2004

2nd quarter

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CECALAIT's NEWSLETTER

LIFE AT CECALAIT – LIFE AT CECALAIT – LIFE AT CECALAIT

CECALAIT's annual general meeting

The AGM was held on 9th June 2004 at the "Maison du Lait" in Paris. For the occasion, the representatives of the administration council and the AGM were re-elected.
The composition of the two authorities is as follows:

Administration council		General Assembly	
1st college : Founding members		**1st college : Founding members**	
CNIEL	- *L. DEVELET*	CNIEL	- *L. DEVELET*
	- *M. DENIEUL*		- *M. DENIEUL*
	- *JP JAMET*		- *JP JAMET*
Institut de l'Elevage	- *V. DAVID*	Institut de l'Elevage	- *V. DAVID*
	- *J. DELACROIX*		- *J. DELACROIX*
	- *M. MARGUET*		- *M. MARGUET*
INRA	- *P. GARNOT*	INRA	- *P. GARNOT*
	- *J. LEONIL*		- *J. LEONIL*
	- *C. MICHEL*		- *C. MICHEL*
2nd College : Associative members		**2nd College : Associative members**	
Private individual Labs	- *E. MALLO*	Private individual Labs	- *B. VOIVENEL*
Society Coop. Labs	- *M. PLACE*	Society Coop. Labs	- *M. PLACE*
Other labs and diverse French organisations	- *V. OVERNEY*	Other labs and diverse French organisations	- *V. OVERNEY*
Interprofessional Labs and milk control	- *D. BOINETTE*	Interprofessional Labs and milk control	- *D. BOINETTE*
Foreign members	- *J. LALOUX*	Foreign members	- *J. LALOUX*

The rest of the morning was spent reviewing the diverse activities of CECALAIT, concerning the production of each service, normalisation at a national and international level, the research and development programmes, as well as the quality surveillance committee report.

The afternoon session was devoted to the presentation of a selection of technical and scientific subjects:

- P. ROLLIER (CECALAIT) presented the latest developments concerning the **methods of enumeration of *Pseudomonas spp* in dairy products.**
- M. PRIEL (LNE) gave a general presentation concerning the **uncertainty of analysis and test result measurements.**
  This intervention was followed by two talks illustrating the different principals:
- B. LOMBARD (AFSSA) presented the **normalisation projects on the uncertainty of microbiological measures that are in process.**
- Ph. TROSSAT (CECALAIT) presented examples of different methods for the **estimation of uncertainty for physico-chemical methods.**

As each year, the day was under the seal of joviality and fruitful exchanges.

The evolution of CECALAIT's website !!

A customer service area will soon be on-line at www.cecalait.fr.
By way of an identity and a password, each laboratory will be able to input all their results on-line, as well as to consult aptitude test participation reports.
On the same principal, CECALAIT's Newsletter will also be available, in full, on-line soon for all the member laboratories.
The finalisation of the instalment of these new functions is scheduled for the end of 2004.
Therefore, each of CECALAIT's members will receive, sometime in September 2004, a letter indicating their identity and password.

A SINGLE REPRESENTATIVE FOR YOUR CLAIMS

Following the creation of a quality assurance assistant / client relations post, CECALAIT has decided to centralise all your claims with Nadine PERROT, who occupies this function at CECALAIT. We invite you therefore to contact her directly by telephone +33 3 84 73 63 12 or by email: n.perrot@cecalait.fr. She will then register your claim and follow it through.

SECONDARY REFERENCE MATERIALS FOR METHOD CONTROL: A WAY OF APPRECIATING THE ACCURACY OF RESULTS

The standard ISO 5725-6 : 1994 (Paragraph 4.2.3: Comparison with a reference value for a laboratory) offers a way of appreciating the accuracy of results during the analysis of reference materials: the calculation of the critical difference with a 95% probability level. This calculation is based on the technical performances of the analytical method described in the corresponding normative documents (reliability parameters: repeatability and reproducibility).

It can be calculated as follows:

That is to say, $\bar{y}$, the mean of n test results (under repeatability conditions) on a SRM presenting a reference value of μ_0. The critical difference, CD, for $(y-\mu_0)$ is determined according to the standard ISO 5725-6: 1994 (paragraph 4.2.3) as follows:

$$CD = (1/\sqrt{2}) \sqrt{[(2.8\ \sigma R)^2 - (2.8\ \sigma r)^2\ ((n-1)/n)]}$$

σr: standard deviation of repeatability
σR: standard deviation of reproducibility
n: number of determinations of the SRM

1) Example in microbiology: TOTAL GERMS SRM

Reference document ISO 4833: 2003 – Food Microbiology – Horizontal method for enumeration of micro-organisms – Colony counting technique at 30°C-

In milk: Repeatability: **r = 0,25 log**
Reproducibility: **R = 0,45 log**

- **Case of duplicate analyses of SRM:**

CD = 0.293 log

In % CFU/ml, in comparison to the reference:
+96% for superior values
-49% for inferior values

For a SRM reference value of 100 000 germs/ml or 5.00 log

The mean log values of the two analyses must be between 4.71 log (-49%) et 5.29 log (+96%).

If, for example, the values obtained are respectively 45 000 and 50 000, two calculations are possible:

1) Log values obtained are 4.65 and 4.70 of which the mean is 4.68 < 4.71 therefore not acceptable.
2) *For the original values in CFU ml: the inferior limit is –49% of the reference value, that is 51 000. The mean of the two results is 48 000, which is inferior to 51 000 therefore not acceptable.*

- **Case of a single SRM analysis:**

CD = 0.318 log

In % CFU/ml, in comparison to the reference:
+108% for superior values
-52% for inferior values

For a SRM reference value of 100 000 germs/ml or 5.00 log

The log value obtained must be between 4.68 log and 5.32 log.

If, for example, the values obtained are respectively 45 000 and 50 000, two calculations are possible:

1) 50 000 (4.70 log) is acceptable; 45 000 (4.65 log) is not acceptable.
2) *For the original values in CFU/ml: the value obtained must be between 48 000 (-52%) and 208 000 (+108%). 50 000 is acceptable; 45 000 is not acceptable.*

3) Example in chemistry: MILK DRY MATTER SRM

Reference document NF V 04 367 : 1985 – Milk, cream and unsweetened concentrated milk: Determination of dry matter

In milk: Repeatability: **r = 0.10g / 100g**
Reproducibility: **R = 0.20g / 100g**

• **Case of duplicate analyses of SRM:**

CD = 0.13g / 100g

For a SRM reference value of 13.00g / 100g, the mean of two analyses must be between 12.87 et 13.13g / 100g

• **Case of a single SRM analysis:**

CD = 0.14g / 100g

For a SRM reference value of 13.00 g /100g, the observed value must be between 12.86 et 13.14 g/100 g

This approach is an existing possibility for the exploration of reference material test results, based on the reliability characteristics of the tested method, described in the corresponding normative documents. It allows a tolerance level to be fixed, beyond which a search for the causes can be engaged and corrective action can be set up. The prerequisite for this approach is a good adequacy of the reliability values with the veritable performance of the method. In all new publications since about 2000, the values are calculated according to the ISO protocol corresponding to CECALAIT's normative sector (8 laboratories from at least two countries, 6 representative samples from the domain of application in blind duplicate and calculated according to the standard ISO 5725 parts 1 et 2). For the older documents, it is better to stay vigilant on the pertinence of the reliability values, and therefore the critical differences obtained by help of this calculation method. It could be thought that this standardised approach is succeeded by the calculation of a maximum limit value, which it would not seem opportune to exceed in this type of application. However, according to the case: calibration and verification of an instrumental method for example, other methods of calculation aiming at the determination of a tolerance level could be set up by the laboratories, as much as it corresponds to their needs.

ASSESSMENT OF THE AFSSA / CECALAIT CRYOSCOPY TESTS With the collaboration of SYNDILAIT (provision of consumer milk), RADIOMETER and FUNKE GERBER (loan of apparatus).

The new standard ISO 5764 concerning the freezing point of milk was published in 2002. In comparison with the previous text, this document clearly defines the reference method as being a plateau finding method (with a new definition of the plateau which is considered as having been reached when the temperature increase has not exceeded 0.5m°C over the last 20 seconds). The fixed time methods are considered to be routine methods and need to be tied to the reference method.

Following the application of the new method, certain countries have observed an interval of approximately 3m°C on samples analysed by means of both an apparatus complying with the new standard and an apparatus complying with the previous text.

Following the presentation of the results to a group of expert chemists (DG AGRI CEE), the president H. GLAESER expressly asked the other member states to voice their conclusions on this analytical comparison.

It seemed therefore essential for us to conduct a comparative study, at the French level, on raw milk and consumer milk, to determine if a significant interval exists between the two versions of the method, or not.

The study consisted in a comparison, in two laboratories, of results obtained between apparatus functioning according to the 1987 version and the 2002 version:
Laboratory 1: one apparatus complying with the standard ISO 5764 : 1987 and one complying with standard ISO 5764: 2002
Laboratory 2: one apparatus complying with the standard ISO 5764: 1987 and two complying with standard ISO 5764: 2002
The study was carried out on 26 raw milk samples conserved with Bronopol (0.02% final), corresponding to bulk milk from different regions in France, and on 35 consumer milk samples, representative of different technological treatments.

The results are as follows:

BY COMPARISON USING RAW MILK (VALUES in m°C x–1)

	ISO 5764 :1987 App 1	ISO 5764:1987 App 2	ISO 5764 :2002 App 3	ISO 5764 :2002 App 4	ISO 5764:2002 App 5
N	26	26	26	26	26
Mean	531,0	530,8	533,0	529,0	532,0
Difference ISO 5764 :1987/ISO 5764 : 2002*	-	-	+2,0	-1,8	+1,2
Standard deviation of the difference ISO 5764:1987 / ISO 5764 : 2002*	-	-	2,0	1,6	2,4

** The deviations are intra laboratory differences, that is:*
Lab 1: ISO 5764: 1987 Apparatus 1 and ISO 5764: 2002 Apparatus 3
Lab 2: ISO 5764: 1987 Apparatus 2 and ISO 5764: 2002 Apparatus 4 and 5

BY COMPARISON USING CONSUMER MILK (VALUES IN m°C x–1)

	ISO 5764 :1987 App 1	ISO 5764 :1987 App 2	ISO 5764 :2002 App 3	ISO 5764 :2002 App 4	ISO 5764:2002 App 5
N	35	35	35	35	35
Mean	519,0	519,1	521,7	517,3	520,9
Difference ISO 5764: 1987 / ISO 5764: 2002*	-	-	+2,7	-1,8	+1,8
Standard deviation of the difference ISO 5764: 1987 / ISO 5764: 2002	-	-	2,2	1,4	3,2

** The deviations are intra laboratory differences, that is:*
Lab 1: ISO 5764: 1987 Apparatus 1 and ISO 5764: 2002 Apparatus 3
Lab 2: ISO 5764: 1987 Apparatus 2 and ISO 5764: 2002 Apparatus 4 and 5

Firstly, it can be observed that the results obtained with raw milk and consumer milk, using the instruments complying with standard ISO 5764: 1987 were equivalent for the two apparatus tested (mean results equivalent).
Then, the presence of an effect "instrument" can be noted for the test results obtained with the samples analysed with the instruments complying with standard ISO 5764: 2002. In effect, the apparatus 3 and 5 present appreciably equivalent results (mean of determinations respectively 533.0 and 532.0 m°C x-1 with raw milk, and 521.7 and 520.9 m°C x-1 with consumer milk). These values are out of line with the results obtained with apparatus 4, presenting the same configuration (mean of determinations respectively 529.0 and 517.3 m°C x–1 with raw milk and consumer milk).
Observation of the analytical test means between the apparatus (including variability "version" and variability "instrument"), show an amplitude of 529 to 533 m°C for raw milk and 517.3 to 521.7 m°C for consumer milk.
There seems to exist a tendency relative to the instrument (all versions included), but, it is necessary to note that the deviations observed can be completely put down to the technical characteristics of the cryoscopic method, such as they are defined in the normative document ISO FIL 5764: a maximum deviation between duplicates (r) = 4 m°C and a reproducibility limit (R) = 6 m°C.
The results obtained in this French study have been transmitted to the group of expert chemists in Brussels, where they will question themselves on the results obtained throughout Europe.
A statistical analysis will be carried out on all the results and will serve as a basis of a general technical reflection on this precise point.

STANDARDS, DRAFT STANDARDS

ISO published standards

MICROBIOLOGIE		
PSYCHROTROPHES / RAPID METHOD / MILK	ISO 8552 : 2004 May 2004	MILK Estimation of psychrotrophic micro-organisms– Colony counting technique at 21°C (rapid method)
PETRI DISH / MILK	ISO 8553 : 2004 May 2004	MILK Enumeration of micro-organisms – Petri Dish Loop Method at 30°C

NEW EU STANDARDS AND REGULATIONS

The classification is established in alphabetic order of the first keyword

AFLATOXINE / INFANTILE MILK

Official Journal L 106, 15 April 2004 – Commission Regulation (EC) No 683/2004 of 13 April 2004 amending Regulation (EC) No 466/2001 as regards aflatoxins and ochratoxin A in foods for infants and young children
http://europa.eu.int/eur-lex/pri/en/oj/dat/2004/l_106/l_10620040415en00030005.pdf

Official Journal L 113, 20 April 2004 – Commission Directive 2004/43/EC of 13 April 2004 amending Directive 98/53/EC and Directive 2002/26/EC as regards sampling methods and methods of analysis for the official control of the levels of aflatoxin and ochratoxin A in food for infants and young children
http://europa.eu.int/eur-lex/pri/en/oj/dat/2004/l_113/l_11320040420en00140016.pdf

DIOXINS / PCB / FOODSTUFFS

Official Journal L 106, 15 April 2004 - Commission Regulation (EC) No 684/2004 of 13 April 2004 amending Regulation (EC) No 466/2001 as regards dioxins
http://europa.eu.int/eur-lex/pri/en/oj/dat/2004/l_106/l_10620040415en00060007.pdf

Official Journal L 113, 20 April 2004 - Commission Directive 2004/44/EC of 13 April 2004 amending Directive 2002/69/EC laying down the sampling methods and the methods of analysis for the official control of dioxins and the determination of dioxin-like PCBs in foodstuffs
http://europa.eu.int/eur-lex/pri/en/oj/dat/2004/l_113/l_11320040420en00170018.pdf

INORGANIC TIN / FOODSTUFFS

Official Journal L 42, 13 February 2004 – Commission Regulation (EC) No 242/2004 of 12 February 2004 amending Regulation (EC) No 466/2001 as regards inorganic tin in foods
http://europa.eu.int/eur-lex/pri/en/oj/dat/2004/l_042/l_04220040213en00030004.pdf

MEASURING INSTRUMENTS

Official Journal L 135, 30 April 2004 – Directive 2004/22/EC of the European Parliament and of the Council of 31 March 2004 on measuring instruments
http://europa.eu.int/eur-lex/pri/en/oj/dat/2004/l_135/l_13520040430en00010080.pdf

MEDICAMENTS VETERINAIRES / RESIDUS / ALIMENTS

Official Journal L 211, 12 June 2004 – Commission Regulation (EC) No 1101/2004 of 10 June 2004 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

http://europa.eu.int/eur-lex/pri/en/oj/dat/2004/l_211/l_21120040612en00030005.pdf

NITRATE / INFANTILE MILK

Official Journal L 104, 8 April 2004 – Commission Regulation (EC) No 655/2004 of 7 April 2004 amending Regulation (EC) No 466/2001 as regards nitrate in foods for infants and young children

http://europa.eu.int/eur-lex/pri/en/oj/dat/2004/l_104/l_10420040408en00480049.pdf

PESTICIDE / MAXIMUM RESIDUE CONTENT

Official Journal L 127, 29 April 2004 – Commission Directive 2004/61/EC of 26 April 2004 amending the Annexes to Council Directives 86/362/EEC, 86/363/EEC and 90/642/EEC as regards maximum residue levels for certain pesticides prohibited for use in the European Community

http://europa.eu.int/eur-lex/pri/en/oj/dat/2004/l_127/l_12720040429en00810091.pdf

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Créatrice : Annette BAPTISTE
Maquette : Annette BAPTISTE, Isabelle BECAR
Responsable de la rédaction : Carine TROUTET - E-mail : c.troutet@cecalait.fr
Ont collaborés à ce numéro : Patricia ROLLIER, Xavier QUERVEL, Philippe TROSSAT, Carine TROUTET
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