CECALAIT-S NEWSLETTER

ACTALIA
CECALAIT

3rd quarter 2013, No. 86

ACTALIA Cecalait

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

ARTICLE

EVALUATION OF THE DELTA INSTRUMENT FTIR 600 LACTOSCOPE INFRARED ANALYSER

The Lactoscope is an infrared spectrophotometer (range µm) used for the determination of the principal components in milk. It is manufactured by Delta Instruments (Advanced group, Netherlands) and commercialised in France by Humeau.

This instrument uses a high resolution industrial infrared spectrophotometer based on Fourier transform (FTIR). The complete infrared spectrum (resolution 8 cm-1) is collected and recorded for each sample. With a MLR or PLS calibration, various criteria (fat, protein, lactose, dry matter, urea, freezing point…) can be determined.

This apparatus, which is connected to a computer that ensures the signal treatment, can be associated to the "Somascope" somatic cells counter to constitute the "Combiscope".

The evaluation tests were performed in ACTALIA Cecalait physico-chemistry laboratory (reference and infrared analyses) from February to June 2013. After preliminary tests of stability of the instrument, contamination between samples, linearity and calibration, the repeatability and accuracy were evaluated for fat (equivalent fat filter B), protein, dry matter, urea and freezing point in cow, goat and ewe milk.

A cleaning solution (aqueous solution of Decon® at 4 %) and a zero solution (aqueous solution of triton X100® at 0,1 %) were necessary for theses tests.

The apparatus was configured for a rate of 600 samples per hour and no correction of contamination.

The appreciation criteria of the estimated parameters were taken from ISO 8196-3 / IDF 128-3:2010, or from the CNIEL handbooks concerning the use of infrared apparatus with the context of milk payment and milk control in France (CNIEL PROC IR 06 et CNIEL PROC CR IR 04).

A. PRELIMINARY TESTS

A.1. Evaluation of the stability

The stability was evaluated by the analysis, in automatic mode, of milk every 20 minutes, representing 20 measurement cycles. To evaluate the stability of the instrument, the repeatability and reproducibility were calculated for each analytical criterion and by level..

The values of standard deviation of reproducibility for fat, protein and urea were below to the limits required in ISO 8196-3 / IDF 128-3 (respectively 0.29, 0.58 g/l and 29 mg/l for the median and high values). As no standardised values exist for freezing point, it can be noted that the reproducibility standard deviation values are lower than the limit value of the CNIEL PROC CR IR 04 handbook (R = 10 m °C → SR lower than 3.6 m °C).

A.2. Evaluation of contamination between samples

This criterion was evaluated in automatic analysis mode, by analysing the same cow milk and distilled water according to the sequence: MILK - MILK – WATER – WATER repeated twenty times for fat, protein and freezing point. The evaluation was carried out on 4 levels for fat, protein and protein.

The contamination level was estimated by the formula: Tc (%) = [(Σ(Eau 1) - Σ(Eau 2)) / (Σ(Lait 2) - Σ(Eau2))] x 100

The contamination rates for fat and protein between successive samples are lower than the maximal limit at 1 % required in the ISO 8196-3 / IDF 128-3 standard and in the CNIEL PROC IR 06 handbook.
The value obtained for freezing point are also lower than the maximal limit at 2 % of the CNIEL PROC IR 06 handbook.

A.3. 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 11 milk samples from 0 to 120 g/l was prepared by mixing cream and skimmed milk. Within this range, the Ar/At ratio (Ar and At: amplitude of residues and amplitude of content respectively) is equal to 2.01 %, that corresponds to the limit of 2 % expressed in ISO 8196-3 / IDF 128-3 standard. A linear regression in the range from 0 to about 100 g/l improves the linearity of the instrument (Ar/At ratio equal to 1 %). A Ar/At ratio equal to 0.34 % is obtained with a linear regression in the range from 20 to about 60 g/l, corresponding to the cow milk.

For protein, a range of 11 milk samples from 0 to 80 g/l was prepared by mixing the proteic retentate and filtrate obtained by tangantial ultrafiltration (cutoff threshold: 10 KD). Each range was analysed three times. The Ar/At ratio within this range studied is equal to 1 %, which is in conformity with the recommendations of 2 % maximum given in ISO 8196-3 / IDF 128-3 standard. A Ar/At ratio equal to 0.53 % is obtained with a linear regression in the range from 20 to about 40 g/l, corresponding to the cow milk.

The linearity of the instrument is therefore satisfactory for fat (range from 0 to 100 g/l)and protein (range from 0 to 80 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 April 2013. Each sample was analysed in duplicate.

Concerning the median range, the residual standard deviations of the linear regression for fat and protein are closed to the standard deviation of deviations. For fat, an optimisation of the lactose residual interaction enables the reduction of this value (SL3 equal to 0.12 g/l).
Concerning the high range, the residual standard deviations for fat is lower than the standard deviation of deviations. As for the median range, it can be optimised. For protein, the mean bias is high.

To conclude, concerning the median range, the mean bias and the regression slope for fat and protein are in accordance with the recommendations of the ISO 8196-3 / IDF 128-3 standard.(respectively 0.5 g/l and 1+/-0.05). Concerning the high range, the mean bias (absolute and relative) and the regression slope for fat are in conformity with the recommendations of the ISO 8196-3 / IDF 128-3 standard (respectively 1 g/l, 1.25 % and 1+/-0.05). The mean bias (absolute and relative) for protein are higher than the standardised limits (1 g/l and 1.5 %), probably due to a preliminary defect of calibration on zone milk.
On the other hand, all the residual standard deviations of linear regression obtained for fat and protein are in accordance with the recommendations of the CNIEL PROC IR 06 handbook (respectively lower than 0.25 and 0.15 g/l for the median range and 0.50 and 0.30 g/l for the high range).

B. EVALUATION OF REPEATABILITY AND ACCURACY

B.1. The samples

The tests were performed on:
- for the cow milk: 100 samples of herd milk from the Franche-Comté region and 130 samples of individual milk from 4 farms in the Jura. Bronopol was added to the individual milk samples to give a final concentration of 0.02 %.
- for the goat milk: 88 samples of herd milk from the Poitou-Charentes region.
- for the ewe milk: 100 samples of herd milk from the Roquefort sur Soulzon region.

B.2. Procedure

The repeatability and accuracy of the instrument were evaluated using all the samples for fat, protein and only using the herd cow milk samples for freezing point. In addition; dry matter and urea were respectively measured using herd cow milk samples and individual cow milk samples. The quantitative analyses were performed in automatic analysis mode, in duplicate for each set of 20 samples according to the following sequence: Set 1 rep 1 - Set 1 rep 2 - Set 2 rep 1 - Set 2 rep 2 … Set n rep 1 - Set n rep 2. A control milk was analysed every 20 samples to verify the stability of the analyser. For fat and protein, the evaluation concerns the values obtained after calibration of the instrument with commercial SRMs produced by ACTALIA Cecalait. For freezing point, the instrumental values are from a calibration carried out by the manufacturer.

The following reference methods were used:
- Fat: Gerber acido-butyrometric method according to NF V 04-210: 2000 (single test and then confirmation if more important residues for the individual milk samples) for cow and goat milk samples, and acido-butyrometric method according to NF V 04-155: 2003 (single test) for ewe milk samples,
- Protein: Amido black method according to NF V 04-216: 2011 (test in duplicate),
- Freezing point: thermistor cryoscopic method according to ISO 5764 / IDF 108: 2009 (single test),
- Dry matter: drying method according to ISO 6731 / IDF 21 : 2011 (single test),
- Urea: differential pH-metry method according to ISO 14637 / IDF 195: 2007 (single test).

B.3. Results

B.3.1. Cow milk

B.3.1.1. Herd milk

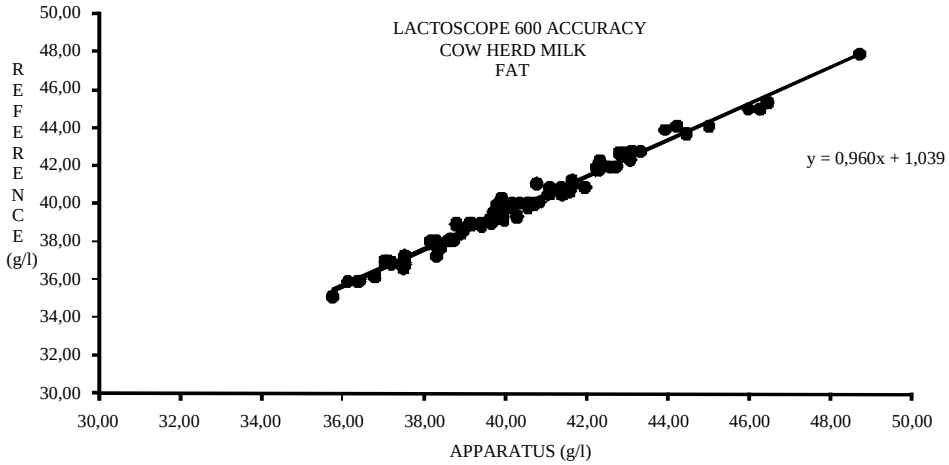
The following tables and figures present the results obtained:

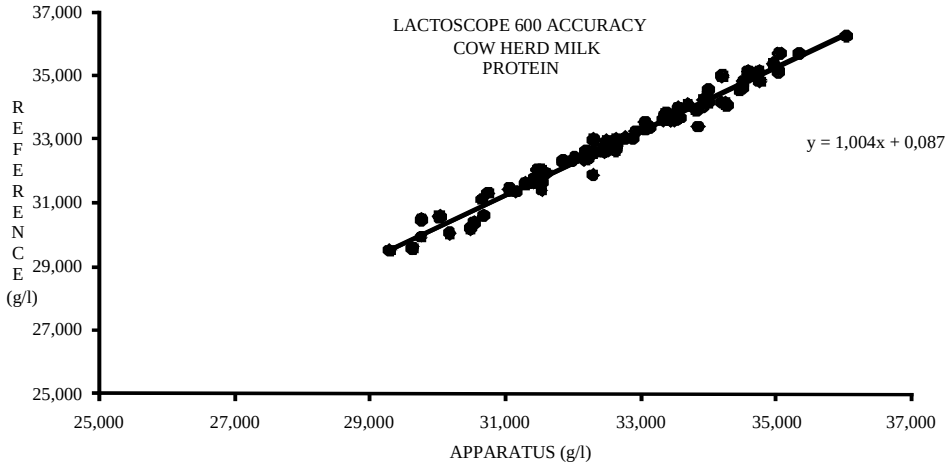
	n	Min	max	M	Sx	Sr	Sr (%)	r
Fat (g/l)	100	35.79	48.75	40.594	2.398	0.055	0.14%	0.150
Protein (g/l)	100	29.32	36.06	32.846	1.502	0.061	0.19%	0.166
Freezing point (m°C x -1)	100	513	534	525.5	3.6	1.1	0.21%	3.1
Dry matter (g/100g)	100	11.98	13.48	12.717	0.327	0.019	0.15%	0.053

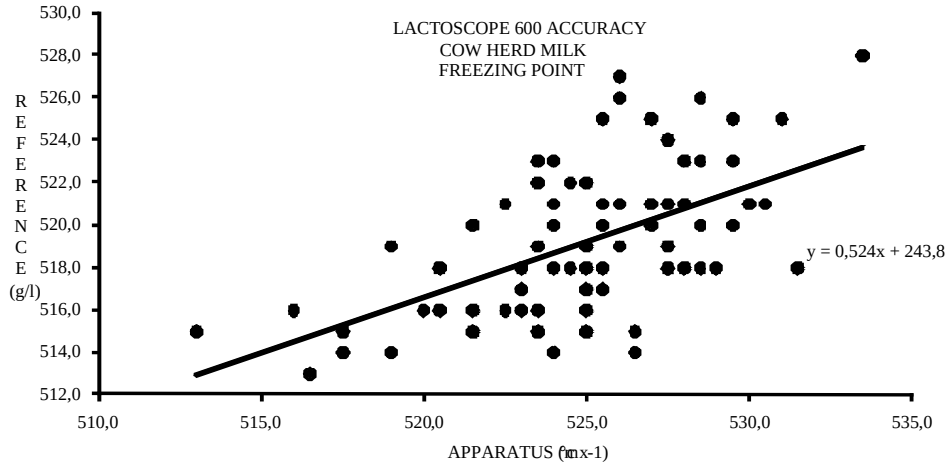
Table 1: Lactoscope repeatability criteria for fat, protein, freezing point and dry matter in herd cow milk samples
n: number of results; min and max: minimum and maximum values; M and Sx: mean and standard deviation of the results; Sr and Sr %: absolute an relative standard deviation of repeatability; r: maximum deviation of repeatability on 95 % of cases

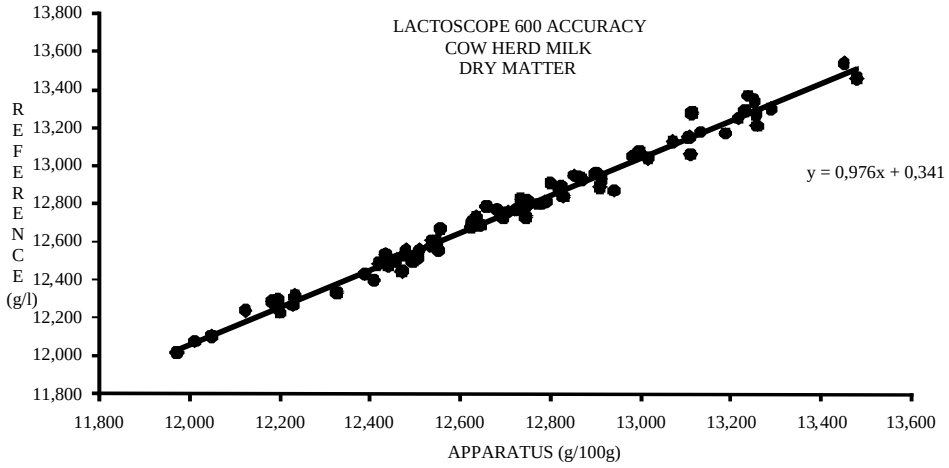
	n	min	max	Y	Sy	d	Sd	Sy,x	Sy,x (%)
Fat (g/l)	79	35.10	47.80	40.054	2.482	0.547	0.321	0.307	0.76
Protein (g/l)	82	29.54	36.28	33.059	1.543	-0.245	0.230	0.231	0.70
Freezing point (m°C x -1)	84	513	528	519.3	3.4	5.8	3.3	2.8	0.53
Dry matter (g/100g)	79	12.02	13.54	12.762	0.339	-0.048	0.043	0.043	0.34

Table 2: Lactoscope accuracy criteria for fat, protein, freezing point and dry matter in herd cow milk samples
n, min, max: number of results, minimum and maximum values; Y: mean results using the reference method; Sy: standard deviation of the results from the reference method; d, Sd: mean and standard deviation of deviations; Sy,x: residual standard deviation.









Figures 1 to 4: Relation between Lactoscope and reference results for fat, protein, freezing point and dry matter in herd cow milk

It can be noted that:

↳ For fat: the mean and the standard deviation of deviations are respectively equal to 0.547 and 0.321 g/l. The regression slope (0.960) and the intercept (1.039) are significantly different from respectively 1.00 and zero (P = 1 %). The residual standard deviation of regression (0.307 g/l) is in conformity with the recommendations of the ISO 8196-3 / IDF 128-3 standard (Sy,x ≤ 0,72 g/l).

↳ For protein: the mean and the standard deviation of deviations are respectively equal to –0.245 and 0.230 g/l. The regression slope (1.004) and the intercept (0.087) are not significantly different from respectively 1.00 and zero (P = 5 %). The residual standard deviation of regression (0.231 g/l) is in conformity with the recommendations of the ISO 8196-3 / IDF 128-3 standard (Sy,x ≤ 0,72 g/l).

↳ For freezing point: the mean and the standard deviation of deviations are respectively equal to 5.8 and 3.3 m °C x -1. The regression slope (0.524) and the intercept (243.8) are significantly different from respectively 1.00 and zero (P = 1 %). The residual standard deviation of regression is equal to 2.8 m °C.

↳ For dry matter: the mean and the standard deviation of deviations are respectively equal to –0.048 and 0.043 g / 100g. The regression slope (0.976) is not significantly different from 1.00 (P = 5 %) and the intercept (0.341) is significantly different from zero (P = 1 %). The residual standard deviation of regression is equal to 0.043 g / 100g.

B.3.1.2. Individual milk

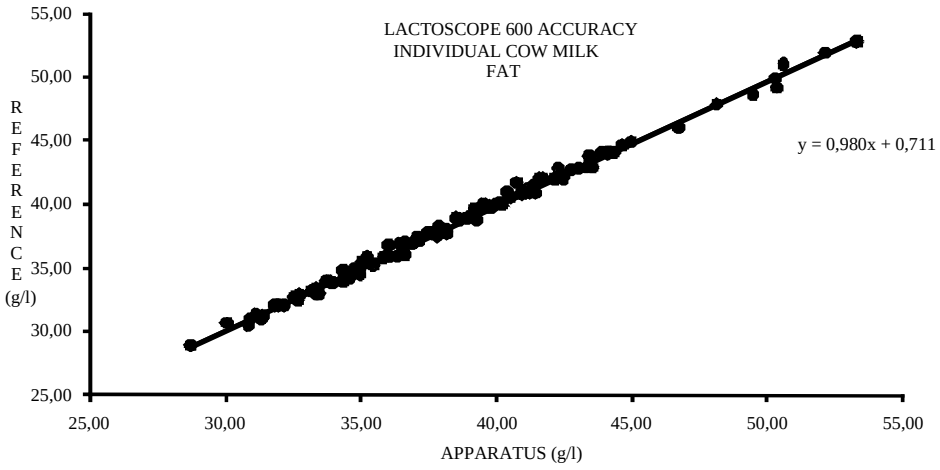
The following tables and figures present the results obtained:

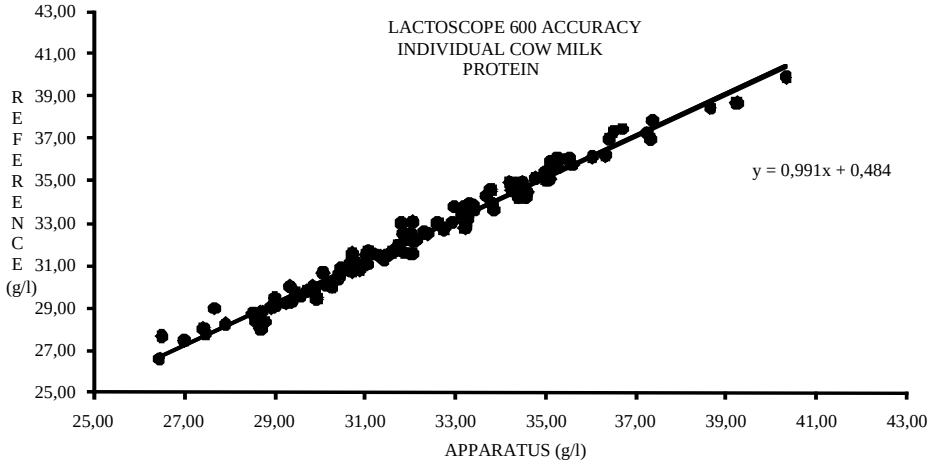
	n	Min	max	M	Sx	Sr	Sr (%)	r
Fat (g/l)	130	27.151	53.349	38.650	5.234	0.056	0.15%	0.153
Protein (g/l)	130	26.440	43.461	32.576	3.043	0.066	0.20%	0.179
Urea (mg/l)	130	125.4	483.1	285.4	64.740	14.1	4.94%	38.3

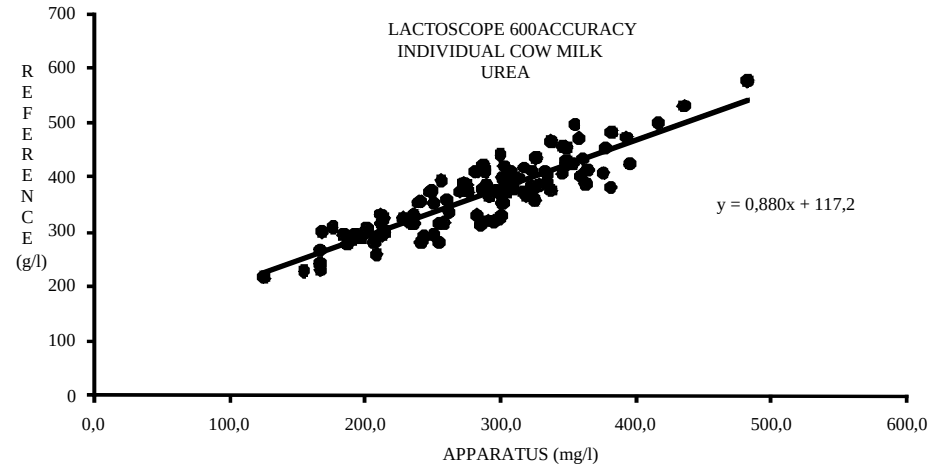
Table 3: Lactoscope repeatability criteria for fat, protein and urea in individual cow milk samples
n: number of results; min and max: minimum and maximum values; M and Sx: mean and standard deviation of the results; Sr and Sr %: absolute an relative standard deviation of repeatability; r: maximum deviation of repeatability on 95 % of cases

	n	min	max	Y	Sy	d	Sd	Sy,x	Sy,x (%)
Fat (g/l)	111	29.00	52.80	38.585	4.937	0.026	0.341	0.328	0.85
Protein (g/l)	111	26.57	39.87	32.585	2.834	-0.205	0.384	0.385	1.19
Urea (mg/l)	109	219	578	369.6	65.0	-82.9	31.3	30.4	10.60

Table 4: Lactoscope accuracy criteria for fat, protein and urea in individual cow milk samples
n, min, max: number of results, minimum and maximum values; Y: mean results using the reference method; Sy: standard deviation of the results from the reference method; d, Sd: mean and standard deviation of deviations; Sy,x: residual standard deviation.







Figures 5 to 7: Relation between Lactoscope and reference results for fat, protein, freezing point and dry matter in individual cow milk

It can be noted that:

↳ For fat: the mean and the standard deviation of deviations are respectively equal to 0.026 and 0.341 g/l. The regression slope (0.980) is significantly different from 1.00 (P = 1 %) and the intercept (0.711) is not significantly different from zero (P = 5 %). The residual standard deviation of regression (0.328 g/l) is in accordance with the recommendations of the ISO 8196-3 / IDF 128-3 standard (Sy,x ≤ 1.03 g/l).

↳ For protein: the mean and the standard deviation of deviations are respectively equal to–0.205 and 0.384 g/l. The regression slope (0.991) and the intercept (0.484) are not significantly different from respectively 1.00 and zero (P = 5 %). The residual standard deviation of regression (0.385 g/l) is in conformity with the recommendations of the ISO 8196-3 / IDF 128-3 standard (Sy,x ≤ 1.03 g/l).

↳ For urea: the mean and the standard deviation of deviations are respectively equal to –82.9 and 31.3 mg/l. The regression slope (0.880) is significantly different from 1.00 (P = 5 %) and the intercept (117.2) is not significantly different from zero (P = 5 %). The residual standard deviation of regression (30.4 mg/l) is in accordance with the recommendations of the ISO 8196-3 / IDF 128-3 standard (Sy,x ≤ 61.8 mg/l).

To conclude, for fat and protein in herd and individual cow milk, the standard deviation of repeatability are in accordance with the recommendations of the ISO 8196-3/IDF 128-3 standard and the CNIEL PROC IR 06 handbook (Sr ≤ 0,14 g/l). For freezing point, the standard deviation of repeatability obtained is in accordance with the recommendations of the CNIEL PROC CR IR 06 handbook (Sr ≤ 2 m °c). For urea, the standard deviation of repeatability is in accordance with the recommendations of the ISO 8196-3/IDF 128-3 standard (Sr ≤ 14.42 mg/l). For dry matter, as no standard exists, it can be noted that the standard deviation of repeatability obtained is lower than the limits of the ISO 6731/IDF 21 :2011 standard, which corresponds to the reference method (Sr ≤ 0,036 g/l).

The results obtained for fat, protein and urea are in accordance with the recommendations of the ISO 8196-3 / IDF 128-3. The residual standard deviation for freezing point (2.8 m °C) and dry matter (0.043 g/100g) enables respectively an accuracy of estimation of +/- 5.6 m °C and +/- 0.086 g/100g.

B.3.2. Goat milk

The following tables and figures present the results obtained:

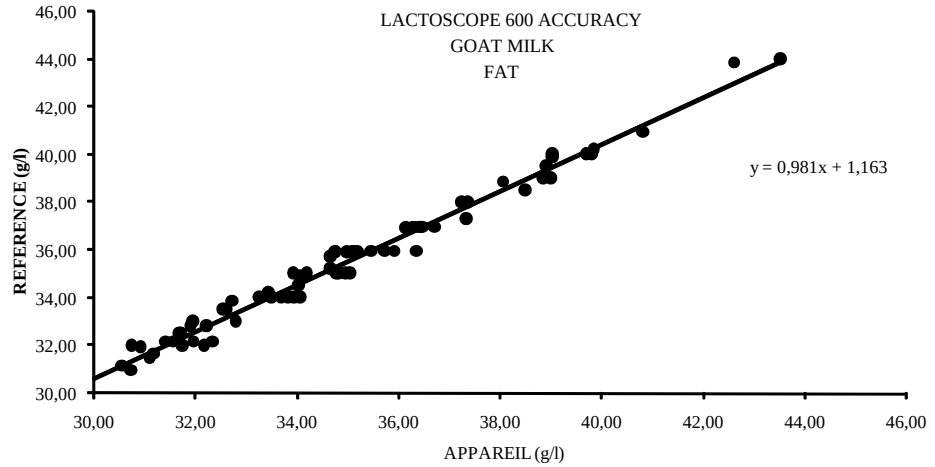
	n	Min	max	M	Sx	Sr	Sr (%)	r
Fat (g/l)	88	28.91	43.56	34.906	3.108	0.057	0.16%	0.154
Protein (g/l)	88	26.37	36.60	30.694	1.918	0.061	0.20%	0.166
Freezing point (m°C x -1)	88	547	569	557.5	4.3	1.1	0.20%	3.2

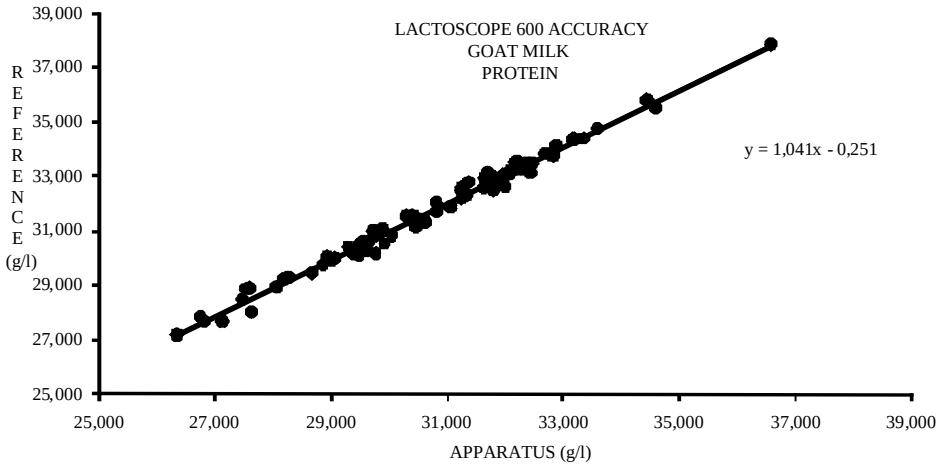
Table 5: Lactoscope repeatability criteria for fat, protein and freezing point in goat milk samples
n: number of results; min and max: minimum and maximum values; M and Sx: mean and standard deviation of the results; Sr and Sr %: absolute an relative standard deviation of repeatability; r: maximum deviation of repeatability on 95 % of cases

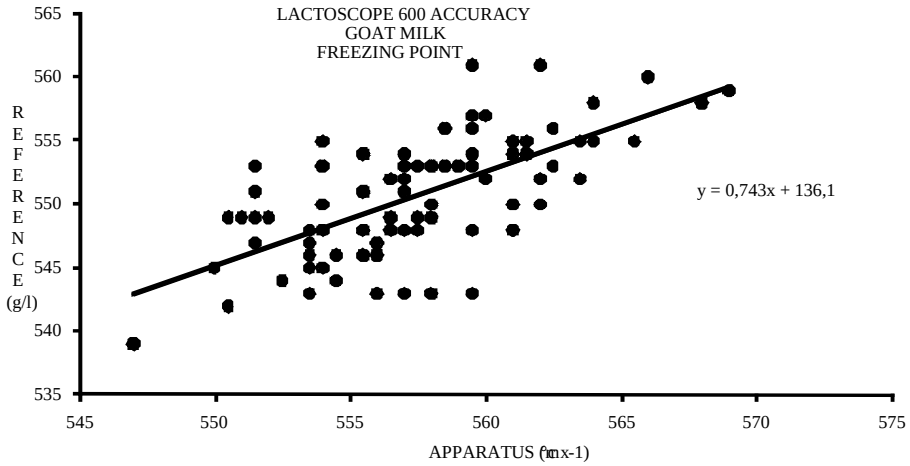
For fat and protein, the standard deviations of repeatability are in accordance with the recommendations of the ISO 8196-3 / IDF 128-3 standard and the CNIEL PROC IR 06 handbook (Sr ≤ 0.14 g/l).
For freezing point, the standard deviation of repeatability is in conformity with the recommendations of the CNIEL PROC CR IR 06 handbook (Sr ≤ 2 m°c).

	n	min	max	Y	Sy	d	Sd	Sy,x	Sy,x (%)
Fat (g/l)	74	29.90	44.00	35.481	2.961	-0.527	0.384	0.383	1.10
Protein (g/l)	84	27.19	37.86	31.753	2.044	-1.026	0.234	0.222	0.72
Freezing point (m°C x -1)	84	539	561	550.7	4.7	6.8	3.7	3.5	0.63

Table 6: Lactoscope accuracy criteria for fat, protein and freezing point in goat milk samples
n, min, max: number of results, minimum and maximum values; Y: mean results using the reference method; Sy: standard deviation of the results from the reference method; d, Sd: mean and standard deviation of deviations; Sy,x: residual standard deviation.







Figures 8 to 10: Relation between Lactoscope and reference results for fat, protein and freezing point in goat milk samples

It can be noted that:

↳ For fat: the mean and the standard deviation of deviations are equal to –0.527 and 0.384 g/l. The regression slope (0.981) is not significantly different from 1.00 (P = 5 %) and the intercept (1.163) is significantly different from zero (P = 1 %). The residual standard deviation of regression (0.383 g/l) is in accordance with the recommendations of the ISO 8196-3 / IDF 128-3 standard (Sy,x ≤ 0.72 g/l).

↳ For protein: the mean and the standard deviation of deviations are equal to –1.026 and 0.234 g/l. The regression slope (1.041) is significantly different from 1 (P = 1 %) and the intercept (-0.251) is not significantly different from zero (P = 5 %). The residual standard deviation of regression (0.222 g/l) is in accordance with the recommendations of the ISO 8196-3 / IDF 128-3 standard (Sy,x ≤ 0.72 g/l).

↳ For freezing point: the mean and the standard deviation of deviations are equal to 6.8 and 3.7 m °C x -1. The regression slope (0.743) and the intercept (136.1) are significantly respectively different from 1.00 and zero (P = 1 %). The residual standard deviation of regression is equal to 3.5 m °C.

To conclude, the results obtained for fat and protein are in accordance with the recommendations of the ISO 8196-3 / IDF 128-3 standard. The high mean bias for protein is probably due to a preliminary defect of calibration on goat and zone milk. Despite the absence of standard criteria for freezing point, the residual standard deviation obtained (3.5 m°C) enables an accuracy of estimation of +/-7 m °C.

B.3.3. Ewe milk

The following tables and figures present the results obtained:

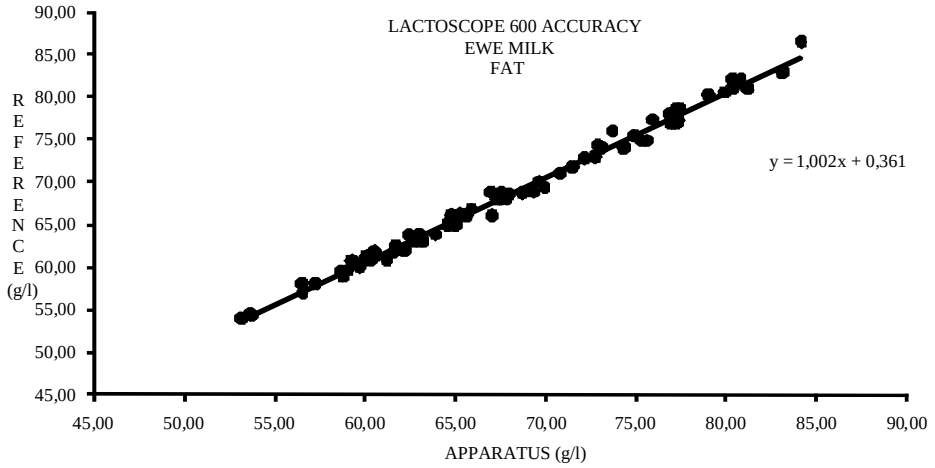
	n	Min	max	M	Sx	Sr	Sr (%)	r
Fat (g/l)	100	53.26	85.64	68.111	7.799	0.104	0.15%	0.282
Protein (g/l)	100	45.23	64.20	53.644	4.441	0.110	0.21%	0.309
Freezing point (m°C x -1)	100	538	563	553.5	4.3	1.0	0.18%	2.8

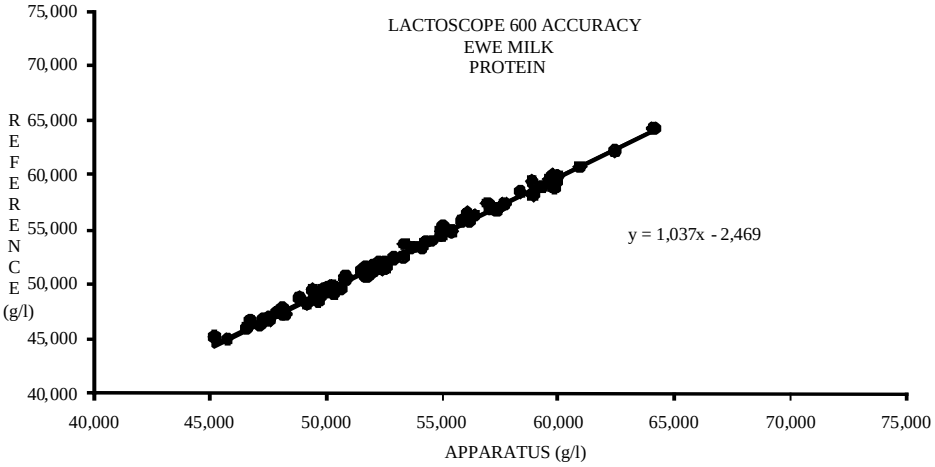
Table 7: Lactoscope repeatability criteria for fat, protein and freezing point in ewe milk samples
n: number of results; min and max: minimum and maximum values; M and Sx: mean and standard deviation of the results; Sr and Sr %: absolute an relative standard deviation of repeatability; r: maximum deviation of repeatability on 95 % of cases

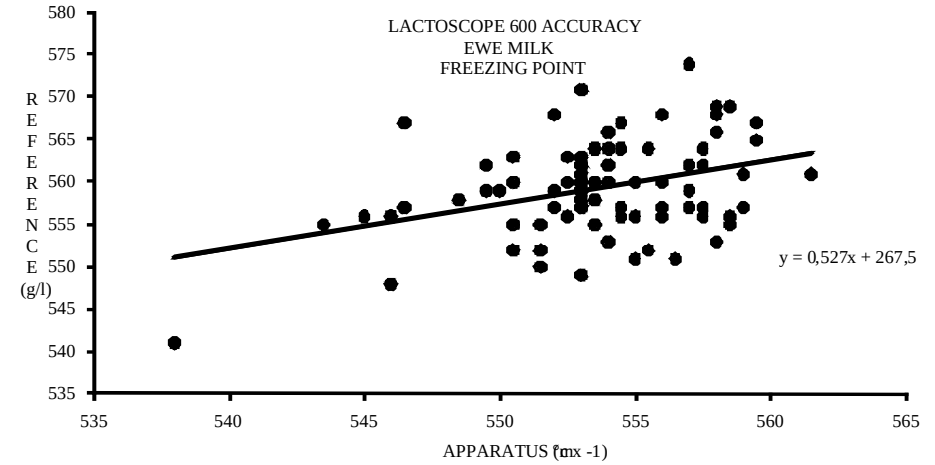
For fat and protein, the absolute and relative standard deviations of repeatability (Sr and Sr %) are in accordance with the recommendations of the ISO 8196-3 / IDF 128-3 standard and the CNIEL PROC IR 06 handbook (respectively lower than 0.29 g/l and 0.35 %).
The standard deviation of repeatability obtained for freezing point is in accordance with the recommendations of the CNIEL PROC CR IR 06 handbook (Sr ≤ 3 m °c).

	n	min	max	Y	Sy	d	Sd	Sy,x	Sy,x (%)
Fat (g/l)	79	54.00	86.50	68.127	7.699	-0.547	0.665	0.669	0.99
Protein (g/l)	79	45.10	64.28	52.589	4.467	0.459	0.397	0.365	0.69
Freezing point (m°C x -1)	80	541	574	559.4	5.8	-5.7	5.7	5.4	0.97

Table 8: Lactoscope accuracy criteria for fat, protein and freezing point in ewe milk samples
n, min, max: number of results, minimum and maximum values; Y: mean results using the reference method; Sy: standard deviation of the results from the reference method; d, Sd: mean and standard deviation of deviations; Sy,x: residual standard deviation.







Figures 11 to 13: Relation between Lactoscope and reference results for fat, protein and freezing point in ewe milk samples

It can be noted that:

↳ For fat: the mean and the standard deviation of deviations are equal to –0.547 and 0.665 g/l. The regression slope (1.002) and the intercept (0.361) are respectively not significantly different from 1.00 and zero (P = 5 %). The absolute and relative residual standard deviation of regression (0.69 g/l and 0.99 %) are in accordance with the recommendations of the ISO 8196-3 / IDF 128-3 standard (respectively lower than 1.45 g/l and 1.75 %).

↳ For protein: the mean and the standard deviation of deviations are equal to 0.459 and 0.397 g/l. The regression slope (1.037) and the intercept (-2.469) are significantly different from 1.00 and zero (P = 1). The absolute and relative residual standard deviation of regression (0.365 g/l and 0.69 %) are in accordance with the recommendations of the ISO 8196-3 / IDF 128-3 standard (respectively lower than 1,45 g/l et 2,0 %).

↳ For freezing point: the mean and the standard deviation of deviations are equal to –5.7 and 5.7 m °C x-1. The regression slope (0.527) and the intercept (267.5) are respectively significantly different from 1.00 and zero (P = 1 %). The residual standard deviation of regression is equal to 5.4 m °C.

ARTICLE

To conclude, the results obtained for fat and protein are in accordance with the recommendations of the ISO 8196-3 / IDF 128-3 standard. Despite the absence of standard criteria for freezing point, the residual standard deviation obtained (5.4 m°C) enables an accuracy of estimation equal to +/- 10.8 m °C.

CONCLUSION

The results obtained for fat, protein and freezing point are in accordance with the recommendations of the ISO 8196-3 / IDF 128-3: 2010 and/or the CNIEL/IE handbooks concerning the use of infrared apparatus with the context of milk payment and milk control in France (CNIEL PROC IR 06, CNIEL PROC CR IR 04) for the three types of milk (cow, goat and ewe).

Moreover, the results obtained for urea (cow individual milk) are in accordance with the recommendations of the ISO 8196-3 / IDF 128-3: 2010 standard. The results obtained for dry matter (cow herd milk) enable accuracy of estimation lower than 0.1 g/100g.

According to the evaluation report of the Lactoscope infrared analyser– X. QUERVEL, P. TROSSAT – September 2013

ACTALIA Cecalait follows its accreditation step

Our «accréditation n° 1-2473, comparaisons interlaboratoires, portée disponible sur www.cofrac.fr», «no 1-2473 accreditation, interlaboratory comparisons, scope available on www.cofrac.fr» as organiser of interlaboratory comparisons has just been extended to the following criteria:

- **enumeration of microorganisms at 30 °C,**
- **enterobacteria,**
- **sulphite-reducing anaerobes,**
- ***Clostridium perfringens,***
- ***Bacillus cereus***

for the proficiency testings on pathogenic flora in milk multi-criteria and pathogenic flora in cheese multi-criteria.

Moreover, our «accréditation n° 1-5577, essais, portée disponible sur www.cofrac.fr», «accreditation no 1-5577, tests, scope available on www.cofrac.fr» concerning the tests has been extended to:

➢ for microbiology:

- the **detection of *Salmonella* spp**. in products intended for human or animal consumption and environmental samples of the food industry (according to ISO 6579 standard),
- the **detection of *Listeria monocytogenes*** in products intended for human or animal consumption (according to ISO 11290-1 standard),
- the **enumeration of *Listeria monocytogenes*** in products intended for human or animal consumption (according to ISO 11290-2 standard),
- the **screening and confirmation of residue at antibiotic activity** in milk (according to an acidification and confirmation by spot step method (2 dishes): internal method using the 6th October 1983 Official Journal of the French Republic).

➢ for physico-chemistry:

- the **counting of somatic cells** by microscope in cow raw milk (according to ISO 13366-1 standard),
- the **determination of moisture content** in dried milk (according to internal method using the totality of the repealed IDF 26A standard).

STANDARDS, DRAFT STANDARDS

Classification in alphabetical order by theme

ISO standards under development

ANIMAL AND VEGETABLE FATS AND OILS	
ISO/DIS 12228-1:2013 September 2013	Determination of individual and total sterols contents – Gas chromatographic method Part 1: Animal and vegetable fats and oils

ISO published standards

MICROBIOLOGY OF FOOD AND ANIMAL FEEDING STUFFS	
ISO 7218:2007/Amd 1:2013 August 2013	MICROBIOLOGY OF FOOD AND ANIMAL FEEDING STUFFS General requirements and guidance for microbiological examinations – Amendment 1
ISO 4833-1 and ISO 4833-2 September 2013	MICROBIOLOGY OF THE FOOD CHAIN Horizontal method for the enumeration of microorganisms Part 1: Colony count at 30 degrees C by the pour plate technique Part 2: Colony count at 30 degrees C by the surface plating technique
MILK AND MILK PRODUCTS	
ISO 9622:2013 (IDF 141) September 2013	MILK AND MILK PRODUCTS Guidelines for the application of mid infrared spectrometry
PROCESSED CHEESE PRODUCTS	
ISO/TS 18083:2013 (IDF/RM 51) July 2013	PROCESSED CHEESE PRODUCTS Calculation of content of added phosphate expressed as phosphorus
QUALITY	
ISO/CEI TS 17023:2013 August 2013	Conformity assessment – Guidelines for determining the duration of management system certification audits

NEW EU REGULATIONS

Classification is established in alphabetical order of the first keyword

ADDITIVES

O.J.E.U. L 202, 27th July 2013 – Commission Regulation (EU) No 724/2013 of 26 July 2013 amending Regulation (EU) No 231/2012 as regards specifications on several polyols
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2013:202:0011:0016:EN:PDF

O.J.E.U. L 230, 29th August 2013 – Commission Regulation (EU) No 817/2013 of 28 August 2013 amending Annexes II and III to Regulation (EC) No 1333/2008 of the European Parliament and of the Council and the Annex to Commission Regulation (EU) No 231/2012 as regards octenyl succinic acid modified gum arabic
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2013:230:0007:0011:EN:PDF

FLAVOURING SUBSTANCES

O.J.E.U. L 273, 15th October 2013 – Commission Regulation (EU) No 985/2013 of 14 October 2013 amending and correcting 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/LexUriServ/LexUriServ.do?uri=OJ:L:2013:273:0018:0024:EN:PDF

STANDARDS - REGULATIONS

HEALTH CLAIMS

O.J.E.U. L 282, 24th October 2013 – Commission Regulation (EU) No 1017/2013 of 23 October 2013 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/LexUriServ/LexUriServ.do?uri=OJ:L:2013:282:0039:0042:EN:PDF

O.J.E.U. L 282, 24th October 2013 – Commission Regulation (EU) No 1018/2013 of 23 October 2013 amending Regulation (EU) No 432/2012 establishing a list of permitted 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/LexUriServ/LexUriServ.do?uri=OJ:L:2013:282:0043:0045:EN:PDF

O.J.E.U. L 289, 31st October 2013 – Commission Regulation (EU) No 1066/2013 of 30 October 2013 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/LexUriServ/LexUriServ.do?uri=OJ:L:2013:289:0049:0055:EN:PDF

HYGIENE

O.J.E.U. L 292, 1st November 2013 – Commission Regulation (EU) No 1079/2013 of 31 October 2013 laying down transitional measures for the application of Regulations (EC) No 853/2004 and (EC) No 854/2004 of the European Parliament and of the Council
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2013:292:0010:0012:EN:PDF

LABELLING

O.J.E.U. L 201, 26th July 2013 – Commission Regulation (EU) No 718/2013 of 25 July 2013 amending Regulation (EC) No 608/2004 concerning the labelling of foods and food ingredients with added phytosterols; phytosterol esters, phytostanols and/or phytostanol esters
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2013:201:0049:0050:EN:PDF

O.J.E.U. L 232, 30th August 2013 – Commission Implementing Decision of 28 August 2013 concerning the Italian draft Decree on the methods for indicating the origin for shelf-stable milk, UHT milk, microfiltered pasteurised milk and high-temperature pasteurised milk
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2013:232:0035:0036:EN:PDF

PESTICIDES

O.J.E.U. L 217, 13th August 2013 – Commission Regulation (EU) No 772/2013 of 8 August 2013 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 diphenylamine in or on certain products
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2013:217:0001:0027:EN:PDF

O.J.E.U. L 221, 17th August 2013 – Commission Regulation (EU) No 777/2013 of 12 August 2013 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 clodinafop, clomazone, diuron, ethalfluralin, ioxynil, iprovalicarb, maleic hydrazide, mepanipyrim, metconazole, prosulfocarb and tepraloxydim in or on certain products
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2013:221:0001:0048:EN:PDF

O.J.E.U. L 233, 31st August 2013 – Commission Regulation (EU) No 834/2013 of 30 August 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 acequinocyl, bixafen, diazinon, difenoconazole, etoxazole, fenhexamid, fludioxonil, isopyrazam, lambda-cyhalothrin, profenofos and prothioconazole in or on certain products
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2013:233:0011:0042:EN:PDF

O.J.E.U. L 279, 19th October 2013 – Commission Regulation (EU) No 1004/2013 of 15 October 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 8-hydroxyquinolone, cyproconazole, cyprodinil, fluopyram, nicotine, pendimethalin, penthiopyrad and trifloxystrobin in or on certain products
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2013:279:0010:0056:EN:PDF

STANDARDS - REGULATIONS

PGI / PDO / TSG

O.J.E.U. C 231, 9th August 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 [Yorkshire Wensleydale (PGI) (cheese)]
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:C:2013:231:0020:0024:EN:PDF

O.J.E.U. C 237, 15th August 2013 – Publication of an application pursuant to Article 50(2)(b) of Regulation (EU) No 1151/2012 of the European Parliament and of the Council on quality schemes for agricultural products and foodstuffs [Zemaitiskas Kastinys (TSG) (sour cream)]
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:C:2013:237:0040:0043:EN:PDF

O.J.E.U. L 238, 6th September 2013 – Commission Implementing Regulation (EU) No 857/2013 of 4 September 2013 approving non-minor amendments to the specification for a name entered in the register of protected designations of origin and protected geographical indications [Mont d'Or / Vacherin du Haut-Doubs (PDO) (cheese)]
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2013:238:0001:0002:EN:PDF

O.J.E.U. L 240, 7th September 2013 – Commission Implementing Regulation (EU) No 862/2013 of 5 September 2013 approving non-minor amendments to the specification for a name entered in the register of protected designations of origin and protected geographical indications [Casatella Trevigiana (PDO) (cheese)]
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2013:240:0015:0016:EN:PDF

O.J.E.U. C 290, 5th October 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 [Strachitunt (PDO) (cheese)]
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:C:2013:290:0005:0008:EN:PDF

O.J.E.U. C 296, 12th October 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 [Valençay (PDO) (cheese)]
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:C:2013:296:0004:0010:EN:PDF

O.J.E.U. L 279, 19th October 2013 – Commission Implementing Regulation (EU) No 1005/2013 of 17 October 2013 approving non-minor amendments to the specification for a name entered in the register of protected designations of origin and protected geographical indications [Emmental français est-central (PGI) (cheese)]
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2013:279:0057:0058:EN:PDF

O.J.E.U. C 312, 26th October 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 [Requeijao da Beira Baixa (PDO) (dairy product)]
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:C:2013:312:0026:0029:EN:PDF

O.J.E.U. C 316, 30th October 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 [Neufchâtel (PDO) (cheese)]
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:C:2013:316:0014:0020:EN:PDF

O.J.E.U. C 318, 1st November 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 [Pecorino Sardo (PDO) (cheese)]
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:C:2013:318:0008:0013:EN:PDF

O.J.E.U. L 293, 5th November 2013 – Commission Implementing Regulation (EU) No 1086/2013 of 30 October 2013 approving non-minor amendments to the specification for a name entered in the register of protected designations of origin and protected geographical indications [Raschera (PDO) (cheese)]
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2013:293:0026:0027:EN:PDF

O.J.E.U. L 293, 5th November 2013 – Commission Implementing Regulation (EU) No 1090/2013 of 4 November 2013 entering a name in the register of protected designations of origin and protected geographical indications [Travia da Beira Baixa (PDO) (dairy product)]
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2013:293:0034:0035:EN:PDF

STANDARDS - REGULATIONS

O.J.E.U. L 298, 8th November 2013 – Commission Implementing Regulation (EU) No 1110/2013 of 5 November 2013 entering a name in the register of protected designations of origin and protected geographical indications [Queso Los Beyos (PGI) (cheese)]
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2013:298:0023:0024:EN:PDF

O.J.E.U. L 298, 8th November 2013 – Commission Implementing Regulation (EU) No 1111/2013 of 5 November 2013 entering a name in the register of protected designations of origin and protected geographical indications [Lietuviskas varskès suris (PGI) (cheese)]
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2013:298:0025:0026:EN:PDF

O.J.E.U. C 326, 12th November 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 [Queso de Murcia al Vino (PDO) (cheese)]
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:C:2013:326:0011:0021:EN:PDF

O.J.E.U. C 329, 13th November 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 [Queso de Murcia (PDO) (cheese)]
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:C:2013:329:0004:0015:EN:PDF

O.J.E.U. L 302, 13th November 2013 – Commission Implementing Regulation (EU) No 1127/2013 of 7 November 2013 approving minor amendments to the specification for a name entered in the register of protected designations of origin and protected geographical indications [Montasio (PDO) (cheese)]
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2013:302:0001:0006:EN:PDF

O.J.E.U. L 302, 13th November 2013 – Commission Implementing Regulation (EU) No 1128/2013 of 7 November 2013 approving minor amendments to the specification for a name entered in the register of protected designations of origin and protected geographical indications [Morbier (PDO) (cheese)]
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2013:302:0007:0013:EN:PDF

O.J.E.U. L 302, 13th November 2013 – Commission Implementing Regulation (EU) No 1132/2013 of 7 November 2013 approving non-minor amendments to the specification for a name entered in the register of protected designations of origin and protected geographical indications [Stelvio/Stilfser (PDO) (cheese)]
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2013:302:0020:0021:EN:PDF

O.J.E.U. L 303, 14th November 2013 – Commission Implementing Regulation (EU) No 1142/2013 of 12 November 2013 approving non-minor amendments to the specification for a name entered in the register of protected designations of origin and protected geographical indications [Chaource (PDO) (cheese)]
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2013:303:0008:0009:EN:PDF

PHARMACOLOGICALLY ACTIVE SUBSTANCES

O.J.E.U. L 288, 30th October 2013 – Commission Implementing Regulation (EU) No 1056/2013 of 29 October 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 neomycin
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2013:288:0060:0062:EN:PDF

O.J.E.U. L 288, 30th October 2013 – Commission Implementing Regulation (EU) No 1057/2013 of 29 October 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 manganese carbonate
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2013:288:0063:0065:EN:PDF

PROTEINS

O.J.E.U. L 230, 29th August 2013 – Commission Directive 2013/46/EU of 28 August 2013 amending Directive 2006/141/EC with regard to protein requirements for infant formulae and follow-on formulae
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2013:230:0016:0019:EN:PDF

AFNOR VALIDATIONS

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

Commercial name	Date	Certificate	Description
RENEWALS OF VALIDATIONS			
RAPID'*L.MONO* ENUMERATION	Validation date: 28 Sep 2001 Renewal: 8 Dec 2005, 2 July 2009 and 3 Oct 2013 Extension: 28 Sep 2006 and 25 Sep 2008 **End of validity: 28 Sep 2017**	**BRD-07/05-09-01**	**Enumeration of *Listeria monocytogenes*** All human food products and environmental samples
TEMPO TC	Validation date: 9 Dec 2005 Renewal: 4 Dec 2009 and 4 Oct 2013 Extension: 3 Feb 2011 **End of validity: 9 Dec 2017**	**BIO-12/17-12/05**	**Enumeration of total coliforms** All human and animal food products (except beverages and cattle food)
CHROMAGAR™ *LISTERIA*	Validation date: 13 Dec 2001 Renewal: 10 Mar 2006, 25 Sep 2009 and 3 Oct 2013 **End of validity: 13 Dec 2017**	**CHR-21/01-12/01**	**Detection of *Listeria monocytogenes*** All human food products and environmental samples
CHROMAGAR™ *LISTERIA* NUMERATION	Validation date: 14 Dec 2006 Renewal: 25 Sep 2009 and 3 Oct 2013 **End of validity: 14 Dec 2017**	**CHR-21/02-12/06**	**Enumeration of *Listeria monocytogenes*** All human food products and environmental samples
EXTENSIONS OF VALIDATIONS			
IQ-CHECK *SALMONELLA* II	Validation date: 1 July 2004 Extension: 24 May 2007, 28 Sep 2009, 25 Sep 2008, 4 Feb 2010, 3 Feb 2011, 1 July 2011, 22 Mar 2012 and 3 Oct 2013 Renewal: 27 Nov 2008 and 10 May 2012 **End of validity: 1 July 2016**	**BRD-07/06-07/04**	**Detection of *Salmonella*** All human and animal food products and environmental samples (including animal faeces and environmental samples from the primary production stage)
IQ-CHECK *LISTERIA MONO- CYTOGENES* II	Validation date: 7 Apr 2005 Extension: 15 Dec 2006, 28 Sep 2009, 4 Feb 2010, 22 Mar 2012 and 3 Oct 2013 Renewal: 26 Mar 2009 and 28 Mar 2013 **End of validity: 7 Apr 2017**	**BRD-07/10-04/05**	**Detection of *Listeria monocytogenes*** All human food products and environmental samples
IQ-CHECK *CRONOBACTER* SPP	Validation date: 31 Jan 2013 Extension: 3 Oct 2013 **End of validity: 31 Jan 2017**	**BRD-07/23-01/13**	**Detection of *Cronobacter* spp.** Powdered infant formula and environmental samples

AFNOR VALIDATIONS

IQ-CHECK *LISTERIA* SPP	Validation date: 24 May 2007 Extension: 28 Sep 2007, 4 Feb 2010,22 Mar 2012 and 3 Oct 2013 Renewal: 13 May 2011 **End of validity: 24 May 2015**	**BRD-07/13-05/07**	**Detection of *Listeria* spp.** All human food products and environmental samples
COMPASS *LISTERIA* AGAR	Validation date: 4 Dec 2007 Extension: 4 Oct 2013 Renewal: 10 May 2012 **End of validity: 4 Dec 2015**	**BKR-23/05-12/07**	**Enumeration of *Listeria monocytogenes*** 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.

FOOD MICROBIOLOGY

SOFOS J. – **Advances in microbial food safety (volume 1)** – Woodhead Publishing – June 2013 – ISBN: 978-0-85709-438-4 – 560 pages

http://www.woodheadpublishing.com/en/book.aspx?bookID=2560

This book summarises new research, outbreaks of foodborne disease and changes to legislation in food microbiology research. It examines also past, present and future food safety management systems and provides updates on specific pathogens including *Salmonella*, *Listeria monocytogenes* and *Bacillus* species. Finally, it explores emerging parasites in food, advances in separation and concentration of microorganisms from food samples, and new approaches in microbial pathogen detection.

POWDER

BHANDARI B.; BANSAL N.; ZHANG M; SCHUCK P. – **Handbook of food powders: Processes and properties** – Woodhead Publishing – August 2013 – ISBN: 978-0-85709-513-8 – 688 pages

http://www.woodheadpublishing.com/en/book.aspx?bookID=2613

This book reviews the processing and handling technologies in the produciton of food powders. It examines also the powder properties, the composition, the shelf life and the techniques used to examine particle size. Finally, it focusses in speciality powders such as dry dairy products, infant formulas, powdered egg, fruit and vegetable, and culinary and speciality products.

SENSORY QUALITY

KILCAST D.. – **Instrumental assessment of food sensory quality: A practical guide** – Woodhead Publishing – September 2013 – ISBN: 978-0-52409-439-1 – 656 pages

http://www.woodheadpublishing.com/en/book.aspx?bookID=2561

This book examines the range et use of instrumental methods for measuring sensory quality. It reviews the principles and practice of the assessment and analysis of food appearance, flavour, texture and viscosoty. Moreover, it reviews the instrumental method of the sensory quality in food, such as beverages, meat, poultry, fish, baked goods, dry products, dairy products and fruit and vegetables.

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, A-G TERPSTRA, W. BEUKEMA
Rédaction achevée le 14 novembre 2013 – Traduction achevée le 19 novembre 2013
Impression : ACTALIA Cecalait, B.P. 70129, 39801 POLIGNY CEDEX
Tél. : 33.(0)3.84.73.63.20 - Fax : 33.(0)3.84.73.63.29
3ème trimestre 2013
Dépôt légal : à parution
ISSN 1298-6976