ARTICLE

EVALUATION OF THE MPA™ INFRARED ANALYSER ON RAW MILK

The MPA™ is a near infrared TF spectrophotometer manufactured by Bruker Optik (Germany, Bruker Group Corporation) and commercialised in France by Bruker Optics. It is used for the determination of the principal components in milk and in liquid (retentate, serum, cream, milk-based drinks…) and solid (powder, cheese, butter, yoghurt) dairy products.

This apparatus allows to analyse:
- liquid samples in a sample compartment with an InGaAs detector cooled thermo-electrically (range 12800 – 4000 cm-1) and,
- solid samples, by transmission module with a Si detector at room temperature (range 15500 – 9000 cm-1), or by reflection module with a PbS detector (range 12800-3600 cm-1). Fiber optic probes can also be installed. The heart of the instrument is a permanent alignment interferometer with gold mirrors.

The apparatus is computer controlled with Opus software, which ensures the signal treatment and the PLS calibrations.

The instrument was already evaluated on homogenised milk, so a complementary module adapted to the analysis of raw milk was installed. It includes a pipetting system, a heater, a HP pump and a homogeniser.

The evaluation tests were performed in Actilait-Cecalait® physico-chemistry laboratory (reference and instrumental analyses) from March to September 2012. After preliminary tests of contamination between samples for fat and crude protein, the repeatability and accuracy on raw milk for fat, dry matter and crude protein were evaluated.

The prediction models used have been developed by the supplier thanks the Opus software.

The calculation parameters are in relation with the ISO 21543/IDF 201 standard.

A – PRELIMINARY TESTS

The objectives of these tests were to evaluation the contamination between samples. Ten sets of whole raw milk were then analysed according the Lait 1 – Lait 2 – Lo1 – Lo2 sequence on 2 levels of different rates. Fat and crude protein parameters were noted. Lo samples (about 15 g/l in fat and crude protein) were used in place of water samples in order to work at constant gain.

The following table summarises the results obtained:

	MILK A		MILK B	
	Fat	Crude protein	Fat	Crude protein
M (g/l)	37.88	31.13	58.6	39.70
Tc (%)	0.25	0.86	0.44	0.68

Table 1: MPA contamination criteria for fat and crude protein on raw milk samples
M: results mean, Tc: contamination rates
The contamination rate was calculated according to the following formula:
*Tc(%) = 100 * [sum(Lo1)-sum(Lo2)] / [sum(Lo2)-sum(Lo2)]*

With no standard criteria, it can be noted that the relative contamination rates obtained vary between 0.25 and 0.86%. The values obtained are in accordance with the ISO 9622/IDF 141 standard, which fixes the maximal limit at 1%.

B – EVALUATION OF THE REPEATABILITY AND THE ACCURACY

B.1 – Samples

The tests were performed on 30 samples of tank milk from Rhône-Alpes region. Bronopol was added to the samples to give a final concentration of 0.02%.

B.2 – Procedure

The repeatability and the accuracy of the instrument for fat, dry matter and crude protein were evaluated using all the milk samples. The infrared quantitative analysis of each sample was carried out in consecutive duplicate. The instrumental values were carried out by a calibration and adjustment of the manufacturer, optimised by the integration of 10 specific samples.

The following reference methods were used:
- Dry matter: drying method according to ISO 6731/IDF 21 (single tests);
- Fat: acido-butyrometric method according to NF V 04-210 (single tests) ;
- Crude protein: Kjeldahl method according to 8968/IDF 20 (single tests), with conversion crude protein = AT x 6.38

B.3 – Results

The tables and figures below summarise the results obtained:

	n	min	max	M	Sx	Sr	Sr (%)	r
Dry matter (g/100g)	30	12.36	12.96	12.776	0.127	0.013	0.11	0.04
Fat (g/l)	30	37.60	44.95	42.872	1.568	0.094	0.22	0.26
Crude protein (g/kg)	30	32.68	34.91	33.834	0.475	0.064	0.19	0.18

Table 2: MPA repeatability criteria for dry matter, fat and crude protein on raw milk samples
n: number of results; min and max: minimum and maximum value; M and Sx: mean and standard deviation of the results; Sr and Sr%: absolute and relative standard deviation of repeatability; r: maximal deviation of repeatability (95% of cases)

	n	min	max	Y	Sy	d	Sd	Sy,x	Sy,x%	b	a
Dry matter (g/100g)	30	12.30	13.00	12.79	0.16	-0.02	0.07	0.066	0.51	1.168	-2.12
Fat (g/l)	30	35.50	42.60	40.78	1.57	2.09	0.29	0.298	0.69	0.985	-1.47
Crude protein (g/kg)	30	32.39	34.77	33.71	0.55	0.12	0.23	0.230	0.68	1.059	-2.12

Table 3: MPA accuracy criteria for dry matter, fat and crude protein on raw milk samples
n, min, max: number of results, minimum and maximum value; Y,X: mean results using reference and instrumental methods; Sy: standard deviation of the results from the reference method; d, Sd: mean and standard deviation of deviations; Sy,x and Sy,x%: absolute and relative standard deviation; b, a: slope and intercept of the linear regression

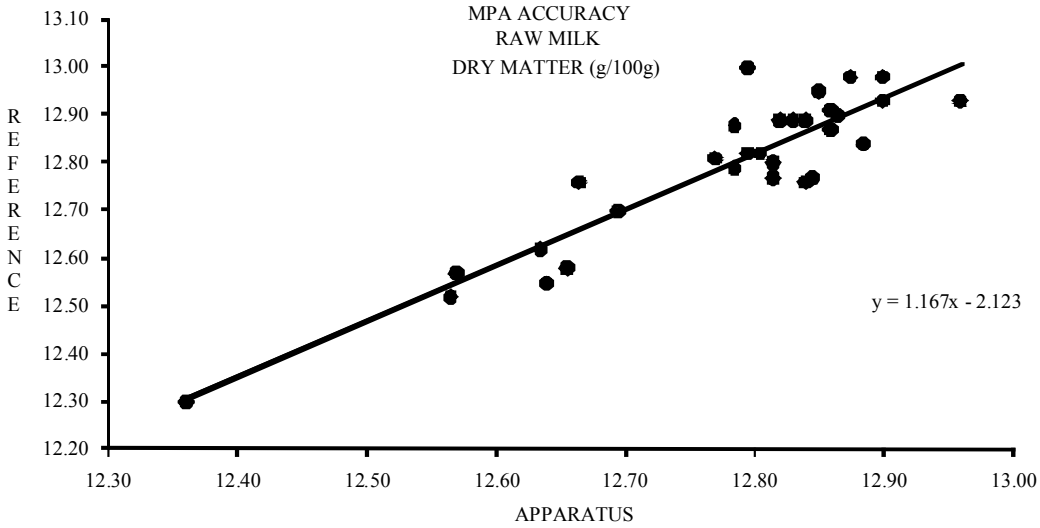

Figure 1: Relation between the MPA and the reference results for dry matter on raw milk samples

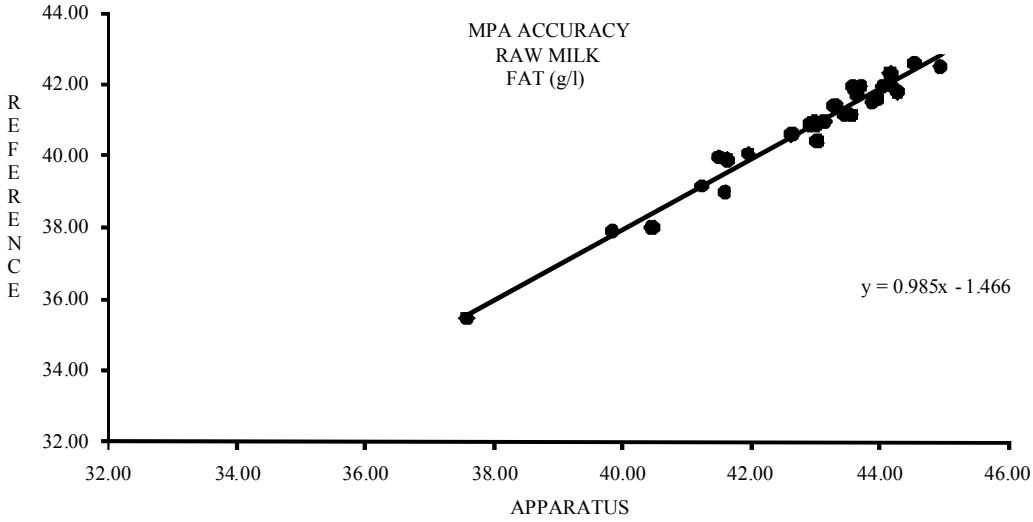

Figure 2: Relation between the MPA and the reference results for fat on raw milk samples

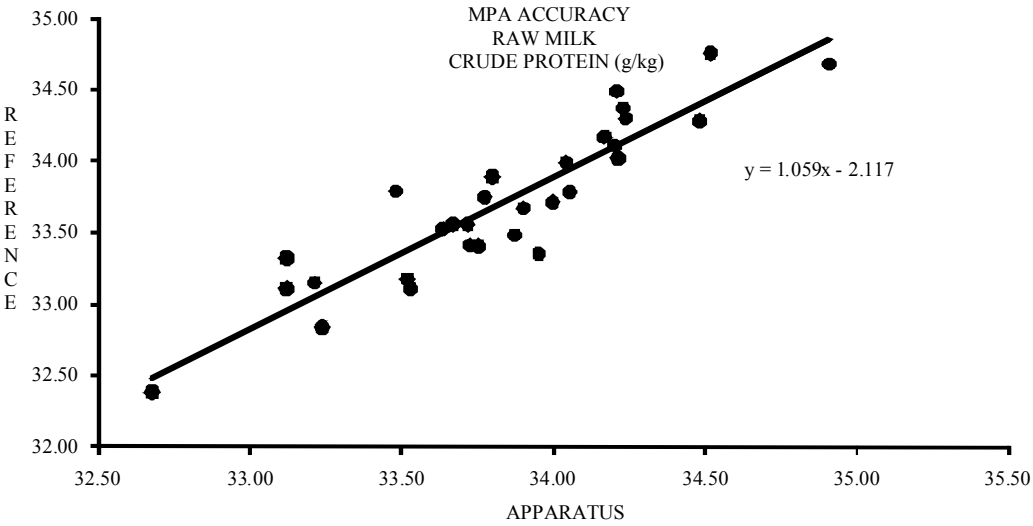

Figure 3: Relation between the MPA and the reference results for crude protein on raw milk samples

It can be noted that:

⇨ For dry matter, the residual standard deviation is equal to 0.066 g/100 g. The slope is equal to 1.167 and is not significantly different from 1.00 (P = 5%),

⇨ For fat, the residual standard deviation is equal to 0.298 g/l.. The slope is equal to 0.985 and is not significantly different from 1.00 (P = 5%),

⇨ For crude protein, the residual standard deviation is equal to 0.230 g/kg. The slope is equal to 1.059 and is not significantly different from 1.00 (P = 5%).

B.4 - Conclusion

Concerning the performance of repeatability, the standard deviations of repeatability are respectively equal to 0.013 g/100 g, 0.064 g/l and 0.094 g/kg for dry matter, fat and crude protein. Despite the absence of standard criteria for the analysis of milk by near infrared, it can be noted that the results obtained are in accordance with the recommendations of the ISO 9622/IDF 141 standard dedicated to the mid infrared analysers on raw milk, which fixes the maximal limit to 0.14 g/l for the standard deviations of repeatability of fat and protein.

Concerning the performance of accuracy, the residual standard deviations of regression observed enable accuracy of estimation (2.Sy,x at 5% risk) equal to 0.132 g/100 g for dry matter, 0.596 g/l for fat and 0.460 g/kg for crude protein. Despite the absence of standard criteria for the analysis of milk by near infrared, it can be noted that the values obtained for fat are in accordance with the maximal limit of the standard deviation of deviations (0.7 g/l) of the ISO 9622/IDF 141 standard.

CONCLUSION

Because of the absence of standard criteria specific to near infrared method, it is difficult to interpret the MPA repeatability and accuracy performances. However, it can be noted that the performances obtained for fat are in accordance with the standardised limits (ISO 9622/IDF 141) and close to the performances published. Moreover, as the evaluation was performed with general manufacturer's calibration, the repeatability and accuracy values observed are most probably maximums and can be improved by enrichment of the model with appropriate samples.

According to the evaluation report of the MPA™ infrared analyser (raw milk) – X. QUERVEL and Ph. TROSSAT – December 2012