

## EVALUATION OF THE NIRMaste<sup>TM</sup> INFRARED ANALYSER

The NIRMaste<sup>TM</sup> is a near infrared TF spectrophotometer manufactured by Buchi (Switzerland, Buchi Group), and commercialised in France by Buchi France (SARL). Among its available applications, it is used for the determination of the principal components in solid dairy products (powder, cheese, butter, yoghurt...).

This instrument uses the diffuse reflection in a spectral range of 12500-4000 cm<sup>-1</sup> with a resolution of 8 cm<sup>-1</sup>, and it is supplied with a polarisation interferometer licensed by BUCHI<sup>®</sup>. NIRMaste<sup>TM</sup> exists in IP 54 and IP 65 configuration, and can be installed in production area, near the industrial plants. 3 types of cupel are available: glass, polystyrene, and quartz unbreakable.

The apparatus is computer controlled with NIRWare<sup>®</sup> software, which ensures the export of the results. The client can also equip its instrument with the NIRCAL<sup>®</sup> software, which ensures the signal treatment and the calibration according to the PLS models (partial least square).



### The tests:

The evaluation tests were performed in Actilait-Cecalait's physico-chemistry laboratory (reference and instrumental analyses) from July to September 2011. After preliminary stability tests performed for dry matter (MS), fat (MG) and crude protein (MAT), the repeatability and accuracy on cheese were evaluated for the same parameters.

The calibrations used have been developed by the supplier thanks the NIRCAL<sup>®</sup> software, and the calculation parameters are in relation with the ISO 21543 / IDF 201 standard.

### **A- PRELIMINARY TESTS: EVALUATION OF THE STABILITY AT SHORT TERM**

#### **A.1- Procedure**

A set of 3 samples of cheese was analysed in consecutive duplicate every 20 minutes for about 3 hours. Fat, dry matter and crude protein parameters were noted.

#### **A.2- Results**

The table below summarise the results obtained:

	CHEESE 1			CHEESE 2			CHEESE 3		
	MS	MG	MAT	MS	MG	MAT	MS	MG	MAT
<b>N</b>	8								
<b>M (g/100g)</b>	64.94	33.15	29.04	63.45	33.98	26.59	58.75	31.39	24.21
<b>SR (g/100g)</b>	0.05	0.12	0.60	0.11	0.10	0.55	0.18	0.12	0.29
<b>SR (%)</b>	0.1	0.4	2.1	0.2	0.3	2.1	0.3	0.4	1.2

**Table 1:** NIRMaste stability criteria for dry matter (MS), fat (MG) and crude protein (MAT)

*N:* number of results; *SR* and *SR%*: absolute and relative standard deviation of reproducibility; *M*: mean of results.

### A.3- Conclusion

With no standard criteria, it can be noted that the relative standard deviations of reproducibility obtained vary between 0.1% and 2.1%.

## B- EVALUATION OF REPEATABILITY AND ACCURACY

### B.1- Samples

The tests were performed on 38 samples of hard cheese sold in supermarkets and hypermarkets (for fat, only 37 samples were evaluated). The samples were analysed after the rind removal and grinding.

### B.2- Procedure

The repeatability and the accuracy of the instrument for dry matter, fat and crude protein were evaluated using all the samples. The quantitative analyses of each sample were carried out on set of 10 samples in consecutive duplicate. The instrumental values are from a calibration carried out by the manufacturer. The measures were realised in transmission through glass petri dishes.

The following reference methods were used:

- Dry matter: drying method according to ISO 5534 (single tests);
- Fat: SBR extraction method according to ISO 1735 (single tests);
- Crude protein: Kjeldahl method according to ISO 8968, conversion crude protein = total nitrogen x 6.38 (single tests).

### B.3- Results

The tables and figures below summarise the results obtained:

	<b>n</b>	<b>min</b>	<b>max</b>	<b>M</b>	<b>Sx</b>	<b>Sr</b>	<b>Sr (%)</b>	<b>r</b>
<b>MS (g/100g)</b>	38	48.63	67.97	60.31	5.40	0.12	0.21	0.34
<b>MG (g/100g)</b>	38	23.11	36.81	31.00	3.69	0.11	0.35	0.30
<b>MAT (g/100g)</b>	38	20.56	29.98	25.68	2.37	0.12	0.45	0.32

**Table 2:** NIRMaste repeatability criteria for dry matter (MS), fat (MG) and crude protein (MAT)  
*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 in 95% of cases.

	<b>MS (g/100g)</b>	<b>MG (g/100g)</b>	<b>MAT (g/100g)</b>
<b>n</b>	38	37	38
<b>min</b>	48.78	22.21	19.87
<b>max</b>	68.48	35.58	29.96
<b>Y</b>	60.67	30.23	25.44
<b>Sy</b>	5.73	3.63	2.57
<b>d</b>	-0.36	0.66	0.24
<b>Sd</b>	0.46	0.35	0.63
<b>Sy,x</b>	0.342	0.345	0.626
<b>Sy,x %</b>	0.57	1.12	2.44
<b>RMSEP</b>	0.58	0.74	0.67
<b>RMSEP %</b>	0.96	2.46	2.63
<b>b</b>	1.059	0.984	1.056
<b>a</b>	-3.20	-0.15	-1.67

**Table 3:** NIRMaste accuracy criteria for dry matter (MS), fat (MG) and crude protein (MAT)  
*n*, *min*, *max*: number of results; minimum and maximum value; *Y,X*: mean results using the 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 point of the linear regression; *RMSEP* and *RMSEP%*: absolute and relative quadratic mean error of prediction.

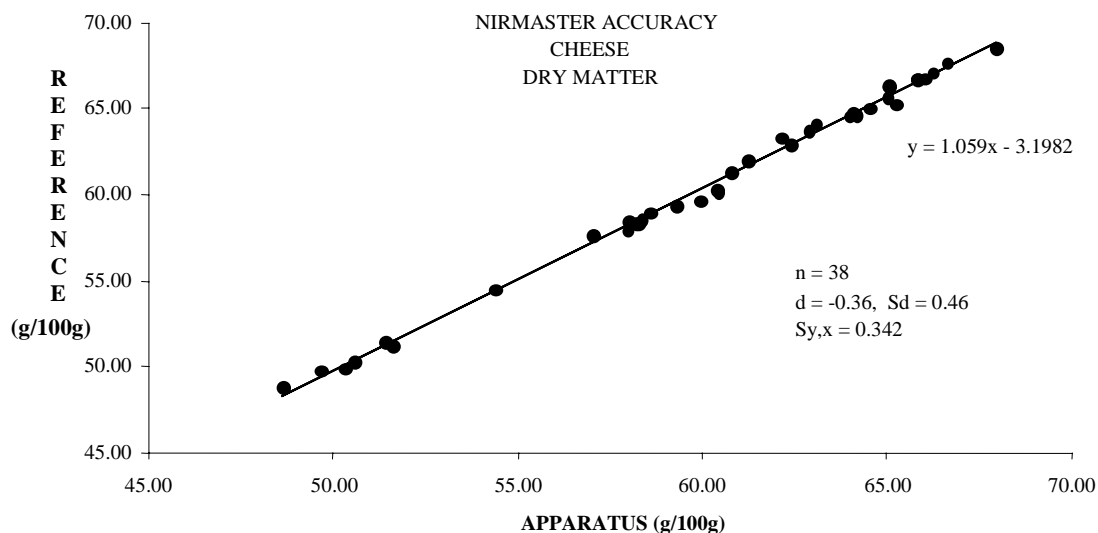


Figure 1: Relation between the NIRMasteR and the reference results for dry matter on cheese samples

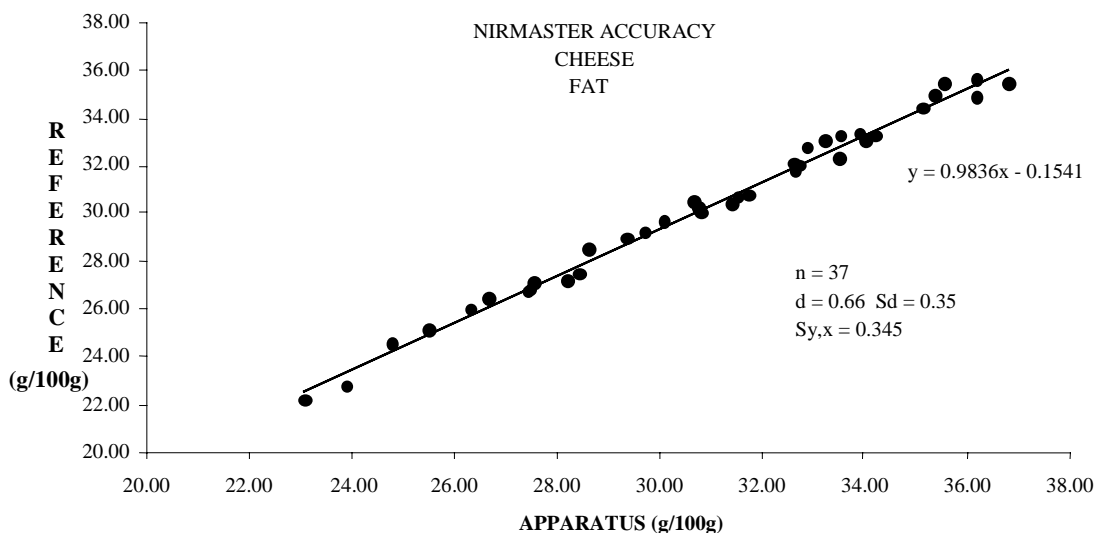


Figure 2: Relation between the NIRMasteR and the reference results for fat on cheese samples

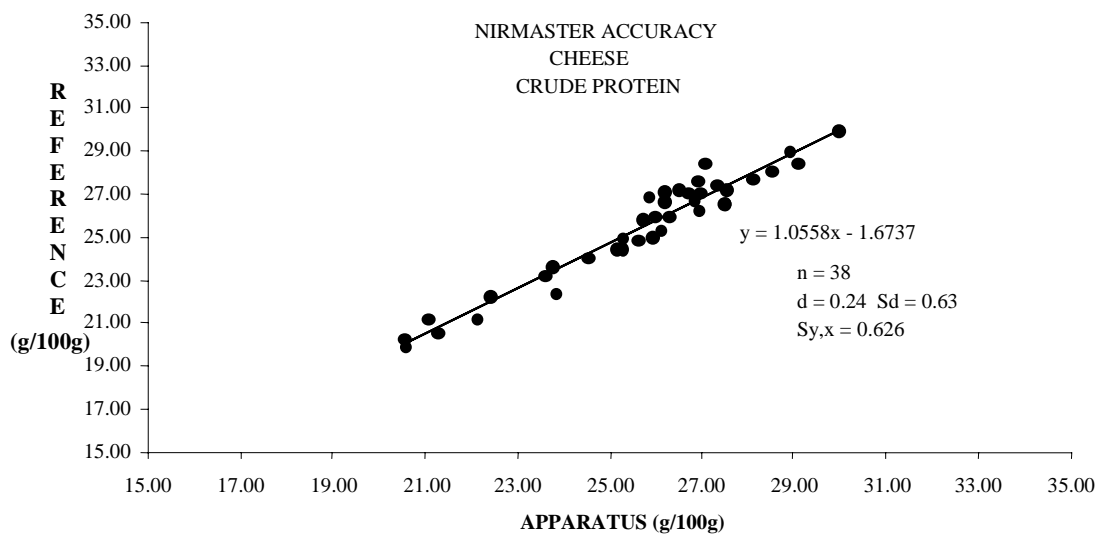


Figure 3: Relation between the NIRMasteR and the reference results for crude protein on cheese samples

It can be noted that:

⇒ For dry matter, the relative residual standard deviation is equal to 0.57%. The slope is equal to 1.059, significantly different from 1 (P = 1%). The relative error of prediction is equal to 0.96%.

⇒ For fat, the relative residual standard deviation is equal to 1.12%. The slope is equal to 0.984, no significantly different from 1 (P = 5%). The relative error of prediction is equal to 2.46%.

⇒ For crude protein, the relative residual standard deviation is equal to 2.44%. The slope is equal to 1.056, no significantly different from 1 (P = 5%). The relative error of prediction is equal to 2.63%.

#### **B.4- Conclusion**

Concerning the performance of repeatability, with no specific standard criteria for the near infrared analysers on these matrixes, it can be noted that the standard deviations of repeatability obtained with the instrument are close to the standard deviations of repeatability of the corresponding reference methods (Sr = 0.12 g/100g against 0.13 for dry matter, 0.11 g/100g against 0.11 for fat, and 0.12 g/100g against 0.11 for crude protein).

Concerning the accuracy, the values observed enable relative accuracy of estimation ( $\pm 2 \cdot \text{RMSEP} \%$  at 5% risk) equal to  $\pm 1.9\%$  for dry matter,  $\pm 4.9\%$  for fat, and  $\pm 5.3\%$  for crude protein.

#### **CONCLUSION**

With no standard criteria on this type of method (instrumental near infrared), the NIRMaster repeatability and accuracy performances cannot be interpreted. However, as the evaluation was performed with a manufacturer's calibration dedicated to a range of cheeses (hard cheese and grated hard cheese), a specific adjustment of the final model equation (slope and intercept point, or only intercept point) on the cheeses analysed would probably enable an improvement of the observed relative accuracy of estimation. Most specific calibrations can also be developed from the global basis.

*According to the evaluation of the NIRMaster™ infrared analyser - X. QUERVEL – October 2011*

---

#### ANNEX

##### List of the cheese samples

#### STABILITY

Cheese 1: Emmental

Cheese 2: Comté

Cheese 3: Gouda

#### REPEATABILITY AND ACCURACY

Chaussée aux moines™

Fol épi™

Gouda

Leerdamer™

St Paulin™

Beaufort

Emmental

Edam

Comté

Tomme du Jura

Appenzeller

Tomme de Savoie