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PUBLICATION OF OUR 2015 CATALOGUE OF SERVICES

As the last years, the Cecalait's catalogue of services has been sent by e-mail last week. We would remind you that you also can find it in on-line in the "Catalogue" section on our website http://www.cecalait.com (Careful, order on-line via our website is not possible. You can only realise your order sheet and calculate its approximate cost. You have then to print your subscription forms realised and send them to us by post, fax or e-mail).

In this catalogue, we propose new services: a proficiency test and standard reference materials on retentate, for dry matter and total nitrogen parameters. Two proficiency tests have been planned in February and October, and as for the other Cecalait's proficiency tests, you can register for one or the both criteria. It is the same for SRMs, which are proposed in monthly dispatch or on request. You can consult further information pages 14-15 and 56 of the catalogue.

We hope that many of you will use these new products and we are at your disposal for any further information.

<u>EVALUATION OF SIGMA REAGENTS</u> – Cecalait's Newsletter No 83 (4th quarter 2012)

An evaluation of the Sigma reagents for the determination of fat by the Gerber method (sulfuric acid and amylic alcohol) was performed in our laboratories in 2012, at the request of the distributor. The results have been published in Cecalait's Newsletter No 83 (4th quarter 2012).

This evaluation was about the comparison between the results obtained with the Sigma reagents and the reagents used in routine in our laboratories for 10 samples of milk and two successive batches of reference materials (4 samples analysed in duplicate) and the reference value. The objective of this study was to verify that no significant differences concerning the accuracy of the evaluated reagents in relation to reagents actually used in routine in the laboratories exist.

It should be noted that these tests were performed after the transmission of the quality prerequisites of the reagents (normative requirements) to the tests enquirer.

In this case, the comparison results to reagents already used in laboratories and the tests with reference materials allowed to observe relatively low and no significant differences for the fat content in milk:

- \bullet Mean deviation of +0.06 g/L with Sd standard deviation of deviations = 0.09 for the comparison of 10 samples of milk
- Mean deviations of -0.03 and +0.07 g/L with Sd standard deviation of deviations respectively equal to 0.08 and 0.05 for the tests on 2 batches of Gerber SRMs.

Contrary to what was initially announced, the amylic alcohol evaluated during these tests (Ref. 59090) was not in conformity with the recommendations of the NF V 04-210 standard concerning the Methyl 3 Butanol 1 and Méthyl 2 Butanol 1 isomers ratio. We have recently learned this information by the description form of this product, which was recently published and clearly indicates that it is composed of 100 % of Méthyl 3 isomer (this was not specified on the conformity certificate of the batch used for the tests).

It can be also noted that the relatively low difference of the concentration in Méthyl 3 Butanol 1 in relation to the recommendation (100 % corresponding to the SIGMA product to 93 % corresponding to the upper limit of the NF V 04-210 standard) may explain that no significant deviations were observed during the comparison tests.

It can be then concluded that, even if low and no significant differences were observed, SIGMA Ref. 59090 amylic alcohol can not be used for the determination of fat in accordance with the NF V 04-210 method. It is also important to note that the other evaluated reagents are not concerned by this technical note.

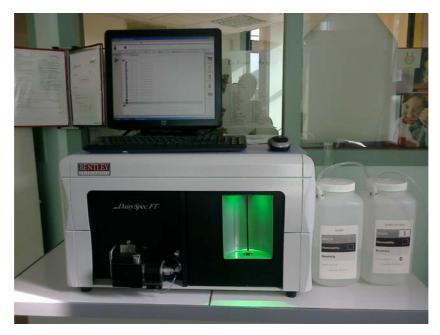
For our part, we are sorry for this late feedback to the laboratories, and to prevent this situation, we have decided, for the next evaluations of reagents, that the evaluation folder will have to contain a formal statement by the enquirer (with documentary evidences) concerning the standardised recommendations of the reagents quality.

EVALUATION OF THE BENTLEY DAIRYSPEC FT® INFRARED ANALYSER

The DairySpec FT is an infrared spectrophotometer (650 to 4000 cm-1) manufactured by Bentley Instruments (USA), and commercialised in France by Bentley Instruments SARL. It is used for the determination of the composition components in milk and liquid dairy products samples (fat, protein, lactose, dry matter, urea, freezing point...).

This instrument uses a mono-bundle with high resolution industrial interferometer Fourier transform-based infrared system (FTIR). The complete infrared spectrum (standard resolution 8 cm-1) collected enables MLR or PLS calibrations, necessary for the quantification of the major or minor components.

The apparatus is computer controlled with a software, which ensures the signal treatment. The back-up of the analysed samples spectrum enables a posteriori re-treatment of the signal. It is possible to equip the instrument with an auto-sampler and to combine it with the "Somacount" somatic cells counter, to achieve an analytical rate of 300 samples / hour.



The tests:

The evaluation tests were performed in ACTALIA Cecalait's physico-chemistry laboratory (reference and infrared analyses) from June to September 2014. After preliminary tests of stability, linearity and calibration performed for dry matter (MS), fat (MG) and protein (MP), the repeatability and accuracy on milk, cream and proteic retentate were evaluated.

The appreciation criteria of the estimated parameters were taken from either ISO 9622/IDF 141: 2013 "Guidelines for the application of mid-infrared spectrometry" and ISO 8196-3/IDF 128-3: 2009 "Milk - Definition and evaluation of the overall accuracy of alternative methods of milk analysis - Part 3", or from the CNIEL/IE PROC IR v06 handbook concerning the use of infrared apparatus within the context of milk payment and milk control in France. A comparison in relation to the manufacturer specifications is also realised..

The instrument was configured for a rate of 60 samples / hour for cream and retentate, 120 samples / hour for milk and non correction of contamination.

A. PRELIMINARY TESTS

A.1 – Evaluation of the stability

The stability was evaluated by the analysis, in automatic mode, of 3 samples of milk at 3 different rates every 15 minutes during a work half-day, representing 20 measurement cycles. The repeatability and reproducibility were calculated for each analytical criterion and by level.

Results:

The following tables 1 to 3 present the results obtained:

	M (g/l)	Sr (g/l)	Sr (%)	SR (g/l)	SR (%)	r (g/l)	R (g/l)
1	19.91	0.04	0.18%	0.04	0.18%	0.10	0.10
2	40.24	0.04	0.10%	0.04	0.10%	0.11	0.11
3	82.61	0.09	0.11%	0.09	0.11%	0.25	0.25

<u>Table 1</u>: DairySpec FT stability criteria for fat

	M (g/l)	Sr (g/l)	Sr (%)	SR (g/l)	SR (%)	r (g/l)	R (g/l)
1	20.06	0.03	0.13%	0.03	0.13%	0.07	0.07
2	30.24	0.03	0.10%	0.03	0.10%	0.08	0.08
3	58.38	0.06	0.10%	0.06	0.10%	0.17	0.17

Table 2: DairySpec FT stability criteria for protein

	M(g/l)	Sr (g/l)	Sr (%)	SR (g/l)	SR (%)	r (g/l)	R (g/l)
1	99.24	0.05	0.05%	0.05	0.05%	0.14	0.14
2	128.41	0.09	0.07%	0.09	0.07%	0.24	0.24
3	193.54	0.17	0.09%	0.17	0.09%	0.49	0.49

Table 3: DairySpec FT stability criteria for dry matter

M: mean; Sr and SR (Sr% and SR%): standard deviation of absolute repeatability and reproducibility (and relative); r and R: maximum deviation of repeatability and reproducibility in 95 % of cases.

The daily mean values of standard deviation of reproducibility (SR) for fat and protein are below the limits required in ISO 8196-3/IDF 128-3 (SR \leq 0.29 g/l for median range or SR \leq 0.58 g/l for high range).

As no standardised or regulatory values exist for dry matter, it can be noted that the reproducibility standard deviation SR is lower than the standardised value of the ISO 6731/IDF 21 : 2010 reference method (R = 2.1 g/l; SR = 0.74 g/l).

A.2 – Evaluation of linearity

Volume/volume dilutions were carried out by corrected weighing of density. This corresponds to the principle of quantitative analysis of infrared spectrophotometry and to the French reference measurements.

For fat, a range of 12 milk samples from 0 to 100 g/l was prepared by mixing cream and skimmed milk. This range was analyse 5 times in the descending order of the fat rates.

The Ar/At ration (Ar and At: amplitude of residues and amplitude of contents respectively) is equal to 1.6 %, that is in accordance with the limit of 2 % expressed in ISO 9622/IDF 141 standard.

3 ranges of optimal (0-20g/l: Ar/At = 0.36 %, 20-70g/l: Ar/At = 0.42 % and 70-100 g/l: Ar/At = 1.56 %) are also identified. Within the 0-20 and 20-70 g/l ranges, the Ar/At ratios are in accordance with the specifications of the ISO 8196-3/IDF 128-3 standard (limit 1 %).

For protein, a range of 12 milk samples from 0 to 130 g/l was prepared by mixing proteic retentate and filtrate obtained by tangential ultrafiltration (cutoff threshold: 10 KD). The range was analysed 5 times in the descending order of the fat rates.

The Ar/At ration within the range studied is equal to 1.3 %, which is in conformity with the recommendations of 2 % maximum given in ISO 9622/IDF 141 standard. The Ar/At ratios within the 0-110 g/l (Ar/At: 0.85 %), 0-40 g/l (Ar/At: 0.18 %) and 40-110 g/l (Ar/At: 0.64 %) ranges are also in accordance with the specification of the 8196-3/IDF 128-3 standard (limit 1 %).

The linearity of this instrument is therefore satisfactory for fat (range from 0 to 100 g/l) and protein (range from 0 to 130 g/l).

A.3 – Evaluation of the calibration

the evaluation of the calibration for fat and protein, initially installed by the manufacturer, was performed with 13 commercial "median" and "high" infrared standard reference materials (SRMs) produced by ACTALIA Cecalait in July and September 2014. Each sample was analysed in duplicate.

Results:

The results are presented in the following table:

	N	Min- max	Sr	d	Sd	Sl1	Sl3
Fat (g/l)-Median	13	22.3 53.7	0.032	0.098	0.194	0.103	0.094
Protein (g/l) -Median	13	24.4 39.9	0.036	-0.045	0.196	0.048	0.029
Dry matter (g/l) -Median	13	104.9 150.8	0.087	-0.159	0.445	0.429	/
Fat (g/l)-High	13	59.6 91.8	0.118	-0.292	0.257	0.262	0.261
Protein (g/l) -High	13	45.2 66.6	0.035	1.575	0.250	0.039	0.037
Dry matter (g/l) -High	13	162.7 212.9	0.086	0.293	0.510	0.531	/

Table 4: DairySpec FT calibration criteria for fat, protein and dry matter

N: number of standards; min and max: minimum and maximum values; Sr: standard deviation of repeatability; d and Sd: mean and standard deviation of deviations (instrument – reference); Sl1 and Sl3: residual standard deviation of simple linear regression (reference vs instrument) or multiple (reference vs fat, protein and lactose)

Conclusion:

The residual standard deviations of the linear regression obtained for fat and protein are in conformity with the recommendations of the CNIEL/IE handbook (respectively lower than 0.25 and 0.15 g/l for the "median" range, corresponding to the cow milk contents, and respectively lower than 0.50 and 0.30 g/l for the "high" range, corresponding to ewe milk contents).

B. EVALUATION OF REPEATABILITY AND ACCURACY

B.1 – The samples

The test were performed on:

- for milk: 40 samples of tank milk from the Rhône-Alpes region.
- for cream: 20 samples of pasteurised cream (obtained by cream skimming) from the Rhône-Alpes region.
- ♦ for proteic retentate: 25 samples of proteic retentate and permeate (obtained by ultrafiltration of skim milk) from the Bretagne region. Many "artificial" samples were realised by mixing retentate and permeate to obtain a variability of protein rates.

Bronopol at 0,02 % final was added to all the samples.

B.2 – Procedure

The repeatability and accuracy of the instrument were evaluated for fat, protein and dry matter in milk, fat and dry matter in cream, and protein and dry matter in retentate. The quantitative analyses were performed in duplicate for each sample. A control milk was analysed at the beginning and the end of each set to verify the stability of the instrument.

The evaluation concerns the values obtained after calibration and adjustment of the instrument with commercial SRMs produced by ACTALIA Cecalait for the milk samples, and after calibration and adjustment of the instrument by the manufacturer for the cream and retentate samples.

The following reference methods were used:

- Fat: Gerber acido-butyrometric method according to NF V 04-210: 2000 for milk and NF V 04-263: 1997 for cream (single tests and then confirmation if more important residues).
- ➤ Protein: Amido black method according to NF V 04-216: 2011 (test in duplicate –volume/volume dilution of samples according to annex A for the retentate).
- ➤ Dry matter: drying method according to ISO 6731/IDF 21: 2010 (single test).

B.2 – Results

B.2.1 – Milk

The following tables and figures present the results obtained:

	n	min	max	M	Sx	Sr	Sr (%)	r
Fat (g/l)	40	38.0	41.8	39.650	0.860	0.022	0.06	0.061
Protein (g/l)	40	31.4	33.3	32.277	0.573	0.023	0.07	0.064
Dry matter (g/l)	40	127.0	130.7	128.958	1.006	0.057	0.04	0.159

<u>Table 1</u>: DairySpec FT repeatability criteria for fat, protein and dry matter in tank milk samples

n: number of results; min et max: minimum and maximum values; M and Sx: mean and standard deviation of the results; Sr and Sr %: absolute and relative standard deviation of repeatability; r: maximum deviation of repeatability on 95 % of cases.

For fat and protein, the standard deviation of repeatability is in conformity with the recommendations of the ISO 8196-3/IDF 129-3 standard and the CNIEL/IE handbook ($Sr \le 0.14 \text{ g/l}$ and $r \le 0.4 \text{ g/l}$).

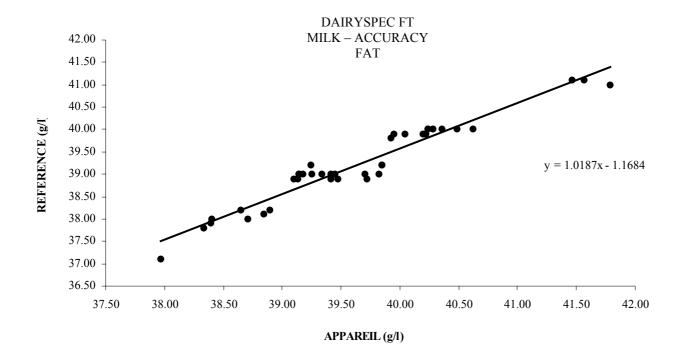
For dry matter, as no values exist in standards or CNIEL/IE handbook, it can be noted that the standard deviation of repeatability obtained is lower than the limits of the ISO 6731/IDF 21 : 2010 reference method (r = 1 g/l - Sr = 0.36 g/l).

The relative standard deviations of repeatability are also in accordance with the specifications (limit at 0.5 % for all the parameters) of the manufacturer.

	n	min (g/l)	max (g/l)	Y (g/l)	Sy (g/l)	d (g/l)	Sd (g/l)	Sy,x (g/l)	Sy,x (%)	b	a
Fat	40	37.10	41.10	39.223	0.907	0.427	0.232	0.235	0.60	1.019	-1.17
Protein	40	31.18	33.24	32.270	0.581	0.007	0.107	0.108	0.34	0.997	0.10
Dry matter	40 (39)	127.00 (127.00)	131.33 (131.12)	129.255 (129.202)	1.065 (1.024)	-0.297 (-0.268)	0.398 (0.357)	0.403 (0.359)	0.31 (0.28)	0.982 (0.951)	2.67 (6.32)

<u>Table 2</u>: DairySpec FT accuracy criteria for fat, protein and dry matter in tank milk samples. For fat and dry matter, the reference results obtained in m/m units have been converted in m/v by the 1.03 g/ml coefficient.

n, min, max: number of results, minimum and maximum values; Y,X: mean results using the reference and instrumental method; Sy: standard deviation of the results from the reference method; d, Sd: mean and standard deviation of deviations; Sy,x: residual standard deviation; b, a: slope and intercept of the linear regression.



ARTICLE 33,50 DAIRYSPEC FT MILK - ACCURACY **PROTEIN** 33.00 REFERENCE (g/l) 32.50 y = 0.9967x + 0.098732,00 31,50 31,00 33.00 31.00 31,50 32.00 32,50 33,50 APPAREIL (g/l) 131.50 DAIRYSPEC FT MILK – ACCURACY 131,00 DRY MATTER 130,50 130.00 REFERENCE (g/l) 129,50 129,00 y = 0.953x + 6.3237128,50 128,00 127,50

Figures 1, 2 and 3: Relation between DairySpec FT and reference for fat, protein and dry matter in tank milk samples.

APPAREIL (g/l)

129,00

129,50

130,00

130,50

131,00

128,50

It can be noted that:

127,00 126,50

126,50

127,00

127,50

128,00

 $rac{1}{2}$ For fat: the mean and the standard deviation of deviations are respectively equal to 0.43 and 0.23 g/l. The regression slope (1.019) is not significantly different from 1.00 (P = 5 %). The residual standard deviation of regression is equal to 0.24 g/l.

 $\$ For protein: the mean and the standard deviation of deviations are respectively equal to 0.01 and 0.11 g/l. The regression slope (0.997) is not significantly different from 1.00 (P = 5 %). The residual standard devaition of regression is equal to 0.11 g/l.

 $rac{l}{l}$ For dry matter: the mean and the standard deviation of deviations are respectively equal to -0.27 and 0.36 g/l. The regression slope (0.953) is not significantly different from 1.00 (P = 5 %). The residual standard devaition of regression is equal to 0.36 g/l.

For fat and protein, the residual standard deviations (respectively 0.24 and 0.11 g/l) are in accordance with the recommendations of the ISO 8196-3/IDF 129-3 standard (limit: 0.7 g/l).

For dry matter, as no standard exists, the residual standard deviation obtained (0.36 g/l) enables an accuracy of estimation of ± 0.72 g/l (risk at 5%).

The relative residual standard deviations are also in accordance with the specifications (limit at 1 % for all the parameters) of the manufacturer.

B.2.2 – Crème

The following tables and figures present the results obtained:

	n	min	max	M	Sx	Sr	Sr (%)	r
Fat (g/100 g)	20	26.4	44.8	40.182	5.242	0.038	0.09	0.104
Dry matter (g/100 g)	20	33.5	51.0	46.529	4.971	0.042	0.09	0.117

Table 3: DairySpec FT repeatability criteria for fat, protein and dry matter in cream samples

n: number of results; min and max: minimum and maximum values; M and Sx: mean and standard deviations of the results; Sr and Sr%: absolute and relative standard deviation of repeatability; r: maximum deviation of repeatability in 95 % of cases.

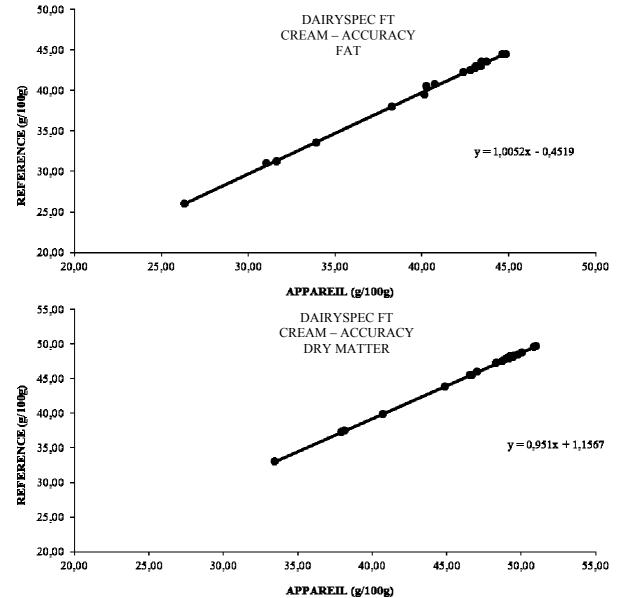
As no standardised or regulatory values exist for fat and dry matter, it can be noted that the standard deviations of repeatability are lower than the limits of the NF V 04-263: 1997 and ISO 6731/FIL 21: 2010 reference methods (respectively 0.14 g/100 g and 0.07 g/100 g).

The relative standard deviation of repeatability are also in accordance with the specifications (limit at 0.5 % for all the parameters) of the manufacturer.

	n	min (g/100g)	max (g/100g)	Y (g/100g)	Sy (g/100g)	d (g/100g)	Sd (g/100g)	Sy,x (g/100g)	Sy,x (%)	b	a
Fat	20	26.0	44.5	39.938	5.273	0.245	0.197	0.201	0.50	1.005	-0.45
Dry matter	20	33.0	49.7	45.404	4.728	1.125	0.256	0.079	0.17	0.951	1.16

Table 4: DairySpec FT accuracy criteria for fat and dry matter in cream samples

n, min, max: number of results, minimum and maximum values; Y,X: mean results using the reference and instrumental method; Sy: standard deviation of the results from the reference method; d, Sd: mean and standard deviation of deviations; Sy,x: residual standard deviation; b, a: slope and intercept of the linear regression.



Figures 4 and 5: Relation between DairySpec FT and reference results for fat and dry matter in cream samples

It can be noted that:

 $rac{1}{2}$ For fat: the mean and the standard deviation of deviations are respectively equal to 0.25 and 0.20 g/100 g. The regression slope (1.005) is not significantly different from 1.00 (P = 5 %). The residual standard deviation of regression is equal to 0.20 g/l.

 $rac{4}{5}$ For dry matter: the mean and the standard deviation of deviations are respectively equal to 1.13 and 0.26 g/100 g. The regression slope (0.951) is significantly different from 1.00 (P = 1 %). The residual standard deviation of regression is equal to 0.08 g/100 g.

As no standardised or regulatory values exist for fat and dry matter, it can be noted that the residual standard deviations obtained (respectively 0.20 and 0.08 g/100 g) enables accuracies of estimation respectively equal to 0.40 and 0.16 g/100 g (risk at 5 %).

The relative residual standard deviations obtained are in accordance with the specifications (limit at 1 % for all the parameters) of the manufacturer.

B.2.3 – Retentate

The following tables and figures present the results obtained:

	n	min	max	M	Sx	Sr	Sr (%)	r
Protein (g/l)	25	87,3	110,1	98,353	7,002	0,111	0,11	0,309
Dry matter (g/l)	25	145,9	169,1	157,157	6,805	0,145	0,09	0,401

<u>Table 5</u>: DairySpec FT repeatability criteria for protein and dry matter in proteic retentate samples n: number of results; min and max: minimum and maximum values; M and Sx: mean and standard deviation of the results; Sr and Sr %: absolute and relative standard deviation of repeatability; r: maximum deviation of repeatability in 95 % of cases.

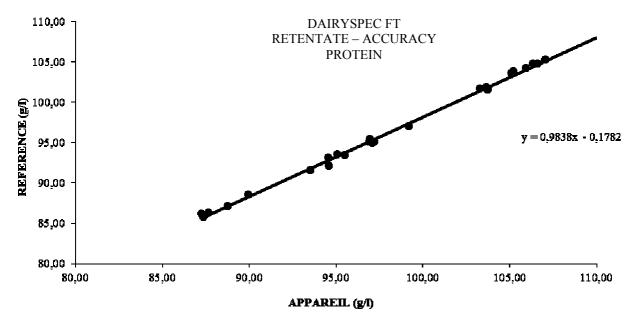
As no standards exist for protein and dry matter, it can be noted that the standard deviations of repeatability obtained are lower or close to the NF V 04-216: 2011 et ISO 6731/IDF 21: 2010 reference methods limits (respectively 0.37 g/l and 0.11 g/l).

The relative standard deviation of repeatability are also in accordance with the specifications (limit at 0.5 % for all the parameters) of the manufacturer.

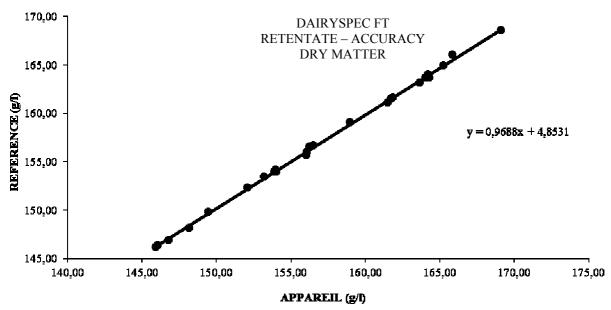
		n	min (g/l)	max (g/l)	Y (g/l)	Sy (g/l)	d (g/l)	Sd (g/l)	Sy,x (g/l)	Sy,x (%)	b	a
I	MP	25	85.79	107.56	96.581	6.897	1.772	0.356	0.345	0.36	0.984	-0.18
I	MS	25	146.2	168.6	157.099	6.595	0.058	0.295	0.209	0.13	0.969	4.85

<u>Table 6</u>: DairySpec FT accuracy criteria for protein and dry matter in proteic retentate

n, min, max: number of results, minimum and maximum values; Y,X: mean results using the reference and instrumental method; Sy: standard deviation of the results from the reference method; d, Sd: mean and standard deviation of deviations; Sy,x: residual standard deviation; b, a: slope and intercept of the linear regression.



ARTICLE



<u>Figures 6 and 7</u>: Relation between DairySpec FT and reference results for protein and dry matter in proteic retentate

It can be noted that:

 $rac{4}{5}$ For protein: the mean and the standard deviation of deviations are respectively equal to 1.77 and 0.36 g/l. The regression slope (0.984) is not significantly different from 1.00 (P = 5 %). The residual standard deviation of regression is equal to 0.35 g/l.

 $rac{4}{5}$ For dry matter: the mean and the standard deviation of deviations are respectively equal to 0.,06 and 0.30 g/l. The regression slope (0.969) is significantly different from 1.00 (P = 5 %). The residual standard deviation of regression is equal to 0.21 g/l.

As no standardised or regulatory values exist for fat and dry matter, it can be noted that the residual standard deviations obtained (respectively 0.36 and 0.21 g/l) enables accuracies of estimation respectively equal to ± 0.72 and 0.42 g/l (risk at 5 %).

The relative residual standard deviations obtained are in accordance with the specifications (limit at 1 % for all the parameters) of the manufacturer.

CONCLUSION GENERALE

The results obtained for fat and protein in milk are in accordance with the recommendations of the ISO 8196-3/IDF 129-3 standard and the CNIEL/IE PROC IR v06 handbook, within the context of milk payment and milk control in France.

Despite the absence of standard criteria for the other parameters (dry matter in milk, fat and dry matter in cream, protein and dry matter in retentate), the results obtained are in conformity with the specifications of the manufacturer.

STANDARDS, DRAFT STANDARDS

ISO standards under development

FERMENTED MILK PRO	DUCTS
ISO/DIS 19344	FERMENTED MILK PRODUCTS
December 2014	Quantification of lactic acid bacteria – Flow cytometry method
MICROBIOLOGY OF TH	E FOOD CHAIN
ISO/DIS 11290-1	MICROBIOLOGY OF THE FOOD CHAIN
March 2015	Horizontal method for the detection and enumeration of <i>Listeria monocytogenes</i> and other <i>Listeria</i> spp Part 1: Detection method
ISO/DIS 11290-2	MICROBIOLOGY OF THE FOOD CHAIN
March 2015	Horizontal method for the detection and enumeration of <i>Listeria monocytogenes</i> and other <i>Listeria</i> spp Part 2: Enumeration method
ISO/DIS 18465	MICROBIOLOGY OF THE FOOD CHAIN
March 2015	Quantitative analysis of emetic toxin (cereulide) using LC-MS/MS
MILK AND CHEESE	
ISO/DIS 27105	MILK AND CHEESE
January 2015	Determination of hen's egg white lysozyme content by high performance liquid chromatography
MILK AND DAIRY PROD	UCTS
ISO/DIS 11816-2	MILK AND DAIRY PRODUCTS
December 2014	Determination of alkaline phosphatase activity - Part 2: Fluorometric method for cheese
QUALITY	
ISO/DIS 9000	QUALITY MANAGEMENT SYSTEMS
October 2014	Fundamentals and vocabulary
SENSORY ANALYSIS	
ISO/DIS 5492/A1	SENSORY ANALYSIS
February 2015	Vocabulary – Amendment 1
ISO/DIS 11056/A2	SENSORY ANALYSIS
December 2014	Methodology – Magnitude estimation method – Amendment 2
ISO/DIS 13299	SENSORY ANALYSIS
February 2015	Methodology – General guidance for establishing a sensory profile

ISO published standards

ANIMAL AND VEGETABLE FATS AND OILS			
ISO 12228-1 July 2014	Determination of individual and total sterols contents - Gas chromatographic method - Part 1: Animal and vegetable fats and oils <i>Revises ISO 12228:1999</i>		
ISO/TS 17383 September 2014	Determination of the triacylglycerol composition of fats and oils - Determination by capillary gas chromatography		
ISO 5555/A1 July 2014	ANIMAL AND VEGETABLE FATS AND OILS Sampling – Amendment 1: Flexitanks		
MICROBIOLOGY			
ISO/TR 6579-3 July 2014	MICROBIOLOGY OF THE FOOD CHAIN Horizontal method for the detection, enumeration and serotyping of <i>Salmonella</i> - Part 3: Guidelines for serotyping of <i>Salmonella</i> spp.		

FANDARDS - REGULATIONS

QUALITY				
ISO 10002 July 2014	QUALITY MANAGEMENT Customer satisfaction - Guidelines for complaints handling in organizations Revises ISO 10002:2004 and ISO 10002:2004/Cor 1:2009			
ISO 22004 September 2014	FOOD SAFETY MANAGEMENT SYSTEMS Guidance on the application of ISO 22000 Revises ISO/TS 22004: 2005			

NEW EU REGULATIONS

Classification is established in alphabetical order of the first keyword

CONTAMINANTS

O.J.E.U. L 271, 12th September 2014 – Commission Recommendation of 10 September 2014 on the monitoring of the presence of 2 and 3-monochloropropane-1,2-diol (2 and 3-MCPD), 2- and 3-MCPD fatty acid esters and glycidyl fatty acid esters in food

http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=urisery:OJ.L .2014.271.01.0093.01.ENG

FOOD ADDITIVES / FLAVOURINGS

O.J.E.U. L 252, 26th August 2014 - Commission Regulation (EU) No 923/2014 of 25 August 2014 amending Annex II to Regulation (EC) No 1333/2008 of the European Parliament and of the Council as regards the use of aluminium lakes of riboflavins (E 101) and cochineal, carminic acid, carmines (E 120) in certain food categories and Annex to Regulation (EU) No 231/2012 as regards the specifications for riboflavins (E 101)

http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=urisery:OJ.L .2014.252.01.0011.01.ENG

O.J.E.U. L 272, 13rd September 2014 – Commission Regulation (EU) No 966/2014 of 12 September 2014 amending Annex to Regulation (EU) No 231/2012 laying down specifications for food additives listed in Annexes II and III to Regulation (EC) No 1333/2008 of the European Parliament and of the Council as regards specifications for calcium propionate

http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L .2014.272.01.0001.01.ENG

O.J.E.U. L 299, 17th October 2014 – Commission Regulation (EU) No 1093/2014 of 16 October 2014 amending and correcting Annex II to Regulation (EC) No 1333/2008 of the European Parliament and of the Council as regards the use of certain colours in flavoured ripened cheese

http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=urisery:OJ.L .2014.272.01.0001.01.ENG

O.J.E.U. L 300, 18th October 2014 – Commission Regulation (EU) No 1098/2014 of 17 October 2014 amending Annex I to Regulation (EC) No 1334/2008 of the European Parliament and of the Council as regards certain flavouring substances

http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L .2014.300.01.0041.01.ENG

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

O.J.E.U. L 234, 7th August 2014 – Commission Implementing Regulation (EU) No 855/2014 of 4 August 2014 approving minor amendments to the specification for a name entered in the register of protected designations of origin and protected geographical indications [Robiola di Roccaverano (PDO) (cheese)]

http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L .2014.098.01.0007.01.ENG

O.J.E.U. C 260, 9th August 2014 - 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 [Beaufort (PDO) (cheese)]

http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.C .2014.260.01.0024.01.ENG

O.J.E.U. C 260, 15th August 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 [Klenovecky Syrec (PGI) (cheese)]
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.C_.2014.269.01.0002.01.ENG

O.J.E.U. L **258**, **29**th **August 2014** – Commission Implementing Regulation (EU) No 928/2014 of 27 August 2014 approving non-minor amendments to the specification for a name entered in the register of protected designations of origin and protected geographical indications [Crottin de Chavignol / Chavignol (PDO) (cheese)]

http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L .2014.258.01.0001.01.ENG

O.J.E.U. L 268, 9th September 2014 – Commission Implementing Regulation (EU) No 954/2014 of 4 September 2014 approving non-minor amendments to the specification for a name entered in the register of protected designations of origin and protected geographical indications [Livarot (PDO) (cheese)]

http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2014.268.01.0001.01.ENG

O.J.E.U. L 271, 12th September 2014 – Commission Implementing Regulation (EU) No 963/2014 of 29 August 2014 entering a name in the register of protected designations of origin and protected geographical indications [Zazrivské vojky (PGI) (cheese)]

http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2014.271.01.0015.01.ENG

O.J.E.U. C **310, 12th September 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 [Toma Piemontese (PDO) (cheese)]

http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=urisery:OJ.C .2014.310.01.0004.01.ENG

O.J.E.U. C 316, 16th September 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 [Brocciu corse / Brocciu (PDO) (cheese)]

http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.C _.2014.316.01.0006.01.ENG

O.J.E.U. C **319, 17th September 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 [Ossau-Iraty (PDO) (cheese)]

http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.C .2014.319.01.0028.01.ENG

O.J.E.U. C 336, 26th September 2014 – Publication of a registration 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 delle Balze Volterrane (PDO) (cheese)]

http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.C .2014.336.01.0023.01.ENG

O.J.E.U. C **340, 30th September 2014** – Publication of a registration 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 [Heumilch / Haymilk / Latte fieno / Lait de foin / Leche de heno (STG) (milk)]

http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.C .2014.340.01.0006.01.ENG

O.J.E.U. C 356, 9th October 2014 – 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 [Comté (PDO) (cheese)]

http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.C .2014.356.01.0054.01.ENG

O.J.E.U. C **364, 15th October 2014** – Publication of a registration 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 [Weisslacker / Allgäuer weisslacker (PDO) (cheese)]

http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.C_.2014.364.01.0043.01.ENG

O.J.E.U. C 387, 1st November 2014 – 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 [Reblochon / Reblochon de Savoie (PDO) (cheese)]

http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.C_.2014.387.01.0017.01.ENG

O.J.E.U. L 318, 5th November 2014 – Commission Implementing Regulation (EU) No 1190/2014 of 24 October 2014 approving non-minor amendments to the specification for a name entered in the register of protected designations of origin and protected geographical indications (Mantequilla de Soria (PDO) (beurre)]

http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L .2014.318.01.0004.01.ENG

PESTICIDES

O.J.E.U. L 300, 18th October 2014 – Commission Regulation (EU) No 1096/2014 of 15 October 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 carbaryl, procymidone and profenofos in or on certain products

http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2014.300.01.0005.01.ENG

STANDARDS - REGULATIONS

O.J.E.U. L 304, 23rd October 2014 – Commission Regulation (EU) No 1119/2014 of 16 October 2014 amending Annex III to Regulation (EC) No 396/2005 of the European Parliament and of the Council as regards maximum residue levels for benzalkonium chloride and didecyldimethylammonium chloride in or on certain products http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L .2014.304.01.0043.01.ENG

O.J.E.U. L 305, 24th October 2014 – Commission Regulation (EU) No 1126/2014 of 17 October 2014 amending Annex II, III and V to Regulation (EC) No 396/2005 of the European Parliament and of the Council as regards maximum residue levels for asulam, cyanamide, dicloran, flumioxazin, flupyrsulfuron-methyl, picolinafen and propisochlor in or on certain products

http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L .2014.305.01.0003.01.ENG

O.J.E.U. L 305, 24th October 2014 – Commission Regulation (EU) No 1127/2014 of 20 October 2014 amending Annex II and III to Regulation (EC) No 396/2005 of the European Parliament and of the Council as regards maximum residue levels for amitrole, dinocap, fipronil, flufenacet, pendi-methalin, propyzamide, and pyridate in or on certain products

http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2014.305.01.0047.01.ENG

O.J.E.U. L 308, 29th October 2014 – Commission Regulation (EU) No 1146/2014 of 23 October 2014 amending Annex 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 anthraquinone, benfluralin, bentazone, bromoxynil, chlorothalonil, famoxadone, imazamox, methyl bromide, propanil and sulphuric acid in or on certain products

http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L .2014.308.01.0003.01.ENG

AFNOR VALIDATIONS

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

Commercial name	Date	Certificate	Description			
NEW VALIDATION						
D-COUNT TVC	Validation date: 2 Oct 2014 End of validity: 2 Oct 2018	BIO-12/36-10/14	Enumeration of mesophilic aerobic flora UHT milk, pasteurised dairy based desserts and meat products			
RENEWALS OF VALIDATIONS						
ТЕМРО ЕВ	Validation date: 14 Dec 2006 Extension: 3 Feb 2011 Renewal: 2 Dec 2010 and 2 Oct 2014 End of validity: 14 Dec 2018	BIO-12/21-12/06	Enumeration of Enterobacteriaceae All human and animal food products (except beverages and cattle food)			
GENEDISC <i>LISTERIA</i> SPP.	Validation date: 2 Jul 2010 Renewal: 2 Oct 2014 End of validity: 2 Jul 2018	GEN-25/07-07/10	Detection of <i>Listeria</i> spp. All human food products and environmental samples			
GENEDISC LISTERIA MONOCYTOGENES	Validation date: 2 Jul 2010 Renewal: 2 Oct 2014 End of validity: 2 Jul 2018	GEN-25/08-07/10	Detection of <i>Listeria monocytogenes</i> All human food products and environmental samples			
HQS CRONOBACTER SPP SYBR	Validation date: 2 Apr 2010 Renewal: 3 Oct 2014 End of validity: 2 Apr 2018	ADN-33/02-04/10	Detection of <i>Cronobacter</i> spp. 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

BOOKSHOP - FORTHCOMING EVENTS - IN THE PRESS-ON THE WEB

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.

MICROBIOLOGY

OZER B., AKDEMIR-EVRENDILEK G. – **Dairy microbiology and biochemistry: Recent developments** –CRC Presss Editions – July 2014 – ISBN: 9781482235029 – 464 pages

http://www.crcpress.com/product/isbn/9781482235029



This book reviews the last developments about types, classifications, genetic characteristics, dairy starter cultures of milk and dairy products. Moreover, it provides strategies for the rapid detection of pathogenic and non-pathogenic organisms in milk and dairy products.

IN THE PRESS – ON THE WEB

Classification in alphabetical order of keywords

ADDITIVES / FOOD INGREDIENTS

Scientific opinion on the re-evaluation of propionic acid (E 280), sodium propionate (E 281), calcium propionate (E 282) and potassium propionate (E 283) as food additives

 $\underline{http://www.efsa.europa.eu/en/efsajournal/pub/3779.htm}$

▶ The Panel concluded that the use of these substances as additives would not be of safety concern at their currently authorised uses and use levels.

Scientific opinion on the safety of astaxanthin-rich ingredients (AstaREAL A1010 and Asta REAL L10) as novel food ingredients

 $\underline{http://www.efsa.europa.eu/en/efsajournal/pub/3757.htm}$

▶ The Panel concluded that the safety of the AstaREAL A1010 et AstaREAL L10 as novel food ingredients at the proposed use and use levels has not been established.

STANDARDISATION

Report of the 37th session of the Codex Alimentarius Commission

http://www.codexalimentarius.org/download/report/807/REP14_CACe.pdf

▶ This report presents the 37th session of the Codex Alimentarius Commission held from 14-18 July 2014 in Geneva, Switzerland. This document presents the list of the amendments, the adopted standard (among which the standard about the maximum level for lead in infant formula and formula for special medical purposes intended for infants and for follow up formula), the draft standards adopted at step 5, the revoked standards and the approved new work (among which a standard for the processed cheese).

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