

# EVALUATION OF THE ADVANCED LACTOSCOPE FTIR™ ANALYSER ON A CHEESE MATRIX

The Lactoscope FTIR™ analyser was evaluated by Actilait-Cecalait for the determination of fat, protein, dry matter and the freezing point in milk, and the fat content in cream. The results are presented in Cecalait's newsletter n°70. The present evaluation concerns the determination of fat, dry matter and total nitrogen in cheese.



The Lactoscope FTIR™ analyser, manufactured by Delta Instruments (Netherlands, Advanced group) and commercialized in France by Humeau Laboratories, is a mid infrared spectrophotometer used for the determination of the principal components (fat, protein, dry matter, freezing point and total nitrogen content) in milk, cream and cheese.

This instrument uses a mono-bundle Fourier transform-based infrared system (FTIR). The apparatus is connected to a computer that ensures the running and the signal treatment. Two mathematical calculations can be carried out: traditional PLS (MLR) and PLS.

## **Consumables:**

The consumables used were:

- "Zero" solution: water + 0.1% triton
- Clean solution: Decon 90™ 4% aqueous solution

## **The tests:**

The evaluation tests were performed in Actilait-Cecalait's physico-chemistry laboratory (reference and infrared analyses) in August and September 2009. The tests concerned the evaluation of repeatability and accuracy for the determination of fat, dry matter and total nitrogen in cheese.

The appreciation criteria of the estimated parameters were taken from ISO 9622 / IDF 141C:2000 "Guide for the operation of mid-infrared instruments", or from the CNIEL/IE handbook concerning the use of infrared apparatus within the context of milk payment and milk control in France.

The following instrumental parameters were used:

- Manual tests at a rate of about 120 samples / hour;
- No correction of contamination;
- MLR calibration (fat and total nitrogen);
- PLS calibration (dry matter).

## **The samples**

The tests were performed on samples of cheese sold in supermarkets and hypermarkets. 20 samples were analysed: 6 samples of "fromage frais" (3 as is from the shops and 3 made up artificially), 7 samples of soft cheese and 7 samples of hard cheese. Bronopol was added to the "fromage frais" samples to give a final concentration of 0.02%. The samples were prepared in accordance with the manufacturer's recommendations: remove the rind and grind the test sample, make a 10% suspension with the "cheese" reagent and grind for one minute in a granulating mill.

## 1- EVALUATION OF REPEATABILITY

### 1.1- Procedure

The repeatability of the instrument for fat, dry matter and protein was evaluated using all the cheese samples. The quantitative analyses were performed in consecutive duplicate (one suspension per sample).

### 1.2- Results

The results obtained are presented in the table below:

	<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>Fat (g/100g)</b>	20	0.10	35.58	20.21	12.40	0.04	0.18	0.10
<b>Dry matter (g/100g)</b>	20	14.26	67.47	43.98	19.35	0.07	0.15	0.18
<b>Total nitrogen (g/100g)</b>	20	7.42	32.76	19.30	8.34	0.04	0.21	0.11

**Table 1:** repeatability criteria FTIR Lactoscope for fat, dry matter and total nitrogen in cheese samples  
*n*: number of results; *min* and *max*: minimum and maximum values, *M* and *Sx*: mean and standard deviation of the results; *Sr* and *Sr%*: absolute and relative standard deviation of repeatability; *r*: maximum deviation of repeatability in 95% of cases.

### 1.3- Conclusion

With no standard criteria or recommendations from the manufacturer for fat, dry matter and total nitrogen content, the FTIR Lactoscope presents a standard deviation of repeatability lower than the maximum acceptable limit laid down in the ISO 1735, ISO 5534 and ISO 8968/IDF 20 (WD 2008) reference methods, equal to 0.07 g/100g, 0.13 g/100g and 0.05 g/100g respectively.

## 2- EVALUATION OF ACCURACY

### 2.1- Procedure

The accuracy of the analyser for fat, dry matter and protein was evaluated using all the cheese samples. The quantitative analyses were performed in compliance with the evaluation of repeatability (cf. 1.2). The instrumental values are from a calibration carried out by the manufacturer.

The following reference methods were used:

- Fat using SBR extraction method according to ISO 1735 (single test);
- Dry matter using drying method according to ISO 5534 (single test);
- Total nitrogen using Kjeldahl method according to ISO 8968 (single test). Conversion using the following calculation:  $MAT = NT \times 6.38$

### 2.2- Results

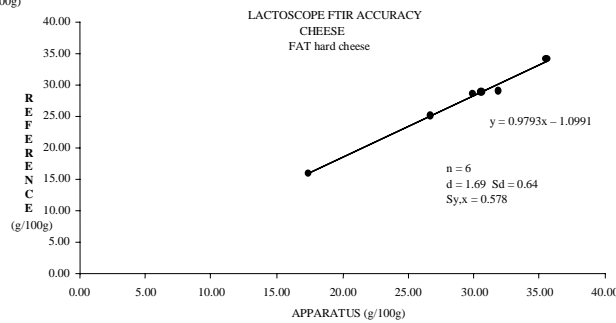
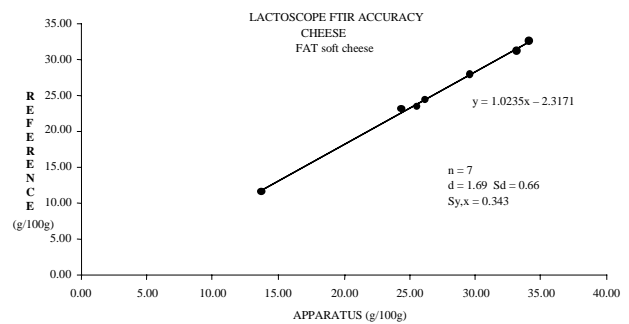
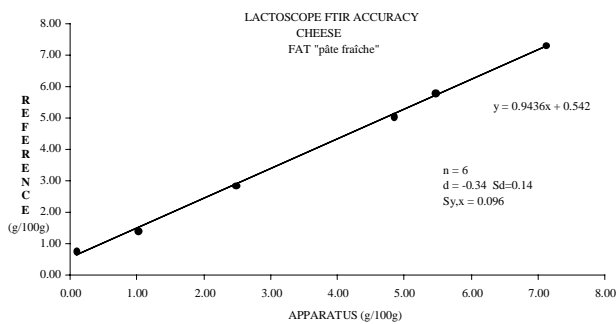
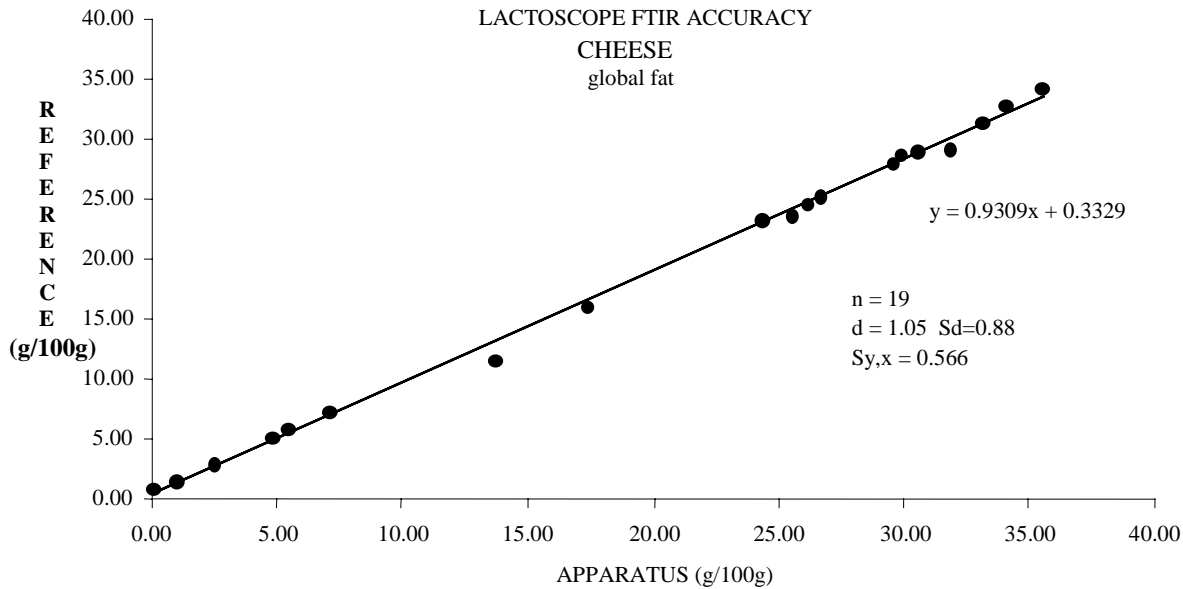
#### 2.2.1- Fat

The results obtained are presented in the following tables and figures.

	<b>n</b>	<b>min</b>	<b>max</b>	<b>Y</b>	<b>Sy</b>	<b>d</b>	<b>Sd</b>	<b>Sy,x</b>	<b>Sy,x %</b>	<b>b</b>	<b>a</b>
<b>Global Fat (g/100g)</b>	19	0.75	34.08	18.93	11.83	1.05	0.88	0.566	3.0	0.931	0.33
<b>Fat (g/100g) "Pâte fraîche"</b>	6	0.75	7.29	3.85	2.59	-0.34	0.14	0.096	2.5	0.944	0.54
<b>Fat (g/100g) Soft cheese</b>	7	11.59	32.68	24.96	6.99	1.69	0.66	0.343	1.4	1.024	-2.32
<b>Fat (g/100g) Hard cheese</b>	6	15.91	34.08	26.97	6.12	1.69	0.64	0.578	2.1	0.979	-1.10

**Table 2:** Accuracy criteria of the FTIR Lactoscope for fat in cheese samples

*n*, *min*, *max*: number of results, minimum and maximum values; *Y*: mean results using the reference; *Sy*: standard deviation of the results from the reference method; *d*, *Sd*: mean and standard deviation of deviations; *Sy,x*: residual standard deviation; *b*, *a*: slope and intercept of the linear regression.



Figures 1, 2, 3 and 4: Relationship between the FTIR Lactoscope and the reference results for fat in "pâte fraîche", soft and hard cheese samples

It can be noted that:

- Overall, the mean and standard deviation of deviations are respectively 1.05 and 0.88 g/100g. The regression slope obtained is significantly different from 1 (P = 1%) and the intercept is significantly different from zero (P = 1%). The residual standard deviation of regression is equal to 0.566 g/100g.
- the slope varies between 0.94 and 1.02 according to the type of cheese, which indicates the necessity for specific adjustments.

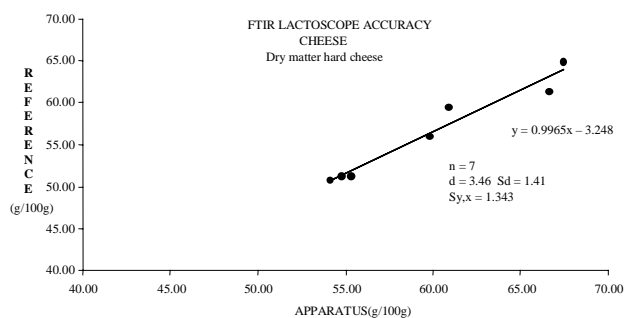
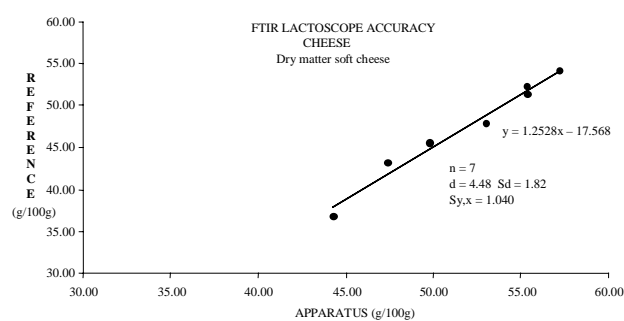
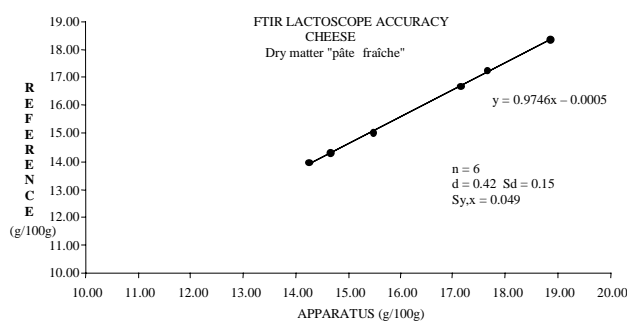
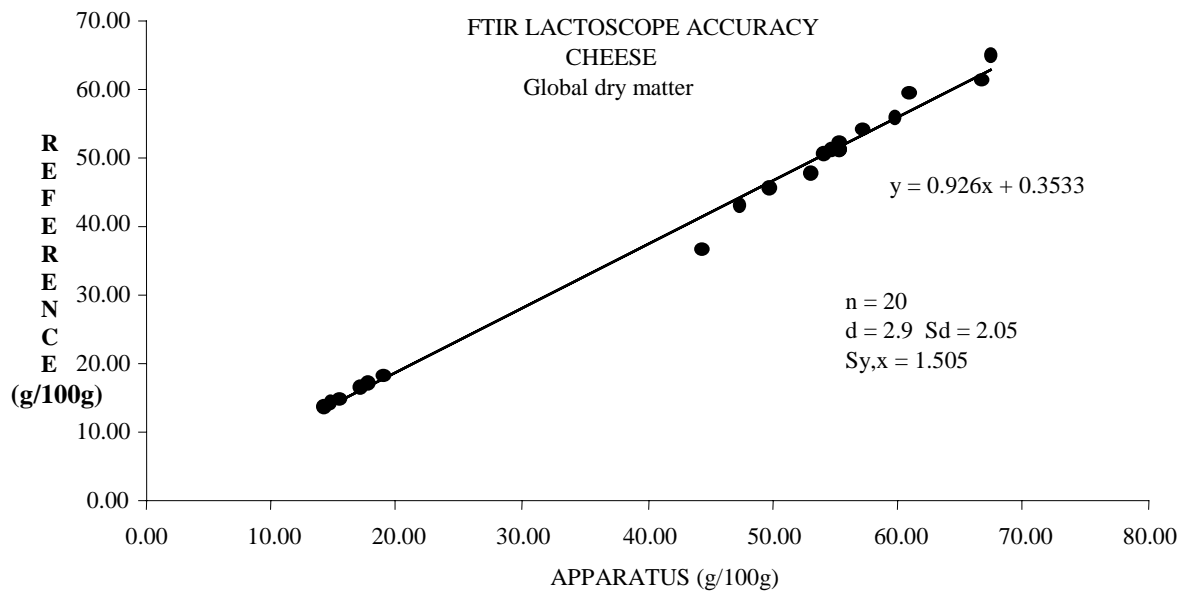
### 2.2.2- Dry matter

The results obtained are presented in the following tables and figures.

	n	min	max	Y	Sy	d	Sd	Sy,x	Sy,x %	b	a
Global dry matter (g/100g)	20	13.94	64.96	41.08	17.98	2.90	2.05	1.505	3.7	0.926	0.35
Dry matter (g/100g) "Pâte fraîche"	6	13.94	18.37	15.92	1.77	0.42	0.15	0.049	0.3	0.975	0.00
Dry matter (g/100g) Soft cheese	7	36.76	54.20	47.31	6.05	4.48	1.82	1.040	2.2	1.253	-17.57
Dry matter (g/100g) Hard cheese	7	50.77	64.96	56.42	5.65	3.46	1.41	1.343	2.4	0.997	-3.25

Table 3: accuracy criteria of the FTIR Lactoscope for dry matter in cheese samples

n, min, max: number of results, minimum and maximum values; Y: mean results using the reference; Sy: standard deviation of the results from the reference method; d, Sd: mean and standard deviation of deviations; Sy,x: residual standard deviation; b, a: slope and intercept of the linear regression.



**Figures 5, 6, 7 and 8:** Relationship between the FTIR Lactoscope and the reference results for dry matter in "pâte fraîche", soft and hard cheese samples

It can be noted that:

- Overall, the mean and standard deviation of deviations are respectively 2.9 and 2.05 g/100g. The regression slope obtained is significantly different from 1 ( $P = 1\%$ ) and the intercept is significantly different from zero ( $P = 1\%$ ). The residual standard deviation is 1.505 g/100g.
- Concerning fat, the slope varies between 0.97 and 1.25 according to the type of cheese, which indicates the necessity for specific adjustments.

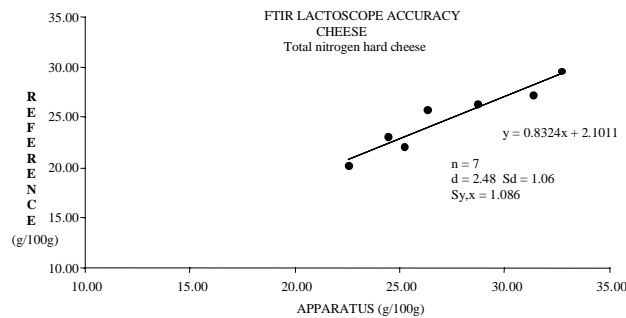
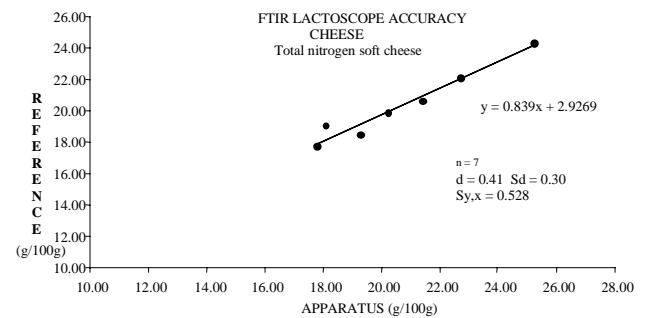
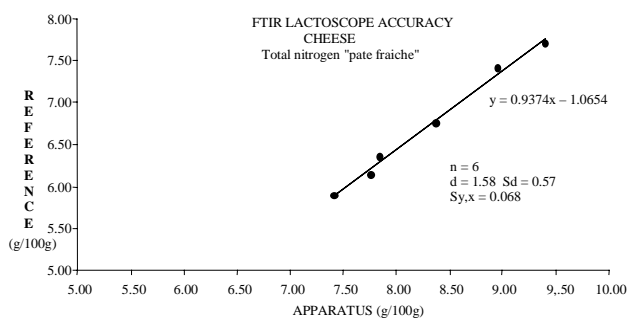
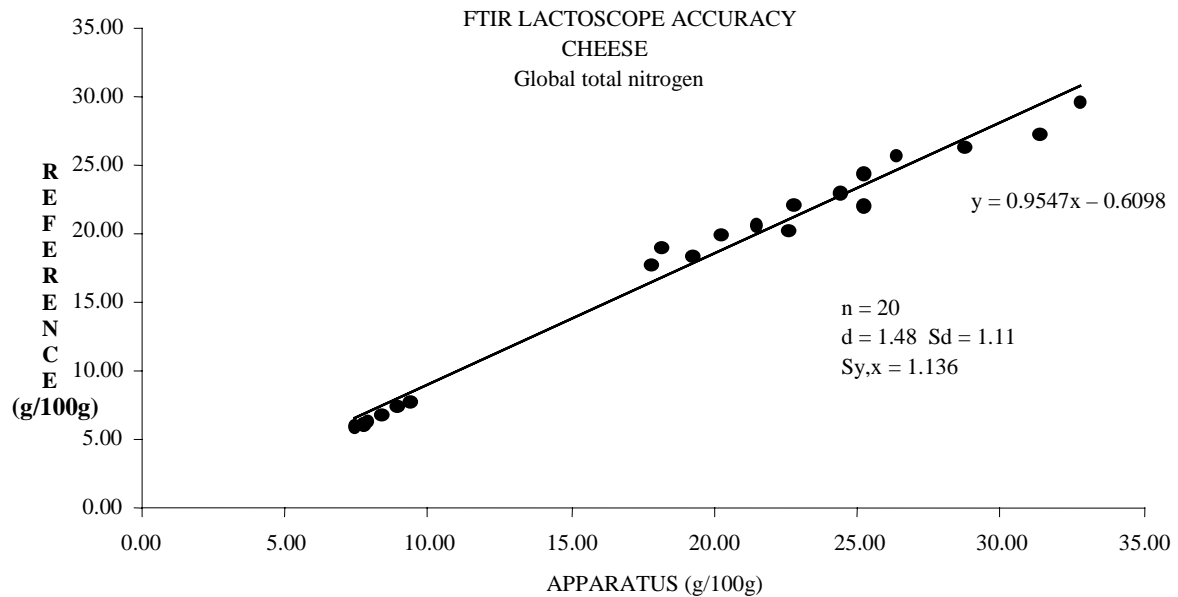
### 2.2.3- Total nitrogen

The results obtained are presented in the following tables and figures.

	n	min	max	Y	Sy	d	Sd	Sy,x	Sy,x%	b	a
<b>Global total nitrogen (g/100g)</b>	20	5.89	29.54	17.81	8.04	1.48	1.11	1.136	6.4	0.955	-0.61
<b>Total nitrogen (g/100g) "Pâte fraîche"</b>	6	5.89	7.70	6.71	0.72	1.58	0.57	0.068	1.0	0.937	-1.07
<b>Total nitrogen (g/100g) soft cheese</b>	7	17.69	24.30	20.29	2.28	0.41	0.30	0.528	2.6	0.839	2.93
<b>Total nitrogen (g/100g) hard cheese</b>	7	20.16	29.54	24.86	3.27	2.48	1.06	1.086	4.4	0.832	2.10

**Table 4:** accuracy criteria of the FTIR Lactoscope for total nitrogen in cheese samples

n, min, max: number of results, minimum and maximum values; Y: mean results using the reference; Sy: standard deviation of the results from the reference method; d, Sd: mean and standard deviation of deviations; Sy,x: residual standard deviation; b, a: slope and intercept of the linear regression.



**Figures 9, 10, 11 and 12:** Relationship between the FTIR Lactoscope and the reference results for total nitrogen in "pâte fraîche", soft and hard cheese samples.

It can be noted that:

- Overall, the mean and standard deviation of deviations are respectively 1.48 and 1.11 g/100g. The regression slope obtained is significantly different from 1 ( $P = 1\%$ ) and the intercept is significantly different from zero ( $P = 1\%$ ). The residual standard deviation of regression is 1.136 g/100g.
- Concerning fat and dry matter, the slope varies between 0.83 and 0.94 according to the type of cheese, which indicates the necessity for specific adjustments. However, the slopes are similar between soft and hard cheese.

### **2.3- Conclusion**

Concerning fat, dry matter and protein, with no standard criteria or recommendations from the manufacturer, the results obtained should enable an accuracy of the overall estimation (in 95% of cases) of approximately +/- 1.1; 3.0 and 2.3 g/100g respectively after adjustment using representative samples. Specific adjustments according to the type of cheese improve the accuracy of the results.