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ARTICLE

EVALUATION OF THE FOSS MILKOSCAN™ MARS INFRARED ANALYSER

The MilkoScan™ Mars is an FTIR infrared analyser manufactured by Foss Analytical A/S (Danmark) and commercialised in France by Foss France SAS. It is used for the determination of the composition components [fat, protein, lactose, dry matter, freezing point (only for milk)] in liquid dairy products as milk, cream and whey.
The apparatus is controlled by an integrated program. All the operations are carried out through a touch screen. The results can be exported to a USB port in .csv file format. The “zero” measurement and the cleaning are automated. The modification of the prediction models can be done by adjusting the slope and bias, the calculations are done directly by the software.

The instrument used in this study was:

- MilkoScan™ Mars
- Serial number: 91840178
- Part: 60062098

The apparatus has « calibration MCF 04 » prediction models.

The instrument was installed by Foss in a temperature controlled room (20-23 °C – air conditioning), without direct sunlight.

Due to an apparatus problem, the tests on the cream matrix (repeatability and accuracy) were carried out on a second apparatus after checking the short-term stability (on milk).
The characteristics of this instrument were:

- MilkoScan™ Mars
- Seril number: 91855805
- Part: 60062098

A cleaning solution [Solution Msc W-960 Cleaning Agent (24 g Msc W-960 in 5 l of demineralised water ISO 3696 grade 3)] and a zero solution [Solution Msc Zero (5 ml Msc Zero in 5 l of demineralised water ISO 3696 grade 3] are required. The reagent vials of the instrument are equipped with a level sensor.

THE TESTS

The evaluation tests were performed in ACTALIA Cecalait’s physico-chemistry laboratory from May to September 2020. After preliminary tests of stability, the repeatability and accuracy on tank raw cow milk, tank raw ewe milk, cream and whey for fat, true protein, dry matter and total nitrogen were evaluated.

The accuracy of the instrument was evaluated according to the following standardised:methods

- Fat in milk:	Gerber method according to ISO 19662|IDF 238
- Fat in ewe milk:	Acido-butyrometric method according to NF V04-155
- Fat in cream:	Röse-Gottlieb method according to ISO 2450|IDF 16
- Fat in whey :	Röse-Gottlieb method according to ISO 1211|IDF 1
- Protein in milk:	Amido black method according to NF V04-216
- Total nitrogen:	Kjeldahl method according to ISO 8968-1|IDF 20-1
- Dry matter:	Oven method according to ISO 6731|IDF 21

1. EVALUATION OF THE SHORT-TERM REPRODUCTIBILITY

The short-term reproductibility was evaluated by analysing 4 samples of raw milk with preservative (Bronopol 0.02 % final), with different concentration levels of fat and protein, in triplicate, every 15-20 minutes to obtain at least 20 sequences.
To evaluate the stability of the instrument, the repeatability and reproducibility were calculated by level.

	Level 1	Level 2	Level 3	Level 4
Fat (g/l)	22	38	54	75
Protein (g/l)	24	35	39	55

Table 1: Content of the samples used for the short-term reproducibility evaluation

The following table presents the results obtained:

		M	Sr	Sr(%)	SR	SR(%)	r	R
Fat (g/l)	**Level 1**	20.765	0.0502	0.242%	0.0649	0.312%	0.139	0.180
	Level 2	35.469	0.0882	0.249%	0.1048	0.295%	0.244	0.290
	Level 3	50.237	0.0932	0.186%	0.1120	0.223%	0.258	0.310
	Level 4	70.999	0.0867	0.122%	0.1143	0.161%	0.240	0.317
True protein (g/l)	**Level 1**	24.749	0.0663	0.268%	0.0813	0.328%	0.184	0.225
	Level 2	31.689	0.0772	0.244%	0.0951	0.300%	0.214	0.263
	Level 3	38.563	0.1051	0.273%	0.1260	0.327%	0.291	0.349
	Level 4	52.611	0.1537	0.292%	0.1810	0.344%	0.426	0.501
Dry matter (g/l)	**Level 1**	97.752	0.1566	0.160%	0.2011	0.206%	0.434	0.557
	Level 2	119.411	0.1715	0.144%	0.2270	0.190%	0.475	0.629
	Level 3	141.187	0.2267	0.161%	0.2717	0.192%	0.628	0.753
	Level 4	176.256	0.2712	0.154%	0.3323	0.189%	0.751	0.921

Table 2: MilkoScan™ Mars stability criteria for fat, true protein and dry matter[1]

The results for levels from 1 to 3 indicate that the standard deviation of repeatability for fat and protein are below the limits required in ISO 8196-3|IDF 128-3 standard for milk with an average fat and protein content (0.28 g/l). For milk with a high fat and protein content (level 4), the results indicate that the standard deviation of repeatability for fat and protein are below the limits required in ISO 8196-3|IDF 128-3 standard for milk with a high fat and protein content (0.56 g/l).

As no standardised value exists for dry matter, it can be noted that the reproducibility of the instrument (R) is lower than the reproducibility of the reference value (0.20 g/100 g).
The short-term stability of the apparatus used for the cream evaluation was verified under the same conditions as for the instrument above. The results obtained are of the same order and comply with the requirements of standard ISO 8196-3.

2. EVALUATION OF THE REPEATABILITY

The repeatability of the instrument was performed by analysing:

- for tank raw cow milk: 39 samples of tank raw milk from a French plant (West of France).
- for ewe milk: 33 samples of ewe milk from a French plant (South-West of France).
- for whey: 24 samples of skimmed whey from a French plant (East-Center of France).
- for cream: 24 samples of cream from a French plant (West of France).

Bronopol was added to the samples to give a final concentration of 0.02 %. They were analysed (after heating at 40 ± 2 °C and 37 ± 2 °C for cream) in non consecutive duplicate according to the following sequence: Set 1 rep 1 – Set 2 rep 2 - … - Set n rep n.

The repeatability was calculted using results in duplicate obtained with all the data and for the criteria:

- Fat for all the matrixes,
- Dry matter for all the matrixes,
- Protein for tank raw cow milk and ewe milk,
- Total nitrogen for whey.

The following table presents the results obtained:

		n	min	max	M	S_r	S_r%	r
Tank raw cow milk	**Fat (g/l)**	39	37.24	41.25	39.75	0.076	0.19%	0.211
	Protein (g/l)	39	31.31	33.31	32.27	0.123	0.38%	0.341
	Dry matter (g/100g)	39	12.46	12.95	12.70	0.025	0.20%	0.069

[1] M: mean; S_r and S_R (S_r% andt S_R%): absolute (and relative) standard deviation of repeatability and reproductibility; r and R: maximum deviation of repeatability and reproductibility in 95 % of cases.

Ewe milk	**Fat (g/l)**	33	71.88	84.54	79.88	0.105	0.13%	0.290
	Protein (g/l)	33	55.61	64.70	60.46	0.125	0.21%	0.347
	Dry matter (g/100g)	33	18.11	19.65	19.12	0.023	0.12%	0.063
Whey	**Fat (g/100g)**	24	0.03	0.05	0.03	0.003	9.50%	0.009
	Total nitrogen (g/100g)	24	0.63	0.85	0.81	0.004	0.47%	0.011
	Dry matter (g/100g)	24	5.21	6.72	6.42	0.006	0.10%	0.017
Cream	**Fat (g/l)**	23	407.66	416.17	413.40	0.735	0.18%	2.035
	Dry matter (g/l)	24	462.08	471.94	468.45	0.850	0.18%	2.353

Table 3: MilkoScan™ Mars repeatability criteria for fat, true protein, dry matter and total nitrogen in tank raw cow milk, ewe milk, whey and cream[2]

It can be noted:

- for tank raw cow milk: for fat and true protein content, the standard deviations of repeatability are lower than the requirements of the ISO 8196-3|IDF 128-3 standard (Sr < 0.14 g/l). For dry matter, the standard deviation of repeatability is in the same order that the results for the other components and lower than the repeatability standard deviation of the reference method (Sr = 0.036 g/100 g).

- for ewe milk: for fat and true protein content, the standard deviations of repeatability are lower than the requirements of the ISO 8196-3|IDF 128-3 standard (Sr < 0.28 g/l). For dry matter, the standard deviation of repeatability is in the same order that the results for the other components and lower than the repeatability standard deviation of the reference method (Sr = 0.036 g/100 g).

- for whey: the standard deviation of repeatability is equivalent for all the criteria. As no standardised value exists for whey, it can be noted that the standard deviations of repeatability obtained with the instrument are lower or near of those obtained with the reference methods when they exist (Sr = 0.001 g/100g vs 0.013 g/100g for fat; 0.006 g/100g vs 0.036 g/100g for dry matter).

- for cream: the standard deviation of repeatability is equivalent for all the criteria. For fat, the relative standard deviation (Sr%) is in accordance with the recommendations of the ISO 8196-3|IDF 128-3 standard for milk with high content (Sr% < 0.35 %).

3. EVALUATION OF THE ACCURACY

The accuracy of the instrument was evaluated by using the same samples than those used for the repeatablity evaluation. The mean of the duplicates of the results obtained in the repeatability evaluation was used for the calculation of the results. Outliers samples (samples whose regression residues are greater than 2 times the standard deviation of deviations: P at 5 %) have been discarded.

The following table presents the results obtained:

		n	min	max	X	Sx	Sy,x	Sy,x%	b	a
Tank raw cow milk	**Fat (g/l)**	37	37.27	41.21	39.71	0.88	0.362	0.92%	0.919	2.842
	True protein (g/l)	37	31.35	33.13	32.24	0.44	0.081	0.25%	0.949	1.690
	Dry matter (g/100 g)	37	12.48	12.95	12.71	0.11	0.034	0.27%	0.860	1.708
Ewe milk	**Fat (g/l)**	32	71.92	84.48	79.97	3.22	0.400	0.50%	1.078	-5.433
	True protein (g/l)	32	55.73	64.56	60.51	2.18	0.259	0.43%	1.077	-4.868
	Dry matter (g/100 g)	31	18.12	19.63	19.11	0.41	0.048	0.25%	1.061	-1.087
Whey	**Fat (g/100g)**	21	0.03	0.05	0.03	0.01	0.004	12.96%	0.756	0.008
	True protein (g/100g)	22	0.63	0.85	0.81	0.05	0.033	3.85%	1.317	-0.216
	Dry matter (g/100 g)	22	5.22	6.72	6.41	0.35	0.025	0.40%	0.954	0.109
Crème	**Fat (g/100 g)**	22	40.83	41.57	41.33	0.20	0.215	0.51%	-0.053	44.190
	Dry matter (g/100 g)	24	46.37	47.08	46.85	0.19	0.148	0.31%	0.370	29.787

Table 4: MilkoScan™ Mars accuracy criteria for fat, true protein and dry matter in tank raw cow milk, ewe milk, whey and cream[3]

[2] N: number of the results; min and max: minimum and maximum values; M : mean of the results; Sr (Sr%): absolute (and relative) standard deviation; r: maximum deviation of repeatability in 95 % of cases

[3] n, min, max: number of results, minimum and maximum values; Y: mean of the 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 (Sy,x%): absolute (and relative) residual standard deviation; b,a: slope and intercept of the linear regression.

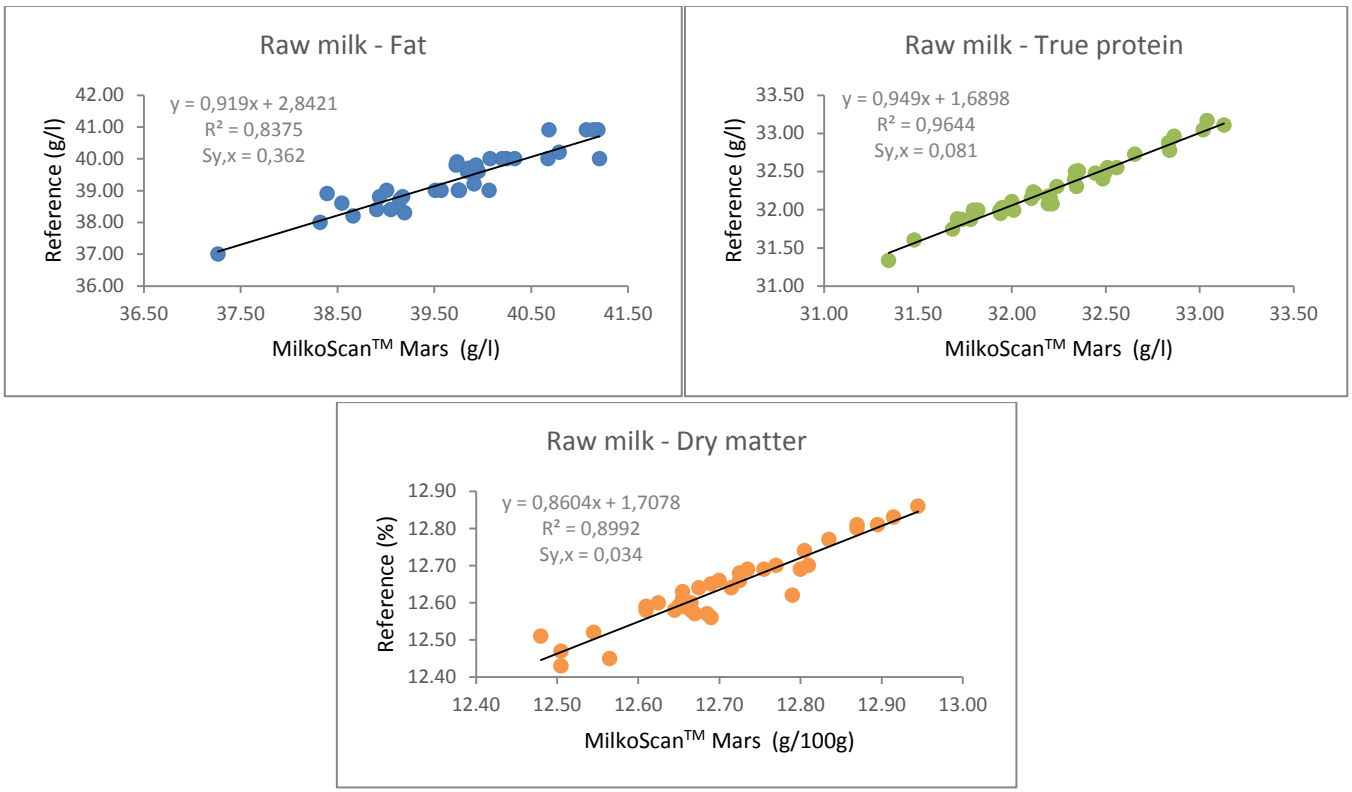


Figure 1: Relation between MilkoScan™ Mars and reference results for fat, true protein and dry matter in tank raw cow milk

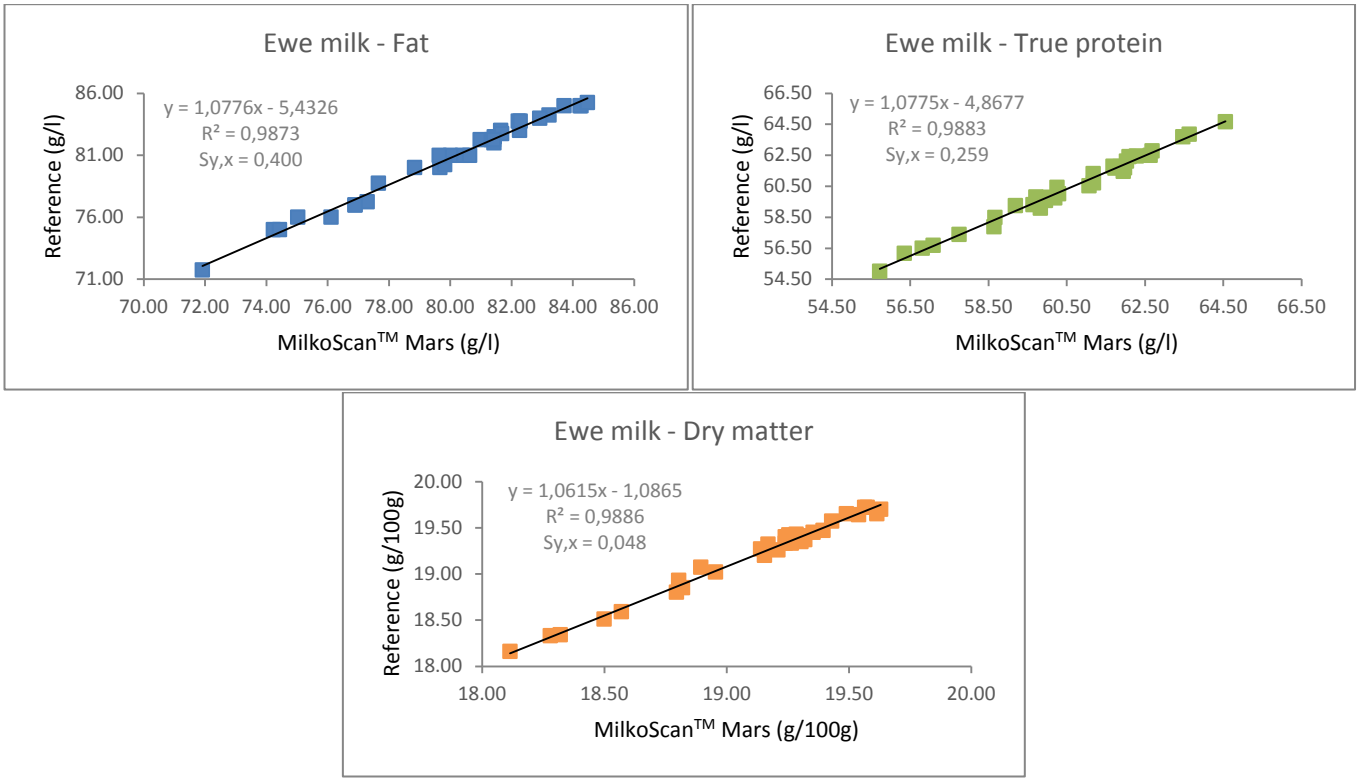


Figure 2: Relation between MilkoScan™ Mars and reference results for fat, true protein and dry matter in ewe milk

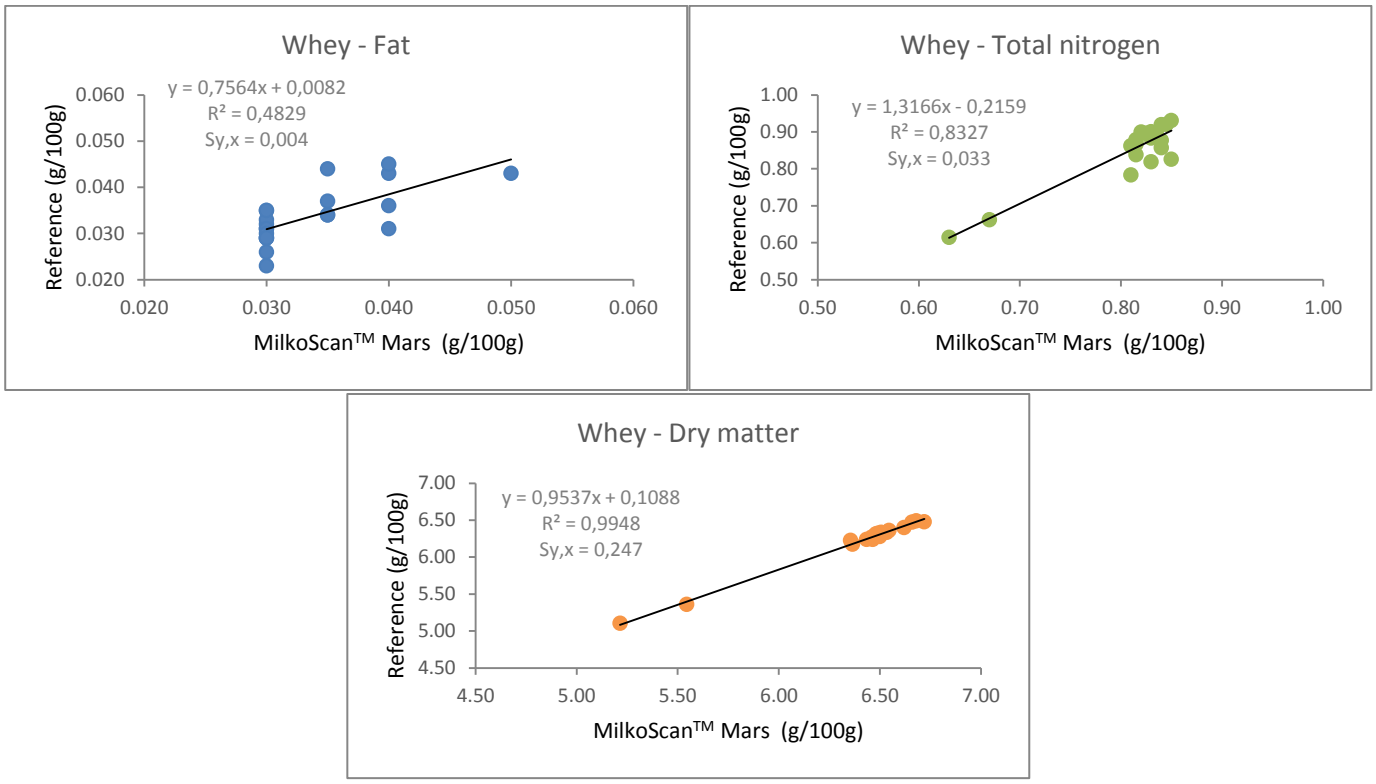


Figure 3: Relation between MilkoScan™ Mars and reference results for fat, total nitrogen and dry matter in whey

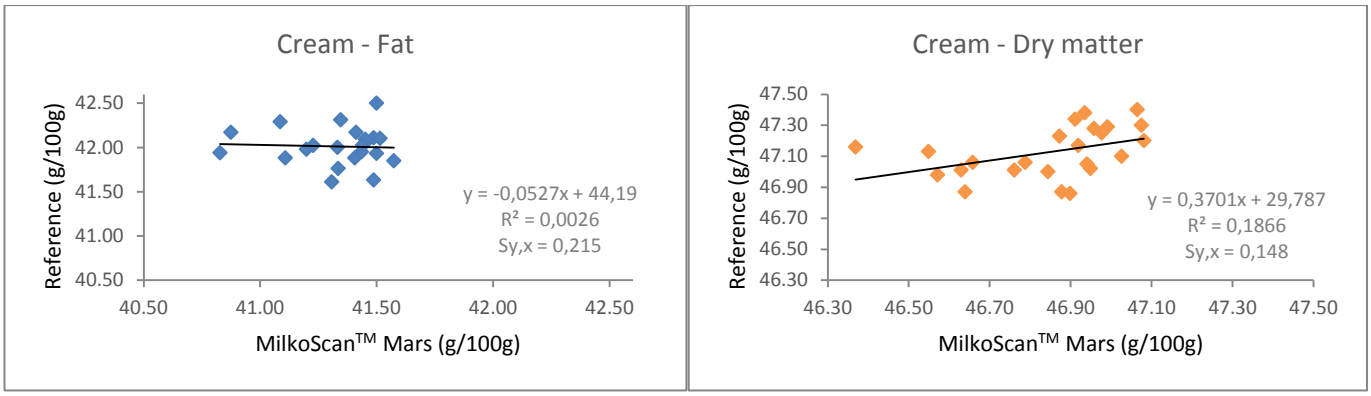


Figure 4: Relation between MilkoScan™ Mars and reference results for fat and dry matter in cream

Concerning the relation between MilkoScan™ Mars reference results, it can be noted that:

- for tank raw cow milk:

The residual standard deviations of linear regression obtained are equal to 0.362 g of fat/l, 0.081 g of protein/l, and 0.034 g of dry matter/100 g. The estimation precision of the instrument is therefore ± 0.72 g/l (± 2 x 0.362 g/l) for fat, ± 0.16 g/l (± 2 x 0.081 g/l) for true protein and ± 0.068 g/100 g (± 2 x 0,034 g/100 g) for dry matter.

- for ewe milk:

The residual standard deviations of linear regression obtained are equal to 0.400 g of fat/l, 0.259 g of protein/l, and 0.048 g of dry matter/100 g. The estimation precision of the instrument is therefore ± 0.80 g/l (± 2 x 0.400 g/l) for fat, ± 0.52 g/l (± 2 x 0.259 g/l) for true protein and ± 0.096 g/100 g (± 2 x 0.048 g/100 g) for dry matter.

- for whey:

The residual standard deviations of linear regression obtained are equal to 0.004 g of fat/100 g, 0.033 g of total nitrogen/100 g, and 0.025 g of dry matter/100 g. The estimation precision of the instrument is therefore ± 0.008 g/100 g (± 2 x 0.004 g/100 g) for fat, ± 0.066 g/100 g (± 2 x 0.033 g/100 g) for total nitrogen and ± 0.050 g/100 g (± 2 x 0.025 g/100g) for dry matter.

- for cream:

The residual standard deviations of linear regression obtained are equal to 0.215 g of fat/100 g and 0.148 g of dry matter/100 g. The estimation precision of the instrument is therefore ± 0.43 g/100 g (± 2 x 0.215 g/100 g) for fat, and ± 0.30 g/100 g (± 2 x 0.148 g/100g) for dry matter.

4. CONCLUSION

We can conclude that the short-term stability of the instrument is in accordance with the requirements of the ISO 8196-3|IDF 128-3 standard.

Concerning the repeatability of the instrument, the results for fat and true protein in milk are in conformity with limits of the ISO 9622IDF 141 standard. For the other products and criteria, the results obtained are in accordance with the recommendations of the ISO 8196-3|IDF 128-3 standard, or lower or near to the repeatability limits of the corresponding reference method.

Concerning the precision, no standardised requirements exist for the products tested during this evaluation (tank raw milk, ewe milk, whey and cream).

According to the evaluation report of the MilkoScan™ Mars – M. ESTEVES, A. OUDOTTE and Ph. TROSSAT – May-September 2020

ARTICLE

EVALUATION OF THE FOSS MILKOSCAN™ FT3 INFRARED ANALYSER

The MilkoScan™ FT3 is a FTIR mid infrared analyser manufactured by Foss Analytical A/S (Danmark) and commercialised in France by Foss France SAS. It is used for the determination of the major and minor composition components in liquid and semi-solid dairy products, such as milk, cream, whey, retentate, chocolated milk…

The instrument is computer controlled with the Nova™ software, which ensures the signal treatment. A mini screen allows the start of a measurement without going through the computer. The control of the humidity in the optical unit is achieved through a patented automatic drying system. The « zero » measurement and the standardisation are simultaneously and automatically realised. The cleaning is defined according to the properties of the programed matrixes. The fluidic system has been designed to handle the analysis of a full range of dairy products, from milk to thick and viscous products like yogurt or chocolate milk with undissolved particles. However, this means that for each product to measure, some settings are required by selecting one or more special features in the software (sample dilution, viscosity, blend).
Within the context of the evaluation, no dilution analysis was performed.

The prediction models can be performed via the « adjustment » program which is designed as a calibration assistant. The calculations are offered directly by the assistant.

The instrument used in this study was:

- MilkoScan™ FT3
- Serial number: 91840178
- Part: 60062098
- Environnement Windows: Windows 10 Pro
- Software: Nova™ MilkoScan™ FT3

Prediction models for many matrixes are available in the instrument.

The instrument was installed by Foss in a temperature controlled room (20-23 °C – air conditioning), without direct sunlight.

A cleaning solution [Solution Msc W-960 Cleaning Agent (24 g Msc W-960 in 5 l of demineralised water ISO 3696 grade 3)] and a zero solution [Solution Msc Zero (5 ml Msc Zero in 5 l of demineralised water ISO 3696 grade 3] are required. The instrument automatically controls the use of solutions: a message appears on the screen when the contenairs are empty and have to be filled.

LES TESTS

The evaluation tests were performed in ACTALIA Cecalait's physico-chemistry laboratory from May to September 2020. After preliminary tests of stability, the repeatability and accuracy on tank raw cow milk, tank raw ewe milk, cream, whey and retentate for fat, true protein, dry matter and total nitrogen were evaluated.

The accuracy of the instrument was evaluated according to the following standardised methods:

- Fat in milk:	Gerber method according to ISO 19662|IDF 238
- Fat in ewe milk:	Acido-butyrometric method according to NF V04-155
- Fat in cream:	Röse-Gottlieb method according to ISO 2450|IDF 16
- Fat in whey :	Röse-Gottlieb method according to ISO 1211|IDF 1
- Protein in milk:	Amido black method according to NF V04-216
- Total nitrogen:	Kjeldahl method according to ISO 8968-1|IDF 20-1
- Dry matter:	Oven method according to ISO 6731|IDF 21

1. EVALUATION OF THE SHORT-TERM REPRODUCTIBILITY

The short-term reproducibility was evaluated by analysing 4 samples of tank raw cow milk with preservative (Bronopol 0.02 % final), with different concentration levels of fat and protein in triplicate, every 15-20 minutes to obtain at least 20 sequences.

To evaluate the stability of the instrument, the repeatability and reproducibility were calculated by level.

	Level 1	Level 2	Level 3	Level 4
Fat (g/l)	22	38	54	75
Protein (g/l)	24	35	39	55

Table 2: Content of the samples used for the short-term reproducibility evaluation

Le tableau suivant présente les résultats obtenus :

		M	Sr	Sr(%)	SR	SR(%)	r	R
Fat (g/l)	**Level 1**	22.121	0.0507	0.229%	0.0693	0.313%	0.141	0.192
	Level 2	38.079	0.0565	0.148%	0.0849	0.223%	0.157	0.235
	Level 3	53.858	0.0611	0.114%	0.0970	0.180%	0.169	0.269
	Level 4	77.526	0.0703	0.091%	0.1039	0.134%	0.195	0.288
True protein (g/l)	**Level 1**	25.548	0.0665	0.271%	0.0792	0.323%	0.184	0.219
	Level 2	31.693	0.0410	0.129%	0.0546	0.172%	0.114	0.151
	Level 3	39.061	0.0657	0.168%	0.0861	0.221%	0.182	0.239
	Level 4	54.343	0.0843	0.155%	0.1019	0.188%	0.233	0.282
Dry matter (g/l)	**Level 1**	10.319	0.0171	0.166%	0.0193	0.187%	0.047	0.054
	Level 2	12.490	0.0071	0.057%	0.0092	0.074%	0.020	0.025
	Level 3	14.640	0.0101	0.069%	0.0133	0.091%	0.028	0.037
	Level 4	18.252	0.0114	0.062%	0.0177	0.097%	0.032	0.049

Table 2: MilkoScan™ FT3 stability criteria for fat, true protein and dry matter [4]

The results for levels from 1 to 3 indicate that the standard deviation of repeatability for fat and protein are below the limits required in ISO 8196-3|IDF 128-3 standard for milk with an average fat and protein content (0.28 g/l). For milk with a high fat and protein content (level 4), the results indicate that the standard deviation of repeatability for fat and protein are below the limits required in ISO 8196-3|IDF 128-3 standard for milk with a high fat and protein content (0.56 g/l).

As no standardised value exists for dry matter, it can be noted that the reproducibility of the instrument is lower than the reproducibility of the reference value (0.20 g/100 g).

2. EVALUATION OF THE REPEATABILITY

The repeatability of the instrument was performed by analysing:

- for tank raw cow milk: 39 samples of tank raw milk from a French plant (West of France).
- for ewe milk: 33 samples of ewe milk from a French plant (South-West of France).
- for whey: 24 samples of skimmed whey from a French plant (East-Center of France).
- for cream: 24 samples of cream from a French plant (West of France).
- for retentate: 20 samples of proteic milk retentate. 5 samples of retentate from a French plant (West of France) are reconstituted with skimmed milk to obtain a range of 10 to 15 g of dry matter/100 g.

Bronopol was added to the samples to give a final concentration of 0.02 %. They were analysed (after heating at 40 ± 2 °C and 37 ± 2 °C for cream) in non consecutive duplicate according to the following sequence: Set 1 rep 1 – Set 2 rep 2 - … - Set n rep n.

The repeatability was calculted using results in duplicate obtained with all the data and for the criteria:

- Fat for all the matrixes except retentate,
- Dry matter for all the matrixes,
- Protein for tank raw milk and ewe milk,
- Total nitrogen for whey and retentate.

[4] M: mean; S_r and S_R (S_r% andt S_R%): absolute (and relative) standard deviation of repeatability and reproductibility; r and R: maximum deviation of repeatability and reproductibility in 95 % of cases.

The following table presents the results obtained:

		n	min	max	M	S_r	S_r%	r
Tank raw cow milk	**Fat (g/l)**	39	37.43	41.39	39.85	0.064	0.16%	0.177
	Protein (g/l)	39	31.18	33.46	32.25	0.051	0.16%	0.141
	Dry matter (g/100g)	39	12.46	12.91	12.66	0.009	0.07%	0.026
Ewe milk	**Fat (g/l)**	33	72.15	85.60	80.57	0.084	0.10%	0.232
	Protein (g/l)	33	55.94	64.99	61.03	0.083	0.14%	0.230
	Dry matter (g/100g)	33	18.19	19.77	19.25	0.014	0.07%	0.039
Whey	**Fat (g/100g)**	24	0.034	0.055	0.040	0.001	3.09%	0.003
	Total nitrogen (g/100g)	24	0.60	0.92	0.87	0.004	0.50%	0.012
	Dry matter (g/100g)	24	5.01	6.56	6.26	0.006	0.10%	0.018
Cream	**Fat (g/100 g)**	24	40.81	41.76	41.42	0.072	0.17%	0.200
	Dry matter (g/100g)	24	46.08	46.85	46.61	0.067	0.14%	0.186
Retentate	**Total nitrogen (g/100 g)**	20	4.60	9.01	7.03	0.009	0.13%	0.025
	Matière sèche (g/100g)	20	10.60	14.45	12.71	0.009	0.07%	0.025

Table 3: MilkoScan™ FT3 repeatability criteria for fat, true protein, dry matter and total nitrogen in tank raw cow milk, ewe milk, whey, cream and retentate[5]

It can be noted that:

- for tank raw cow milk: for fat and true protein content, the standard deviations of repeatability are lower than the requirements of the ISO 8196-3|IDF 128-3 and ISO 9622|IDF 141 standards (Sr < 0.14 g/l). For dry matter, the standard deviation of repeatability is in the same order that the results for the other components and lower than the repeatability standard deviation of the reference method (Sr = 0.036 g/100 g).

- for ewe milk: for fat and true protein content, the standard deviations of repeatability are lower than the requirements of the ISO 8196-3|IDF 128-3 standard (Sr < 0.28 g/l). For dry matter, the standard deviation of repeatability is in the same order that the results for the other components and lower than the repeatability standard deviation of the reference method (Sr = 0.036 g/100 g).

- for whey: the standard deviation of repeatability is equivalent for all the criteria. As no standardised value exists for whey, it can be noted that the standard deviations of repeatability obtained with the instrument are lower or near of those obtained with the reference methods when they exist (Sr = 0.001 g/100g vs 0.013 g/100g for fat; 0.006 g/100g vs 0.036 g/100g for dry matter).

- for cream: the standard deviation of repeatability is equivalent for all the criteria. For fat, the relative standard deviation (Sr%) is in accordance with the recommendations of the ISO 8196-3|IDF 128-3 standard for milk with high content (Sr% < 0.35 %).
As no standardised value exists for whey, it can be noted that the standard deviation of repeatability obtained with the instrument is lower or near of those obtained with the reference methods (Sr = 0.067 g/100 g vs 0.072 g/100 g).

- for retentate: the standard deviation of repeatability is equivalent for all the criteria. For total nitrogen, the relative standard deviation (Sr%) is in accordance with the recommendations of the ISO 8196-3|IDF 128-3 standard for milk with high content (Sr% < 0,40% for protein).
As no standardised value exists for dry matter, it can be noted that the standard deviations of repeatability obtained with the instrument are lower or near of those obtained with the reference methods (Sr = 0.025 g/100g vs 0.108 g/100g).

3. EVALUATION OF THE ACCURACY

The accuracy of the instrument was evaluated by using the same samples than those used for the repeatablity evaluation. The mean of the duplicates of the results obtained in the repeatability evaluation was used for the calculation of the results. Outliers samples (samples whose regression residues are greater than 2 times the standard deviation of deviations: P at 5 %) have been discarded.

The following table presents the results obtained:

[5] N: number of the results; min and max: minimum and maximum values; M : mean of the results; Sr (Sr%): absolute (and relative) standard deviation; r: maximum deviation of repeatability in 95 % of cases

		n	min	max	X	Sx	Sy,x	Sy,x%	b	a
Tank raw cow milk	Fat (g/l)	36	37.47	41.32	39.79	0.83	0.304	0.77%	0.963	0.987
	True protein (g/l)	37	31.24	33.45	32.26	0.46	0.092	0.28%	0.903	3.166
	Dry matter (g/100 g)	37	12.46	12.90	12.66	0.12	0.031	0.24%	0.855	1.808
Ewe milk	Fat (g/l)	33	72.21	85.57	80.57	3.36	0.463	0.57%	1.017	-1.231
	True protein (g/l)	30	56.01	64.95	61.24	2.09	0.207	0.34%	1.091	-6.294
	Dry matter (g/100 g)	31	18.20	19.76	19.25	0.43	0.033	0.17%	1.019	-0.406
Whey	Fat (g/100g)	21	0.036	0.054	0.040	0.005	0.006	16.78%	0.466	0.015
	Total nitrogen (g/100g)	22	0.61	0.92	0.87	0.08	0.033	3.91%	0.879	0.087
	Dry matter (g/100 g)	22	5.02	6.56	6.26	0.36	0.023	0.36%	0.922	0.455
Cream	Fat (g/100 g)	22	40.99	41.69	41.41	0.16	0.183	0.44%	0.884	0.986
	Dry matter (g/100 g)	23	46.33	46.84	46.62	0.13	0.104	0.22%	0.986	1.147
Retentate	Total nitrogen (g/100g)	19	4.60	9.01	7.00	1.44	0.015	0.20%	1.064	0.205
	Dry matter (g/100 g)	18	11.00	14.44	12.85	1.18	0.039	0.28%	1.159	-1.040

Table 4: MilkoScan™ FT3 accuracy criteria for fat, true protein, dry matter and total nitrogen in tank raw cow milk, ewe milk, whey , cream and retentate [6]

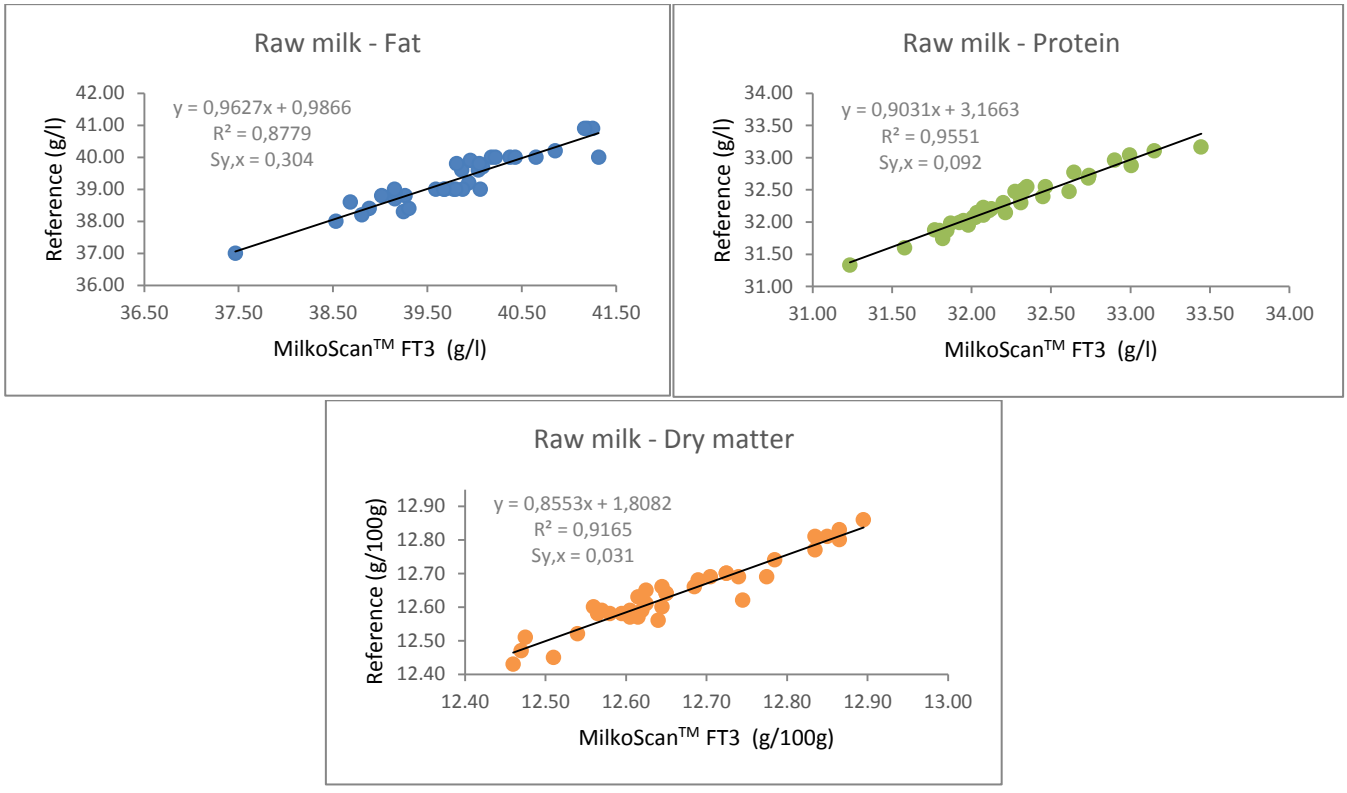
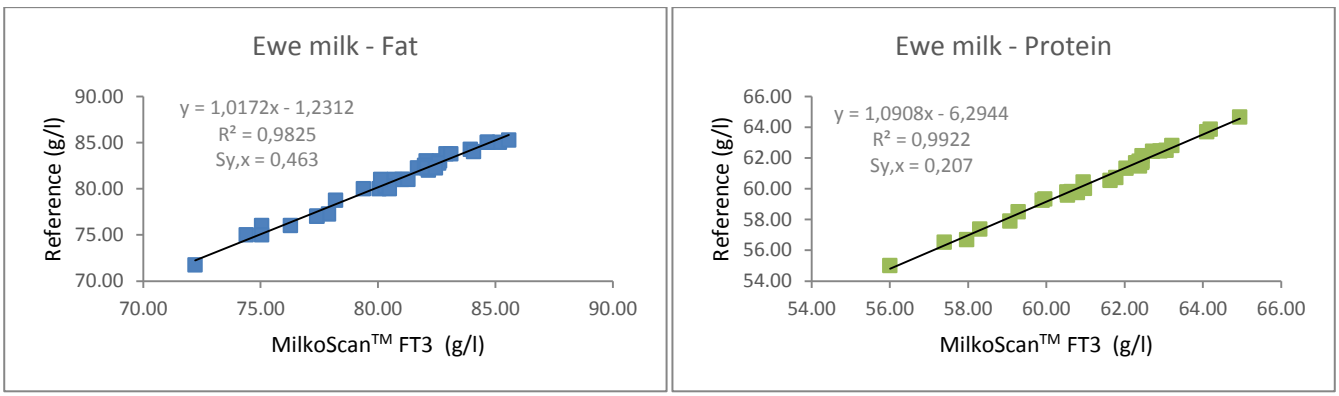


Figure 1: Relation between MilkoScan™ FT3 and reference results for fat, true protein and dry matter in tank raw cow milk



[6] n, min, max: number of results, minimum and maximum values; Y: mean of the 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 (Sy,x%): absolute (and relative) residual standard deviation; b,a: slope and intercept of the linear regression.

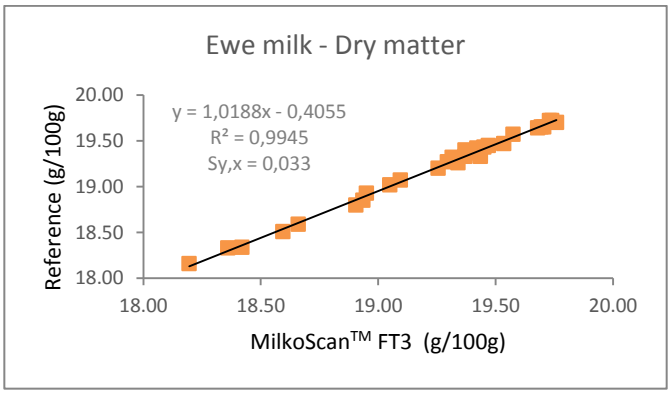


Figure 2: Relation between MilkoScan™ FT3 and reference results for fat, true protein and dry matter in ewe milk

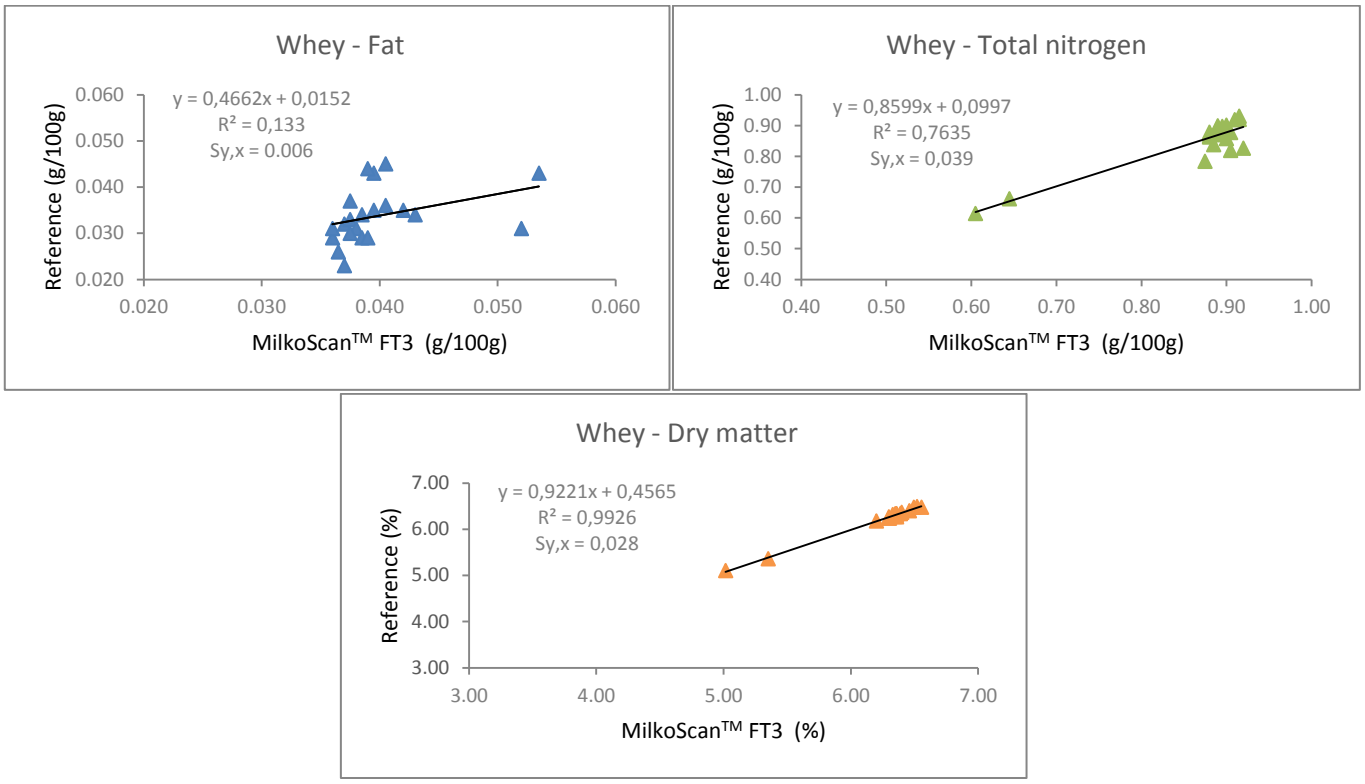


Figure 3: Relation between MilkoScan™ FT3 and reference results for fat, total nitrogen and dry matter in whey

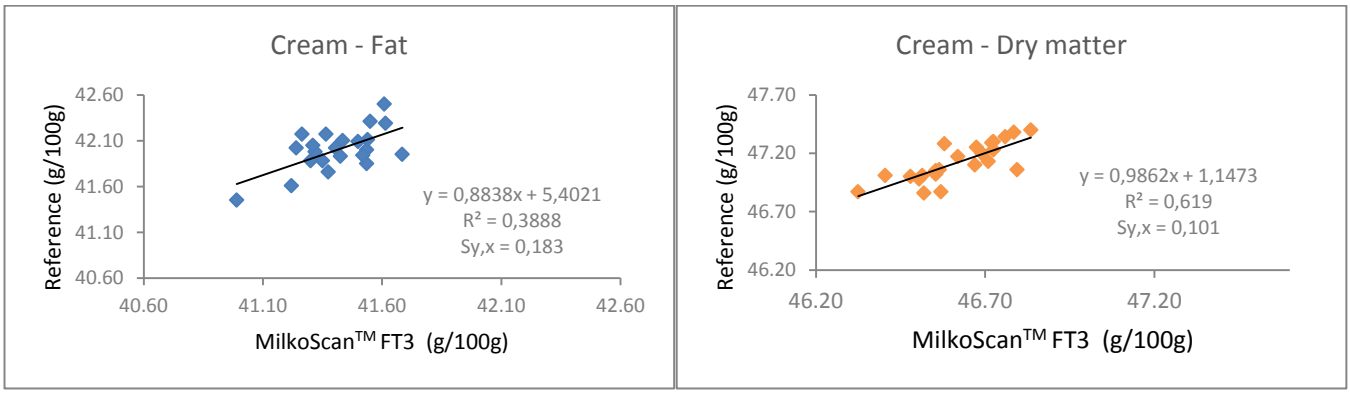


Figure 4: Relation between MilkoScan™ FT3 and reference results for fat and dry matter in cream

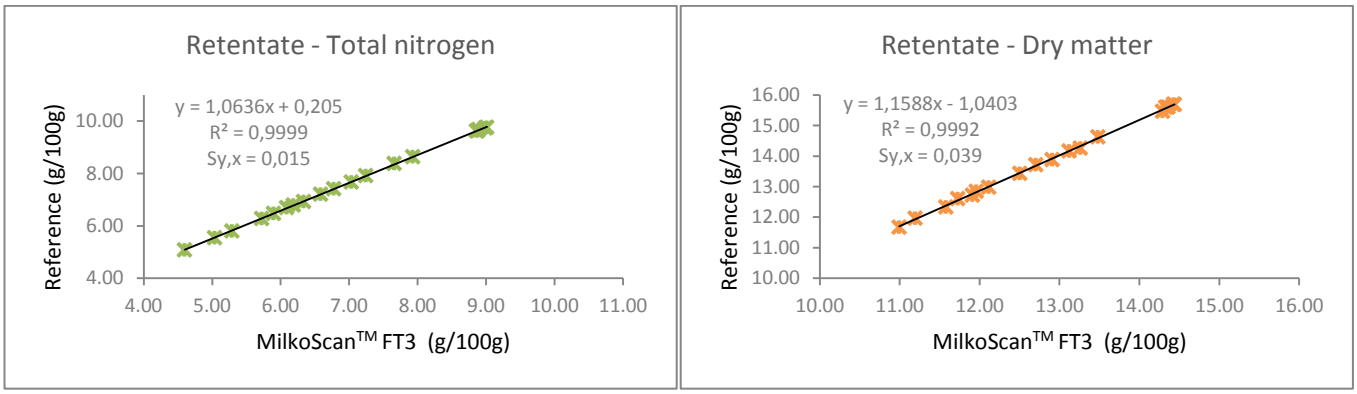


Figure 5: Relation between MilkoScan™ FT3 and reference results for total nitrogen and dry matter in retentate

Concerning the relation between MilkoScan™ FT3 and the reference results, it can be noted that:

- for tank raw cownmilk:

The residual standard deviations of linear regression obtained are equal to 0.304 g of fat/l, 0.092 g of protein/l, and 0.031 g of dry matter/100 g. The estimation precision of the instrument is therefore ± 0.61 g/l (± 2 x 0.304 g/l) for fat, ± 0.18 g/l (± 2 x 0.092 g/l) for true protein and ± 0.062 g/100 g (± 2 x 0.031 g/100 g) for dry matter.

- for ewe milk:

The residual standard deviations of linear regression obtained are equal to 0.463 g of fat/l, 0.207 g of protein/l, and 0.033 g of dry matter/100 g. The estimation precision of the instrument is therefore ± 0.93 g/l (± 2 x 0.463 g/l) for fat, ± 0.41 g/l (± 2 x 0.207 g/l) for true protein and ± 0.066 g/100 g (± 2 x 0.033 g/100 g) for dry matter.

- for whey:

The residual standard deviations of linear regression obtained are equal to 0.006 g of fat/100 g, 0.033 g of total nitrogen/100 g, and 0.023 g of dry matter/100 g. The estimation precision of the instrument is therefore ± 0.012 g/100 g (± 2 x 0.006 g/100 g) for fat, ± 0.066 g/100 g (± 2 x 0.033 g/100 g) for total nitrogen and ± 0.046 g/100 g (± 2 x 0.023 g/100g) for dry matter.

- for cream:

The residual standard deviations of linear regression obtained are equal to 0.183 g of fat/100 g and 0.104 g of dry matter/100 g. The estimation precision of the instrument is therefore ± 0.37 g/100 g (± 2 x 0,183 g/100 g) for fat and ± 0.21 g/100 g (± 2 x 0.104 g/100g) for dry matter.

- for retentate:

The residual standard deviations of linear regression obtained are equal to 0.015 g of total nitrogen/100 g and 0,039 g of dry matter/100 g. The estimation precision of the instrument is therefore ± 0.030 g/100 g (± 2 x 0.015 g/100 g) for total nitrogen and ± 0.078 g/100 g (± 2 x 0.039 g/100g) for dry matter.

4. CONCLUSION

We can conclude that the short-term stability of the instrument is in accordance with the requirements of the ISO 8196-3|IDF 128-3 standard.

Concerning the repeatability of the instrument, the results for fat and true protein in milk are in conformity with limits of the ISO 9622IDF 141 standard. For the other products and criteria, the results obtained are in accordance with the recommendations of the ISO 8196-3|IDF 128-3 standard, or lower or near to the repeatability limits of the corresponding reference method.

Concerning the precision, no standardised requirements exist for the products tested during this evaluation (tank raw milk, ewe milk, whey and cream).

According to the evaluation report of the MilkoScan™ FT3 – M. ESTEVES, A. OUDOTTE and Ph. TROSSAT – May-September 2020

STANDARDS, DRAFT STANDARDS

Classification in alphabetical order by theme

ISO published standards

INFANT FORMULA	
ISO 22579 (IDF 241) September 2020	INFANT FORMULA AND ADULT NUTRITIONALS Determination of fructans – High perfromance anion exchange chromatography with pulsed amperometric detection (HPAEC-PAD) after enzymatic treatment

NEW EU REGULATIONS

Classification is established in alphabetical order of the first keyword

NOVEL FOOD

O.J.E.U. L 357, 27th October 2020 – Commission Implementing Regulation (EU) 2020/1559 of 26 October 2020 amending Implementing Regulation (EU) 2017/2470 establishing the Union list of novel foods
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2020.357.01.0007.01.ENG

O.J.E.U. L 406, 3rd December 2020 – Commission Implementing Regulation (EU) 2020/1820 of 2 December 2020 authorising the placing on the market of dried *Euglena gracilis* as a novel food under Regulation (EU) 2015/2283 of the European Parliament and of the Council and amending Commission Implementing Regulation (EU) 2017/2470
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2020.406.01.0029.01.ENG

PESTICIDES

O.J.E.U. L 358, 28th October 2020 – Commission Regulation (EU) 2020/1566 of 27 October 2020 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 bupirimate, carfentrazone-ethyl, ethirimol, and pyriofenone in or on certain products
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2020.358.01.0030.01.ENG

O.J.E.U. L 367, 5th November 2020 – Commission Regulation (EU) 2020/1633 of 27 October 2020 amending Annexes 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 azinphos-methyl, bentazone, dimethomorph, fludioxonil, flufenoxuron, oxadiazon, phosalone, pyraclostrobin, repellants: tall oil and teflubenzuron in or on certain products
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2020.367.01.0001.01.ENG

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

O.J.E.U. C 347, 19th October 2020 – Publication of the amended single document following the approval of a minor amendment pursuant to the second subparagraph of Article 53(2) of Regulation (EU) No 1151/2012 [Queso los Beyos (cheese) (PGI)]
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.C_.2020.347.01.0016.01.ENG

O.J.E.U. L 353, 23th October 2020 – Commission Implementing Regulation (EU) 2020/1539 of 16 October 2020 approving non-minor amendments to the specification for a name entered in the register of protected designations of origin and protected geographical indications [Sainte-Maure de Touraine (cheese) (PDO)]
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2020.353.01.0003.01.ENG

O.J.E.U. L 386, 18th November 2020 – Commission Implementing Regulation (EU) 2020/1719 of 11 November 2020 approving non- minor amendments to the specification for a name entered in the register of protected designations of origin and protected geographical indications [Casciotta d'Urbino (cheese) (PDO)]
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2020.386.01.0005.01.ENG

O.J.E.U. C 408, 27th November 2020 – Publication of an application for approval of an amendment, which is not minor, to a product specification 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 [Burrata di Andria (cheese) (PGI)]
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.C_.2020.408.01.0009.01.ENG

O.J.E.U. C 424, 8th December 2020 – Publication of an application for approval of an amendment, which is not minor, to a product specification 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 [Mozzarella (cheese) (TGS)]
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.C_.2020.424.01.0039.01.ENG

STANDARDS - REGULATIONS

O.J.E.U. L 415, 10th December 2020 – Commission Implementing Regulation (EU) 2020/2018 of 9 December 2020 entering a name in the register of protected designations of origin and protected geographical indications [Mozzarella di Gioia del Colle (cheese) (PDO)]

http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2020.415.01.0046.01.ENG

O.J.E.U. L 431, 21st December 2020 – Commission Implementing Regulation (EU) 2020/2158 of 14 December 2020 approving non- minor amendments to the specification for a name entered in the register of protected designations of origin and protected geographical indications [Chabichou du Poitou (cheese) (PDO)]

http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2020.431.01.0032.01.ENG

O.J.E.U. L 433, 22nd December 2020 – Commission Implementing Regulation (EU) 2020/2178 of 15 December 2020 correcting Implementing Regulation (EU) 2020/1433 approving non-minor amendments to the specification for a name entered in the register of protected designations of origin and protected geographical indications [Pouligny-Saint-Pierre (cheese) (PDO)]

http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2020.433.01.0031.01.ENG

PHARMACOLOGICALLY ACTIVE SUBSTANCES

O.J.E.U. L 384, 17th November 2020 – Commission Implementing Regulation (EU) 2020/1712 of 16 November 2020 amending Regulation (EU) No 37/2010 to classify the substance lidocaine as regards its maximum residue limit

http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2020.384.01.0003.01.ENG

AFNOR VALIDATIONS

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

Commercial name	Date	Certificate	Description
NEW VALIDATIONS			
THERMO SCIENTIFIC™ SURETECT™ STEC SCREENING PCR ASSAY AND THERMO SCIENTIFIC™ SURETECT™ STEC IDENTIFICATION PCR ASSAY	Validation date: 9 Nov 2020 **End of validity: 9 Nov 2024**	**UNI-03/13-10/20**	**Detection of shiga-toxin-producing *Escherichia coli* (STEC)** Raw meats (excluding poultry meats), dairy products and vegetables
IDEXX SNAP DUO ST PLUS TEST	Validation date: 2 Oct 2020 **End of validity: 2 Oct 2024**	**IDX-33/08-10/20**	**Detection of antibiotics** Raw cow's milk and raw commingled cow's milk
RENEWALS OF VALIDATIONS			
3M™ PETRIFILM™ RAPID AEROBIC COUNT PLATE	Validation date: 25 Nov 2016 Renewal: 1 Oct .2020 **End of validity: 25 Nov 2024**	**3M-01/17-11/16**	**Enumeration of mesophilic aerobic flora** Milk powders and dairy products
3M™ MOLECULAR DETECTION ASSAY 2 - *SALMONELLA*	Validation date: 25 Nov 2016 Renewal: 2 Oct .2020 Extension: 22 Mar 2019 **End of validity: 25 Nov 2024**	**3M-01/16-11/16**	**Detection of *Salmonella*** All human food products, pet food and animal feed and industrial production environmental samples and primary production samples
TRANSIA PLATE *SALMONELLA* GOLD	Validation date: 23 Mar 2001 Renewal: 3 Feb 2005, 2 Jul 2009, 29 Nov 2012, 25 Jan 2017 and 1 Oct.2020 Extension: 12 May 2021 and 18 Mar 2016 **End of validity: 3 Feb 2025**	**TRA-02/08-03/01**	**Detection of *Salmonella* spp.** All human and animal food products and production environmental samples (except primary production environment)
ASSURANCE GDS *SALMONELLA*	Validation date: 26 Jan 2009 Renewal: 29 Nov 2012, 25 Jan 2018 and 1 Oct 2020 Extension: 2 Jul 2018 **End of validity: 26 Jan 2025**	**TRA-02/12-01/09**	**Detection of *Salmonella* spp.** All human food products (except sprouts), pet food and production environmental samples (except primary production environment)
RAPID'*E. COLI* 2	Validation date: 19 Nov 1997 Renewal: 7 Mar 2002, 2 Dec 2004, 28 Nov 2008, 29 Nov 2012, 22 Nov 2017 and 2 Oct 2020 **End of validity: 2 Dec 2024**	**BRD-07/01-07/93**	**Enumeration at 44 °C of β-glucuronidase positive *E. coli*** All human food products
RAPID'*E. COLI* 2	Validation date: 2 Dec 2004 Renewal: 28 Nov 2008, 29 Nov 2012, 22 Nov 2017 and 2 Oct 2020 **End of validity: 2 Dec 2024**	**BRD-07/07-12/04**	**Enumeration at 37 °C of β-glucuronidase positive *E. coli*** All human food products

AFNOR VALIDATIONS

RAPID'*E. COLI* 2	Validation date: 2 Dec 2004 Renewal: 28 Nov 2008, 29 Nov 2012, 22 Nov 2017 and 2 Oct 2020 **End of validity: 2 Dec 2024**	**BRD-07/08-12/04**	**Enumeration at 37 °C of coliforms** All human food products
GENE-UP *LISTERIA MONOCYTOGENES*	Validation date: 24 Nov 2016 Renewal: 1 Oct 2020 Extension: 27 Jan 2014, 3 Jul 2017 and 4 Dec 2018 **End of validity: 24 Nov 2024**	**BIO-12/40-11/06**	**Detection of *Listeria monocytogenes*** All human food products and production environmental samples
EXTENSIONS OF VALIDATIONS			
***SALMONELLA* PRECIS™**	Validation date: 4 Dec 2007 Renewal: 6 Oct 2011, 6 Jul 2015 and 30 Jan 2020 Extension: 1 Oct 2020 **End of validity: 4 Dec 2023**	**UNI-03/06-12/07**	**Detection of *Salmonella* spp.** All human and animal food products and production environmental samples (except primary production environment)
THERMO SCIENTIFIC SURETECT *SALMONELLA* SPECIES PCR ASSAY	Validation date: 4 Nov 2013 Renewal: 22 Mar 2018 Extension: 30 Jan 2014, 21 Mar 2014, 30 Jun 2016, 24 Mar 2017, 3 Dec 2018, 16 May 2019, 2 Jul 2020 and 1 Oct 2020 **End of validity: 4 Nov 2021**	**UNI-03/07-11/13**	**Detection of *Salmonella* spp.** All human food products, pet food and production environmental samples (except primary production environment)
RAPID'*SALMONELLA*	Validation date: 9 Dec 2005 Renewal: 24 Sep 2009, 29 Nov 2013 & 22 Mar 2018 Extension: 3 Jul 2009, 21 May 2010, 3 Feb 2011, 4 Oct 2012, 6 Jul 2015, 22 Mar 2018 & 1 Oct 2020 **End of validity: 9 Dec 2021**	**BRD-07/11-12/05**	**Detection of *Salmonella* spp.** All human and animal food products, and industrial production environmental samples
EXTENSION OF VALIDITY			
DELVOTEST®T	Validation date: 3 Feb 2012 Renewal: 28 Jan .2016 **End of validity: 3 Feb 2020** **Validity extended till: 16 Feb 2021**	**DSM-28/02-02/12**	**Detection des antibiotics** Cow, goat and sheep milk (with or without azidiol)

The validation certificates and the recapitulative list are available at the following website address:
http://www.afnor-validation.com/afnor-validation-validated-methods/validated-methods.html

IN THE PRESS – ON THE WEB

Classification in alphabetical order of keywords

FOOD ADDITIVES

Opinion on the re-evaluation of lecithins (E 322) as a food additive in foods for infants below 16 weeks of age and follow-up of its re-evaluation as food additive for uses in foods for all population groups

https://efsa.onlinelibrary.wiley.com/doi/10.2903/j.efsa.2020.6266

▸ Lecithins (E 322) were re-evaluated in 2017 by the former EFSA Panel on Food Additives and Sources of Nutrients Added to Food (ANS). As part of the follow-up to this assessment, the Scientific Panel on Food Additives and Flavorings (FAF) was invited to assess the safety of lecithins (E 322) used as a food additive in foods intended for infants under 16 weeks. . Based on the information submitted in response to the call for data from the previous assessment, the FAF group considered that it was possible to modify the EU specifications, in particular for the toxic elements arsenic, lead, mercury and to introduce new specifications for cadmium and microbiological criteria. The safety issue identified by the ANS group in 2017 concerned potential neuro-developmental effects. Since choline is a precursor to the neurotransmitter acetylcholine, the Panel considered it appropriate to review the safety of lecithins (E 322) as a food additive in infant formula used in infants under 16 weeks of age. comparing the concentration of choline in breast milk with that in the formula. The Panel concluded that the ingestion of lecithins (E 322) as a food additive in infant formulas does not raise any safety concerns up to the maximum permitted limit.

NOVEL FOOD

Safety of *Schizochytrium* sp. Oil as a novel food pursuant to Regulation (EU) 2015/2283

https://efsa.onlinelibrary.wiley.com/doi/10.2903/j.efsa.2020.6242

▸ The EFSA Panel on Nutrition, Novel Foods and Food Allergens evaluated the safety of oil from *Schizochytrium* sp. as a novel food, in accordance with Regulation (EU) 2015/2283, in infant formulas and follow-on formulas. On the basis of the available data, the scientific panel considers that there are no toxicity problems. The Panel concludes that this new food is safe under the proposed conditions of use.

Safety of lacto-*N*-neotetraose (LNnT) produced by derivative strains of *E. coli* BL 21 as a novel food pursuant to Regulation (EU) 2015/2283

https://efsa.onlinelibrary.wiley.com/doi/10.2903/j.efsa.2020.6305

▸ Following a request from the European Commission, the EFSA Scientific Panel on Nutrition, Novel Foods and Food Allergens (NDA) was asked to deliver an opinion on the change in the production process and specifications of lacto-*N*-neotetraose (LNnT) as a novel food (NF) pursuant to Regulation (EU) 2015/2283. The Panel concludes that lacto-*N*-neotetraose (LNnT) as a novel food when produced by fermentation with two genetically modified strains of *E. coli* BL21 is safe under the proposed conditions of use.

La Lettre de CECALAIT est éditée par ACTALIA Cecalait, B.P. 70129, 39801 POLIGNY CEDEX
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