EVALUATION OF THE FT6000® INFRARED ANALYSER LIPOLYSIS CRITERION

The FT 6000® has previously been evaluated by CECALAIT for the following composition parameters : fat matter, protein matter and lactose, as well as for determination of freezing point equivalent and urea content. The present evaluation concerns the determination of lypolysis in milk, which could constitute an alternative method to those used at present. The evaluation of repeatability indicates that the standard deviation is higher than that obtained with the reference method BDI, but is of the same order of size as that of the routine method using copper soaps. The accuracy performances are in accordance with the values given by FOSS.

PRINCIPLE AND DESCRIPTION

The FT 6000® is an automated instrument for milk analysis, manufactured and marketed by FOSS. It allows determination of certain parameters in milk (fat matter, protein matter, lactose) and freezing point equivalent. These criteria having previously been evaluated and were the subject of an article in CECALAIT's Newsletter, n°33. The instrument also allows the analysis of other criteria such as the determination of fat matter acidity (FMA).

On the technical side, the apparatus uses a single beam Fourier transform infrared system. The apparatus, coupled to a computer which assures the running and the signal treatment, allows two types of mathematical calculation: traditional PLS for the major components and spectrum PLS for all the components.

The tests

The evaluation tests were carried out in CECALAIT's physico-chemistry laboratory (reference analyses and infrared analyses) from March to July 2002 and concerned the fat matter acidity (FMA) criterion.
The following points were focused on:
▪ Evaluation of repeatability.
▪ Evaluation of accuracy.

❶ Repeatability

Procedure

The repeatability of the apparatus was evaluated using 260 milk samples from herds in the Massif Central region in France. In view of the wide calibration set used by FOSS for this application, only milk samples presenting a value inferior to 1.5 meq/100g were conserved for evaluation of repeatability (that is to say 217 samples). The samples were taken in series of 20 each and dispatched by express in refrigerated packages. The analyses were carried out the day after sample taking. The measurements were carried out in automatic analysis mode, in non consecutive duplicate, for each series of 20 samples.

The evaluation of repeatability was carried out on a population covering levels ranging from 0.458 to 1.485 meq/100g (0.173 to 0.672 meq/l).

Table 1 :Evaluation of repeatability

	n	M	Sx	Sr	Sr (%)	r
AMG / FMA (meq/100g)	217	1.046	0.221	0.044	4.17	0.121
AMG (meq/l)		0.427	0.093	0.018	4.15	0.049

With : n: number of samples
M : mean
Sx : standard deviation of the results
Sr and Sr% : relative and absolute standard deviation of repeatability
r : maximal difference between duplicates in 95 % of occurrences

Conclusion

The standard deviation of repeatability (Sr) is higher than that of the reference method BDI (doc FIL 265/1991 r $\leq$ 0.05 meq/100g MG either Sr $\leq$ 0.018 meq/100g MG or 0,0072 meq/l).
However, Sr is of the same order of size as that of the copper soaps method used by the interprofessional laboratories (CNIEL PROC LIP 0402/00 Sr $\leq$ 0.018 meq/l).

2 Accuracy

Procedure

The apparatus was evaluated using the same milk samples as those used for the evaluation of repeatability. Immediately after the instrumental analyses, the samples were cooled to 20°C and then analysed using the BDI method.
Only samples for which the reference values were lower than 1.5 meq/100g fat were retained for evaluation of accuracy (that is to say 189 samples).

Infrared analyses

The infrared analyses were carried out in non consecutive duplicate. The mean of two repeats was calculated and used to determine the results.
The evaluation concerned the values obtained from the non adjusted calibration carried out by FOSS using local milk samples.

Reference analyses

The determination of fat matter acidity was carried out, in duplicate, using the BDI method according to IDF bulletin 265: 1991. The value taken into account for evaluation of this criterion corresponds to the mean of two non consecutive replicates.

Statistical analysis

The accuracy was appreciated by means of the deviations, the standard deviation of the deviations, the residual standard deviation and the estimated equation for the regression line, by using, for the ordinate Y, the reference method, and for the abscissas X, the Milkoscan® FT6000.

Table 2: : Evaluation of accuracy

|	AMG (meq/100g)	AMG (meq/l)
n	189	
Min	0.336	0.134
Max	1.479	0.598
Y	0.783	0.319
X	1.046	0.426
Sy	0.247	0.103
d	0.263	0.107
Sd	0.1439	0.0598
Sy,x	0.1411	0.0584
b	0.870	0.864
a	-0.127	-0.049

With : n:number of samples
M : mean
r : maximal difference between duplicates in 95 % of occurrences
Sx : standard deviation of the results
Sr and Sr% : relative and absolute standard deviation of accuracy

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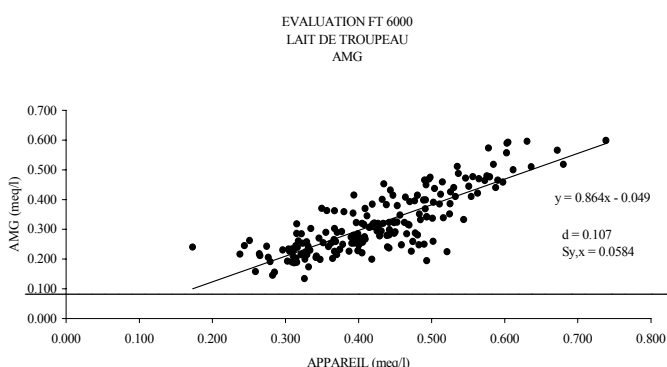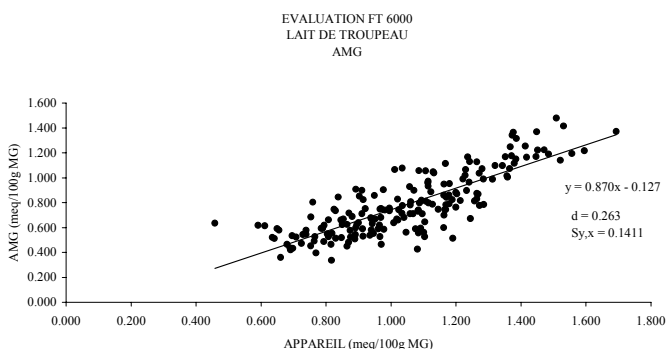
The above table and figures show the results and the regression lines obtained.

The slope and the intercept obtained (b = 0,864; a = -0,049) are significantly different from 1 and 0 (P=1%). The residual standard deviation of regression obtained is 0,0584 meq/litre.

Conclusion

The accuracy performances obtained during this evaluation are slightly better than the previous tests carried out by FOSS in comparison with the BDI method (Sy,x = 0,184 meq/100 g) and the copper soaps method (Sy,x = 0,066 meq/litre).

The mean difference observed (+ 0,26 meq/100 g), as well as the significant difference in the slope compared to 1 (b = 0,870), are, in all likelihood, linked to the BDI method used (to date there exists different protocols that lead to different results, notably according to the nature and the pH of the extractant). The influence of the geographical origin of the milk (notably concerning the choice of samples for the calibration set) is a parameter yet to be determined.

GENERAL CONCLUSION

The infrared FT6000® analyser evaluation results confirm the results obtained from the different tests carried out and communicated by FOSS.

To date, the results have been transmitted to the scientific and technical commission of the ministry of agriculture and to the CNIEL, who will rule on this alternative method and, if necessary, will define the modalities of its use within the context of milk payment according to quality.

Authors : QUERVEL X. et TROSSAT Ph.
Translation Helen LAMPRELL

Abbreviations

FMA: fat matter acidity
CNIEL: Centre National Interprofessionnel de l'Economie Laitière / Interprofessional Center for the Dairy Economy
FIL: Fédération Internationale de Laiterie / IDF : International Dairy Federation
PLS: partial least square / moindres carrés partiels

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