EVALUATION OF PANREAC® REAGENTS

The objective of this study was to evaluate the adequacy of certain reagents, proposed by PANREAC, for the realisation of tests on milk and cheese. These tests, realised in CECALAIT's physico-chemical laboratory from January to June 2006, were performed on the 3 following test-reagent couples:

- **Determination of fat in milk according to the acido-butyrometric method NF V 04-210 (1) (Gerber): sulphuric acid 90% (ref. 121010) and amylic acid (ref 125715)**

- **Determination of fat in cheese according to the acido-butyrometric method NF V 04-287 (2) (Heiss): acetic acid (ref. 131008) and perchloric acid 60% (ref. 131054)**

- **Determination of fat in milk according to the Röse-Gottlieb method NF ISO 1211(3): ammoniac 25% (ref 121129), ethanol (ref 12086), petroleum ether 40-60 (ref 131315) and diethyl ether (ref 212770).**

1/- Acido-butyrometric method NF V 04-210 (Gerber)

1.1/- Procedure

The following two types of test were performed:

- Test, in duplicate, on 4 reference samples (Gerber SRMs) over 2 consecutive months (January and February 2006).

- Comparative test, in duplicate, on 10 samples of mixtures of raw milk in relation to the reagents used by CECALAIT.

1.2/- Results

Reference samples (SRMs)

Table 1 presents the results obtained. The results observed correspond to the mean of two repetitions performed in repeatability conditions (deviations between duplicates < 0.5 g/l). The reference value corresponds to the SRM's assigned value.

SRM 01 LGER	OBSERVED (g/l)	REFERENCE (g/l)
X	41,19	41,33
d	-0.14	
t	2,57	
SRM 02 LGER	OBSERVED (g/l)	REFERENCE (g/l)
X	41,06	41,16
d	-0,10	
t	3,10	

Table 1: Results of the "Gerber" tests realised on 2 consecutive SRMs
X: arithmetic mean of the results, d: mean deviations between the results observed and the reference value, t: t-Student value between the mean of the results observed and the reference value.

The mean deviations observed are low and not statistically significant (risk 5%).

Samples of mixtures of milk

Table 2 presents the results obtained. The results observed correspond to the mean of two repetitions performed in repeatability conditions (deviations between duplicates < 0.5 g/l).

ID	OBSERVED (g/l)	CECALAIT (g/l)
X	41,25	41,25
Sx	1,92	1,91
d	< 0,01	
t	< 0,01	
F	1,01	
Sd	0,14	

Table 2*:* : Results of the "Gerber" tests realised on milk samples
X et Sx: arithmetic mean and standard deviation of the results, d: mean deviation between the results observed and CECALAIT's results, t: t-Student value between the means, F: F value of Snedecor between the variances

The mean deviation between the two sets is quasi-nul (< 0,01 g/l). The parameters F and t are not significant (risk 5%).

2/- Acido-butyrometric method NF V 04-287 (Heiss)

2.1/- Procedure

A comparative test, in duplicate, on 9 samples of cheese, in relation to the reagents used by CECALAIT, was performed.

2.2/- Results

Table 3 presents the results obtained. The results observed and CECALAIT's values correspond to the mean of two repetitions performed in repeatability conditions (deviations between duplicates < 0.5 g/100g).

N°	ID	OBSERVED (g/100g)	CECALAIT (g/100g)
1	Soft cheese	29,5	29,5
2	"Carré de l'est"	31,00	31,13
3	Soft cheese	32,00	31,63
4	Light soft cheese	11,75	11,75
5	"Camembert"	24,75	24,86
6	"Brie"	31,63	31,63
7	"Comté"	36,75	36,75
8	"Emmental"	29,63	29,5
9	Hard cheese	15,38	15,5
X		26,93	26,92
Sx		8,24	8,19
d		0,01	
t		< 0,01	
F		1,00	
Sd		0,16	

Table 3*:* : Results of the "Heiss" tests realised on cheese samples
X and Sx: arithmetic mean and standard deviation of the results, d: mean deviation between the results observed and CECALAIT's results, t: t-Student value between the means, F: F value of Snedecor between the variances

The mean deviation between both sets is very low (0,01g/100g). The parameters F and t are not significant (risk 5%).

3/- Röse-Gottlieb method NF ISO 1211

3.1/- Procedure

Two types of test were performed:

- a test, in duplicate, on 7 reference samples (SRM EXTRACTION 05/2006)

- a comparative test, in duplicate, on 13 samples of mixtures of milk in relation to the reagents used by CECALAIT.

3.2/- Results

↳ Reference samples

Table 4 presents the results obtained. The results observed and CECALAIT's values correspond to the mean of two repetitions performed in repeatability conditions (deviations between duplicates < 0,20 g/kg). The reference value corresponds to the SRM's assigned value.

SRM 05 LEXT	OBSERVED (g/kg)	REFERENCE (g/kg)
X	39,99	39,99
d	< 0,01	
t	< 0,01	

Table 4: Results of the "Röse-Gottlieb" tests realised on SRMs
X: arithmetic mean of the results, d: mean deviation between the results observed and the reference values, t: t-Student value between the mean of the results observed and the reference value.

The mean deviations observed are very low (< 0,01 g/kg) and not significant (risk 5%).

↳ Milk samples

Table 4 presents the results obtained. The results observed and CECALAIT's values correspond to the mean of two repetitions performed in repeatability conditions (deviations between duplicates < 0,20 g/kg).

N°	OBSERVED (g/kg)	CECALAIT (g/kg)
X	38,60	38,56
Sx	1,93	1,93
d	0,04	
t	0,03	
F	1,00	
Sd	0,17	

Table 5*:* Results of the "Röse-Gottlieb" tests realised on milk samples
X and Sx: arithmetic mean and standard deviation of the results, d: mean deviation between the results observed and CECALAIT's results, t: t-Student value between the means, F: F value of Snedecor between the variances

The mean deviation between the sets is low (0,04 g/kg) and the F and t tests are not significant (risk 5%).

4/- Conclusion

Concerning both the acido-butyrometric methods (NF V 04-210 and NF V 04-287) and the extraction method (NF ISO 1211), the results obtained are not statistically different from the reference values.
However, all the reagents tested (sulphuric acid 90% ref 121010, amylic alcohol ref 125715, acetic acid ref 131008, perchloric acid 60% ref 131054, ammoniac 25% ref 121129, ethanol ref 12086, petroleum ether 40-60 ref 131315 and diethyl ether ref 212770) permit to obtain results equivalent to these obtained with other reagents available on the market.

Bibliography:

(1) AFNOR NF V 04-210 standard: 2000 «Lait - Détermination de la teneur en matière grasse - Méthode acido-butyrométrique»

(2) AFNOR NF V 04-287 standard: 2002 «Fromages - Détermination de la teneur en matière grasse - Méthode acido-butyrométrique»

(3) NF EN ISO 1211 standard: 2001 «Lait - Détermination de la teneur en matière grasse - Méthode gravimétrique»