

EVALUATION OF THE FOSS MILKOSCAN™ FT3 INFRARED ANALYSER

The MilkoScan™ FT3 is a FTIR mid infrared analyser manufactured by Foss Analytical A/S (Denmark) 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, chocolate 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 programmed 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 containers are empty and have to be filled.

LES TESTS

The evaluation tests were performed in ACTALIA Cevalait'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 REPRODUCIBILITY

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 calculated 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% and S_R%): absolute (and relative) standard deviation of repeatability and reproducibility; r and R: maximum deviation of repeatability and reproducibility 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 retentate⁵

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 repeatability 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 retentate⁶

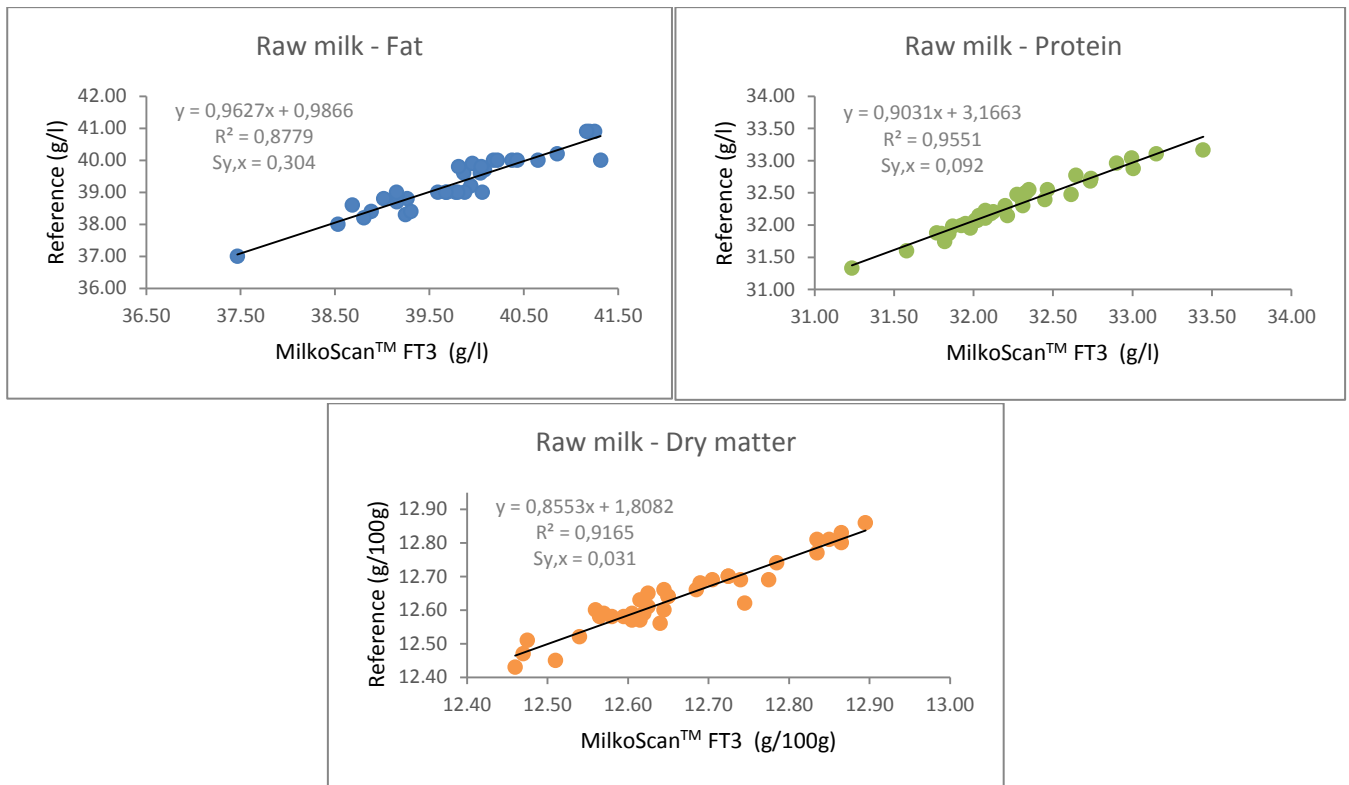
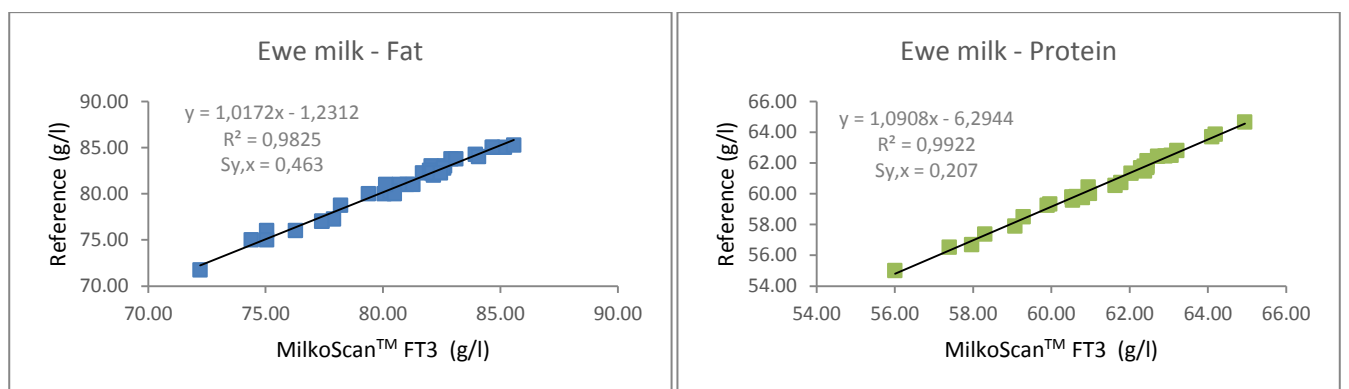


Figure 1: Relation between MilkoScan™ FT3 and reference results for fat, true protein and dry matter in tank raw cow milk



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.

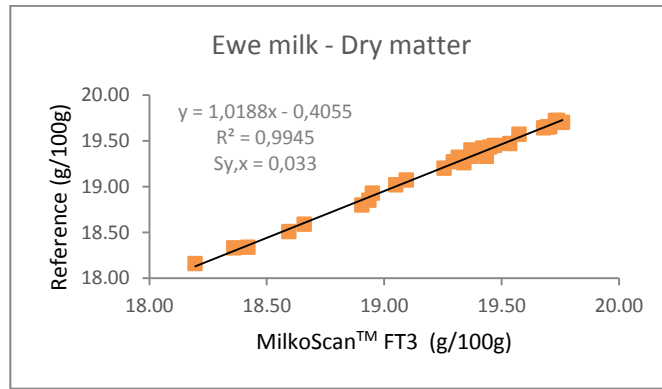


Figure 2: Relation between MilkoScan™ FT3 and reference results for fat, true protein and dry matter in ewe milk

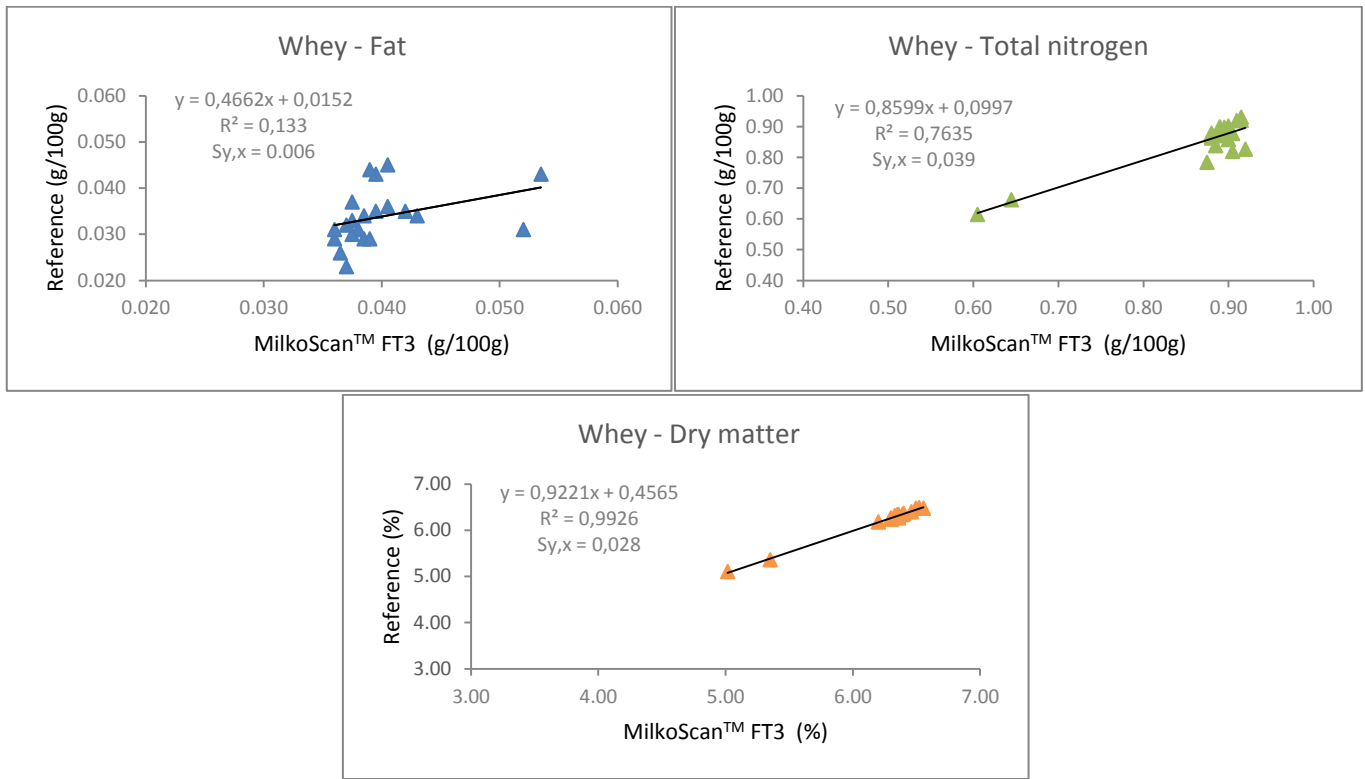


Figure 3: Relation between MilkoScan™ FT3 and reference results for fat, total nitrogen and dry matter in whey

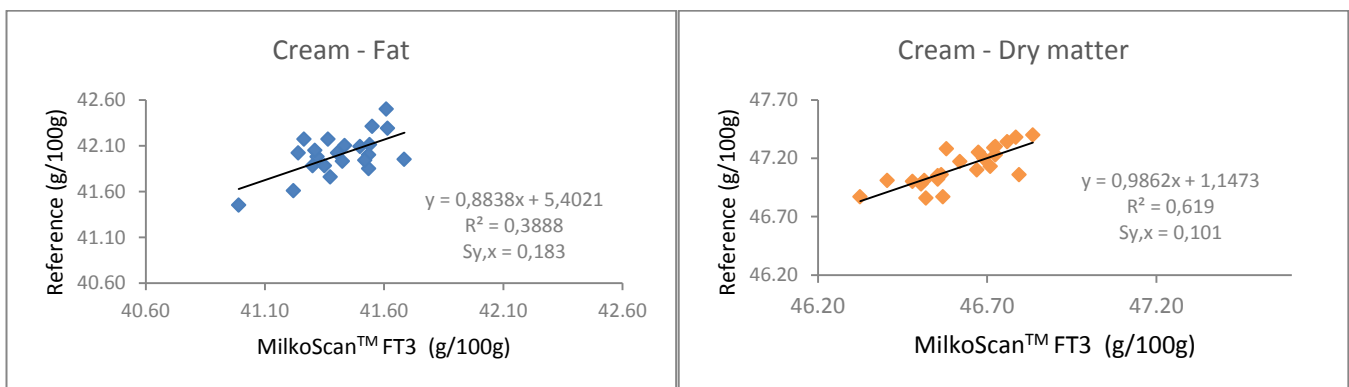


Figure 4: Relation between MilkoScan™ FT3 and reference results for fat and dry matter in cream

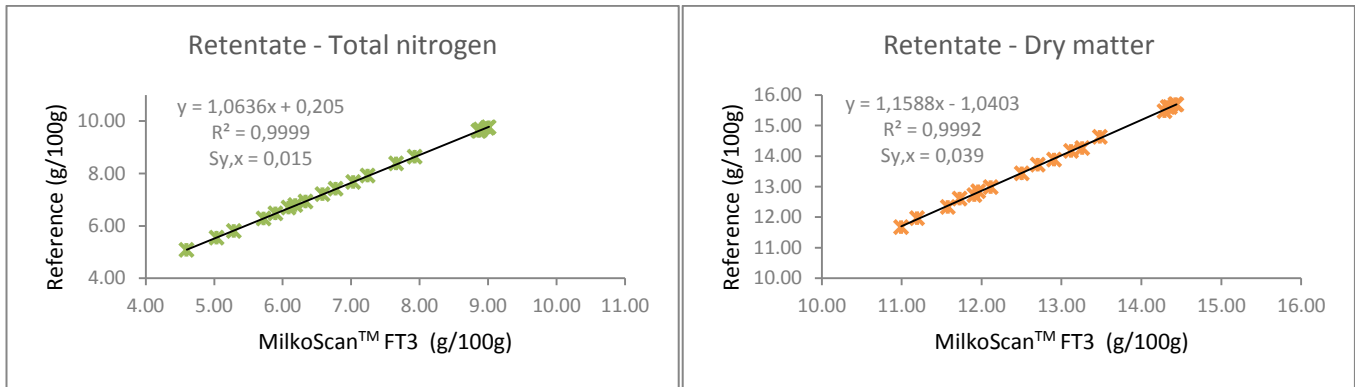


Figure 5: Relation between MilkoScan™ FT3 and reference results for total nitrogen and dry matter in retentate

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

- for tank raw cowmilk:

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 ($\pm 2 \times 0.304$ g/l) for fat, ± 0.18 g/l ($\pm 2 \times 0.092$ g/l) for true protein and ± 0.062 g/100 g ($\pm 2 \times 0.031$ 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 ($\pm 2 \times 0.463$ g/l) for fat, ± 0.41 g/l ($\pm 2 \times 0.207$ g/l) for true protein and ± 0.066 g/100 g ($\pm 2 \times 0.033$ 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 ($\pm 2 \times 0.006$ g/100 g) for fat, ± 0.066 g/100 g ($\pm 2 \times 0.033$ g/100 g) for total nitrogen and ± 0.046 g/100 g ($\pm 2 \times 0.023$ 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 ($\pm 2 \times 0,183$ g/100 g) for fat and ± 0.21 g/100 g ($\pm 2 \times 0.104$ 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 ($\pm 2 \times 0.015$ g/100 g) for total nitrogen and ± 0.078 g/100 g ($\pm 2 \times 0.039$ 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 9622|IDF 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