and salted butter) in consecutive duplicate for each sample. A control milk was analysed before each set to verify the stability of the analyser.

1.3- Results

The following table present the results obtained:

PRODUCT	PARAMETER	n	min	max	M	Sx	Sr	Sr (%)	r
UNSALTED BUTTER	Moisture (g/100g)	15 (13)	13.18 (15.35)	16.31 (16.31)	15.562 (15.843)	0.821 (0.303)	0.067 (0.048)	0.43 (0.31)	0.185 (0.134)
SALTED BUTTER	Moisture (g/100g)	15 (13)	12.98 (15.12)	16.17 (16.17)	15.354 (15.684)	0.913 (0.278)	0.142 (0.099)	0.93 (0.63)	0.394 (0.273)
	NaCl (g/100g)	30 (29)	1.45 (1.45)	2.51 (2.51)	2.053 (2.047)	0.236 (0.237)	0.046 (0.040)	2.26 (1.94)	0.129 (0.110)

Table 1: Quant repeatability criteria for moisture and NaCl in butter samples
n, min, max: number of results, minimum and maximum value; M and Sx: mean and standard deviation of results; Sr and Sr%: absolute and relative standard deviation of repeatability; r: maximum deviation of repeatability (95% of cases)

The values in brackets correspond to values recalculated after elimination (deviations between duplicate higher than 3Sr and values out of calibration range 15-16.5% for moisture).

1.4- Conclusion

Despite the absence of standard criteria, it can be noted that the relative standard deviations of repeatability vary between 0.31% and 1.94% according to the parameter measured. For moisture, the performances are better for unsalted butter than for salted butter (0.31% against 0.63%).
If we compare to the reference methods used to evaluate the accuracy, the Sr values obtained (0.048 g/100 g and 0.099 g/100 g) are higher (moisture ISO 3727-1, Sr limit equal to 0.036 g/100 g).

2- EVALUATION OF THE ACCURACY

2.1- Procedure

The accuracy of the instrument was evaluated according to the evaluation of the repeatability. The instrumental values were carried out by a calibration of the manufacturer. The following reference methods were used :

- Moisture: drying method according to ISO 3727-1/IDF 80-1: 2001 (test in duplicate for unsalted butter and single test for salted butter),

Salt: chloruremeter method (single test).

The results obtained for moisture in unsalted butter samples by the reference method correspond to the mean of two replicates performed in repeatability condition.

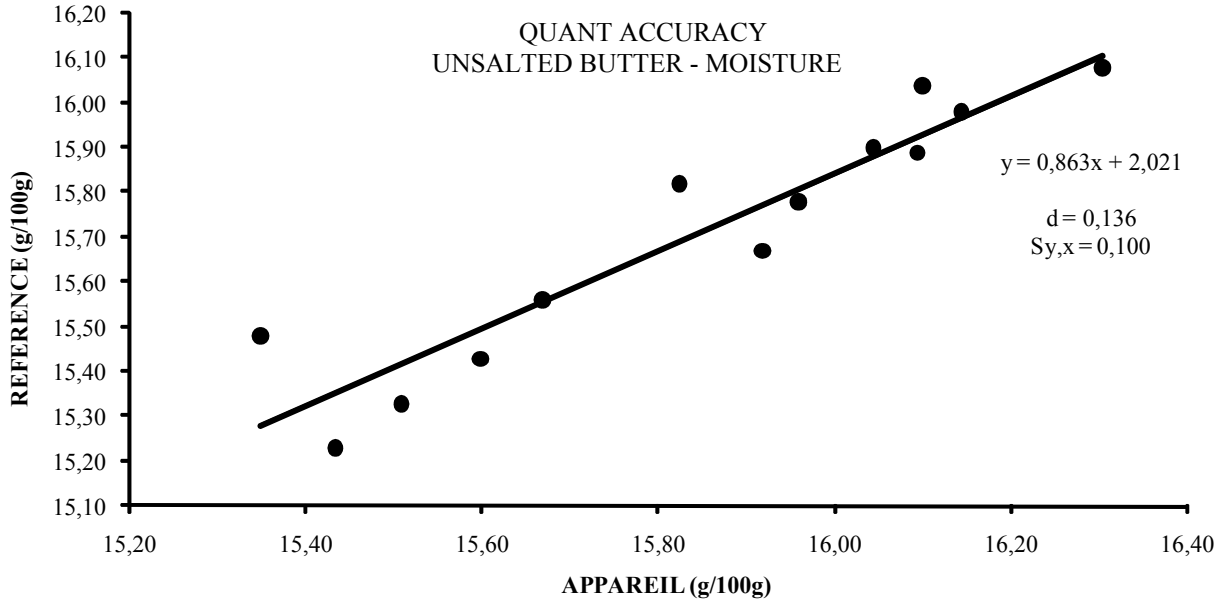
2.2- Results

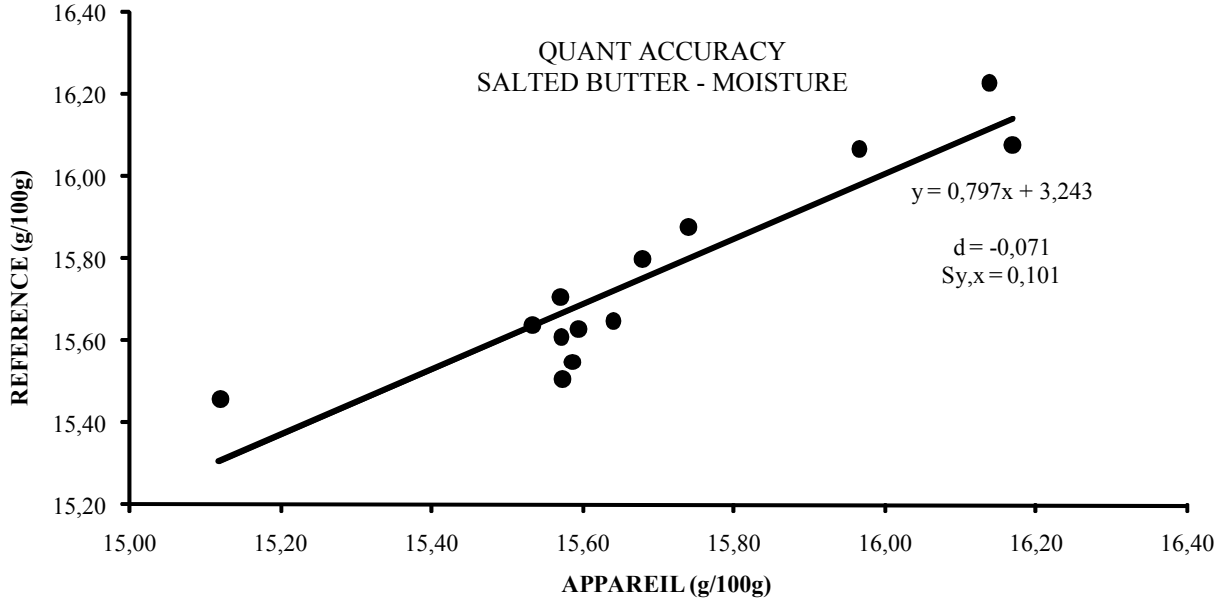
The following table and figures present the results obtained:

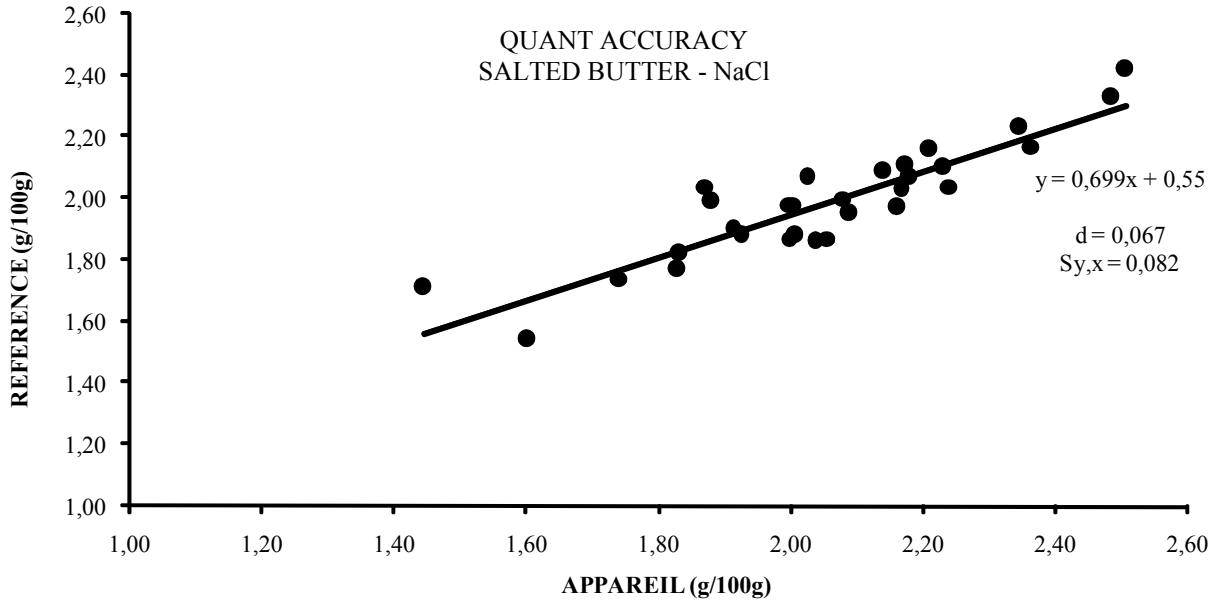
		n	min	Max	Y	Sy	d	Sd	Sy,x	Sy,x (%)	RMSEP
UNSALTED BUTTER	Moisture (g/100g)	15 (13)	14.18 (15.23)	16.08 (16.08)	15.587 (15.707)	0.472 (0.279)	-0.026 (0.136)	0.439 (0.104)	0.204 (0.100)	1.31 (0.63)	0.425 (0.169)
SALTED BUTTER	Moisture (g/100g)	15 (13)	14.46 (15.46)	16.23 (16.23)	15.595 (15.755)	0.481 (0.242)	-0.241 (-0.071)	0.462 (0.111)	0.122 (0.101)	0.80 (0.64)	0.507 (0.129)
	NaCl (g/100g)	30	1.54	2.42	1.986	0.183	0.067	0.107	0.082	4.00	0.125

Table 2: Quant accuracy criteria for moisture and NaCl in butter samples
n, min, max: number of results, minimum and maximum value; Y: mean results using reference method; Sy: standard deviation of the results from the reference method; d, Sd: mean and standard deviation of deviations; Sy,x, Sy,x (%): residual and relative standard deviation; RMSEP: prediction error.

The values in brackets correspond to values recalculated after elimination of the abnormal values according to Grubbs 5%, and the out of calibration range results (15-16.5% for moisture).







Figures 1,2 and 3: Relation between the reference and instrumental results for moisture and NaCl in butter samples

It can be noted that:

- the RMSEP values for moisture in unsalted and salted butter are respectively equal to 0.169 g/100 g and 0.129 g/100 g. The linear regression slopes (respectively 0.897 and 0.763) are not different from 1 (P = 5%).

- the RMSEP value for salt is equal to 0.125 g/100 g. The slope (0.699) of the linear regression is significantly different from 1 (P = 1%).

2.3- Conclusion

Despite the absence of standard criteria, the performances observed enable RMSEP predictions respectively equal to 0.169 g/100 g and 0.129 g/100 g (1.1% and 0.8%) for moisture in unsalted and salted butter, and 0.,125 g/100 g (6,3%) for salt. The RMSEP values observed are close to the manufacturer specifications (RMSECV limit: 0.1 g/100 g for moisture and 0.05 g/100 g for NaCl). The residual standard deviations of linear regression obtained are in accordance (moisture) or nearest (salt) of these specifications.

CONCLUSION

Because the absence of standard criteria, it is difficult to interpret the results obtained. As the evaluation was performed using manufacturer calibrations optimised for samples from a unique production entity, the RMSEP performances observed for each parameter measured are considered as maximum.
Consequently, according to the regression parameters obtained, the performances can be improved by a specific adjustment of the calibrations (slope and intercept according to the final equation Y = b.X + a) in local samples or by development of calibrations specific to the site.

According to the evaluation report of the FT-NIR DairyQuant B4 analyser – X. QUERVEL and Ph. TROSSAT – October 2013

STANDARDS, DRAFT STANDARDS

Classification in alphabetical order by theme

ISO standards under development

LABORATORY GLASSWARE	
ISO/DIS 3819 November 2013	LABORATORY GLASSWARE Beakers

ISO published standards

MICROBIOLOGY OF THE FOOD CHAIN	
ISO 4833-2/Cor1:2014 February 2014	MICROBIOLOGY OF THE FOOD CHAIN Horizontal method for the enumeration of microorganisms - Part 2: Colony count at 30 °C by the surface plating technique – Corrigendum 1
MILK AND MILK PRODUCTS	
ISO 8968-1:2014 (IDF 20-1) February 2014	MILK AND MILK PRODUCTS Determination of nitrogen content – Part 1: Kjeldahl principle and crude protein calculation *Replace ISO 5549 (1978), ISO 8968-1 (2001), ISO 8968-2 (2001) and ISO/TS 17837 (2008)*
STATISTICS	
ISO 16269-6:2014 January 2014	Statistical interpretation of data - Part 6: Determination of statistical tolerance intervals

NEW EU REGULATIONS

Classification is established in alphabetical order of the first keyword

CONTAMINANTS

O.J.E.U. L 65, 5th March 2014 – Commission Recommendation of 3 March 2014 on the monitoring of traces of brominated flame retardants in food
http://eur-lex.europa.eu/legal-content/EN/TXT/?uri=uriserv:OJ.L_.2014.065.01.0039.01.ENG

FLAVOURING SUBSTANCES

O.J.E.U. L 74, 14th March 2014 – Commission Regulation (EU) No 246/2014 of 13 March 2014 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/EN/TXT/?uri=uriserv:OJ.L_.2014.074.01.0058.01.ENG

FOOD SUPPLEMENTS

O.J.E.U. L 39, 8th February 2014 – Commission Regulation (EU) No 119/2014 of 7 February 2014 amending Directive 2002/46/EC of the European Parliament and of the Council and Regulation (EC) No 1925/2006 of the European Parliament and of the Council as regards chromium enriched yeast used for the manufacture of food supplements and chromium (III) lactate tri-hydrate added to foods
http://eur-lex.europa.eu/legal-content/EN/TXT/?uri=uriserv:OJ.L_.2014.039.01.0044.01.ENG

HEALTH CLAIM

O.J.E.U. L 14, 18th January 2014 – Commission Regulation (EU) No 40/2014 of 17 January 2014 authorising a health claim made on foods, other than those referring to the reduction of disease risk and to children's development and health and amending Regulation (EU) No 432/2012
http://eur-lex.europa.eu/legal-content/EN/TXT/?uri=uriserv:OJ.L_.2014.014.01.0008.01.ENG

STANDARDS - REGULATIONS

O.J.E.U. L 56, 26th February 2014 – Commission Regulation (EU) No 175/2014 of 25 February 2014 refusing to authorise certain health claims made on foods, other than those referring to the reduction of disease risk and to children's development and health
http://eur-lex.europa.eu/legal-content/EN/TXT/?uri=uriserv:OJ.L_.2014.056.01.0007.01.ENG

HYGIENE OF FOOD

O.J.E.U. L 66, 6th March 2014 – Commission Regulation (EU) No 209/2014 of 5 March 2014 amending Regulation (EU) No 605/2010 as regards animal and public health and veterinary certifications for the introduction of colostrum and colostrum-based products intended for human consumption into the Union
http://eur-lex.europa.eu/legal-content/EN/TXT/?uri=uriserv:OJ.L_.2014.066.01.0011.01.ENG

IMPORTS

O.J.E.U. L 87, 22nd March 2014 – Commission Implementing Decision of 20 March 2014 repealing lists of third country establishments from which Member States authorise imports of certain products of animal origin adopted on the basis of Council Decision 95/408/EC
http://eur-lex.europa.eu/legal-content/EN/TXT/?uri=uriserv:OJ.L_.2014.087.01.0104.01.ENG

METROLOGY

O.J.E.U. L 96, 29th March 2014 – Directive 2014/31/EU of the European Parliament and of the Council of 26 February 2014 on the harmonisation of the laws of the Member States relating to the making available on the market of non-automatic weighing instruments
http://eur-lex.europa.eu/legal-content/EN/TXT/?uri=uriserv:OJ.L_.2014.096.01.0107.01.ENG

O.J.E.U. L 96, 29th March 2014 – Directive 2014/32/EU of the European Parliament and of the Council of 26 February 2014 on the harmonisation of the laws of the Member States relating to the making available on the market of measuring instruments (recast)
http://eur-lex.europa.eu/legal-content/EN/TXT/?uri=uriserv:OJ.L_.2014.096.01.0149.01.ENG

P.D.O. / PG.I. / T.S.G.

O.J.E.U. L 8, 11th January 2014 – Commission Implementing Regulation (EU) No 18/2014 of 10 January 2014 entering a name in the register of traditional specialities guaranteed [Zemaitiskas kastinys (TSG) (dairy product)]
http://eur-lex.europa.eu/legal-content/EN/TXT/?uri=uriserv:OJ.L_.2014.008.01.0016.01.ENG

O.J.E.U. C 20, 21st January 2014 – 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 [Havarti (PGI) (cheese)]
http://eur-lex.europa.eu/legal-content/EN/TXT/?uri=uriserv:OJ.C_.2014.020.01.0009.01.ENG

O.J.E.U. C 28, 31st January 2014 – 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 [Charolais (PDO) (cheese)]
http://eur-lex.europa.eu/legal-content/EN/TXT/?uri=uriserv:OJ.C_.2014.028.01.0016.01.ENG

O.J.E.U. L 41, 12th February 2014 – Commission Implementing Regulation (EU) No 130/2014 of 10 February 2014 approving non-minor amendments to the specification for a name entered in the register of protected designations of origin and protected geographical indications [Selles-sur-Cher (PDO) (cheese)]
http://eur-lex.europa.eu/legal-content/EN/TXT/?uri=uriserv:OJ.L_.2014.041.01.0001.01.ENG

O.J.E.U. L 74, 14th March 2014 – Commission Implementing Regulation (EU) No 244/2014 of 7 March 2014 entering a name in the register of protected designations of origin and protected geographical indications [Strachitunt (AOP) (fromage)]
http://eur-lex.europa.eu/legal-content/EN/TXT/?uri=uriserv:OJ.L_.2014.074.01.0031.01.ENG

O.J.E.U. L 89, 25th March 2014 – Commission Implementing Regulation (EU) No 296/2014 of 20 March 2014 approving non-minor amendments to the specification for a name entered in the register of protected designations of origin and protected geographical indications [Neufchâtel (PDO) (cheese)]
http://eur-lex.europa.eu/legal-content/EN/TXT/?uri=uriserv:OJ.L_.2014.089.01.0032.01.ENG

O.J.E.U. L 89, 25th March 2014 – Commission Implementing Regulation (EU) No 297/2014 of 20 March 2014 approving non-minor amendments to the specification for a name entered in the register of protected designations of origin and protected geographical indications [Valençay (PDO) (cheese)]
http://eur-lex.europa.eu/legal-content/EN/TXT/?uri=uriserv:OJ.L_.2014.089.01.0034.01.ENG

STANDARDS - REGULATIONS

O.J.E.U. L 91, 27th March 2014 – Commission Implementing Regulation (EU) No 313/2014 of 26 March 2014 approving non-minor amendments to the specification for a name entered in the register of protected designations of origin and protected geographical indications [Pecorino Sardo (PDO) (cheese)]
http://eur-lex.europa.eu/legal-content/EN/TXT/?uri=uriserv:OJ.L_.2014.091.01.0036.01.ENG

PESTICIDES

O.J.E.U. L 22, 25th January 2014 – Commission Regulation (EU) No 61/2014 of 24 January 2014 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 cyromazine, fenpropidin, formetanate, oxamyl and tebuconazole in or on certain products
http://eur-lex.europa.eu/legal-content/EN/TXT/?uri=uriserv:OJ.L_.2014.022.01.0001.01.ENG

O.J.E.U. L 27, 30th January 2014 – Commission Regulation (EU) No 79/2014 of 29 January 2014 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 bifenazate, chlorpropham, esfenvalerate, fludioxonil and thiobencarb in or on certain products
http://eur-lex.europa.eu/legal-content/EN/TXT/?uri=uriserv:OJ.L_.2014.027.01.0009.01.ENG

O.J.E.U. L 35, 5th February 2014 – Commission Regulation (EU) No 87/2014 of 31 January 2014 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 acetamiprid, butralin, chlorotoluron, daminozide, isoproturon, picoxystrobin, pyrimethanil and trinexapac in or on certain products
http://eur-lex.europa.eu/legal-content/EN/TXT/?uri=uriserv:OJ.L_.2014.035.01.0001.01.ENG

O.J.E.U. L 87, 22nd March 2014 – Commission Regulation (EU) No 289/2014 of 21 March 2014 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 foramsulfuron, azimsulfuron, iodosulfuron, oxasulfuron, mesosulfuron, flazasulfuron, imazosulfuron, propamocarb, bifenazate, chlorpropham and thiobencarb in or on certain products
http://eur-lex.europa.eu/legal-content/FR/TXT/PDF/?uri=OJ:JOL_2014_087_R_0049_01&from=EN

PHARMACOLOGICALLY ACTIVE SUBSTANCES

O.J.E.U. L 8, 11th January 2014 – Commission Implementing Regulation (EU) No 19/2014 of 10 January 2014 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 chloroform
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2014:008:0018:0019:EN:PDF

O.J.E.U. L 8, 11th January 2014 – Commission Implementing Regulation (EU) No 20/2014 of 10 January 2014 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 butafosfan
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2014:008:0020:0021:EN:PDF

O.J.E.U. L 62, 4th March 2014 – Commission Implementing Regulation (EU) No 200/2014 of 3 March 2014 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 triptorelin acetate
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2014:062:0008:0009:EN:PDF

O.J.E.U. L 62, 4th March 2014 – Commission Implementing Regulation (EU) No 201/2014 of 3 March 2014 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 tildipirosin
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2014:062:0010:0012:EN:PDF

AFNOR VALIDATIONS

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

Commercial name	Date	Certificate	Description
NEW VALIDATION			
RAPID' *CAMPYLOBACTER*	Validation date: 31 Jan 2014 **End of validity: 31 Jan 2018**	**BRD-07/25-01/14**	**Enumeration of *Campylobacter* spp.** Meat products and production environment samples
RENEWALS OF VALIDATIONS			
VIDAS *LISTERIA* DUO	Validation date: 9 Mar 2006 Extension: 30 Jun 2011 Renewal: 3 Dec 2009 and 30 Jan 2014 **End of validity: 9 Mar 2018**	**BIO-12/18-03/06**	**Detection of *Listeria monocytogenes* and *Listeria* spp.** All human food products and environmental samples
TEMPO STA	Validation date: 1 Apr 2010 Renewal: 30 Jan 2014 **End of validity: 1 Apr 2018**	**BIO-12/28-04/10**	**Enumeration of coagulase positive staphylococci** All human food products and animal feeding
EXTENSIONS OF VALIDATIONS			
THERMOSCIENTIFIC SURETECT SALMONELLA SPECIES PCR ASSAY	Validation date: 4 Nov 2013 Extension: 30 Jan 2014 **End of validity: 4 Nov 2017**	**UNI-03/07-11/13**	**Detection of *Salmonella*** All human food products and pet food
RAPID' *ENTEROBACTERIACEAE*	Validation date: 29 Nov 2013 Extension: 31 Jan 2014 **End of validity: 29 Nov 2017**	**BRD-07/24-11/13**	**Enumeration of *Enterobacteriaceae*** All human food, animal feeding and production environment samples
VIDAS EASY *SALMONELLA*	Validation date: 20 Sep 2005 Renewal: 2 Jul 2009 and 4 Jul 2013 Extension: 30 Jun 2011 and 30 Jan 2014 **End of validity: 20 Sep 2017**	**BIO-12/16-09/05**	**Detection of *Salmonella*** All human food products and production environment samples (except primary production stage environment)
VIDAS CAM	Validation date: 21 May 2010 Extension: 30 Jun 2011 and 30 Jan 2014 **End of validity: 21 May 2014**	**BIO-12/29-05/10**	**Detection of *Campylobacter* spp.** Meat products and samples of production environment
VIDAS UP *SALMONELLA*	Validation date: 6 Oct 2011 Extension: 2 Feb 2012, 6 Jul 2012, 31 Jan 2013 and 30 Jan 2014 **End of validity: 6 Oct 2015**	**BIO-12/32-10/11**	**Detection of *Salmonella*** All human food products, animal feeding stuffs and production environment samples (including animal faeces and environmental samples from the primary production stage)

AFNOR VALIDATIONS

CAMPYFOOD AGAR	Validation date: 21 May 2010 Extension: 30 Jan 2014 **End of validity:** **21 May 2014**	**BIO-12/30-05/10**	**Detection of *Campylobacter* spp.** Meat products and samples of production environment
IRIS *SALMONELLA*	Validation date: 7 Oct 2011 Extension: 30 Jan 2014 **End of validity:** **7 Oct 2015**	**BKR-23/07-10/11**	**Detection of *Salmonella*** All human food products, animal feeding stuffs and production environment samples (except primary production stage environment)
PROLONGATION OF VALIDATION			
HQS E. COLI O157:H7	Validation date: 5 Feb 2010 **End of validity:** **5 Feb 2014** **Prolongation till** **5 May 2014**	**ADN-33/01-02/10**	**Detection of *E. coli* O157:H7** Meat products and dairy 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

IN THE PRESS – ON THE WEB

Classification in alphabetical order of keywords

METHODS OF ANALYSIS AND SAMPLING

Report of the thirty-fifth session of the Codex Committee on methods of analysis and sampling

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

► This report presents the 35th session of the Codex Committee on methods of analysis and sampling held from 3-7 March 2014 in Budapest, Hungary. The conclusions will be submit for adoption / examination by the 37th session of the Codex Alimentarius on 14-18 July 2014 in Geneva, Switzerland.

FORTHCOMING EVENTS

Classified in chronological order

8-10 April 2014 Baltimore, Maryland, United States	Food safety summit	http://www.foodsafetysummit.com/
15-20 May2014 Berlin, Germany	ISO/IDF analytical week 2014	http://www.idf-iso-analytical-week.org/
24-26 June 2014 Budapest, Hungary	International scientific conference on probiotics and prebiotics - 2014	http://www.probiotic-conference.net/

La Lettre de Cecalait est éditée par ACTALIA Cecalait, B.P. 70129, 39801 POLIGNY CEDEX
ACTALIA : association. Président : Patrick RAMET ; Directeur : Vincent OVERNEY
Directeur de la publication : Vincent OVERNEY
Créatrice : Annette BAPTISTE
Maquette : A. BAPTISTE, I. BECAR
Responsable de la rédaction : Carine TROUTET - E-mail : c.troutet@actalia.eu
A collaboré à ce numéro : Ph. TROSSAT, X. QUERVEL
Relecture : Ph. TROSSAT, X. QUERVEL, T. CADIOU
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