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.

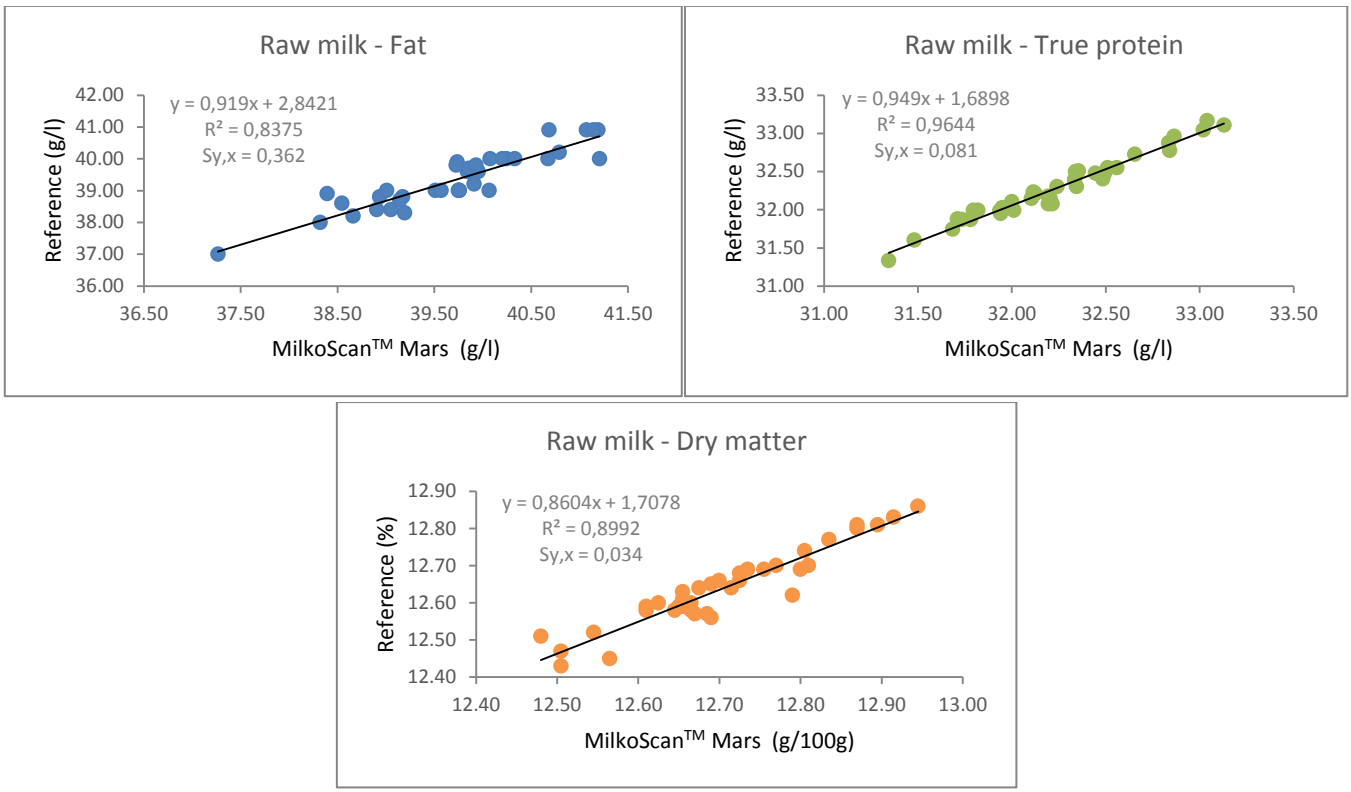

Figure 1: Relation between MilkoScan™ Mars and reference results for fat, true protein and dry matter in tank raw cow milk

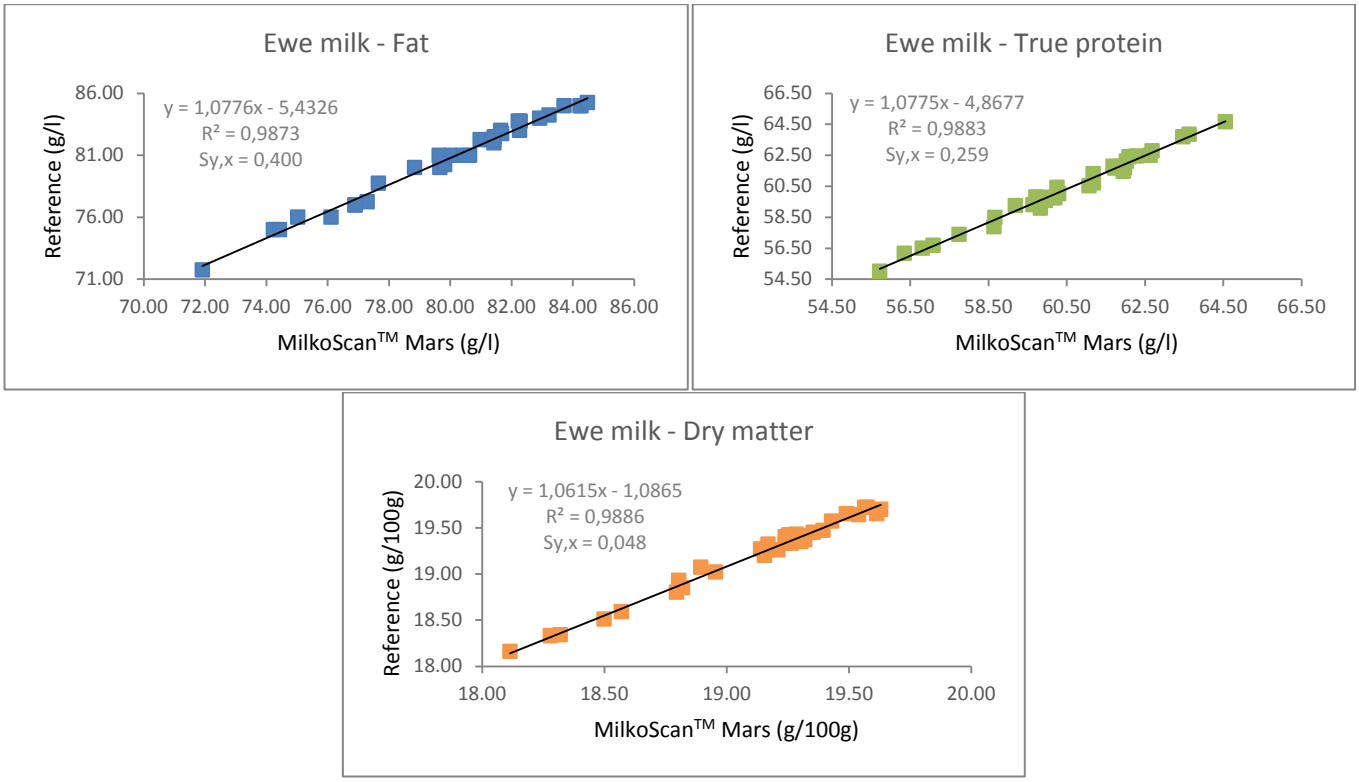

Figure 2: Relation between MilkoScan™ Mars and reference results for fat, true protein and dry matter in ewe milk

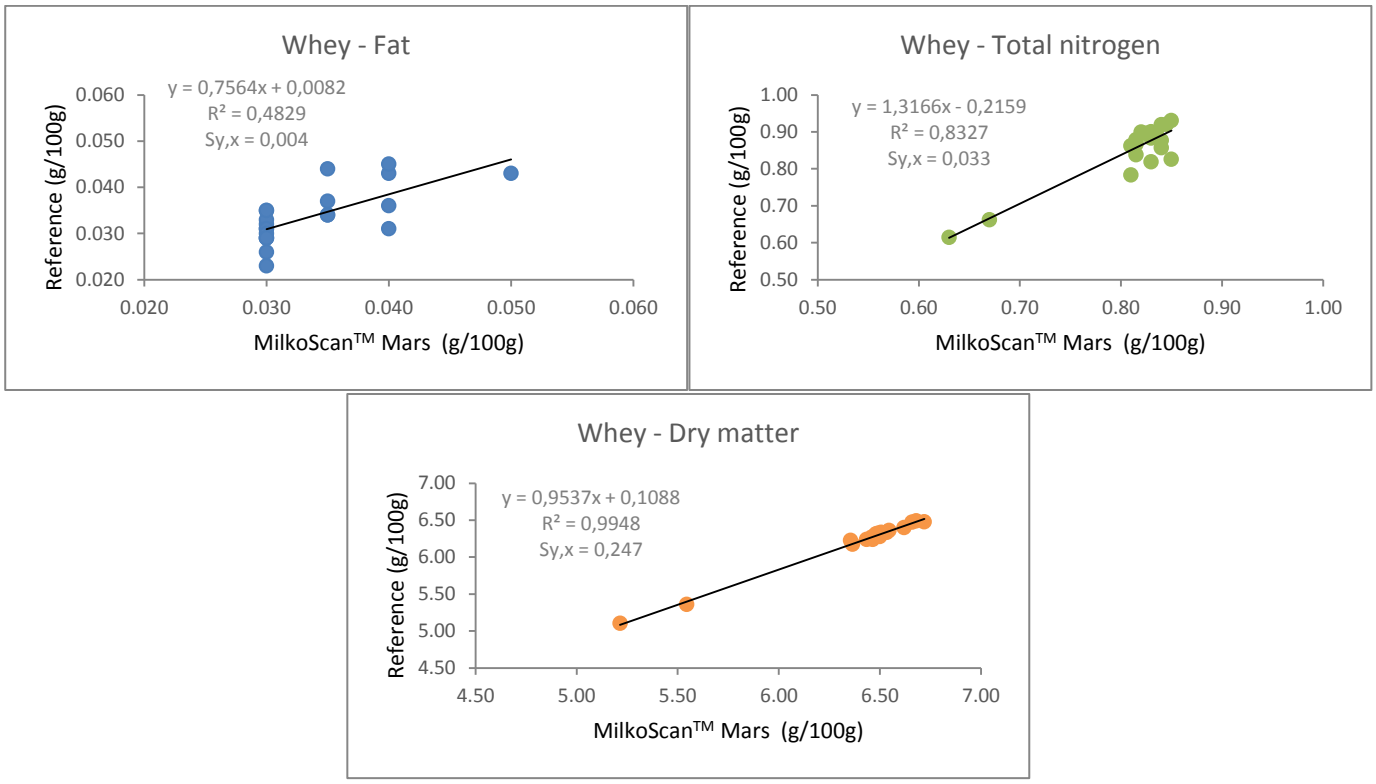

Figure 3: Relation between MilkoScan™ Mars and reference results for fat, total nitrogen and dry matter in whey

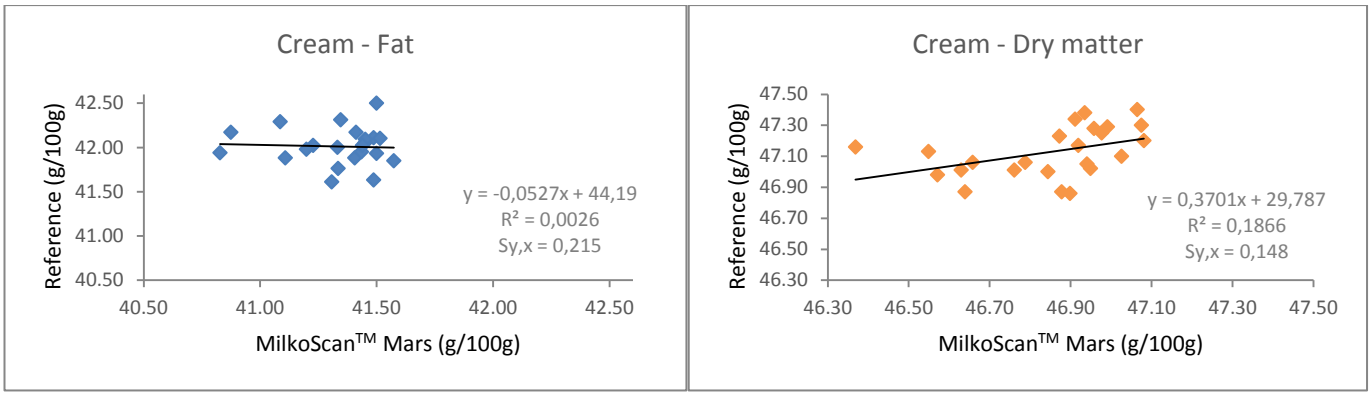

Figure 4: Relation between MilkoScan™ Mars and reference results for fat and dry matter in cream

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