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EVALUATION OF THE NIRMasteTM INFRARED ANALYSER

The NIRMasteTM is a near infrared TF spectrophotometer manufactured by Buchi (Switzerland, Buchi Group), and commercialised in France by Buchi France (SARL). Among its available applications, it is used for the determination of the principal components in solid dairy products (powder, cheese, butter, yoghurt...).

This instrument uses the diffuse reflection in a spectral range of 12500-4000 cm⁻¹ with a resolution of 8 cm⁻¹, and it is supplied with a polarisation interferometer licensed by BUCHI[®]. NIRMasteTM exists in IP 54 and IP 65 configuration, and can be installed in production area, near the industrial plants. 3 types of cupel are available: glass, polystyrene, and quartz unbreakable.

The apparatus is computer controlled with NIRWare[®] software, which ensures the export of the results. The client can also equip its instrument with the NIRCAL[®] software, which ensures the signal treatment and the calibration according to the PLS models (partial least square).



The tests:

The evaluation tests were performed in Actilait-Cecalait's physico-chemistry laboratory (reference and instrumental analyses) from July to September 2011. After preliminary stability tests performed for dry matter (MS), fat (MG) and crude protein (MAT), the repeatability and accuracy on cheese were evaluated for the same parameters.

The calibrations used have been developed by the supplier thanks the NIRCAL[®] software, and the calculation parameters are in relation with the ISO 21543 / IDF 201 standard.

A- PRELIMINARY TESTS: EVALUATION OF THE STABILITY AT SHORT TERM

A.1- Procedure

A set of 3 samples of cheese was analysed in consecutive duplicate every 20 minutes for about 3 hours. Fat, dry matter and crude protein parameters were noted.

A.2- Results

The table below summarise the results obtained:

	CHEESE 1			CHEESE 2			CHEESE 3		
	MS	MG	MAT	MS	MG	MAT	MS	MG	MAT
N	8								
M (g/100g)	64.94	33.15	29.04	63.45	33.98	26.59	58.75	31.39	24.21
SR (g/100g)	0.05	0.12	0.60	0.11	0.10	0.55	0.18	0.12	0.29
SR (%)	0.1	0.4	2.1	0.2	0.3	2.1	0.3	0.4	1.2

Table 1: NIRMaste stability criteria for dry matter (MS), fat (MG) and crude protein (MAT)

N: number of results; *SR* and *SR%*: absolute and relative standard deviation of reproducibility; *M*: mean of results.

A.3- Conclusion

With no standard criteria, it can be noted that the relative standard deviations of reproducibility obtained vary between 0.1% and 2.1%.

B- EVALUATION OF REPEATABILITY AND ACCURACY

B.1- Samples

The tests were performed on 38 samples of hard cheese sold in supermarkets and hypermarkets (for fat, only 37 samples were evaluated). The samples were analysed after the rind removal and grinding.

B.2- Procedure

The repeatability and the accuracy of the instrument for dry matter, fat and crude protein were evaluated using all the samples. The quantitative analyses of each sample were carried out on set of 10 samples in consecutive duplicate. The instrumental values are from a calibration carried out by the manufacturer. The measures were realised in transmission through glass petri dishes.

The following reference methods were used:

- Dry matter: drying method according to ISO 5534 (single tests);
- Fat: SBR extraction method according to ISO 1735 (single tests);
- Crude protein: Kjeldahl method according to ISO 8968, conversion crude protein = total nitrogen x 6.38 (single tests).

B.3- Results

The tables and figures below summarise the results obtained:

	n	min	max	M	Sx	Sr	Sr (%)	r
MS (g/100g)	38	48.63	67.97	60.31	5.40	0.12	0.21	0.34
MG (g/100g)	38	23.11	36.81	31.00	3.69	0.11	0.35	0.30
MAT (g/100g)	38	20.56	29.98	25.68	2.37	0.12	0.45	0.32

Table 2: NIRMaste repeatability criteria for dry matter (MS), fat (MG) and crude protein (MAT)

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 in 95% of cases.

	MS (g/100g)	MG (g/100g)	MAT (g/100g)
n	38	37	38
min	48.78	22.21	19.87
max	68.48	35.58	29.96
Y	60.67	30.23	25.44
Sy	5.73	3.63	2.57
d	-0.36	0.66	0.24
Sd	0.46	0.35	0.63
Sy,x	0.342	0.345	0.626
Sy,x %	0.57	1.12	2.44
RMSEP	0.58	0.74	0.67
RMSEP %	0.96	2.46	2.63
b	1.059	0.984	1.056
a	-3.20	-0.15	-1.67

Table 3: NIRMaste accuracy criteria for dry matter (MS), fat (MG) and crude protein (MAT)

n, *min*, *max*: number of results; minimum and maximum value; *Y,X*: mean results using the 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 point of the linear regression; *RMSEP* and *RMSEP%*: absolute and relative quadratic mean error of prediction.

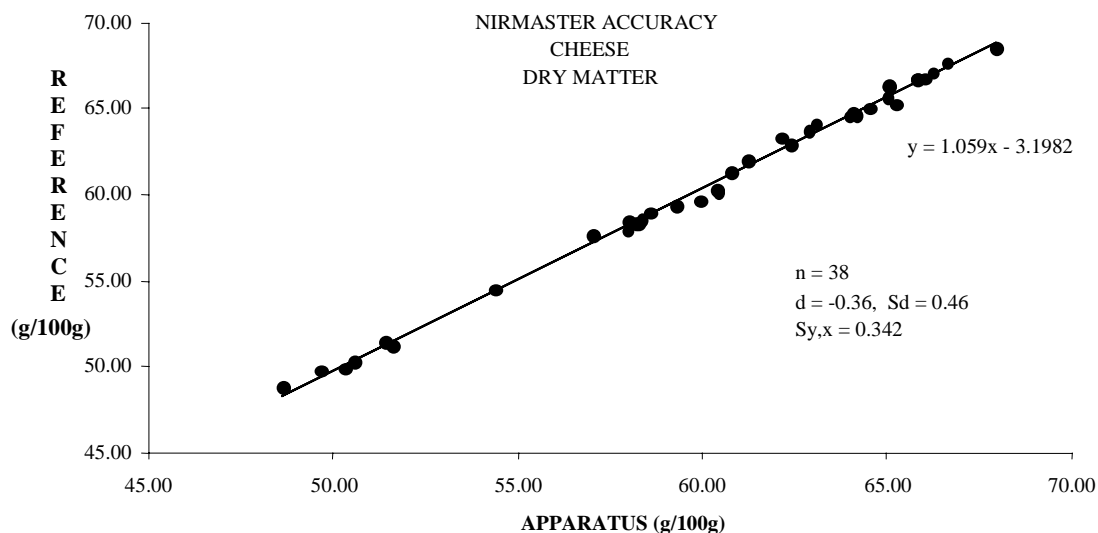


Figure 1: Relation between the NIRMasteR and the reference results for dry matter on cheese samples

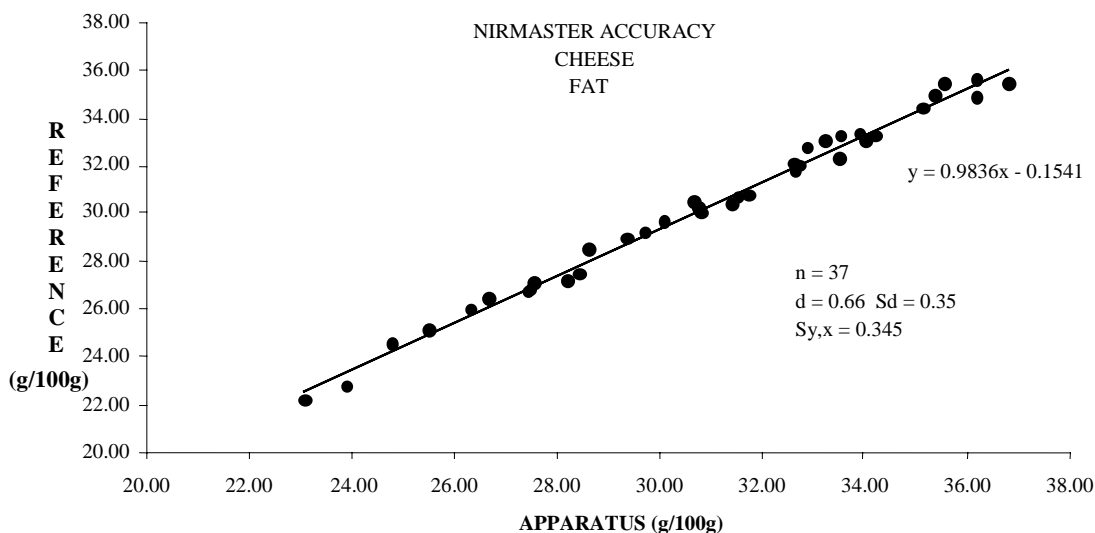


Figure 2: Relation between the NIRMasteR and the reference results for fat on cheese samples

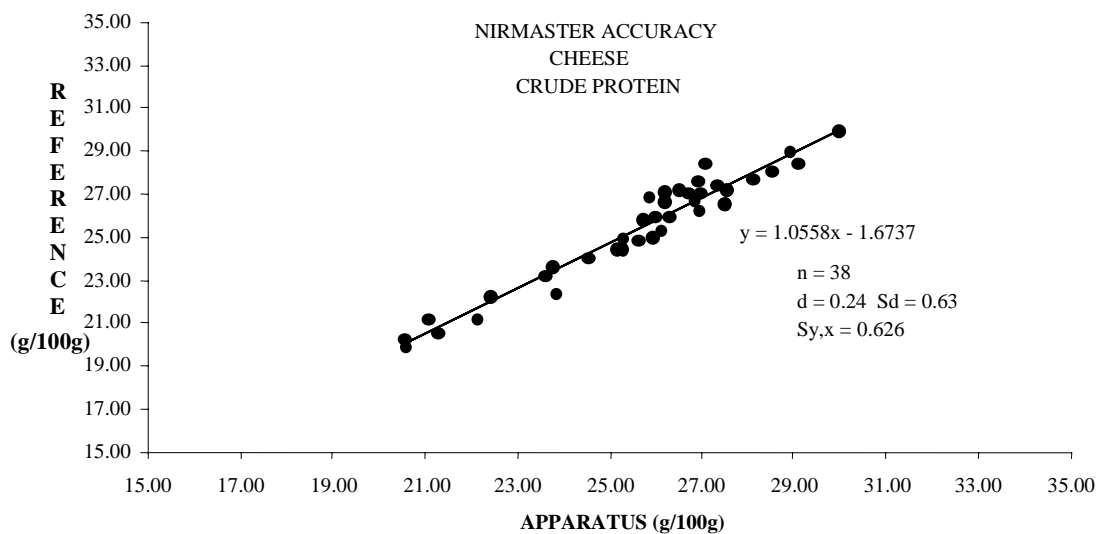


Figure 3: Relation between the NIRMasteR and the reference results for crude protein on cheese samples

It can be noted that:

⇒ For dry matter, the relative residual standard deviation is equal to 0.57%. The slope is equal to 1.059, significantly different from 1 (P = 1%). The relative error of prediction is equal to 0.96%.

⇒ For fat, the relative residual standard deviation is equal to 1.12%. The slope is equal to 0.984, no significantly different from 1 (P = 5%). The relative error of prediction is equal to 2.46%.

⇒ For crude protein, the relative residual standard deviation is equal to 2.44%. The slope is equal to 1.056, no significantly different from 1 (P = 5%). The relative error of prediction is equal to 2.63%.

B.4- Conclusion

Concerning the performance of repeatability, with no specific standard criteria for the near infrared analysers on these matrixes, it can be noted that the standard deviations of repeatability obtained with the instrument are close to the standard deviations of repeatability of the corresponding reference methods (Sr = 0.12 g/100g against 0.13 for dry matter, 0.11 g/100g against 0.11 for fat, and 0.12 g/100g against 0.11 for crude protein).

Concerning the accuracy, the values observed enable relative accuracy of estimation ($\pm 2 \cdot \text{RMSEP} \%$ at 5% risk) equal to $\pm 1.9\%$ for dry matter, $\pm 4.9\%$ for fat, and $\pm 5.3\%$ for crude protein.

CONCLUSION

With no standard criteria on this type of method (instrumental near infrared), the NIRMaster repeatability and accuracy performances cannot be interpreted. However, as the evaluation was performed with a manufacturer's calibration dedicated to a range of cheeses (hard cheese and grated hard cheese), a specific adjustment of the final model equation (slope and intercept point, or only intercept point) on the cheeses analysed would probably enable an improvement of the observed relative accuracy of estimation. Most specific calibrations can also be developed from the global basis.

According to the evaluation of the NIRMaster™ infrared analyser - X. QUERVEL – October 2011

ANNEX

List of the cheese samples

STABILITY

Cheese 1: Emmental

Cheese 2: Comté

Cheese 3: Gouda

REPEATABILITY AND ACCURACY

Chaussée aux moines™

Fol épi™

Gouda

Leerdamer™

St Paulin™

Beaufort

Emmental

Edam

Comté

Tomme du Jura

Appenzeller

Tomme de Savoie

STANDARDS, DRAFT STANDARDS

Classification in alphabetical order by theme

ISO standards under development

SENSORY ANALYSIS	
ISO/DIS 8586 September 2011	SENSORY ANALYSIS General guidances for the selection, training and monitoring of selected assessors and expert sensory assessors

ISO published standards

MICROBIOLOGY OF FOOD AND ANIMAL FEEDING STUFFS	
ISO 16140:2003/Amd 1 August 2011	MICROBIOLOGY OF FOOD AND ANIMAL FEEDING STUFFS Protocol for the validation of alternative methods – Amendment 1: interlaboratory study on quantitative methods
ISO 22118 July 2011	MICROBIOLOGY OF FOOD AND ANIMAL FEEDING STUFFS Polymerase chain reaction (PCR) for the detection and quantification of food-borne pathogens – Performance characteristics
ISO 22119 July 2011	MICROBIOLOGY OF FOOD AND ANIMAL FEEDING STUFFS Polymerase chain reaction (PCR) for the detection of food-borne pathogens – General requirements and definitions
SENSORY ANALYSIS	
ISO 11037 July 2011	SENSORY ANALYSIS Guidelines for sensory assessment of the colour of products
ISO 29842 July 2011	SENSORY ANALYSIS Methodology – Balanced incomplete block designs

NEW EU REGULATIONS

Classification is established in alphabetical order of the first keyword

CONTAMINANTS
O.J.E.U. L 215, 20th August 2011 – Commission Regulation (EU) No 836/2011 of 19 August 2011 amending Regulation (EC) No 333/2007 laying down the methods of sampling and analysis for the official control of the levels of lead, cadmium, mercury, inorganic tin, 3-MCPD and benzo(a)pyrene in foodstuffs http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2011:215:0009:0016:EN:PDF
O.J.E.U. L 218, 24th August 2011 – Commission Recommendation of 23 August 2011 on the reduction of the presence of dioxins, furans and PCBs in feed and food http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2011:218:0023:0025:EN:PDF
FOOD INGREDIENTS
O.J.E.U. L 215, 20th August 2011 – Commission Implementing Decision of 19 August 2011 authorising the placing on the market of phosphatidylserine from soya phospholipids as a novel food ingredient under Regulation (EC) No 258/97 of the European Parliament and of the Council http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2011:215:0020:0022:EN:PDF

HEALTH CLAIMS

O.J.E.U. L 182, 12th July 2011 – Commission Regulation (EU) No 666/2011 of 11 July 2011 refusing to authorise certain health claims made on foods, other than referring to the reduction of disease risk and to children's development and health

<http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2011:182:0008:0009:EN:PDF>

PESTICIDES

O.J.E.U. L 208, 13th August 2011 – Commission Regulation (EU) No 813/2011 of 11 August 2011 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 acequinocyl, emamectin benzoate, ethametsulfuron-methyl, flubendiamide, fludioxonil, kresoxim-methyl, methoxyfenozide, novaluron, thiacloprid and trifloxystrobin in or on certain products

<http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2011:208:0023:0079:EN:PDF>

O.J.E.U. L 258, 4th October 2011 – Commission Regulation (EU) No 978/2011 of 3 October 2011 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 acetamiprid, biphenyl, captan, chlorantraniliprole, cyflufenamid, cymoxanil, dichlorprop-P, difenoconazole, dimethomorph, dithiocarbamates, epoxiconazole, ethephon, flutriafol, fluxapyroxad, isopyrazam, propamocarb, pyraclostrobin, pyrimethanil and spirotetramat in or on certain products

<http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2011:258:0012:0069:EN:PDF>

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

O.J.E.U. L 200, 3rd August 2011 – Commission Implementing Regulation (EU) No 766/2011 of 29 July 2011 entering a name in the register of protected designations of origin and protected geographical indications [Xygaló Siteias (cheese) (PDO)]

<http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2011:200:0012:0013:EN:PDF>

O.J.E.U. L 204, 9th August 2011 – Commission Regulation (EU) No 794/2011 of 8 August 2011 approving amendments to the specification for a name entered in the register of protected designations of origin and protected geographical indications [Parmigiano Reggiano (cheese) (PDO)]

<http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2011:204:0019:0020:EN:PDF>

O.J.E.U. C 247, 25th August 2011 – Publication of an amendment application pursuant to Article 6(2) of Council Regulation (EC) No 510/2006 on the protection of geographical indications and designations of origin for agricultural products and foodstuffs [Langres (cheese) (PDO)]

<http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:C:2011:247:0011:0019:EN:PDF>

HEALTH SECURITY

O.J.E.U. L 237, 14th September 2011 – Commission Implementing Regulation (EU) No 914/2011 of 13 September 2011 amending Regulation (EU) No 605/2010 laying down animal and public health and veterinary certification 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:2011:237:0001:0016:EN:PDF>

O.J.E.U. L 242, 20th September 2011 – Commission Implementing Regulation (EU) No 931/2011 of 19 September 2011 on the traceability requirements set by Regulation (EC) No 178/2002 of the European Parliament and of the Council for food of animal origin

<http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2011:242:0002:0003:EN:PDF>

AFNOR VALIDATIONS

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

Commercial name	Date	Certificate	Description
NEW VALIDATION			
IBISA	Validation date: 01.07.2011 End of validity: 01.07.2015	AES 10/11-07/11	Detection of <i>Salmonella</i> All human food products, animal feeding stuffs and production environment samples (except primary production stage environment)
RENEWALS OF VALIDATIONS			
SALMONELLA RAPID TEST	Validation date: 30.05.1991 Renewal: 08.09.1995, 07.09.1999, 11.12.2003, 04.12.2007 and 01.07.2011 End of validity: 07.09.2015	UNI 03/01-05/91	Detection of <i>Salmonella</i> All human food products and production environment samples (except primary production stage environment)
TAQMAN SALMONELLA	Validation date: 28.09.2007 Renewal: 01.07.2011 End of validity: 28.09.2015	ABI 29/01-09/07	Detection of <i>Salmonella</i> All human and animal food products
EXTENSIONS OF VALIDATIONS			
IQ-CHECK SALMONELLA II	Validation date: 01.07.2004 Renewal: 27.11.2008 Extension: 24.05.2007, 28.09.2007, 25.09.2008, 04.02.2010, 03.02.2011 and 01.07.2011 End of validity: 01.07.2012	BRD 07/6-07/04	Detection of <i>Salmonella</i> All human and animal food products and environmental samples (including animal faeces and environmental samples from the primary production stage)
VIDAS SALMONELLA (DUAL SELECTIVE ENRICHMENT PROTOCOL)	Validation date: 06.04.1994 Renewal: 09.06.1998, 18.09.2002, 15.09.2006 and 20.05.2010 Extension: 30.06.2011 End of validity: 09.06.2014	BIO 12/01-04/94	Detection of <i>Salmonella</i> All human food and pet food products
VIDAS SALMONELLA (SINGLE SELECTIVE ENRICHMENT PROTOCOL)	Validation date: 18.09.2002 Renewal: 15.09.2006 and 20.05.2010 Extension: 30.06.2011 End of validity: 18.09.2014	BIO 12/10-09/02	Detection of <i>Salmonella</i> All human food and pet food products
VIDAS EASY SALMONELLA	Validation date: 20.09.2005 Renewal: 02.07.2009 Extension: 30.06.2011 End of validity: 20.09.2013	BIO 12/16-09/05	Detection of <i>Salmonella</i> All human food products and production environment samples (except primary production stage environment)
VIDAS IMMUNO-CONCENTRATION SALMONELLA II (VIDAS ICS2 + SLM)	Validation date: 24.05.2007 Renewal: 12.05.2011 Extension: 30.06.2011 End of validity: 24.05.2015	BIO 12/22-05/07	Detection of <i>Salmonella</i> All human products (except raw milk) and pet food products

AFNOR VALIDATIONS

VIDAS LISTERIA MONOCYTOGENES XPRESS (VIDAS LMX)	Validation date: 04.02.2010 Extension: 30.06.2011 End of validity: 04.02.2014	BIO 12/27-02/10	Detection of <i>Listeria monocytogenes</i> All human food products and environmental samples
VIDAS LISTERIA MONOCYTOGENES 2 (SAME PROTOCOL AS VIDAS LISTERIA – WITH A 30°C ENRICHMENT STEP)	Validation date: 03.07.2002 Renewal: 15.06.2006 and 21.05.2010 Extension: 14.12.2006 and 30.06.2011 End of validity: 03.07.2014	BIO 12/09-07/02	Detection of <i>Listeria monocytogenes</i> All human food products (except raw products) and environmental samples
VIDAS LISTERIA MONOCYTOGENES 2 (WITH A 37°C ENRICHMENT STEP)	Validation date: 12.03.2004 Renewal: 17.01.2008 Extension: 02.12.2004, 14.12.2006 and 30.06.2011 End of validity: 12.03.2012	BIO 12/11-03/04	Detection of <i>Listeria monocytogenes</i> All human food products and environmental samples
VIDAS LISTERIA DUO	Validation date: 09.03.2006 Renewal: 03.12.2009 Extension: 30.06.2011 End of validity: 09.03.2014	BIO 12/18-03/06	Detection of <i>Listeria monocytogenes</i> and <i>Listeria spp.</i> All human food products and environmental samples
VIDAS LISTERIA	Validation date: 17.06.1994 Renewal: 09.06.1998, 03.07.2002, 04.05.2006 and 21.05.2010 Extension: 18.09.2002, 02.04.2003 and 30.06.2011 End of validity: 09.06.2014	BIO 12/02-06/94	Detection of <i>Listeria spp.</i> All human food products and environmental samples
VIDAS LISTERIA SPECIES XPRESS	Validation date: 01.07.2004 Renewal: 01.07.2008 Extension: 02.12.2004, 20.09.2005 and 30.06.2011 End of validity: 01.07.2012	BIO 12/12-07/04	Detection of <i>Listeria spp.</i> Milk products, meat products, vegetable products and environmental samples
VIDAS ECO	Validation date: 05.07.2000 Renewal: 23.09.2004 and 23.05.2008 Extension: 24.05.2007, 17.01.2008 and 30.06.2011 End of validity: 05.07.2012	BIO 12/08-07/00	Detection of <i>E. coli</i> O157 All human food products (+ specific operating rules for raw beef meat)
VIDAS UP <i>E. COLI</i> O157 INCLUDING H7 (VIDAS ECPT)	Validation date: 18.05.2009 Extension: 03.12.2009 and 30.06.2011 End of validity: 18.05.2013	BIO 12/25-05/09	Detection of <i>E. coli</i> O157 All human food products and 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>

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.

MILK AND DAIRY PRODUCTS

FUQUAY J.W., FOX P.F., McSWEENEY P.H.L. – **Encyclopedia of Dairy Sciences – 2nd Edition – Four volume set** –Elsevier Editions – March 2011 – ISBN : 978-0-12-374402-9 – 4170 pages

<http://www.elsevierdirect.com/ISBN/9780123744029/Encyclopedia-of-Dairy-Sciences-2nd-Edition-FourVolume-set>



This second edition was fully reviewed, revised and updated with the last developments in dairy science. The biological, chemical, physical, microbiological and technological aspects of milk and milk-derived food products are studied.

IN THE PRESS – ON THE WEB

Classification in alphabetical order of keywords

ANTIBIOTICS

Radox unveils multi-analyte milk testing platform

<http://www.laboratorytalk.com/news/ran/ran230.html>

► Radox Food Diagnostics has developed and manufactured a multi-analyte milk testing platform enable to detect sulphonamide, trimethoprim, quinolone, tetracycline, penicillin, cephalosporin compounds...

HEALTH CLAIMS

EFSA publishes fifth series of evaluations of "general function" health claims

<http://www.efsa.europa.eu/en/press/news/110630.htm>

► On 30 June 2011, EFSA's NDA panel finalised the evaluation of all "general function" health claims due to be adopted by that date. 536 new claims have been also added to the 2187 claims published to date. A remaining of 35 claims has been published in July 2011.

PESTICIDES

Reasoned opinion of EFSA: Modification of the existing MRLs for amidosulfuron in bovine fat, kidney, liver and milk

<http://www.efsa.europa.eu/en/efsajournal/pub/2325.htm>

► EFSA received from Bayer CropScience an application to modify the existing MRL for the active substance amidosulfuron in bovine fat, kidney, liver and milk.

STANDARDISATION

Codex Alimentarius Commission

<http://www.codexalimentarius.net/web/archives.jsp?lang=en>

► Report from the 34th session of the Codex Alimentarius Commission held from 4-9 July in Geneva, Switzerland.

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