

## EVALUATION OF THE MIRA<sup>®</sup> INFRARED ANALYSER

The MIRA<sup>®</sup> is a spectrophotometer manufactured by Bruker. It is used for the determination of the composition components (fat, dry matter, lactose and optionally freezing point) in liquid dairy products.

The apparatus is computer controlled with the OPUS/LSM2 software, which ensures the signal treatment. Cleaning and zero measurements are completely automated. The spectrophotometer uses a DLaTGS detector type. Several filters can be used for the determination of a product. They present correction factors unequal to zero.

The instrument used in this study was:

- Type of instrument: MIRA<sup>®</sup>
- Internal Bruker part number: 1804289
- Instrument serial number: 100036
- Software version: OPUS 8.2.21



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

Cleaning solution (Decon solution at 4 %) and zero solution (Triton X100 solution at 0.1%) are standard solutions delivered with the instrument and required for the usage.

### THE TESTS

The evaluation tests were performed in ACTALIA Cevalait's physico-chemistry laboratory from September 2019 to February 2020. After preliminary tests of stability, the repeatability and accuracy on cow raw milk, cream, skimmed and semi-skimmed milk for fat, true protein and dry matter were evaluated.

The accuracy of the instrument was evaluated according to the following standards:

- Fat content in milk: Gerber method according to ISO 19662|IDF 238 (2018)
- Fat content in skimmed milk: Röse-Gottlieb method according to ISO 1211|IDF 1 (2010)
- Fat content in cream: Röse-Gottlieb method according to ISO 2450|IDF 16 (2008)
- True protein content in milk and skimmed milk: Amido black method according to NF V04-216 (2011)
- Dry matter content in milk, skimmed milk and cream: Oven method according to ISO 6731|IDF 21 (2011).

### 1. EVALUATION OF SHORT-TERM REPRODUCIBILITY

The short term reproducibility was evaluated by analysing 3 samples of milk, with different levels of fat and protein content, in triplicate, every 15 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
<b>Fat (g/100 g)</b>	2.2	3.7	5.1
<b>Protein (g/100 g)</b>	2.3	3.1	3.9

**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
<b>Fat (g/100 g)</b>	<b>Level 1</b>	2.159	0.0081	0.374%	0.0229	1.060%	0.022	0.063
	<b>Level 2</b>	3.671	0.0066	0.179%	0.0204	0.555%	0.018	0.056
	<b>Level 3</b>	5.135	0.0085	0.166%	0.0174	0.339%	0.024	0.048
<b>True protein (g/100 g)</b>	<b>Level 1</b>	2.341	0.0087	0.372%	0.0252	1.078%	0.024	0.070
	<b>Level 2</b>	3.143	0.0073	0.234%	0.0218	0.696%	0.020	0.060
	<b>Level 3</b>	3.928	0.0069	0.176%	0.0196	0.499%	0.019	0.054
<b>Dry matter (g/100 g)</b>	<b>Level 1</b>	10.294	0.0199	0.193%	0.0494	0.480%	0.055	0.137
	<b>Level 2</b>	12.258	0.0190	0.155%	0.0447	0.365%	0.053	0.124
	<b>Level 3</b>	14.187	0.0187	0.132%	0.0336	0.271%	0.052	0.107

**Table 2: MIRA® stability criteria for fat, true protein and dry matter content<sup>1</sup>**

The results indicate that the standard deviations of repeatability for fat and protein are below the limits (0.028 g/100 g) required in ISO 8196-3|IDF 128-3 standard.

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

## 2. EVALUATION OF THE REPEATABILITY

The repeatability of the instrument was performed by analysing:

- **for trucks raw milk:** 44 samples of trucks raw milk from a French plant (West of France).
- **for cream:** 34 samples of cream from a French plant (West of France).
- **for skimmed and semi-skimmed milk:** 20 samples of skimmed and semi-skimmed milk. Final samples have been reconstituted from skimmed and whole milk samples in order to obtain fat ranging from around 0.05 to 1.6 g of fat/100 g.

Bronopol was added to the samples to give a final concentration of 0.02 %. The quantitative analyses were performed in duplicate according to the following sequence: Set 1 rep 1 – Set2 rep 2 - ... - Set n rep n.

The following table presents the results obtained:

		n	min	max	M	S <sub>r</sub>	S <sub>r</sub> %	r
<b>Trucks raw milk</b>	<b>Fat (g/100 g)</b>	44	3.98	4.44	4.20	0.006	0.14%	0.016
	<b>True protein (g/100 g)</b>	44	3.26	3.51	3.36	0.005	0.14%	0.013
	<b>Dry matter (g/100 g)</b>	44	12.70	13.24	12.95	0.008	0.06%	0.023
<b>Cream</b>	<b>Fat (g/100g)</b>	34	31.69	34.90	33.81	0.113	0.33%	0.313
	<b>Dry matter (g/100 g)</b>	34	44.10	48.65	47.11	0.150	0.32%	0.414
<b>Skimmed and semi-skimmed milk</b>	<b>Fat (g/100 g)</b>	19	-0.10	2.27	1.07	0.009	0.83%	0.025
	<b>True protein (g/100 g)</b>	19	3.33	3.48	3.42	0.009	0.25%	0.024
	<b>Dry matter (g/100 g)</b>	19	9.08	11.27	10.19	0.018	0.17%	0.049

**Table 3: MIRA® repeatability criteria for fat, true protein and dry matter in trucks raw milk, cream and skimmed and semi-skimmed milk<sup>2</sup>**

It can be noted that:

- **for trucks raw milk:** for fat and true protein content, the standard deviations of repeatability are in accordance with the recommendations of the ISO 8196-3|IDF 128-3 standard (S<sub>r</sub> < 0.014 g/100 g).

For dry matter, the absolute standard deviation of repeatability (S<sub>r</sub>) of the instrument is in the same order that the results for the other components and significantly lower than the repeatability standard deviation of the reference method (S<sub>r</sub> = 0.036 g/100g).

- **for cream:** For fat the relative standard deviation of repeatability is in accordance with the recommendations of the ISO 8196-3|IDF 128-3 standard (S<sub>r</sub>% < 0.35 %).

For dry matter, the relative repeatability (S<sub>r</sub> %) of the instrument is equivalent to results obtained for fat and the absolute standard deviation is higher to the limit of the reference method (S<sub>r</sub> = 0.072 g/100 g).

<sup>1</sup> M: mean; S<sub>r</sub> and S<sub>R</sub> (S<sub>r</sub>% and S<sub>R</sub>%): absolute (and relative) standard deviation of repeatability and reproducibility; r and R: maximum deviation of repeatability and reproducibility in 95 % of cases.

<sup>2</sup> n: number of results; min and max: minimum and maximum values; M: mean of the results; S<sub>r</sub> (S<sub>r</sub>%): absolute (and relative) standard deviation of repeatability; r: maximum deviation of repeatability in 95 % of cases.

• for skimmed and semi-skimmed milk: for fat and true protein, the standard deviations of repeatability are in accordance with the recommendations of the ISO 8196-3 | IDF 128-3 standard ( $S_r < 0.014$  g/100 g). For dry matter, the absolute standard deviation of repeatability ( $S_r$ ) of the instrument is significantly lower than the limit of the reference method ( $S_r = 0.036$  g/100 g).

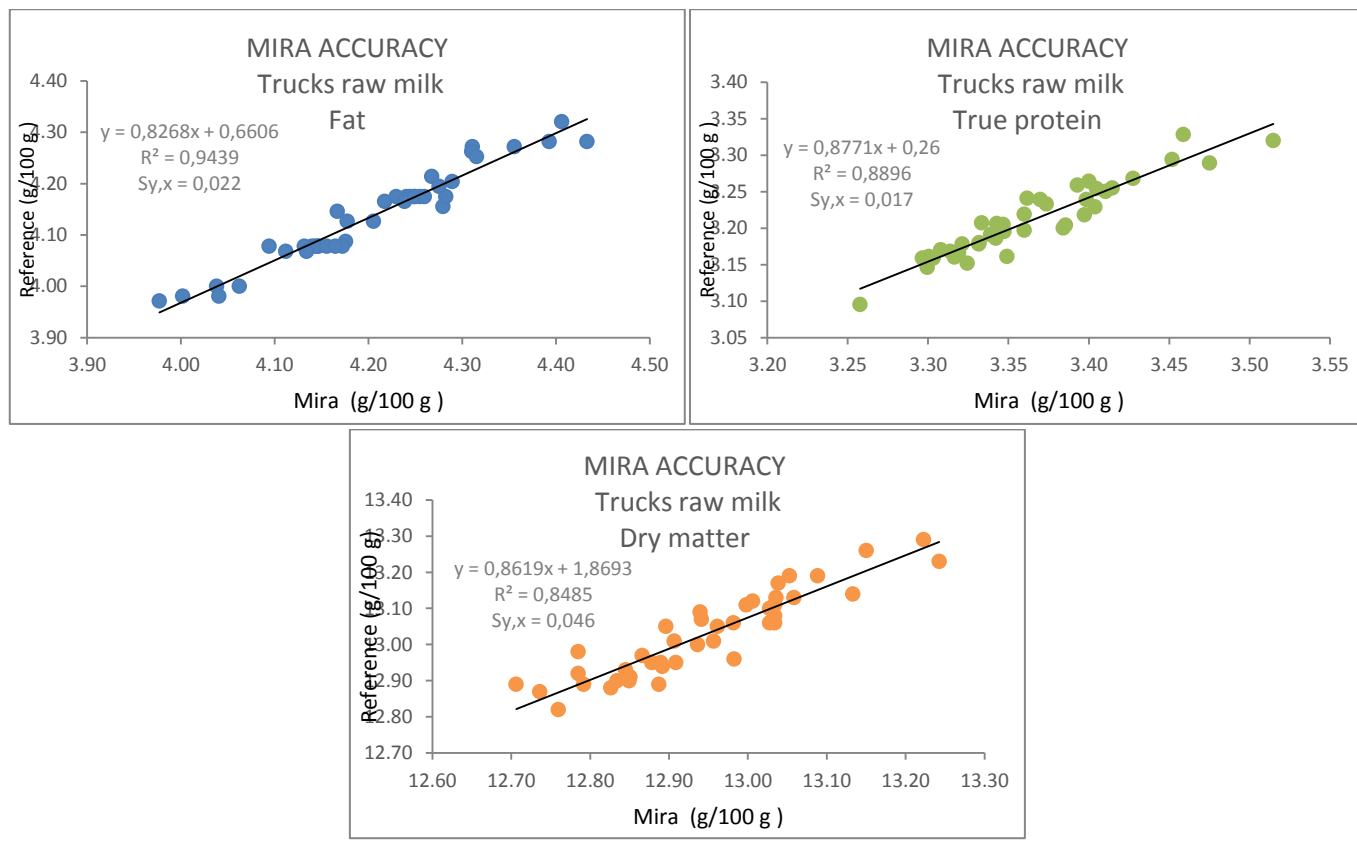
**3. EVALUATION OF THE ACCURACY**

The accuracy of the instrument was evaluated by using the mean of the instrument results from the repeatability test. Outliers samples (samples whose regression residues are greater than 2 times the type of deviation: P at 5 %) have been discarded.

The following table presents the results obtained:

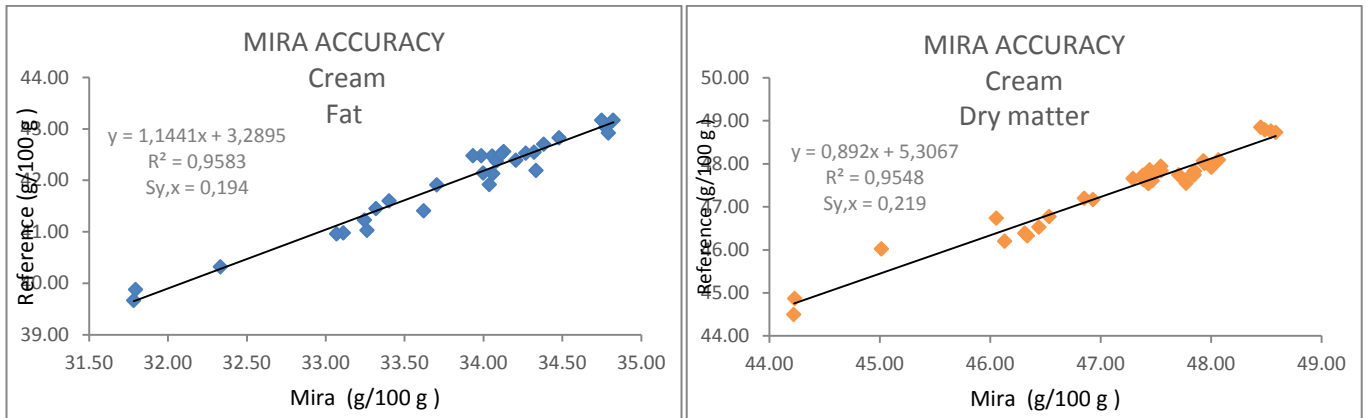
		n	min	max	Y	Sy	d	Sd	Sy,x	Sy,x%
Trucks raw milk	Fat (g/100g)	41	3.98	4.43	4.14	0.09	0.068	0.028	0.022	0.52%
	True protein (g/100g)	40	3.26	3.51	3.21	0.05	0.15	0.018	0.017	0.53%
	Dry matter (g/100g)	42	12.71	13.24	13.03	0.05	-0.08	0.049	0.046	0.35%
Cream	Fat (g/100g)	30	31.78	34.82	41.97	0.93	-8.16	0.190	0.194	0.46%
	True protein (g/100g)	33	44.22	48.59	47.40	1.01	-0.21	0.216	0.219	0.46%
Skimmed and semi-skimmed milk	Fat (g/100g)	19	-0.10	2.26	0.84	0.53	0.229	0.024	0.025	2.99%
	True protein (g/100g)	18	3.34	3.47	3.42	0.03	-0.003	0.013	0.014	0.40%
	Dry matter (g/100g)	18	9.09	11.27	10.13	0.45	0.105	0.060	0.062	0.61%

**Table 4** MIRA® accuracy criteria for fat, true protein and dry matter in trucks raw milk, cream and skimmed and semi-skimmed milk<sup>3</sup>

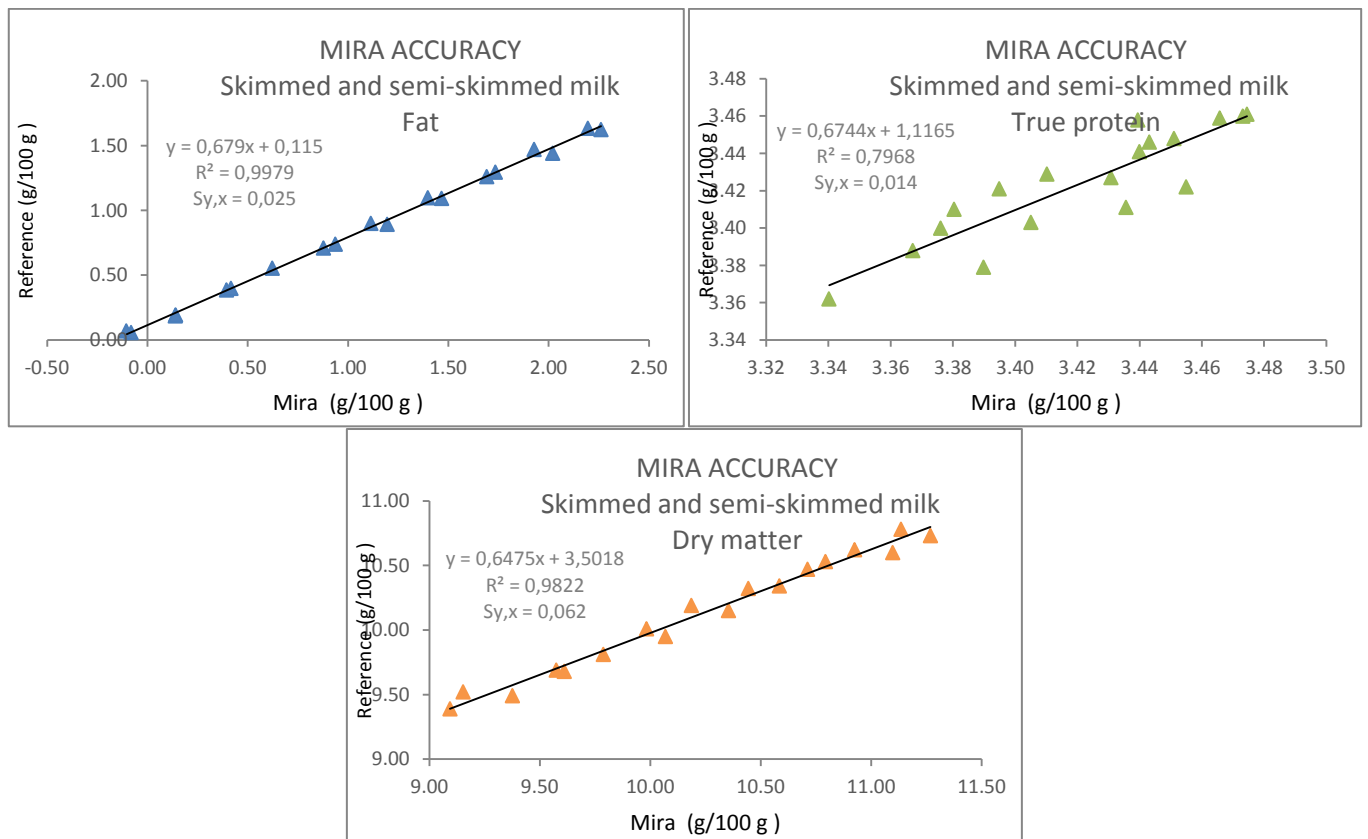


**Figure 1:** Relation between MIRA® and reference results for fat, true protein and dry matter in trucks raw milk

<sup>3</sup> 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 deviations of deviations; Sy,x (Sy,x%): absolute (and relative) residual standard deviation.



**Figure 2:** Relation between MIRA® and reference results for fat and dry matter in cream



**Figure 3:** Relation between MIRA® and reference results for fat, true protein and dry matter in skimmed and semi-skimmed milk

Concerning the relation between MIRA® and reference results, it can be noted that:

- **for trucks raw milk:**

The means and standard deviations of deviation are respectively equal to 0.068 and 0.028 g of fat/100 g, 0.154 and 0.018 g of protein/100 g, and -0.082 and 0.046 g of dry matter/100 g.

The regression slopes (fat: 0.827; protein: 0.877; dry matter: 0.862) are significantly different from 1.00. The intercepts for fat (0.661), protein (0.260) and dry matter (1.869) are significantly different from 0.00. These results can be explained by the milks used for this evaluation, which may have a fine composition different from those used to calibrate the instrument as well as the range of contents of the tested samples, which is not very wide.

The residual standard deviations of linear regression are equal to 0.022 g of fat/100 g, 0.017 g of protein/100 g, and 0.046 g of dry matter/100 g.

- **for cream:**

The means and standard deviations of deviation are respectively equal to -8.16 and 0.190 g of fat/100 g, and -0.21 and 0.216 g of dry matter/100 g.

The regression slopes (fat: 1.144; dry matter: 0.892) and the intercepts (fat: 3.289; dry matter: 5.307) are significantly different from 1.00 and 0.00 ( $P = 5\%$ ). These results can be explained by the creams used for this evaluation, which may have a fine composition different from those used to make the model as well as the range of fat contents of the samples tested which is not very wide.

The residual standard deviations of linear regression are equal to 0.19 g of fat/100 g and 0.22 g of dry matter/100 g.

- for skimmed and semi-skimmed milk:

The means and standard deviations of deviation are respectively equal to 0.229 and 0.024 g of fat/100 g, -0.003 and 0.013 g of protein/100 g, and 0.105 and 0.060 g of dry matter/100 g.

The regression slopes (fat: 0.679; protein: 0.674; dry matter: 0.647) and the intercepts (fat: 0.115; protein: 1.117; dry matter: 3.502) are significantly different from 1.00 and 0.00 (P = 5 %). These results can be explained by the milks used for this evaluation, which may have a fine composition different from the milks used to make the model, as well as the range of fat contents of the samples tested which is not very wide.

The residual standard deviations of linear regression are equal to 0.025 g of fat/100 g, 0.014 g of protein/100 g, and 0.062 g of dry matter/100 g.

#### 4. CONCLUSION

We can conclude that the stability of the instrument is in accordance with the requirements of the ISO 8196-3 standard.

Concerning the repeatability of the instrument, the results for fat and true protein in milk are in conformity with the requirements of the ISO 9622 standard.

The performance of the instrument for the fat determination on cream is in accordance with the relative standard deviation (0,35 %) prescribed in the ISO 9622 standard.

As no standardised value exists for dry matter content, the reproducibility values of the instrument have been compared to the reproducibility values of the reference methods.

Concerning the accuracy of the instrument, no standardised requirements exist for these types of dairy products (trucks raw milk, cream and skimmed milk).

*According to the evaluation report of the MIRA<sup>®</sup> analyser - A. OUDOTTE and Ph. TROSSAT – September 2019 – February 2020*