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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»*

PRESENTATION OF THE NEW EUROPEAN DIRECTIVE CONCERNING HYGIENE

Summary of the talk by Mrs VION (DGAL) at CECALAIT's AGM 2006

The food "hygiene package" corresponds to many European regulations, based on the food law (regulation (EC) n° 178/2002 laying down the general principles and requirements of food law, establishing the European food safety authority and laying down procedures in matters of food safety). Following the publication of this regulation, many specific texts, addressed to professionals and control services, have been published. Indeed, the regulations (EC) n° 852/2004 on the hygiene of foodstuffs and (EC) n° 853/2004 concerning specific hygiene rules for food of animal origin, constituting a regulatory basis, apply to professionals. To complete these documents, other texts such as the regulations (EC) n° 2073/2005, (EC) n° 2074/2005, (EC) n° 2075/2005 and (EC) n° 2076/2005 have come into effect. This regulatory modification also implied the set up of new national arrangements, and repercussions on the different sectors, in particular for the raw milk – dairy products sector.

THE BASIC DOCUMENTS

- Regulation (EC) n° 852/2004

This text applies to all stages of the food chain, including primary production (except for private domestic use) and its related activities (transport, storage, handling...).

This regulation establishes, for food business operators, general rules in matters of food hygiene, such as:

- the declaration of all the establishments and agreements,
- the primary responsibility of the food business operators in matters of food safety,
- the respect of the guidelines on good hygiene practices (maintenance of the cold chain...),
- the procedures based on the principles of HACCP,
- the guidelines on good hygiene practices and application of the HACCP,
- the training of staff.

- Regulation (EC) n° 853/2004

This text, which applies to foodstuffs of animal origin, completes the regulation (EC) n° 852/2004 using specific requirements. It does not apply to:

- food containing both products of plant origin and processed products of animal origin,
- small quantities of primary products,
- for private domestic consumption,
- local retail establishments (however, a Member State may adopt national measures to apply this requirement).

The annex of this text establishes the definitions, the requirements concerning many products of animal origin (identification marking, procedures based on the HACCP principles; information concerning the food chain) and the specific conditions of each type of establishment (section IX: raw milk and dairy products).

COMPLEMENTARY TEXTS

To complete both these regulations, new texts have come into effect. Thus, the regulation (EC) n° 2073/2005 concerning the microbiological criteria for foodstuffs became operative on 1st January 2006. This text establishes:

- Food safety criteria defining the acceptability of a product or a batch of products. These criteria are applicable to products on the market. When testing of these criteria provides unsatisfactory results, the product or batch of foodstuffs is withdrawn or recalled.
- Process hygiene criteria defining the acceptability of the processes. These criteria are applicable mainly at the end of the manufacturing process. When testing of these criteria provides unsatisfactory results, the food business operators must take corrective measures.
- Rules for sampling.

Other regulations, such as (EC) n° 2074/2005 concerning implementation of measures for certain products, (EC) n° 2075/2005 concerning specific rules on official controls for *Trichinella*, and (EC) n° 2076/2005 concerning transitional arrangements (of which one of them concerns the verification of the maximum plate count for raw cows' milk), have also come into force. The measures established in the regulation (EC) n° 2074/2005 derogate from regulation (EC) n° 852/2004 for foodstuffs with traditional characteristics (a ministerial decree and a note to the European Commission are in the process of being written, in order to define the conditions of attribution of the derogations for the use of locals or traditional materials in the context of manufacturing of traditional products).

THE NECESSITY OF NATIONAL ARRANGEMENTS

The regulations mentioned above are a statutory base, which can be completed by texts from national regulations, if the community regulations give permission or foresee them.

- National regulation

It was necessary to establish national arrangements in several cases:

- for the areas for which the regulations do not apply,
- when national arrangements are explicitly foreseen by the regulation (traditional method, geographic constraint, national rules until publication of community rules),
- when the regulation is silent (precisions may be absolutely necessary).

Thus, the national regulation will be simplified, compatible with the community regulation, and the redundant or contrary arrangements to the European regulation will be repealed.

The above texts are applicable to all foodstuffs, implying evolutions in the different sectors.

IMPACTS ON THE RAW MILK – DAIRY PRODUCTS SECTOR

This sector is subject to the regulations mentioned above, particularly the regulation (EC) n° 853/2004. Indeed, many definitions are recovered or specified in annex I of this text. Therefore, the definition of "raw milk" stays the same, whereas other definitions such as "milk production holding" or "dairy products" are specified.

Chapter I of annex III section IX (raw milk and dairy products) specifies arrangements for raw milk-primary production. It specifies:

- health requirements for raw milk production (with regard to brucellosis and tuberculosis),
- hygiene on milk production holdings (premises and equipment, hygiene rules during milking, collection and transport, and staff hygiene rules),
- criteria for raw milk:
 - milk quality: these criteria apply to milk from all species. The criteria concerning micro-organisms and somatic cell contents are the same as the criteria mentioned in the regulation (EC) n° 92/46, but the *Staphylococcus* criterion has disappeared.
 - the checking of these criteria may be carried out by the food business operator producing the milk, the food business operator collecting or processing the milk, a group of food business operators, or in the context of a national or regional control scheme.

Chapter II establishes requirements concerning dairy products. It specifies:

- temperature requirements: milk must be quickly cooled and stored at 6°C, except if its processing begins immediately after milking, or within four hours of delivery, or if a competent authority authorises a higher temperature for technological reasons concerning the manufacture of certain dairy products,
- requirements for heat treatment: the requirements described in this part are very general but precisions are described in the regulation (EC) n° 2074/2005 (definition of pasteurisation and heat treatment),

- criteria for raw cows' milk before processing: the micro-organisms content must be < 300000 / ml if dairy products are prepared using raw cows' milk, and < 100000 / ml if using processed cows' milk (see transitional arrangements in the regulation (EC) n° 2076/2005),
- rules for conditioning, packaging, labelling (mention of "raw milk" or "made with raw milk") and identification marking.

To complete this text, the regulation (EC) n° 2073/2005 presents the food safety and process hygiene criteria for milk and dairy products (see tables in "La Lettre de CECALAIT" n° 56, 1st quarter 2006). The exceeding of indicator process criteria

induces the setting up of controls concerning safety criteria. It is notably the case for the detection of staphylococcal enterotoxins in dairy products, of *Salmonella* spp. and *Enterobacter sakazakii* in dried infant formulae and dried dietary foods for special medical purposes intended for infants below 6 months of age.

The enumeration of *Enterobacteriaceae*, *Escherichia coli* and coagulase positive staphylococci have now been introduced in the process hygiene criteria.

You will find the complete texts mentioned in this article on the website <http://eur-lex.europa.eu/en/index.htm>

NEW EU STANDARDS AND REGULATIONS

Classification is established in alphabetical order of the first keyword

ADDITIVES / SWEETENERS
<p>O.J.E.U. L 204, 26th July 2006 – Directive 2006/52/EC of the European Parliament and of the Council of 5 July 2006 amending Directive 95/2/EC on food additives other than colours and sweeteners and Directive 94/35/EC on sweeteners for use in foodstuffs http://eur-lex.europa.eu/LexUriServ/site/en/oj/2006/l_204/l_20420060726en00100022.pdf</p>
DESIGNATION OF ORIGIN
<p>O.J.E.U. C 211, 2nd September 2006 – Publication of an application pursuant to Article 6(2) of Council Regulation (EC) No 510/2006 on the protection of geographical indications and designations of origin for agricultural products and foodstuffs ("Tome des Bauges") http://eur-lex.europa.eu/LexUriServ/site/en/oj/2006/c_211/c_21120060902en00080011.pdf</p>
PESTICIDES / RESIDUE LEVELS
<p>O.J.E.U. L 175, 29th June 2006 – Commission Directive 2006/59/EC of 28 June 2006 amending Annexes to Council Directives 76/895/EEC, 86/362/EEC, 86/363/EEC and 90/642/EEC as regards maximum residue levels for carbaryl, deltamethrin, endosulfan, fenithrothion, methidathion and oxamyl http://eur-lex.europa.eu/LexUriServ/site/en/oj/2006/l_175/l_17520060629en00610076.pdf</p>
<p>O.J.E.U. L 206, 27th July 2006 – Commission Directive 2006/61/EC of 7 July 2006 amending the Annexes to Council Directives 86/362/EEC, 86/363/EEC and 90/642/EEC as regards maximum residue levels for atrazine, azinphos-ethyl, cyfluthrin, ethephon, fenthion, methamidophos, methomyl, paraquat and triazophos http://eur-lex.europa.eu/LexUriServ/site/en/oj/2006/l_206/l_20620060727en00120026.pdf</p>
<p>O.J.E.U. L 206, 27th July 2006 – Commission Directive 2006/62/EC of 12 July 2006 amending the Annexes to Council Directives 76/895/EEC, 86/362/EEC, 86/363/EEC and 90/642/EEC as regards maximum residue levels for desmedipham, phenmedipham and chlorfenvinphos http://eur-lex.europa.eu/LexUriServ/site/en/oj/2006/l_206/l_20620060727en00270035.pdf</p>
VETERINARY MEDICINAL PRODUCTS / RESIDUE / FOODSTUFFS
<p>O.J.E.U. L 225, 17th August 2006 - Commission Regulation (EC) No 1231/2006 of 16 August 2006 amending Annexes I and II to Council Regulation (EEC) No 2377/90 laying down a Community procedure for the establishment of maximum residue limits of veterinary medicinal products in foodstuffs of animal origin, as regards ceftiofur and polyoxyethylene sorbitan monooleate and trioleate http://eur-lex.europa.eu/LexUriServ/site/en/oj/2006/l_225/l_22520060817en00030004.pdf</p>

STANDARDS, DRAFT STANDARDS

Classification in alphabetic order by theme

ISO published standards

CHEESE		
CHEESE / RHEOLOGICAL PROPERTIES	ISO 17996:2006 (IDF 205)	CHEESE Determination of rheological properties by uniaxial compression at constant displacement rate
MICROBIOLOGY OF FOOD AND ANIMAL FEEDING STUFFS		
COLIFORMS / HORIZONTAL METHOD	ISO 4831 August 2006	MICROBIOLOGY OF FOOD AND ANIMAL FEEDING STUFFS Horizontal method for the detection and enumeration of coliforms -- Most probable number technique
MILK, MILK PRODUCTS AND MESOPHILIC STARTER CULTURES		
MILK / MILK PRODUCTS / MESOPHILIC STARTER CULTURES / LACTIC ACID BACTERIA	ISO 17792 (IDF 180) August 2006	MILK, MILK PRODUCTS AND MESOPHILIC STARTER CULTURES Enumeration of citrate-fermenting lactic acid bacteria - Colony-count technique at 25 degrees
MILK POWDER		
MILK POWDER / SOY AND PEA PROTEINS	ISO 17129 (IDF 206) July 2006	MILK POWDER Determination of soy and pea proteins using capillary electrophoresis in the presence of sodium dodecyl sulfate (SDS-CE) – Screening method

IN THE PRESS – ON THE WEB

Classification in alphabetical order of keywords

MILK / DAIRY PRODUCTS / STANDARDS

Report of the seventh session of the Codex Alimentarius on milk and dairy products – Queenstown, New Zealand, 27 March – 1 April 2006

http://www.codexalimentarius.net/download/report/654/al29_11e.pdf

► This report resume the questions which will be considered by the 29th session of the Codex Alimentarius Commission.

Report of the 29th session of the Codex Alimentarius Commission – Geneva, Switzerland, 3-7 July 2006

ftp://ftp.fao.org/codex/CAC/CAC29/al29_41e_adv.pdf

► This report give for each committee the different points adopted or amended, the draft standard proposed during this session.

SALMONELLA / DETECTION

Sensitive salmonella detection

<http://www.foodproductiondaily.com/news/printNewsBis.asp?id=69918>

► Oxoid developed a kit of salmonella detection giving results in less than 24 hours. This kit can be used with raw meat and poultry, dairy products, eggs and other fresh products.

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