

**EVALUATION OF THE DELTA INSTRUMENT  
LACTOSCOPE INFRARED ANALYSER**



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## CONTENTS

	PAGE
INTRODUCTION :	
The instruments	
The consumables	
The tests	
1- EVALUATION OF THE STABILITY	
2- EVALUATION OF THE CONTAMINATION	
3 -EVALUATION OF THE LINEARITY	
4- EVALUATION OF THE CALIBRATIONS	
5- EVALUATION OF THE REPEATABILITY AND ACCURACY	
5.1- Cow milk	
5.2- Goat milk	
5.3- Ewe milk	
CONCLUSION	

### Thank to:

- LIAL MC - Aurillac (15), LILCO - Surgères (17) and LIAL FC - Rioz (70) for the supplying of herd milk samples.  
OCL of Jura for the supplying of individual milk samples.

## **The instrument**

The LactoScope is an infrared spectrophotometer (range 4000-400 cm<sup>-1</sup>) used for the determination of the principal components in milk. It is manufactured by Delta Instruments (Advanced group, Netherlands) and commercialised in France by Humeau.

This instrument uses a high resolution industrial infrared spectrophotometer based on Fourier transform (FTIR). The complete infrared spectrum (resolution 8 cm<sup>-1</sup>) is collected and recorded for each sample. With a MLR or PLS calibration, various criteria (fat, protein, lactose, dry matter, urea, freezing point...) can be determined. A conductimeter for the measurement of the freezing point is also present in this instrument.

This apparatus, which is connected to a computer that ensures the signal treatment, can be associated to the "Somscope" somatic cells counter to constitute the "Combiscope".

## **The consumables:**

The necessary consumables were:

- Cleaning solution: aqueous solution of Decon® at 4%
- Zero solution: aqueous solution of triton X100 ® at 0,1%.

## **The tests:**

The evaluation tests concerning fat (equivalent fat filter B), protein, dry matter, urea and freezing point were performed in ACTALIA Cecalait physico-chemistry laboratory (reference and infrared analyses) from February to June 2013.

The stability of the instrument, the contamination between samples, the linearity, the calibrations, the repeatability and the accuracy were evaluated.

The appreciation criteria of the estimated parameters were taken from ISO 8196-3 / IDF 128-3:2010, or from the CNIEL handbooks concerning the use of infrared apparatus with the context of milk payment and milk control in France (CNIEL PROC IR 06 et CNIEL PROC CR IR 04).

The apparatus was configured for a rate of 600 samples per hour and no correction of contamination.

## 1- EVALUATION OF THE STABILITY OF THE INSTRUMENT

### 1.1- Procedure

The stability was evaluated by the analysis, in automatic mode, of milk every 20 minutes, representing 20 measurement cycles. The evaluation was performed on 4 levels of composition in fat and protein with 3 replicates by samples. To evaluate the stability of the instrument, the repeatability and reproducibility were calculated for each analytical criterion and by level.

### 1.2- Results

The following tables present the results obtained:

LEVEL	M (g/l)	Sr (g/l)	Sr (%)	SR (g/l)	SR (%)	r (g/l)	R (g/l)
1	19.780	0.127	0.64	0.127	0.64	0.356	0.356
2	40.187	0.049	0.12	0.050	0.12	0.138	0.140
3	60.635	0.047	0.08	0.067	0.11	0.132	0.189
4	80.434	0.053	0.07	0.087	0.11	0.148	0.244

Table 1: LactoScope stability criteria of fat

LEVEL	M (g/l)	Sr (g/l)	Sr (%)	SR (g/l)	SR (%)	r (g/l)	R (g/l)
1	20.141	0.086	0.426	0.101	0.503	0.240	0.284
2	30.129	0.055	0.183	0.091	0.302	0.154	0.254
3	38.949	0.046	0.117	0.080	0.206	0.128	0.224
4	57.527	0.063	0.109	0.101	0.175	0.175	0.282

Table 2: LactoScope stability criteria for protein

LEVEL	M (g/l)	Sr (g/l)	Sr (%)	SR (g/l)	SR (%)	r (g/l)	R (g/l)
1	98.963	0.265	0.27	0.265	0.27	0.742	0.742
2	128.698	0.194	0.15	0.231	0.18	0.543	0.646
3	156.225	0.162	0.10	0.203	0.13	0.454	0.569
4	193.416	0.223	0.12	0.267	0.14	0.625	0.746

Table 3: LactoScope stability criteria for dry matter

LEVEL	M (m°C x -1)	Sr (m°C x -1)	Sr (%)	SR (m°C x -1)	SR (%)	r (m°C x -1)	R (m°C x -1)
1	550,2	1,2	0,23	1,2	0,23	3,5	3,5
2	568,7	0,7	0,12	0,8	0,13	2,0	2,1
3	576,7	0,6	0,11	0,7	0,13	1,8	2,0
4	597,0	0,6	0,11	0,7	0,12	1,8	2,0

Table 4: LactoScope stability criteria for freezing point

LEVEL	M (mg/l)	Sr (mg/l)	Sr (%)	SR (mg/l)	SR (%)	r (mg/l)	R (mg/l)
1	179,15	11,41	6,37	20,35	11,36	31,94	56,98
2	206,62	12,24	5,92	19,83	9,60	34,27	55,54
3	213,97	17,53	8,19	21,33	9,97	49,08	59,73
4	244,90	15,92	6,50	21,43	8,75	44,58	60,00

Table 5: LactoScope stability criteria for urea

M: mean; Sr and SR (Sr% and SR%): absolute (and relative) standard deviations of repeatability and reproducibility; r and R: maximum deviation of repeatability and reproducibility in 95 % of cases.

### 1.3- Conclusion

The values of standard deviation of reproducibility for fat, protein and urea were below to the limits required in ISO 8196-3 / IDF 128-3 (respectively 0.29, 0.58 g/l and 29 mg/l for the median and high values).

As no standardised values exist for freezing point, it can be noted that the reproducibility standard deviation values are lower than the limit value of the CNIEL PROC CR IR 04 handbook ( $R = 10 \text{ m}^\circ\text{C} \rightarrow \text{SR lower than } 3.6 \text{ m}^\circ\text{C}$ ).

## 2- EVALUATION OF THE CONTAMINATION BETWEEN SAMPLES

### 2.1- Procedure

This criterion was evaluated in automatic analysis mode, by analysing the same cow milk and distilled water according to the sequence: MILK - MILK – WATER – WATER repeated twenty times for fat, protein and freezing point. The evaluation was carried out on 4 levels for fat and protein.

The contamination level was estimated by the formula:

$$Tc (\%) = [ (\Sigma(Eau 1) - \Sigma(Eau 2)) / (\Sigma(Lait 2) - \Sigma(Eau2)) ] \times 100$$

### 2.2- Results

The following table present the results obtained:

LEVEL	Mean values Fat (g/l)	Mean values Protein (g/l)	Mean values Freezing point (m°C)	TC(%) Fat	TC(%) Protein	TC(%) Freezing point
1	19.6	20.1	514.2	0.57	0.69	0.65
2	40.1	30.2	529.4	0.54	0.70	0.83
3	60.5	39.0	534.7	0.55	0.75	0.87
4	80.3	57.6	550.2	0.64	0.80	0.99

Table 6: LactoScope contamination criteria for fat, protein and freezing point

### 2.3- Conclusion

The contamination rates for fat and protein between successive samples are lower than the maximal limit at 1 % required in the ISO 8196-3 / IDF 128-3 standard and in the CNIEL PROC IR 06 handbook.

The value obtained for freezing point are also lower than the maximal limit at 2 % of the CNIEL PROC IR 06 handbook.

## 3- EVALUATION OF THE LINEARITY

### 3.1- Constitution of the samples

Volume/volume dilutions were carried out by corrected weighing of density. This corresponds to the principle of quantitative analysis of infrared spectrophotometry and to the French reference measurements.

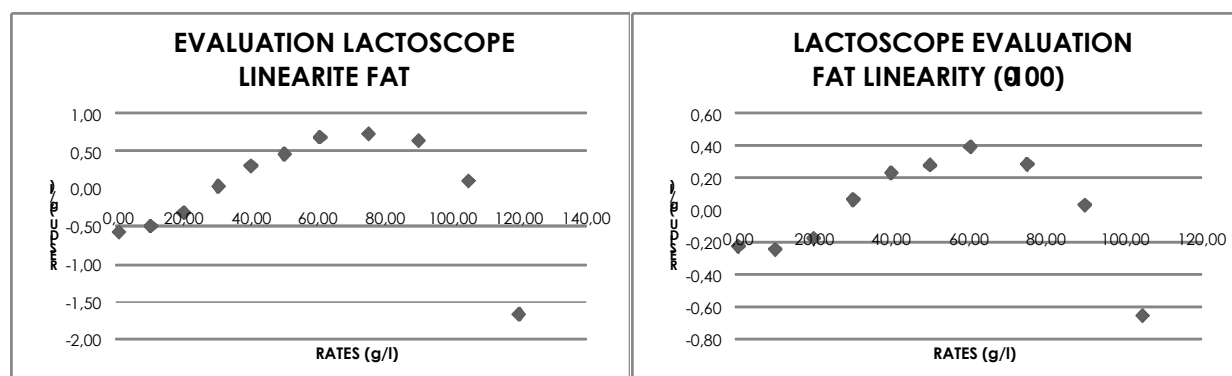
### 3.2- Fat

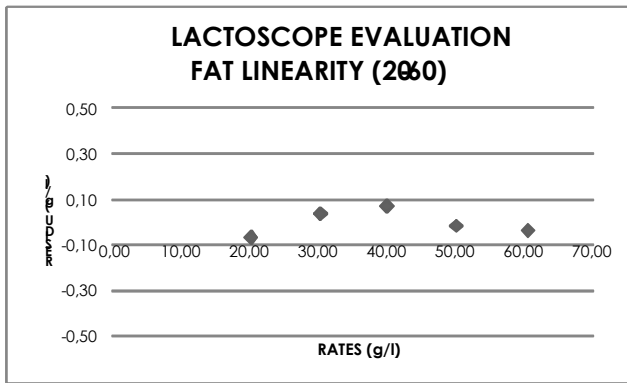
#### 3.2.1- Procedure

A range of 11 milk samples from 0 to 120 g/l was prepared by mixing cream and skimmed milk. Each level was replicated three times.

#### 3.2.2- Results

The following figures illustrate the linearity of the instrument. We can observe the deviations according to the theoretical rates of cream and skimmed milk dilutions.





Figures 1,2 and 3: LactoScope linearity criteria for fat (linear regressions 0-120 g/l,0-100 g/l and 20-60 g/l)

Within the range 0-120 g/l, the Ar/At ratio (Ar and At: amplitude of residues and amplitude of content respectively) is equal to 2.01 %, that corresponds to the limit of 2 % expressed in ISO 8196-3 / IDF 128-3 standard. A linear regression in the range from 0 to about 100 g/l improves the linearity of the instrument (Ar/At ratio equal to 1 %). A Ar/At ratio equal to 0.34 % is obtained with a linear regression in the range from 20 to about 60 g/l, corresponding to the cow milk.

### 3.3- Protein

#### 3.3.1- Procedure

A range of 11 milk samples from 0 to 80 g/l was prepared by mixing the proteic retentate and filtrate obtained by tangential ultrafiltration (cutoff threshold: 10 KD). Each range was analysed three times.

#### 3.3.2- Results

The following figures illustrate the linearity of the instrument. We can observe the deviations according to the theoretical rates of retentate in the ultrafiltrated filtrate.

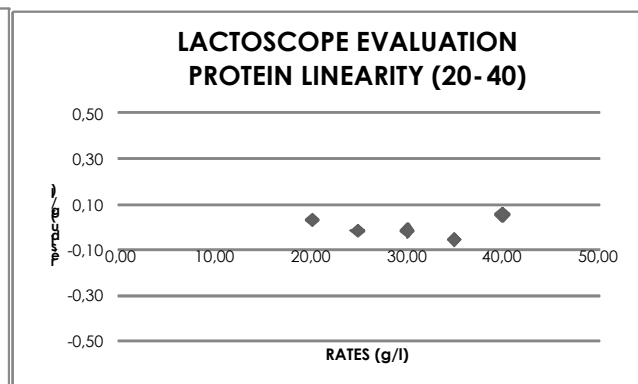
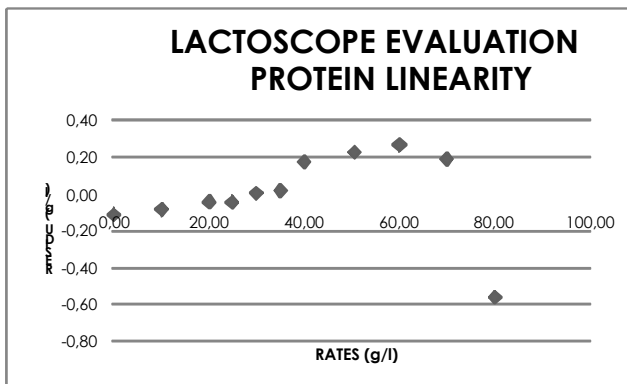


Figure 4 and 5: LactoScope linearity criteria for protein (linear regressions 0-80 g/l and 20-40 g/l)

The Ar/At ratio within the range 0-80 g/l is equal to 1 %, which is in conformity with the recommendations of 2 % maximum given in ISO 8196-3 / IDF 128-3 standard. A Ar/At ratio equal to 0.53 % is obtained with a linear regression in the range from 20 to about 40 g/l, corresponding to the cow milk.

### 3.4- Conclusion

The linearity of the instrument is therefore satisfactory for fat (range from 0 to 100 g/l) and protein (range from 0 to 80 g/l).

## 4- EVALUATION OF THE CALIBRATION

### 4.1- Procedure

The evaluation of the calibration for fat and protein, initially installed by the manufacturer, was performed with 13 commercial "median" and "high" infrared standard reference materials (SRMs) produced by ACTALIA Cecalait in April 2013. Each sample was analysed in duplicate.

#### 4.2- Results

The following table present the results obtained:

	<b>N</b>	<b>Min max</b>	<b>d d%</b>	<b>Sd</b>	<b>SI1</b>	<b>SI3</b>	<b>b a</b>
<b>Fat (g/l)-Median</b>	13	21.8 53.9	0.01	0.20	0.19	0.12	0.992 0.30
<b>Protein (g/l) Median</b>	13	24.2 39.2	-0.24	0.06	0.06	0.04	0.998 0.29
<b>Fat (g/l) High</b>	13	59.8 90.9	-0.06 -0.08%	0.32	0.20	0.11	1.024 -1.72
<b>Protein (g/l) High</b>	13	45.5 66.4	1.41 2.52%	0.11	0.12	0.05	1.001 -1.48

**Table 7:** LactoScope calibration criteria for fat and protein

*N: number of standards, min and max: minimum and maximum values, Sr: standard deviation of repeatability, d and d%: absolute and relative mean deviations (instrument –reference), Sd: standard deviation of deviations (instrument –reference), SI1 and SI3: residual standard deviation of the simple (reference vs instrument) or multiple (reference vs fat, protein and lactose) linear regression, b and a: slope and intercept of the linear regression (reference vs instrument)*

It can be noted that:

- Concerning the median range, the residual standard deviations of the linear regression for fat and protein are closed to the standard deviation of deviations. For fat, an optimisation of the lactose residual interaction enables the reduction of this value (SL3 equal to 0.12 g/l).

- Concerning the high range, the residual standard deviations for fat is lower than the standard deviation of deviations. As for the median range, it can be optimised. For protein, the mean bias is high.

#### 4.3- Conclusion

To conclude, concerning the median range, the mean bias and the regression slope for fat and protein are in accordance with the recommendations of the ISO 8196-3 / IDF 128-3 standard (respectively 0.5 g/l and 1+/-0.05). Concerning the high range, the mean bias (absolute and relative) and the regression slope for fat are in conformity with the recommendations of the ISO 8196-3 / IDF 128-3 standard (respectively 1 g/l, 1.25 % and 1+/-0.05). The mean bias (absolute and relative) for protein are higher than the standardised limits (1 g/l and 1.5 %), probably due to a preliminary defect of calibration on zone milk.

On the other hand, all the residual standard deviations of linear regression obtained for fat and protein are in accordance with the recommendations of the CNIEL PROC IR 06 handbook (respectively lower than 0.25 and 0.15 g/l for the median range and 0.50 and 0.30 g/l for the high range).

### **5- EVALUATION OF THE REPEATABILITY AND ACCURACY**

#### 5.1- Cow milk

##### 5.1.1- Samples

The tests were performed on 100 samples of herd milk from the Franche-Comté region and 130 samples of individual milk from 4 farms in the Jura. Bronopol was added to the individual milk samples to give a final concentration of 0.02 %.

##### 5.1.2- Repeatability

###### 5.1.2.1- Procedure

The repeatability of the instrument was evaluated using all the samples for fat, protein and only using the herd cow milk samples for freezing point. In addition; dry matter and urea were respectively measured using herd cow milk samples and

individual cow milk samples. The quantitative analyses were performed in automatic analysis mode, in duplicate for each set of 20 samples according to the following sequence: Set 1 rep 1 - Set 1 rep 2 - Set 2 rep 1 - Set 2 rep 2 ... Set n rep 1 - Set n rep 2. A control milk was analysed every 20 samples to verify the stability of the analyser.

### 5.1.2.2- Results

The following tables and figures present 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>Fat (g/l)</b>	100	35.79	48.75	40.594	2.398	0.055	0.14%	0.150
<b>Protein (g/l)</b>	100	29.32	36.06	32.846	1.502	0.061	0.19%	0.166
<b>Freezing point (m°C x -1)</b>	100	513	534	525.5	3.6	1.1	0.21%	3.1
<b>Dry matter (g/100g)</b>	100	11.98	13.48	12.717	0.327	0.019	0.15%	0.053

**Table 8:** LactoScope repeatability criteria for fat, protein, freezing point and dry matter in herd cow milk 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 on 95 % of cases

	<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/l)</b>	130	27.151	53.349	38.650	5.234	0.056	0.15%	0.153
<b>Protein (g/l)</b>	130	26.440	43.461	32.576	3.043	0.066	0.20%	0.179
<b>Urea (mg/l)</b>	130	125.4	483.1	285.4	64.740	14.1	4.94%	38.3

**Table 9:** LactoScope repeatability criteria for fat, protein and urea in individual cow milk 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 on 95 % of cases

### 5.1.2.3- Conclusion

For fat and protein in herd and individual cow milk, the standard deviation of repeatability are in accordance with the recommendations of the ISO 8196-3/IDF 128-3 standard and the CNIEL PROC IR 06 handbook ( $Sr \leq 0,14$  g/l). For freezing point, the standard deviation of repeatability obtained is in accordance with the recommendations of the CNIEL PROC CR IR 06 handbook ( $Sr \leq 2$  m°C). For urea, the standard deviation of repeatability is in accordance with the recommendations of the ISO 8196-3/IDF 128-3 standard ( $Sr \leq 14.42$  mg/l). For dry matter, as no standard exists, it can be noted that the standard deviation of repeatability obtained is lower than the limits of the ISO 6731/IDF 21 :2011 standard, which corresponds to the reference method ( $Sr \leq 0,036$  g/l).

### 5.1.3- Accuracy

#### 5.1.3.1- Procedure

The accuracy of the instrument was evaluated in accordance with the repeatability. For fat and protein, the evaluation concerns the values obtained after calibration of the instrument with commercial SRMs produced by ACTALIA Cecalait. For freezing point, the instrumental values are from a calibration carried out by the manufacturer.

The following reference methods were used:

- Fat: Gerber acido-butyrometric method according to NF V 04-210: 2000 (single test and then confirmation if more important residues for the individual milk samples)
- Protein: Amido black method according to NF V 04-216: 2011 (test in duplicate),
- Freezing point: thermistor cryoscopic method according to ISO 5764 / IDF 108: 2009 (single test),
- Dry matter: drying method according to ISO 6731 / IDF 21 : 2011 (single test),
- Urea: differential pH-metry method according to ISO 14637 / IDF 195: 2007 (single test).

### 5.1.3.2- Results

#### 5.1.3.2.1 – Herd milk

The following tables and figures present the results obtained:

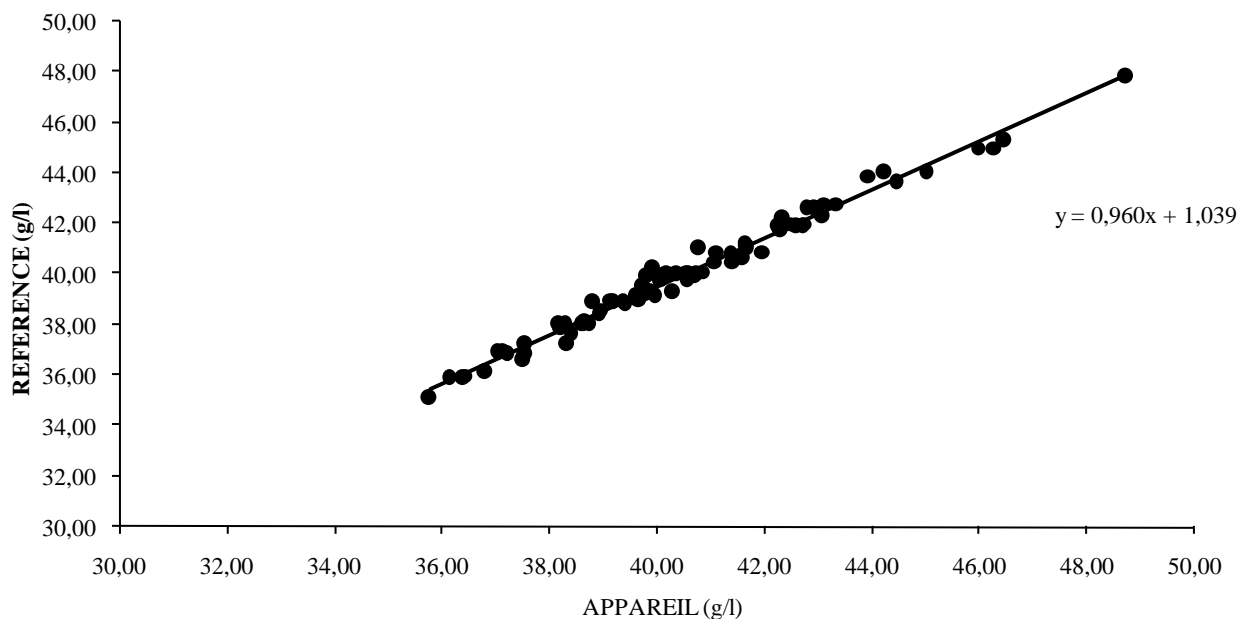
	<b>FAT (g/l)</b>	<b>PROTEIN (g/l)</b>	<b>FREEZING POINT (m°C x -1)</b>	<b>DRY MATTER (g/100g)</b>
<b>n</b>	79	82	84	79



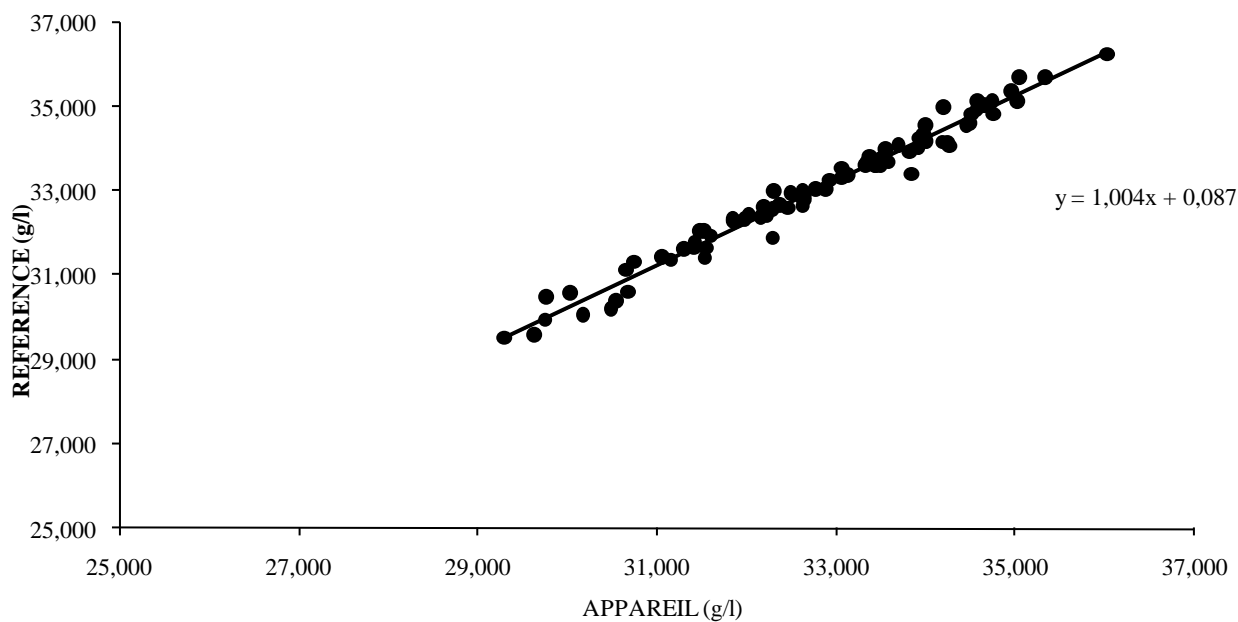
<b>min</b>	35,10	29,54	513	12,02
<b>Max</b>	47,80	36,28	528	13,54
<b>Y</b>	40,054	33,059	519,3	12,762
<b>Sy</b>	2,482	1,543	3,4	0,339
<b>d</b>	0,547	-0,245	5,8	-0,048
<b>Sd</b>	0,321	0,230	3,3	0,043
<b>Sy,x</b>	0,307	0,231	2,8	0,043
<b>Sy,x (%)</b>	0,76	0,70	0,53	0,34

**Table 10:** LactoScope accuracy criteria for fat, protein, freezing point and dry matter in herd cow milk samples  
*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 deviation of deviations; *Sy,x:* residual standard deviation.

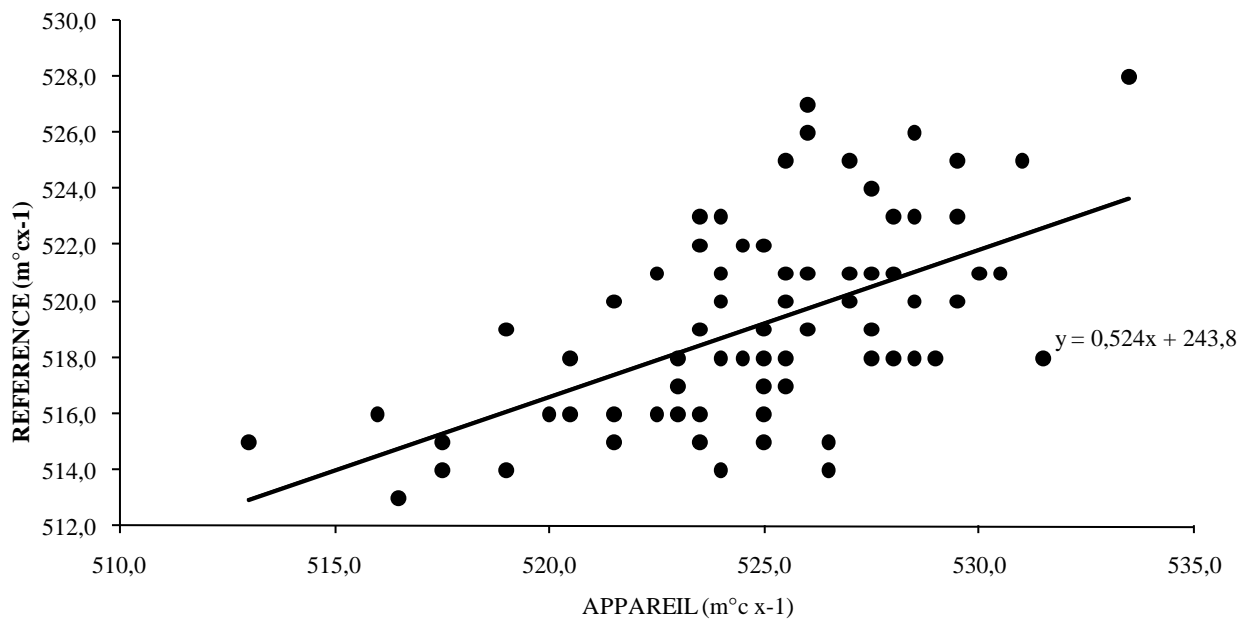
JUSTESSELACTOSCOPE 600  
 LAIT DE MELANGE VACHE  
 MG



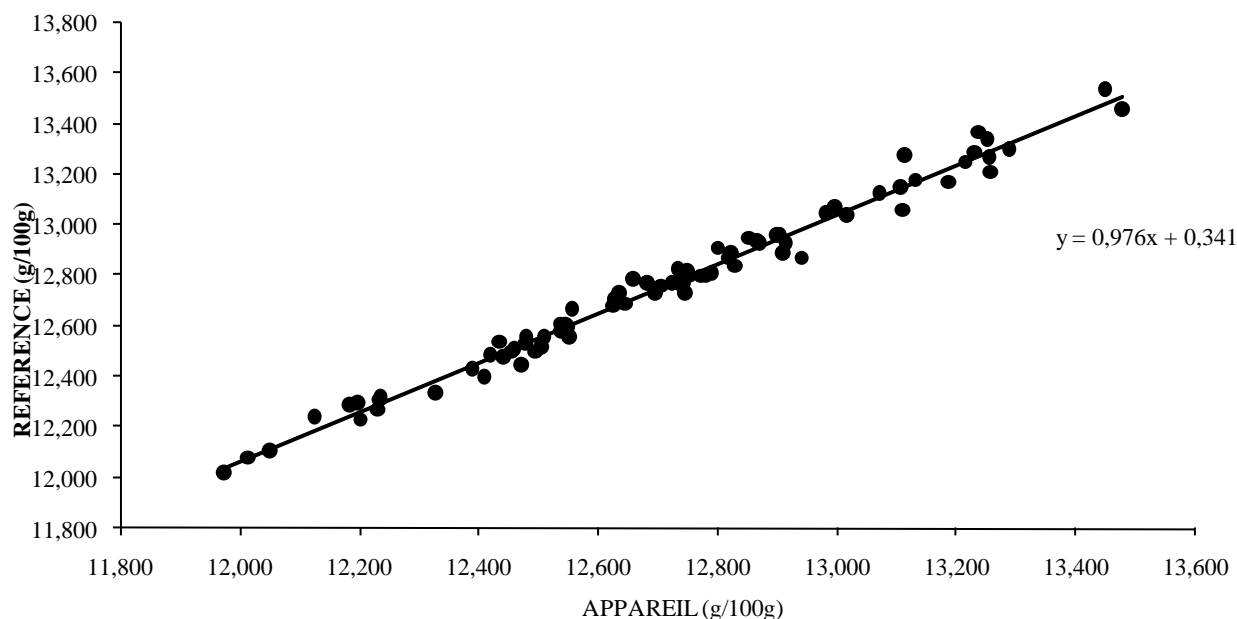
JUSTESSELACTOSCOPE 600  
LAIT DE MELANGE VACHE  
MP



JUSTESSELACTOSCOPE 600  
LAIT DE MELANGE VACHE  
FPD



JUSTESSELACTOSCOPE 600  
LAIT DE MELANGE VACHE  
MS



Figures 6 to 9: Relation between LactoScope and reference results for fat, protein, freezing point and dry matter in herd cow milk

It can be noted that:

↳ **For fat:** the mean and the standard deviation of deviations are respectively equal to 0.547 and 0.321 g/l. The regression slope (0.960) and the intercept (1.039) are significantly different from respectively 1.00 and zero ( $P = 1\%$ ). The residual standard deviation of regression (0.307 g/l) is in conformity with the recommendations of the ISO 8196-3 / IDF 128-3 standard ( $S_{y,x} \leq 0,72$  g/l).

↳ **For protein:** the mean and the standard deviation of deviations are respectively equal to  $-0.245$  and  $0.230$  g/l. The regression slope (1.004) and the intercept (0.087) are not significantly different from respectively 1.00 and zero ( $P = 5\%$ ). The residual standard deviation of regression (0.231 g/l) is in conformity with the recommendations of the ISO 8196-3 / IDF 128-3 standard ( $S_{y,x} \leq 0,72$  g/l).

↳ **For freezing point:** the mean and the standard deviation of deviations are respectively equal to  $5.8$  and  $3.3$   $m^{\circ}C \times -1$ . The regression slope (0.524) and the intercept (243.8) are significantly different from respectively 1.00 and zero ( $P = 1\%$ ). The residual standard deviation of regression is equal to  $2.8$   $m^{\circ}C$ .

↳ **For dry matter:** the mean and the standard deviation of deviations are respectively equal to  $-0.048$  and  $0.043$  g / 100g. The regression slope (0.976) is not significantly different from 1.00 ( $P = 5\%$ ) and the intercept (0.341) is significantly different from zero ( $P = 1\%$ ). The residual standard deviation of regression is equal to  $0.043$  g / 100g.

#### 5.1.3.2.2 – Individual milk

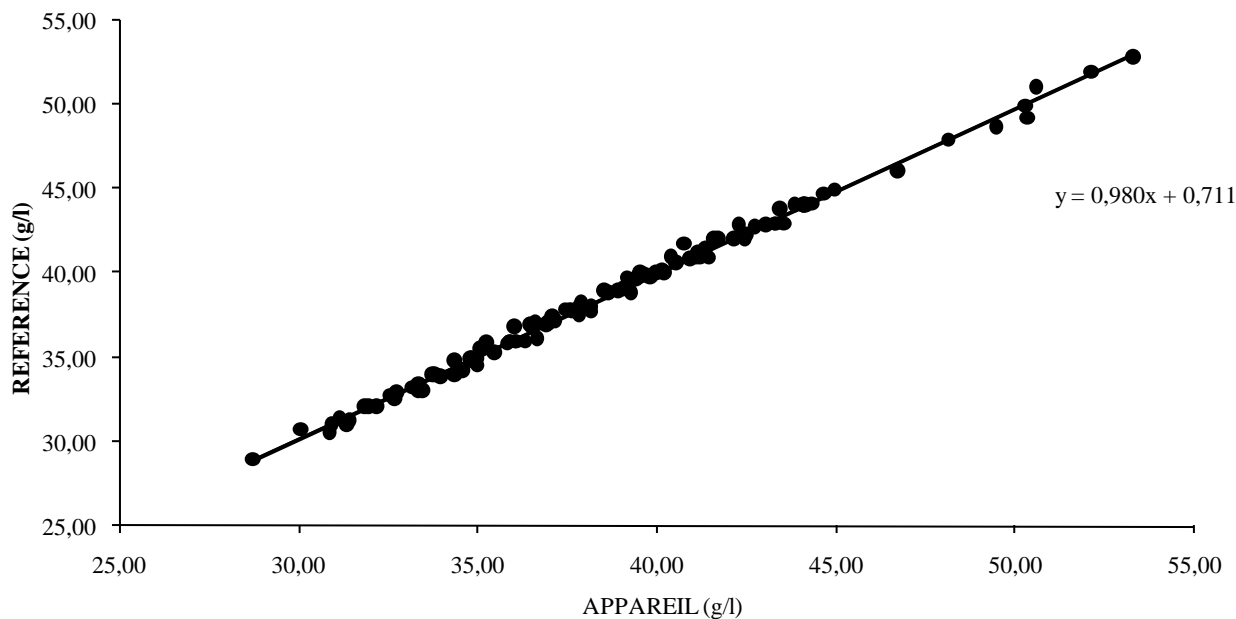
The following tables and figures present the results obtained:

	<b>FAT (g/l)</b>	<b>PROTEIN (g/l)</b>	<b>UREA (mg/l)</b>
<b>n</b>	111	111	109
<b>min</b>	29,00	26,57	219
<b>Max</b>	52,80	39,87	578
<b>Y</b>	38,585	32,585	369,6
<b>Sy</b>	4,937	2,834	65,0
<b>d</b>	0,026	-0,205	-82,9

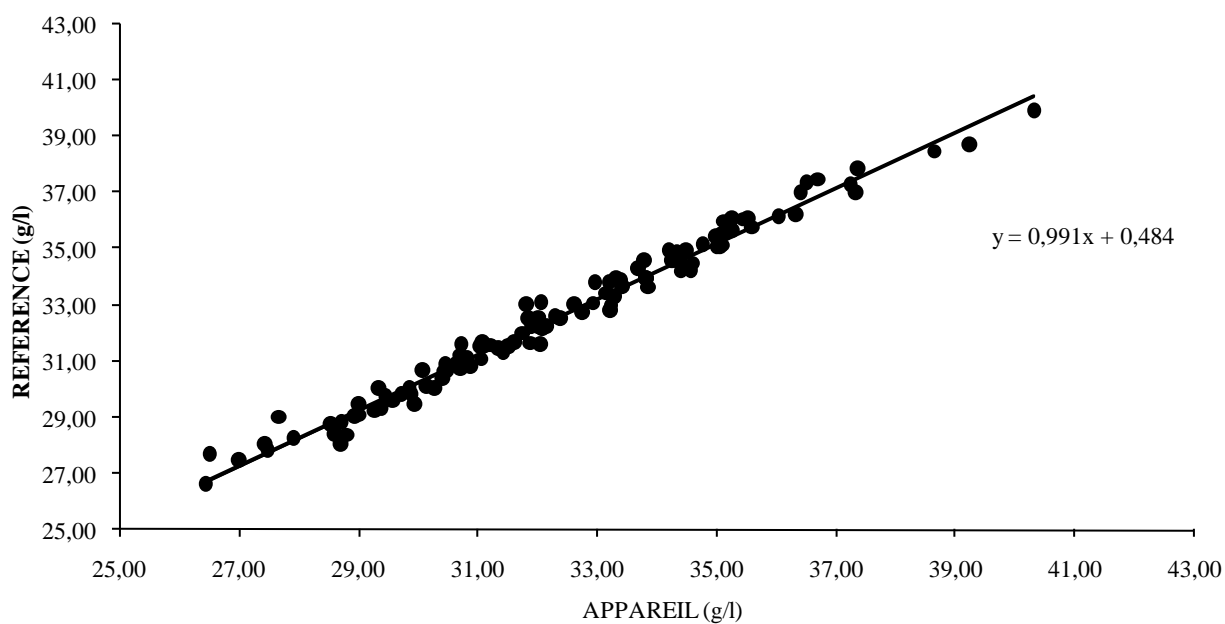
<b>Sd</b>	0,341	0,384	31,3
<b>Sy,x</b>	0,328	0,385	30,4
<b>Sy,x (%)</b>	0,85	1,19	10,60

**Tableau 11:** LactoScope accuracy criteria for fat, protein and urea in individual cow milk samples  
*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 deviation of deviations; Sy,x: residual standard deviation.*

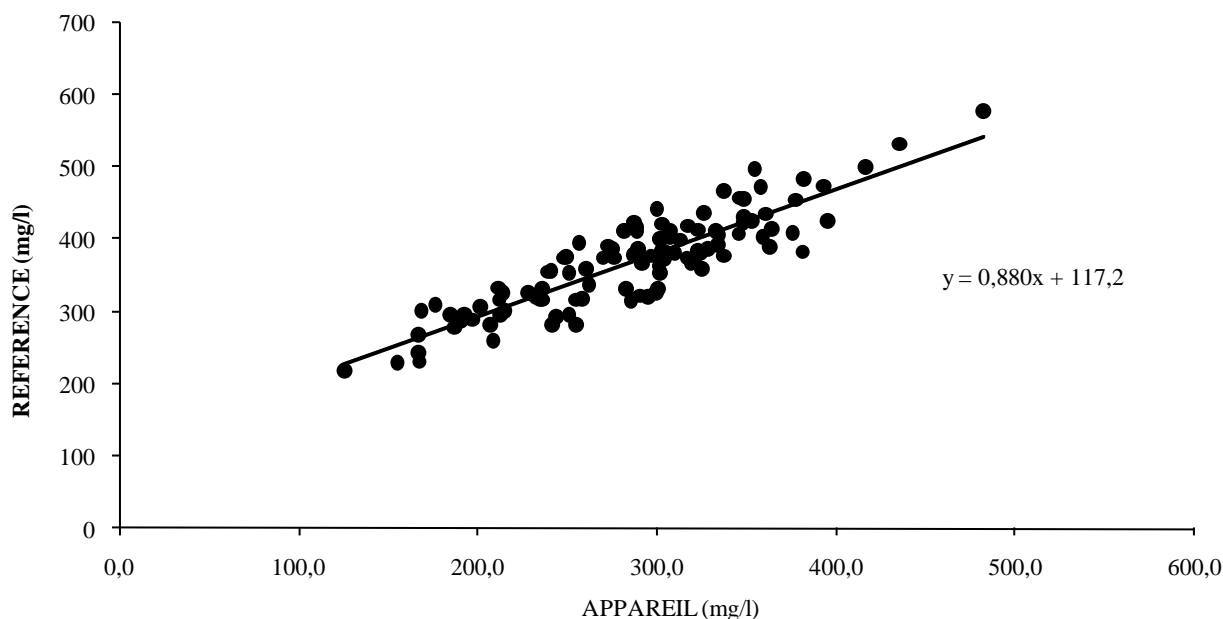
JUSTESSELACTOSCOPE 600  
 LAIT INDIVIDUEL VACHE  
 MG



JUSTESSELACTOSCOPE 600  
 LAIT INDIVIDUEL VACHE  
 MP



JUSTESSELACTOSCOPE 600  
LAIT INDIVIDUEL VACHE  
UREE



Figures 10 to 12: Relation between LactoScope and reference results for fat, protein, freezing point and dry matter in individual cow milk

It can be noted that:

↳ For fat: the mean and the standard deviation of deviations are respectively equal to 0.026 and 0.341 g/l. The regression slope (0.980) is significantly different from 1.00 (P = 1 %) and the intercept (0.711) is not significantly different from zero (P = 5 %). The residual standard deviation of regression (0.328 g/l) is in accordance with the recommendations of the ISO 8196-3 / IDF 128-3 standard ( $S_{y,x} \leq 1.03$  g/l).

↳ For protein: the mean and the standard deviation of deviations are respectively equal to -0.205 and 0.384 g/l. The regression slope (0.991) and the intercept (0.484) are not significantly different from respectively 1.00 and zero (P = 5 %). The residual standard deviation of regression (0.385 g/l) is in conformity with the recommendations of the ISO 8196-3 / IDF 128-3 standard ( $S_{y,x} \leq 1.03$  g/l).

↳ For urea: the mean and the standard deviation of deviations are respectively equal to -82.9 and 31.3 mg/l. The regression slope (0.880) is significantly different from 1.00 (P = 5 %) and the intercept (117.2) is not significantly different from zero (P = 5 %). The residual standard deviation of regression (30.4 mg/l) is in accordance with the recommendations of the ISO 8196-3 / IDF 128-3 standard ( $S_{y,x} \leq 61.8$  mg/l).

#### 5.1.3.3- Conclusion

The results obtained for fat, protein and urea are in accordance with the recommendations of the ISO 8196-3 / IDF 128-3. The residual standard deviation for freezing point (2.8 m°C) and dry matter (0.043 g/100g) enables respectively an accuracy of estimation of +/- 5.6 m°C and +/- 0.086 g/100g.

#### 5.2- Goat milk

##### 5.2.1- Samples

The tests were performed on 88 samples of herd milk from the Poitou-Charentes region.

##### 5.2.2- Repeatability

### 5.2.2.1- Procedure

The repeatability of the instrument was evaluated using all the samples for fat, protein and freezing point. The quantitative analyses were performed in automatic analysis mode, in duplicate for each set of 20 samples according to the following sequence: Set 1 rep 1 - Set 1 rep 2 - Set 2 rep 1 - Set 2 rep 2 ... Set n rep 1 - Set n rep 2. A control milk was analysed every 20 samples to verify the stability of the analyser.

### 5.2.2.2- Results

The following table present 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>FAT (g/l)</b>	88	28,91	43,56	34,906	3,108	0,057	0,16%	0,154
<b>PROTEIN (g/l)</b>	88	26,37	36,60	30,694	1,918	0,061	0,20%	0,166
<b>FREEZING POINT (m°C x -1)</b>	88	547	569	557,5	4,3	1,1	0,20%	3,2

Tableau 12: LactoScope repeatability criteria for fat, protein and freezing point in goat milk 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 on 95 % of cases

### 5.2.2.3- Conclusion

For fat and protein, the standard deviations of repeatability are in accordance with the recommendations of the ISO 8196-3 / IDF 128-3 standard and the CNIEL PROC IR 06 handbook ( $Sr \leq 0.14$  g/l).

For freezing point, the standard deviation of repeatability is in conformity with the recommendations of the CNIEL PROC CR IR 06 handbook ( $Sr \leq 2$  m°C).

### 5.2.3-Accuracy

#### 5.2.3.1- Procedure

The accuracy of the instrument was evaluated in accordance with the repeatability. For fat and protein, the evaluation concerns the values obtained after calibration of the instrument with commercial SRMs produced by ACTALIA Cecalait. (cf §4). For freezing point, the instrumental values are from a calibration carried out by the manufacturer.

The following reference methods were used:

- Fat: Gerber acido-butyrometric method according to NF V 04-210: 2000 (single test)
- Protein: Amido black method according to NF V 04-216: 2011 (test in duplicate),
- Freezing point: thermistor cryoscopic method according to ISO 5764 / IDF 108: 2009 (single test).

#### 5.2.3.2- Results

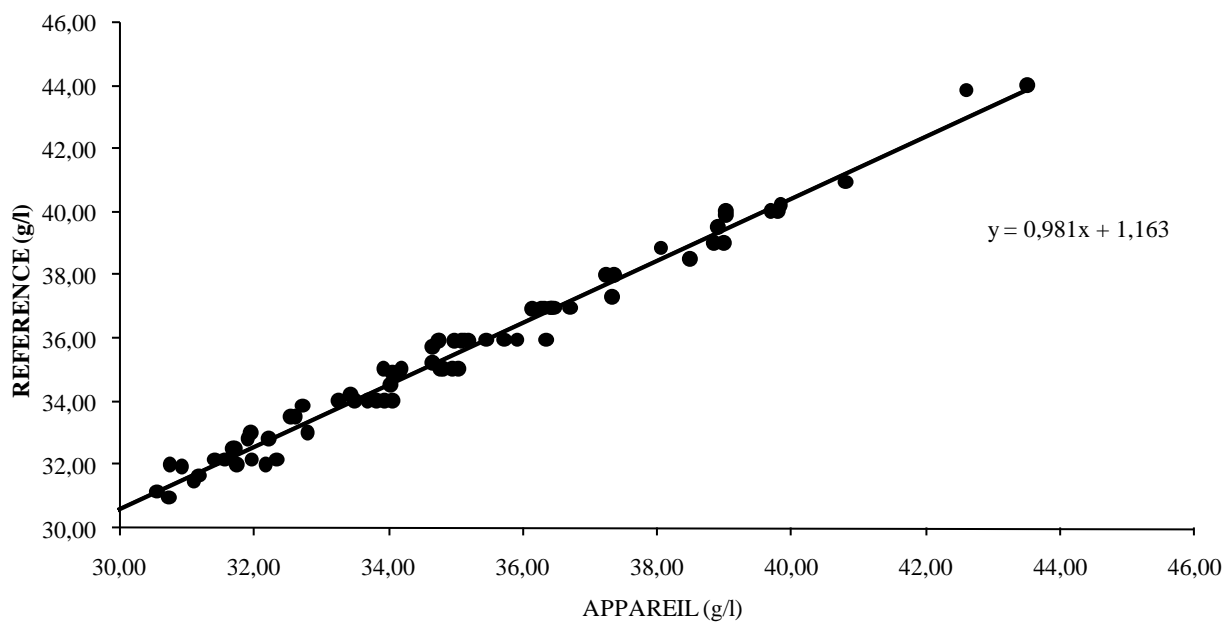
The following tables and figures present the results obtained:

	<b>FAT (g/l)</b>	<b>PROTEIN (g/l)</b>	<b>FREEZING POINT (m°C x -1)</b>
<b>n</b>	74	84	84
<b>min</b>	29,90	27,19	539
<b>Max</b>	44,00	37,86	561
<b>Y</b>	35,481	31,753	550,7
<b>Sy</b>	2,961	2,044	4,7
<b>d</b>	-0,527	-1,026	6,8
<b>Sd</b>	0,384	0,234	3,7
<b>Sy,x</b>	0,383	0,222	3,5
<b>Sy,x (%)</b>	1,10	0,72	0,63

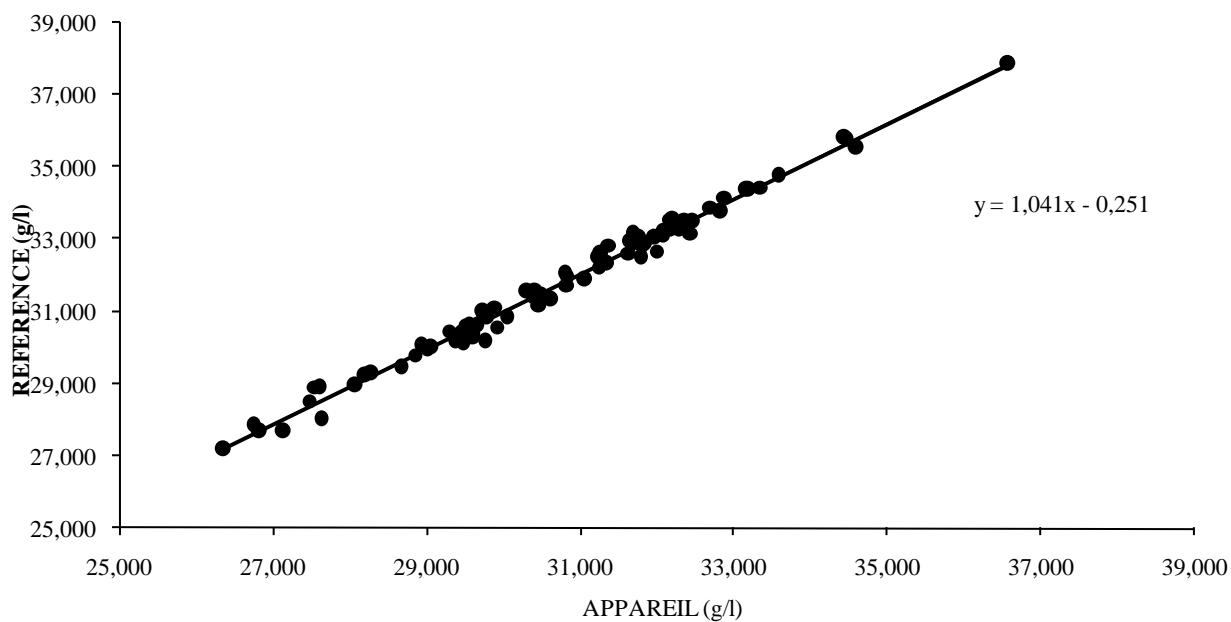
Tableau 13: LactoScope accuracy criteria for fat, protein and freezing point in goat milk samples

*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 deviation of deviations; *Sy,x*: residual standard deviation

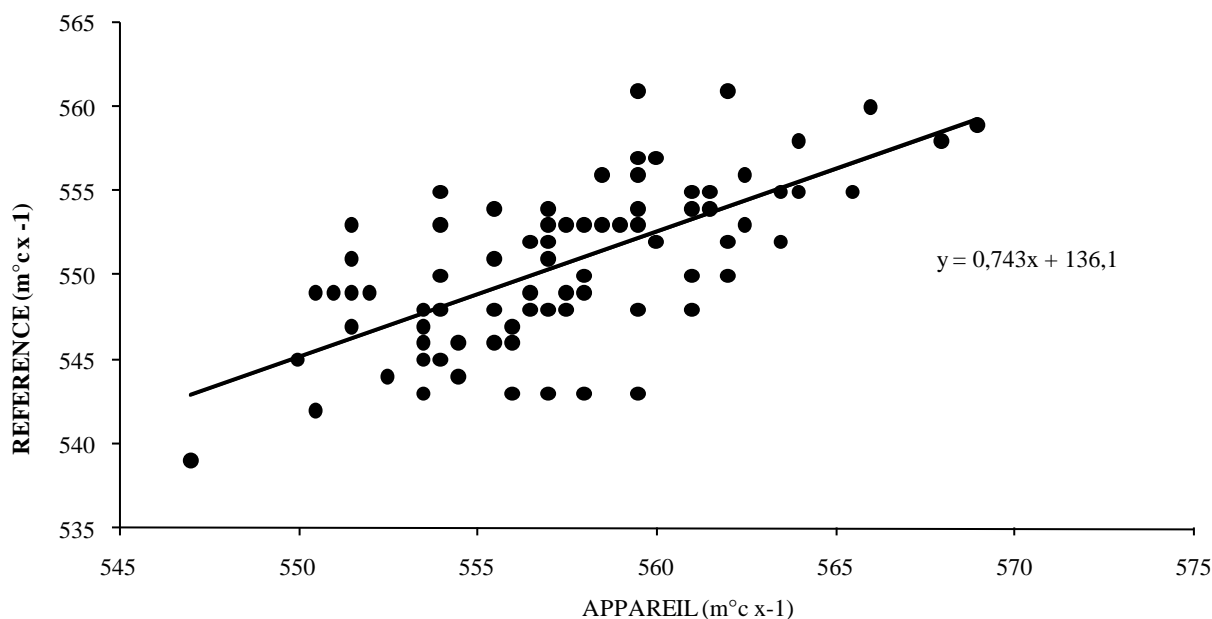
JUSTESSE LACTOSCOPE 600  
LAIT DE MELANGE CHEVRE  
MG



JUSTESSE LACTOSCOPE 600  
LAIT DE MELANGE CHEVRE  
MP



JUSTESSE LACTOSCOPE 600  
LAIT DE MELANGE CHEVRE  
FPD



Figures 13 to 15: Relation between LactoScope and reference results for fat, protein and freezing point in goat milk samples

It can be noted that:

↳ For fat: the mean and the standard deviation of deviations are equal to  $-0.527$  and  $0.384$  g/l. The regression slope ( $0.981$ ) is not significantly different from  $1.00$  ( $P = 5\%$ ) and the intercept ( $1.163$ ) is significantly different from zero ( $P = 1\%$ ). The residual standard deviation of regression ( $0.383$  g/l) is in accordance with the recommendations of the ISO 8196-3 / IDF 128-3 standard ( $S_{y,x} \leq 0.72$  g/l).

↳ For protein: the mean and the standard deviation of deviations are equal to  $-1.026$  and  $0.234$  g/l. The regression slope ( $1.041$ ) is significantly different from  $1$  ( $P = 1\%$ ) and the intercept ( $-0.251$ ) is not significantly different from zero ( $P = 5\%$ ). The residual standard deviation of regression ( $0.222$  g/l) is in accordance with the recommendations of the ISO 8196-3 / IDF 128-3 standard ( $S_{y,x} \leq 0.72$  g/l).

↳ For freezing point: the mean and the standard deviation of deviations are equal to  $6.8$  and  $3.7$  m°C x  $-1$ . The regression slope ( $0.743$ ) and the intercept ( $136.1$ ) are significantly respectively different from  $1.00$  and zero ( $P = 1\%$ ). The residual standard deviation of regression is equal to  $3.5$  m°C.

### 5.2.3.3- Conclusion

The results obtained for fat and protein are in accordance with the recommendations of the ISO 8196-3 / IDF 128-3 standard. The high mean bias for protein is probably due to a preliminary defect of calibration on goat and zone milk. Despite the absence of standard criteria for freezing point, the residual standard deviation obtained ( $3.5$  m°C) enables an accuracy of estimation of  $\pm 7$  m°C.

## 5.3- Ewe milk

### 5.3.1- Samples

The tests were performed on 100 samples of herd milk from the Roquefort sur Soulzon region.

### 5.3.2- Repeatability

#### 5.3.2.1- Procedure



The repeatability of the instrument was evaluated using all the samples for fat, protein and freezing point. The quantitative analyses were performed in automatic analysis mode, in duplicate for each set of 20 samples according to the following sequence: Set 1 rep 1 - Set 1 rep 2 - Set 2 rep 1 - Set 2 rep 2 ... Set n rep 1 - Set n rep 2. A control milk was analysed every 20 samples to verify the stability of the analyser.

### 5.3.2.2- Results

The following table present 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>FAT (g/l)</b>	100	53,26	85,64	68,111	7,799	0,104	0,15%	0,282
<b>PROTEIN (g/l)</b>	100	45,23	64,20	53,644	4,441	0,110	0,21%	0,309
<b>FREEZING POINT (m°C x -1)</b>	100	538	563	553,5	4,3	1,0	0,18%	2,8

**Tableau 14:** LactoScope repeatability criteria for fat, protein and freezing point in ewe milk 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 on 95 % of cases

### 5.3.2.3- Conclusion

For fat and protein, the absolute and relative standard deviations of repeatability (*Sr* and *Sr%*) are in accordance with the recommendations of the ISO 8196-3 / IDF 128-3 standard and the CNIEL PROC IR 06 handbook (respectively lower than 0.29 g/l and 0.35 %).

The standard deviation of repeatability obtained for freezing point is in accordance with the recommendations of the CNIEL PROC CR IR 06 handbook ( $Sr \leq 3 \text{ m}^\circ\text{c}$ ).

### 5.3.3- Accuracy

#### 5.3.3.1- Procedure

The accuracy of the instrument was evaluated in accordance with the repeatability. For fat and protein, the evaluation concerns the values obtained after calibration of the instrument with commercial SRMs produced by ACTALIA Cecalait. (cf §4). For freezing point, the instrumental values are from a calibration carried out by the manufacturer.

The following reference methods were used:

- Fat: Acido-butyrometric method according to NF V 04-155: 2003 (single test)
- Protein: Amido black method according to NF V 04-216: 2011 (test in duplicate),
- Freezing point: thermistor cryoscopic method according to ISO 5764 / IDF 108: 2009 (single test),

#### 5.3.3.2- Results

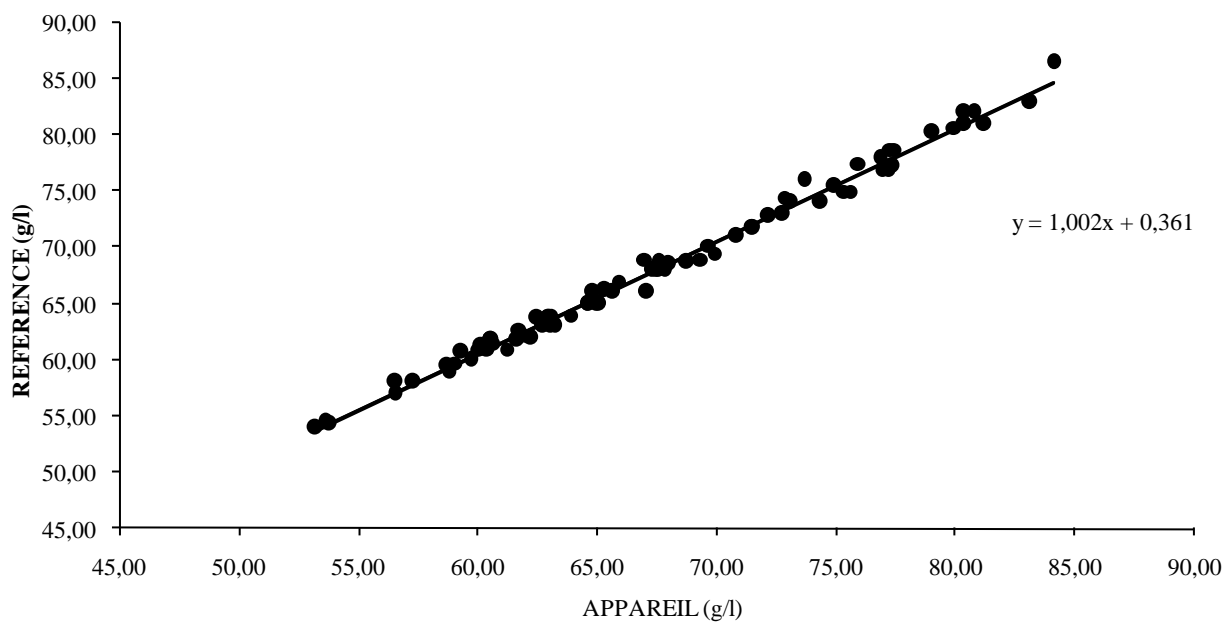
The following tables and figures present the results obtained:

	<b>FAT (g/l)</b>	<b>PROTEIN (g/l)</b>	<b>FREEZING POINT (m°C x -1)</b>
<b>n</b>	79	79	80
<b>min</b>	54,00	45,10	541
<b>Max</b>	86,50	64,28	574
<b>Y</b>	68,127	52,589	559,4
<b>Sy</b>	7,699	4,467	5,8
<b>d</b>	-0,547	0,459	-5,7
<b>Sd</b>	0,665	0,397	5,7
<b>Sy,x</b>	0,669	0,365	5,4
<b>Sy,x (%)</b>	0,99	0,69	0,97

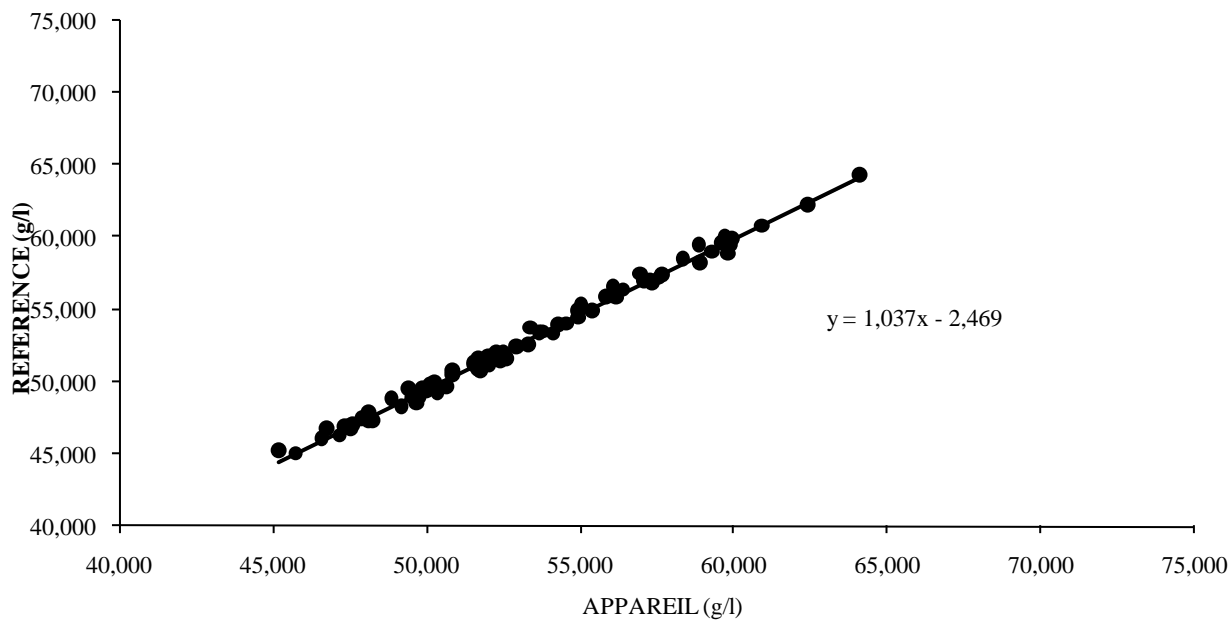
**Tableau 15:** LactoScope accuracy criteria for fat, protein and freezing point in ewe milk samples

*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 deviation of deviations; *Sy,x*: residual standard deviation.

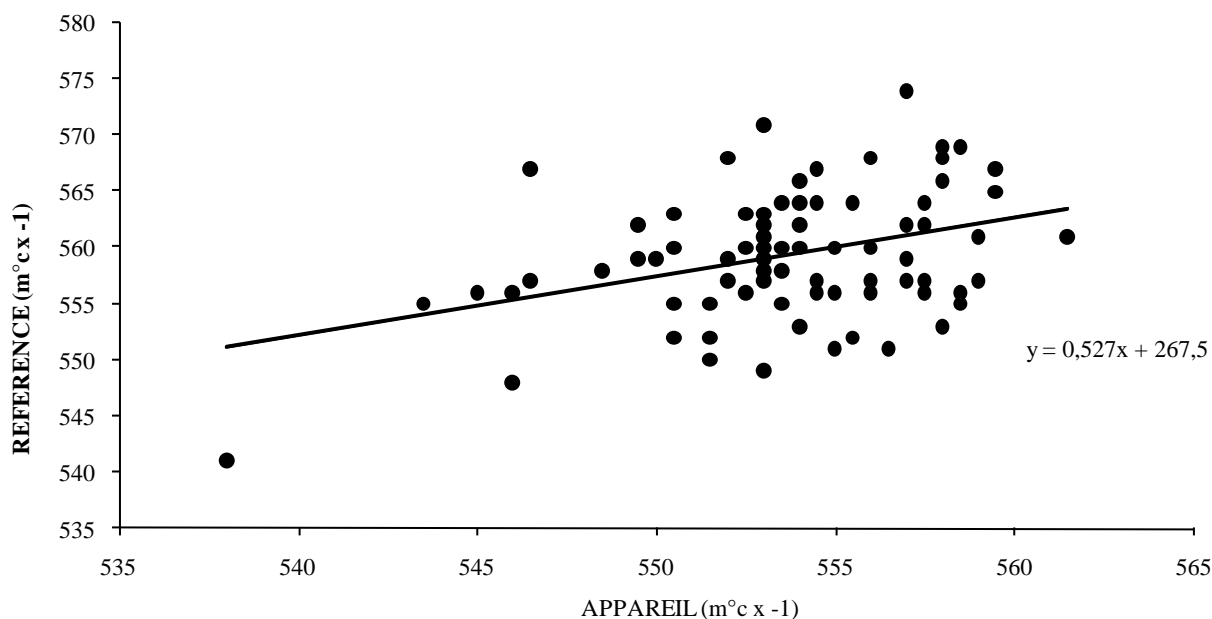
JUSTESSE LACTOSCOPE 600  
LAIT DE MELANGE BREBIS  
MG



JUSTESSE LACTOSCOPE 600  
LAIT DE MELANGE BREBIS  
MP



JUSTESSELACTOSCOPE 600  
LAIT DE MELANGE BREBIS  
FPD



Figures 16 to 18: Relation between LactoScope and reference results for fat, protein and freezing point in ewe milk samples

It can be noted that:

↳ For fat: the mean and the standard deviation of deviations are equal to  $-0.547$  and  $0.665$  g/l. The regression slope ( $1.002$ ) and the intercept ( $0.361$ ) are respectively not significantly different from  $1.00$  and zero ( $P = 5\%$ ). The absolute and relative residual standard deviation of regression ( $0.69$  g/l and  $0.99\%$ ) are in accordance with the recommendations of the ISO 8196-3 / IDF 128-3 standard (respectively lower than  $1.45$  g/l and  $1.75\%$ ).

↳ For protein: the mean and the standard deviation of deviations are equal to  $0.459$  and  $0.397$  g/l. The regression slope ( $1.037$ ) and the intercept ( $-2.469$ ) are significantly different from  $1.00$  and zero ( $P = 1$ ). The absolute and relative residual standard deviation of regression ( $0.365$  g/l and  $0.69\%$ ) are in accordance with the recommendations of the ISO 8196-3 / IDF 128-3 standard (respectively lower than  $1,45$  g/l et  $2,0\%$ ).

↳ For freezing point: the mean and the standard deviation of deviations are equal to  $-5.7$  and  $5.7$  m°C x-1. The regression slope ( $0.527$ ) and the intercept ( $267.5$ ) are respectively significantly different from  $1.00$  and zero ( $P = 1\%$ ). The residual standard deviation of regression is equal to  $5.4$  m°C.

### 5.3.3.3- Conclusion

The results obtained for fat and protein are in accordance with the recommendations of the ISO 8196-3 / IDF 128-3 standard. Despite the absence of standard criteria for freezing point, the residual standard deviation obtained ( $5.4$  m°C) enables an accuracy of estimation equal to  $\pm 10.8$  m°C.

## CONCLUSION

The results obtained for fat, protein and freezing point are in accordance with the recommendations of the ISO 8196-3 / IDF 128-3: 2010 and/or the CNIEL/IE handbooks concerning the use of infrared apparatus with the context of milk payment and milk control in France (CNIEL PROC IR 06, CNIEL PROC CR IR 04) for the three types of milk (cow, goat and ewe).

Moreover, the results obtained for urea (cow individual milk) are in accordance with the recommendations of the ISO 8196-3 / IDF 128-3: 2010 standard. The results obtained for dry matter (cow herd milk) enable accuracy of estimation lower than  $0.1$  g/100g.