EVALUATION OF THE FT 6000 ® INFRARED ANALYSER: UREA CRITERION
(according to the evaluation report -phase I- from CECALAIT)

The Fourier transform infrared analyser, FT 6000® manufactured and marketed by FOSS, has previously been evaluated by CECALAIT for determination of fat matter, protein matter and lactose content in milk, as well as for determination of the freezing-point equivalent (see Cecalait's Newsletter n° 33). In July 2002, the apparatus was evaluated for determination of urea content. The repeatability and accuracy values, as much for individual milk as for herd milk, confirm the manufacturer's specifications and the improvement in performances in relation to the MSC 4000® .

PRINCIPLE AND DESCRIPTION

The FT 6000® uses a single beam Fourier transform infrared system. The apparatus is coupled to a computer that assures the running and the signal treatment. It allows two types of mathematical calculation: traditonnal 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, who carried out the reference analyses as well as the infrared analyses.

The tests concerned urea and focused on the following points:
- Evaluation of repeatability
- Evaluation of accuracy

❶ Repeatability

The repeatability of the apparatus was evaluated in automatic analysis mode, using 146 individual cow's milk samples taken from 7 Jura cattle farms and 55 herd milk samples from the Franche-comté region. The samples covered a range of urea levels from 172 to 696 mg/l. The measurements were carried out in duplicate for each series of 20 samples.

The samples were conserved with bronopol (0,02 %).

Tableau/Table 1 : Evaluation de la répétabilité/ Evaluation of repeatability

Lait / milk	n	M	Sx	Sr	Sr (%)	r
individuel individual milk	146	444	98,1	9,2	2,06	25,4
troupeau herd milk	55	369	62,2	8,7	2,35	24,0
			UREE (mg/l) UREA (mg/l)			

Avec / with : n: nombre de résultats / number of samples
M : moyenne des résultats / mean

Sx : écart-type des résultats / standard deviation of the results
Sr et Sr% : écart-type de répétabilité absolu et relatif / relative and absolute standard deviation of repeatability
r : écart maximal de répétabilité dans 95% des cas / maximal difference between duplicates in 95% of occurrences

The standard deviation of repeatability, Sr, was conform with the manufacturer's specifications (Sr < 15 mg/l). As a matter of interest, the standard deviation of repeatability of the reference method specified in the draft standard ISO / FIL 195 is Sr = 5,4 mg/l (r = 15 mg/l).

❷ Accuracy

The apparatus was evaluated using the same individual and herd milk samples as those used to evaluate repeatability.

The infrared analyses were carried out in non consecutive duplicate. A standard milk sample was inserted every 20 samples to verify the stability of the signal. The value taken into account for evaluation of this criterion corresponds to the mean of two non consecutive repetitions.

The evaluation concerned the values obtained from the unadjusted calibration carried out by FOSS with local milk samples.

The method used here for determination of urea content is the enzymatic method by differential pHmetry according to the draft standard ISO FIL 195.

The value used as a reference corresponds to the mean of two repetitions.

The accuracy is appreciated by the mean deviations, the standard deviation of deviations, the residual standard deviation and the estimated equation for the regression line taking into account the explained variable Y, the reference method and the explicative variable X, the Milkscan FT6000®.
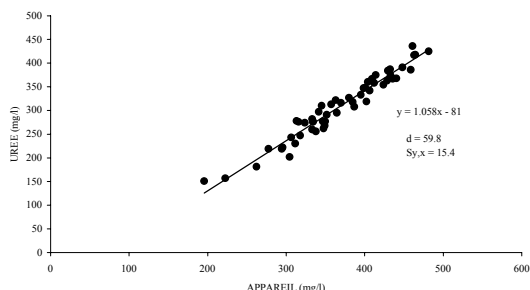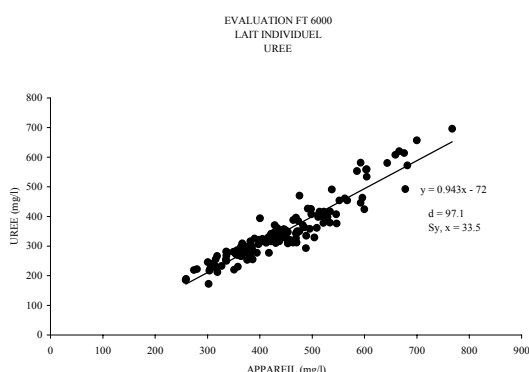
y = 1.058x - 81

d = 59.8
Sy,x = 15.4

Figure 2 : Lait de troupeau / herd milk

Table 2 and figures 1 and 2 present the results and the regression lines for all the individual milk samples and the herd milk samples. Concerning :

- the individual milk samples, the slope and the intercept obtained from the regression (b = 0,943 ; a = - 72) are significantly different from 1 and 0 (P=1%). The residual standard deviation of regression is 33,5 mg/l.

- the herd milk samples, the slope and the intercept obtained from the regression (b = 1,058 ; a = - 81) are significantly different from 1 (P = 5%) and from zero (P = 1%). The residual standard deviation of regression is 15,4 mg/l.

The residual standard deviations of regression obtained are conform with the manufacturer's specifications (individual milk Sy, x < 35 mg/l; herd milk Sy, x < 30 mg/l). The mean difference observed, from - 70 to - 80 mg/l for individual and herd milk, between the infrared and reference methods, is in all likelihood linked to either the origin of the milk samples used for the calibration carried out by FOSS, or to the chemical method used for determination of the reference values.

GENERAL CONCLUSION

The evaluation results of the FT 6000® infrared analyser confirm the specifications announced by FOSS as far as repeatability and accuracy are concerned.

For routine use, an adjustment for local milk seems to be necessary to improve the accuracy of the calibration.

Finally, an improvement in accuracy in relation to the MSC 4000 (see: CECALAIT's Newsletter, n° 21) is appreciable since the residual standard deviations of regression have decreased from 48,3 to 33,5 mg/l for individual milk and from 37,9 to 15,4 mg/l for herd milk.

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Tableau/ Table 2 : évaluation de la justesse / evaluation of accuracy

laits / milks	individuel / individual	troupeau / herd
n	146	55
min (mg/l)	172	151
max (mg/l)	696	436
Y (mg/l)	347.0	309.3
X (mg/l)	444.1	369.2
Sy (mg/l)	98.4	67.6
d (mg/l)	97.1	59.8
Sd (mg/l)	33.8	15.7
Sy,x (mg/l)	33.5	15.4
b	0.943	1.058
a	- 72	- 81

n : nombre de résultats / number of results
min : valeur minimum / minimum value
max: valeur maximum / maximum value
Y,X : moyenne des résultats par méthode de référence et instrumentale / mean results using the reference and instrumental methods
Sy : écart-type des résultats par méthode de référence / standard deviation of the results using the reference method
d, Sd : moyenne et écart-type des écarts / mean and standard deviation of the differences
Sy,x : écart-type résiduel / residual standard deviation
b, a : pente et ordonnée à l'origine de la régression linéaire / slope and intercept of the regression line.

Figure 1 : Lait individuel / individual milk

y = 0.943x - 72

d = 97.1
Sy, x = 33.5

Abbreviations

FPD : Freezing Point Detection / point de congélation
IRTF : infra-rouge à transformée de Fourier
FTIR : Fourier transform Infra Red
PLS : Partial Least Square / moindres carrés partiels

Bibliography

▪ **Projet de norme ISO / FIL 195 -**
Lait - Détermination de la teneur en urée - Méthode enzymatique par pHmétrie différentielle (Méthode de référence)
Milk - Determination of urea content - Enzymatic method using the difference in pH measurement (Reference method)

▪ **Evaluation : le MILKOSCAN 4000 (lactose, urée, point de congélation)**. La Lettre de CECALAIT, n° 21, janvier 1997, p.1-4

▪ **Evaluation : le MILKOSCAN 6000 (matière grasse, protéines, lactose, point de congélation)**. La Lettre de CECALAIT, n° 33, 2ème trim. 2000, p. 2-7.

▪ QUERVEL, X. TROSSAT, P. - **Rapport d'évaluation de l'analyseur infra-rouge FT 6000 ® critère urée**, CECALAIT, 2002, Poligny, 5 p.