

CENTRE D'EXPERTISE ET DE CONTROLE DES ANALYSES LAITIÈRES



# CECALAIT'S NEWSLETTER

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# LIFE AT CECALAIT

#### ➤ The evolution of CECALAIT's website :

In 2006, to favour efficiency and to improve services to the laboratories, the following will be developed on our website :

- **On-line consultation of proficiency test reports:** As from the March proficiency tests, the laboratories will be able to access the results of their statistical treatment by consulting the CECALAIT website. Each laboratory will receive by mail a login and a password which will permit them to consult the individual reports before receiving the paper version. Nevertheless, CECALAIT will continue to send them by post. As soon as the results are available, a note will be put on the front page menu.

- **Internationalisation of the website:** In 2006, we will acquire the domain name "cecalait.com" and keywords in English, which will permit to develop our layout on the search engines. The positioning of the website abroad will also be strengthened by the acquisition of the domain name "cecalait.eu".

The objective of these actions is to be known better beyond our borders and to develop our export activity.

#### > CECALAIT follows its accreditation step:

In 2005, CECALAIT asked COFRAC for an accreditation concerning its activity as a proficiency test organiser according to the LAB-CIL Ref 02 and for an extension of its accreditation according to the NF EN ISO/CEI 17025 standard.

So, COFRAC recently carried out two audits at CECALAIT:

- in December 2005 an initial audit for the organisation of proficiency tests,
- in February 2006 an inspection audit for the program n° 61 "physico-chemistry on milk and dairy products" and an extension audit for the program n° 59 "food microbiology".

We are awaiting for the decisions of COFRAC in the course of the first semester 2006.

# **NUCLEAR MAGNETIC RESONANCE** A possible answer to actual and future analytical problems

Summary: Within a context of production and internationalised marketing, the development of analysis methods must respond to the needs of more and more numerous, specific and precise analyses. The spectroscopic methods, which are non-destructive and permit simultaneous multiple determinations, are privileged tools for these needs. In Particular, high-resolution NMR spectroscopy presents characteristics and a potential capable of answering the dairy industry's needs. This method is not yet very widespread in control laboratories, but, to date, the results obtained seem promising.

#### New analytical objectives :

The needs in analytical methods for measuring the components of milk and dairy products, are now covered as much by the reference methods as by the fast routine methods.

However, research and development in matters of analysis methods has to face up to new needs relating to product quality. The dairy sector cannot escape from this trend.

Indeed, with increasing international exchanges and consumer protection, a more and more complex international regulation is being set up (CE, Codex) concerning :

- nutritional allegations and health,
- the absence of chemical, biochemical and microbiological contamination,
- product conformity (falsification, adulteration),
- AOP product authenticity.

Moreover, the needs for control concern essentially consumption products, but they can also concern raw materials, milk in this case, whose quality is regularly verified by the dairy inter-profession through its milk payment laboratories network. For example, the increased knowledge of fat could lead to a differential classification of milk on the basis of its health and nutritional quality.

Considering the diversity and frequency of the imposed controls due to increasing commercial exchanges, a regular control of product quality and conformity is hardly feasible with reference methods, which are often long, expensive and sometimes require a high technicality. To be precise, the identification and the quantification of particular molecules in a complex mixture (i.e. food matrix) require a previous separation of the analytes before analysis. The reference methods are established on this principle. The control of the product composition in relation to contract conditions multiplies the analyses by the number of analytes concerned, generating a very important cost of the collected information.

The development of fast analysis methods proves to be necessary.

#### Spectroscopic methods

Spectroscopic methods became the privileged tools to analyse the composition of dairy products thanks to their rapidity, their non-destructive and non-polluting character, and their aptitude to measure simultaneously several analytes or components of the matrix.

In the dairy sector, the actual fast routine analysis methods are principally based on mid and near infrared spectrometry. As they are not very specific, they are limited to a restrictive number of components or component families (e.g. fat, protein, sugar) and they generally present a low precision for the minor components when they can be measured. Low-resolution nuclear magnetic resonance is less used because of its limited number of specific applications (water and fat).

Considering these limits, it seems necessary to identify other fast spectroscopic analytical methods to permit the specific measures of a large diversity of molecules in food products, particularly in dairy products, and to develop applications.

# High-resolution nuclear magnetic resonance spectroscopy (HR-NMR)

### A fast method with potential

NMR spectroscopy addresses the atomic nucleus, permitting obtention of a characteristic spectral signature of the atomic and electronic environment of each atom.

<u>Principle</u>: NMR measures the difference between the resonance frequency and that of a reference standard. This is called the chemical shift. Each molecule can therefore be characterised, in theory, by all the chemical shifts of each nucleus considered, and so constituting a characteristic spectrum of the molecule. In a molecular mixture, the spectrum obtained reflects the sum of the individual spectra.

Born in the United States in 1945, the NMR principle obtained the Nobel Prize for physics in 1952 for its two principal inventors, Edward M. Purcell and Felix Bloch. Since then, this method has been stated at many levels according to the applications sought after. Principally, two types of NMR can be distinguished : low-resolution and high-resolution.

Not all the elements are visible in NMR, but the principal elements that constitute living matter (C, H, O, N, P) are, either directly, indirectly according to their environment or via one of their isotopes .

Low-resolution NMR : this method uses low intensity fields and the resonance of the hydrogen nucleus, for which the maximum intensity of the signal is obtained. The cost of this method is not very high and its instrumentation not yet very bulky. It is applied to the direct quantitative analysis of certain elements (water and fat) and is used in routine analyses for product control. It also permits to distinguish the different forms of water and to study the repartition of water in the organic matrix by nuclear imaging.

**High- resolution NMR :** this method uses high intensity fields and has been for a long time reserved to research laboratories, within the context of structural and dynamic study of organic components and the reactional mechanisms. It is potentially applicable to all the "sensitive" nuclei, that is to say nuclei with a magnetic moment. Apart from the expensive material, this method requires a high technicity facing the various techniques used to exploit the spectral information.

The state of mobility of the elements in the matrices allows distinction of NMR with solids, for which a high-resolution is less easy to obtain than with liquids and for which particular techniques have had to be developed. It is more particularly via sample presentation modalities, that a very significant gain in resolution can be obtained, with the introduction of magic angle rotation (MAR). A rotation of the sample according to an angle of 54°44" cancels out many effects, especially interactions between nuclei (direct coupling effects) and sample heterogeneity . In the favourable case of products with partial molecular mobility (semi-solids), it is possible to reach a resolution near to that obtained with liquids. This technique is called high-resolution NMR magic angle rotation (HR-MAR). It is particularly efficient for polyphasic heterogeneous matrices (e.g. paste and gel). This technique permits to envisage a fine analysis of food and foodstuffs.

#### A constantly improved tool :

Improvements in high-resolution NMR have been developed thanks to the progress in supra-conductor electromagnet technology, in electronics and in the treatment of the signal and the spectral information. Thanks to scientific, technical and technological developments, the sensitivity and the resolution of the method have seen considerable improvements. The method is, therefore, more and more adapted to direct quantitative and qualitative analysis of complex products :

- improvement in magnet technology (more regular, powerful and protected fields),
- taller sample racks (better signal),
- low temperature probe (better signal/noise ratio)
- multiple probes permitting simultaneous scrutiny of several nuclei (increase in specificity),
- improved electronics (better signal/noise ratio),
- quicker signal treatment (deconvolution, Fourier transformation),
- treatment of the spectral information thanks to advances in data processing and software.

To be more accessible to a larger number of users, user-friendly automated systems with microcomputing and software dedicated to the treatment have been perfected (e.g. ADVANCE INCA<sup>™</sup> and Metabolic Profiler, by Bruker; Varian NMR System, by Varian Inc.).

More particularly, integrated systems have been developed to associate chromatographic separations before the NMR measures, for example, LC-NMR (Liquid chromatography NMR, Spraul and al., 2005).

Now, applications dedicated to routine analysis laboratories in the food industry are being developed through collaborations with research and development laboratories.

To date, work in the area of food analysis is still rare and oriented to a more qualitative rather than quantitative aspect. It is often associated with product characterisation. Examples of work on quantitative analyses are rare.

#### **Development for specific quantitative analysis :**

The work by De Angelis Curtis and al. (2000) gives a possible example of application in the dairy industry. Indeed, other than proposing a method for following the ripening of Grana Padano cheese they have shown that amino acid profiles during ripening can be established by  ${}^{1}$ H (proton) NMR at 500 MHz.

The amino acid composition is a major element of food nutritional value and, in the case of foodstuffs dedicated to sensitive populations (e.g. infant foods), its control is, to date, limited by the lack of quick or economic analytical techniques. NMR could be an option to establish an aminogram by direct scrutiny of a sample in the context of product control.

# **Development for qualitative analysis and characterisation :**

In the dairy industry, NMR had its first success in the 80s, with the isotopic hydrogen ratio measurement technique for the detection of chaptalization during vinification (SNIF-NMR®). The method, that has since been used for other applications, is based on the preliminary identification of a tracer (for example, ethanol), for which the spectral fingerprint is determined, and that varies according to the factor to be identified (e.g. nature and origin of the fermented sugars).

More global approaches, using multidimensional statistical treatments to exploit the NMR spectra, have appeared. They permit recognition and classification according to various combinations of spectral zones that can be linked to various biochemical tracers, which together, characterise the products.

NMR is efficient in the characterisation of various products in the food industry. In the dairy industry, recent work by the TRACES laboratory (Universities of Provence and Paul Cezanne in Marseille) has shown the possibility to characterise different steps during ripening of Parmigino Reggiano cheese by the application of HR-MAR using <sup>1</sup>H NMR at 400 MHz. In parallel, it is possible to assign the spectral lines to the principal tracer element during ripening (Shintu et al., 2004). Another study, led by the same team, on 20 Emmental cheeses from 5 European countries and using the same technique, permits to establish a distinct discrimination of the cheeses according to the origin of country. It also allows cheeses made from heat treated milk to be identified (Shintu et al., 2006).

For the protection of Registered Designation of Origin (RDO) products, this method is an alternative to the necessary traditional methods for establishing the authenticity of a product's origin and its conformity to the RDO type contract. The need for rapid controls has increased with the development in the commercialisation of portions, for which the reference of the cheese round is no longer possible for the consumer.

# Hypotheses and conjectures in relation to emerging needs

With competition for market shares, nutritional and health allegations are more and more important. Those relating to a particular composition must be verifiable by the manufacturer and the regulation control services. The incidence of food fat quality on human health is part of this problem, to regulate the maximal transfatty acid content and to guarantee minimal unsaturated and polyunsaturated fatty acid content. In the future, it will be possible to have the definition of new quality criteria of fat.

The fast specific quantitative analysis of these fatty acids could become necessary to establish characteristic fatty acid profiles.

To date, for many fatty acids, tables of chemical shifts for <sup>1</sup>H and <sup>13</sup>C nuclei in various positions of the molecules have been set up. It is notably possible to appreciate the spectral differences between fatty acids.

At the development of analytical methods level, various authors summarise the state of quantitative analyses on unsaturated fatty acids (AGM, AGPI,  $\omega$ -3) in different fat matrices (oil and fish fat) by <sup>1</sup>H and <sup>13</sup>C NMR (Knothe, 2005).

However, the work published to date only concerns a limited number of particular fatty acids in relatively homogenous and simple matrices.

The technical developments realised with NMR, associated with possible couplings with separative techniques, permit to expect increased possibilities relating to molecule recognition and quantification.

Therefore, it is not excluded that NMR brings fast alternative solutions applicable to dairy fat, to partially substitute the long and costly methods actually required for the establishment of fatty acid profiles.

#### Conclusion

NMR is still ill-known and little used in the dairy sector. However, since 2000, various studies have opened up applications to dairy products. The method presents an attractive character by its versatility so much as for analysis of dairy matrices as for analysis of measurable elements in these products. The qualitative approach in relation with the characterisation of product quality seems to be more advanced than the developments in quantitative analysis.

Lines for development and priorities will therefore have to be defined.

#### Bibliographic references:

De Angelis Curtis S., Curini R., Delfini M., Brosio E., D'Ascenzo F., Bocca B., (2000) : Amino acid profil in the ripening of Grana Padano cheese : A NMR study. *Food Chemistry*, Vol. 71, 4, 495-502.

Knothe G., (2005): <sup>1</sup>H NMR Spectroscopy of Fatty Acids and therir derivatives – Quantitation by <sup>1</sup>H NMR. The Lipid Library, www.lipidlibrary.co.uk.

Knothe G., Kenar J.A., (2004) : Determination of the fatty acid profil by <sup>1</sup>H NMR spectroscopy. *Eur. J. Lipid Sci. Technol.*, 106, 88–96 (2004).

Shintu L. , Caldarelli S., (2006) : Towards the determination of the geographical origin of Emmental cheese via High Resolution MAS NMR : A preliminary investigation. (envoyé pour publication).

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Spraul M., Humpfer E., Schäfer H., (2005): Application if hyphenated NMR to the study of food. From « Magnetic Resonance in Food Science : The Multivariate Challenge. The Royal Society of Chemistry 2005. ISBN 0-85404-648-8.

Royer A., Naulet N., Lees M., Martin G.H., (1997) : Isotopic analysis for the authentification of extra-virgin oils. Food Authenticity 4<sup>th</sup> European Symposium, La Baule, 4-6 juin.

#### NEW MICROBIOLOGICAL CRITERIA

In order to standardise the regulation, to prevent differing interpretations and to contribute to the public health protection, harmonised microbiological criteria have been defined in the European level. Indeed, since the  $1^{st}$  January 2006, the Commission regulation (EC) n° 2073/2005 of  $15^{th}$  November 2005 concerning the microbiological criteria for foodstuffs is effective.

In fact, some national or community decrees relating to milk and dairy products are in process of repeal. Then, safety criteria for foodstuffs and process hygiene criteria have been elaborated in order to define their acceptability.

You will find, in the following tables, a summary of the criteria concerning milk and dairy products.

## Food safety criteria

	Micro-organisms/	Sampling plan <sup>(1)</sup>		Limits <sup>(2)</sup>		Analytical reference	Stage where the criterion
Food category	their toxins, metabolites	n	с	m	М	method <sup>(3)</sup>	applied
Ready-to-eat foods intended for infants and ready-to-eat foods for special medical purposes <sup>(4)</sup>	Listeria monocytogenes	10	0	Absence in	1 25 g	EN/ISO 11290-1	Products placed on the market during their shelf-life
Ready-to-eat foods able to support the		5	0	100 ufc/§	g <sup>(5)</sup>	EN/ISO 11290-2 <sup>(6)</sup>	Products placed on the market during their shelf-life
growth of <i>L. monocytogenes</i> , other than those intended for infants and for special medical purposes	Listeria monocytogenes	5	0	Absence in 2	25 g <sup>(7)</sup>	EN/ISO 11290-1	Before the food has left the immediate control of the food business operator, who has produced it
Ready-to-eat foods unable to support the growth of <i>L. monocytogenes</i> , other than those intended for infants and for special medical purposes	Listeria monocytogenes	5	0	100 ufc	/g	EN/ISO 11290-2 <sup>(6)</sup>	Products placed on the market during their shelf-life
Cheeses, butter and cream made from raw milk or milk that has undergone a lower heat treatment than pasteurisation <sup>(10)</sup>	Salmonella	5	0	Absence in	1 25 g	EN/ISO 6579	Products placed on the market during their shelf-life
Milk powder and whey powder <sup>(10)</sup>	Salmonella	5	0	Absence in	1 25 g	EN/ISO 6579	Products placed on the market during their shelf-life
Ice cream <sup>(11)</sup> , excluding products where the manufacturing process or the composition of the product will eliminate the salmonella risk	Salmonella	5	0	Absence in	1 25 g	EN/ISO 6579	Products placed on the market during their shelf-life
Cheeses, milk powder and whey powder, as referred to in the coagulase-positive staphylococci criteria in the table below	Staphylococcal enterotoxins	5	0	Not detected	in 25 g	European screening method of the CRL for milk <sup>(13)</sup>	Products placed on the market during their shelf-life
Dried infant formulae and dried dietary foods for special medical purposes intended for infants below six months of age, as referred to in the Enterobacteriaceae criterion in the table below	Salmonella	30	0	Absence in	a 25 g	EN/ISO 6579	Products placed on the market during their shelf-life

	Micro-organisms/	Sampling plan <sup>(1)</sup>		Limits <sup>(2)</sup>		Analytical reference	Stage where the criterion
Food category	their toxins,	n	с	m	М	method <sup>(3)</sup>	applied
	metabolites						
Dried infant formulae and dried dietary foods for special medical purposes intended for infants below six months of age, as referred in the Enterobacteriaceae criterion in the table below	Enterobacter sakazakii	30	0	Absence ir	n 10 g	ISO/DTS 22964	Products placed on the market during their shelf-life

(1) n = number of units comprising the sample; c = number of sample units giving values over m or between m and M

(2) m = M

- (3) The most recent edition of the standard shall be used
- (4) Regular testing against the criterion is not useful in normal circumstances for the following ready-to-eat foods:
  - those whose have received heat treatment or other processing effective to eliminate *L. monocytogenes*, when recontamination is not possible after this treatment (e.g. products heat treated in their final package)

(5) This criterion applies if the manufacturer is able to demonstrate, to the satisfaction of the competent authority, that the product will not exceed the limit 100 cfu/g throughout the shelf-life. The operator may fix intermediate limits during the process that should be low enough to guarantee that the limit of 100 cfu/g is not exceeded at the end of the shelf-life.

(6) 1 ml of inoculum is plated on a Petri dish of 140 mm diameter or on three Petri dishes of 90 mm diameter.

(7) This criterion applies to products before they have left the immediate control of the producing food business operator, when he is not able to demonstrate, to the satisfaction of the competent authority, that the product will not exceed the limit of 100 cfu/g throughout the shelf-life.

(8) Products with  $pH \le 4,4$  or  $a_w \le 0,92$ , products with  $pH \le 5,0$  and  $a_w \le 0,94$ , products with a shelf-life of less than five days are automatically considered to belong to this category. Other categories of products can also belong to this category, subject to scientific justification.

(10) Excluding products when the manufacturer can demonstrate to the satisfaction of the competent authorities that, due to the ripening time and  $a_w$  of the product where appropriate, there is no salmonella risk

(11) Only ice creams containing milk ingredients.

(13) Reference : Hennekinne et al., J. AOAC Internat. Vol 86, N° 2, 2003.

#### Interpretation of the test results

The limits given refer to each sample unit tested.

The test results demonstrate the microbiological quality of the batch tested <sup>(1)</sup>.

L. monocytogenes in ready-to-eat foods intended for infants and for special medical purposes:

- satisfactory, if all the values observed indicate the absence of the bacterium,
- unsatisfactory, if the presence of the bacterium is detected in any of the samples units.

*L. monocytogenes* in ready-to-eat foods able to support the growth of *L. monocytogenes* before the food has left the immediate control of the producing food business operator when he is not able to demonstrate that the product will not exceed the limit of 100 cfu/g throughout the shelf-life:

- satisfactory, if all the values observed indicate the absence of the bacterium,
- unsatisfactory, if the presence of the bacterium is detected in any of the sample units.

*L. monocytogenes* in other ready-to-eat foods:

- satisfactory, if all the values observed are  $\leq$  the limit,
- unsatisfactory, if any of the values are > the limit.

Salmonella in different food categories:

- satisfactory, if all the values observed indicate the absence of the bacterium,
- unsatisfactory, if the presence of the bacterium is detected in any of the sample units.

Staphylococcal enterotoxins in dairy products:

- satisfactory, if in all the sample units the enterotoxins are not detected,
- unsatisfactory, if the enterotoxins are detected in any of the sample units.

Enterobacter sakazakii in dried infant formulae and dried dietary foods for special medical purposes intended for infants below 6 months of age:

- satisfactory, if all the values observed indicate the absence of the bacterium,
- unsatisfactory, if the presence of the bacterium is detected in any of the sample units.

(1) The test results can be used also for demonstrating the effectiveness of the HACCP or good hygiene procedure of the process

## Process hygiene criteria – Milk and dairy products

Food astagomy	Micro-	Sampling	g plan <sup>(1)</sup>	Limit	ts <sup>(2)</sup>	Analytical reference	Stage where the criterion	Action in case of unsatisfactory
Food category	organisms	n	c	m	М	method <sup>(3)</sup>	applies	results
Pasteurised milk and other pasteurised liquid dairy products <sup>(4)</sup>	Entero- bacteriaceae	5	2	< 1 ufc/ml	5 ufc/ml	ISO 21528-1	End of the manufacturing process	Check on the efficiency of heat- treament and prevention of recontamination as well as the quality of raw materials
Cheeses made from milk or whey that has undergone heat treatment	E. coli <sup>(5)</sup>	5	2	100 ufc/g	1000 ufc/g	ISO 16649-1 ou 2	At the time during the manufacturing process when the <i>E. coli</i> count is expected to be highest <sup>(6)</sup>	Improvements in production hygiene and selection of raw materials
Cheeses made from raw milk	Coagulase- positive staphylococci	5	2	10 <sup>4</sup> ufc/g	10 <sup>5</sup> ufc/g	EN/ISO 6888-2		
Cheeses made from milk that has undergone a lower heat treatment than pasteurisation <sup>(7)</sup> and ripened cheeses made from milk or whey that has undergone pasteurisation or a stronger heat treatment <sup>(7)</sup>	Coagulase- positive staphylococci	5	2	100 ufc/g	1000 ufc/g	EN/ISO 6888-1 ou 2	At the time during the manufacturing process when the number of staphylococci is expected to be highest	Improvements in production hygiene and selection of raw materials. If values $> 10^5$ cfu/g are detected, the cheese batch has to be tested for staphylococcal enterotoxins
Unripened soft cheeses (fresh cheeses) made from milk or whey that has undergone pasteurisation or a stronger heat treatment	Coagulase- positive staphylococci	5	2	10 ufc/g	100 ufc/g	EN/ISO 6888-1 ou 2	End of the manufacturing process	Improvements in production hygiene. If values $> 10^5$ cfu/g are detected, the cheese batch has to be tested for staphylococcal enterotoxins
Butter and cream made from raw milk or milk that has undergone a lower heat treatment than pasteurisation	E. coli <sup>(5)</sup>	5	2	10 ufc/g	100 ufc/g	ISO 16649-1 ou 2	End of the manufacturing process	Improvements in production hygiene and selection of raw materials

Food catagory	Micro-	Samplin	g plan <sup>(1)</sup>	Limits <sup>(2)</sup>		Limits <sup>(2)</sup>		Analytical reference	Stage where the criterion	Action in case of unsatisfactory
Food category	organisms	n	с	m	М	method <sup>(3)</sup>	applies	results		
	Entero- bacteriaceae	5	0	10 ut	fc/g	ISO 21528-1	End of the manufacturing process	Check on the efficiency of heat treatment and prevention of recontamination		
powder <sup>(4)</sup>	Coagulase- positive staphylococci	5	2	10 ufc/g	100 ufc/g	EN/ISO 6888-1 ou 2	End of the manufacturing process	Improvements in production hygiene. If values $> 10^5$ cfu/g are detected, the batch has to be tested for staphylococcal enterotoxins		
Ice cream <sup>(8)</sup> and frozen dairy desserts	Entero- bacteriaceae	5	2	10 ufc/g	100 ufc/g	ISO 21528-2	End of the manufacturing process	Improvements in production hygiene		
Dried infant formulae and dried dietary foods for special medical purposes intended for infants below six months of age	Entero- bacteriaceae	10	0	Absence in 10 g		ISO 21528-1	End of the manufacturing process	Improvements in production hygiene to minimise contamination. If <i>Enterobacteriaceae</i> are detected in any of the sample units, the batch has to be tested for <i>E. sakazakii</i> and <i>Salmonella</i>		

(1) n = number of units comprising the sample; c = number of sample units giving values between m and M

(2) For *Enterobacteriaceae* in milk powder and whey powder, m = M

- (3) The most recent edition of the standard shall be used
- (4) The criterion does not apply to products intended for further processing in the food industry
- (5) E. coli is used here as an indicator for the level of hygiene
- (6) For cheeses which are not able to support the growth of *E. coli*, the *E. coli* count is usually the highest at the beginning of the ripening period, and for cheeses which are able to support the growth of *E. coli*, it is normally at the end of the ripening period
- (7) Excluding cheeses where the manufacturer can demonstrate, to the satisfaction of the competent authorities, that the product does not pose a risk of staphylococcal enterotoxins
- (8) Only ice creams containing milk ingredients

#### **Interpretation of the test results**

The limits given refer to each sample unit tested.

The test results demonstrate the microbiological quality of the process tested.

Enterobacteriaceae in dried infant formulae and dried dietary foods for special medical purposes intended for infants below six months of age:

- satisfactory, if all the values observed indicate the absence of the bacterium,
- unsatisfactory, if the presence of the bacterium is detected in any of the samples unit.

E. coli, enterobacteriaceae (other food categories) and coagulase-positive staphylococci:

- satisfactory, if all the values observed are < m,
- acceptable, if a maximum of c/n values are between m and M, and the rest of the values observed are < m,
- unsatisfactory, if one or more of the values observed are > M or more than c/n values are between m and M.

# STANDARDS, DRAFT STANDARDS

Classification in alphabetic order by theme

# **ISO published standards**

ANHYDROUS MILK FA	T					
ANHYDROUS MILK FAT / STEROL / REFERENCE METHOD	ISO 12078: 2006 (IDF 159) February 2006	ANHYDROUS MILK FAT Determination of sterol composition by gas liquid chromatography (Reference method)				
ANHYDROUS MILK FAT / STEROL	ISO 18252: 2006 (IDF 200) February 2006	ANHYDROUS MILK FAT Determination of sterol composition by gas liquid chromatography (Routine method)				
DRIED MILK / DRIED D	DAIRY PRODUCTS					
DRIED MILK / DRIED DAIRY PRODUCTS	ISO 8967: 2005 (IDF 134)	DRIED MILK AND DRIED DAIRY PRODUCT				
BULK DENSITY	December 2005	Determination of bulk density				
MICROBIOLOGY OF F	OOD AND ANIMAL FI	EEDING STUFFS				
COLIFORMS / HORIZONTAL	ISO 4832: 2006	MICROBIOLOGY OF FOOD AND ANIMAL FEEDING STUFFS				
METHOD	redituary 2000	Colony-count technique				
CAMPYLOBACTER /	ISO 10272-1: 2006	MICROBIOLOGY OF FOOD AND ANIMAL FEEDING STUFFS				
HORIZONTAL METHOD	January 2006	Campylobacter spp. Part 1: Detection method				
<i>BACILLUS CEREUS /</i> HORIZONTAL METHOD	ISO 21871: 2006 January 2006	MICROBIOLOGY OF FOOD AND ANIMAL FEEDING STUFFS Horizontal method for the determination of low numbers of presumptive <i>Bacillus cereus</i> - Most probable number technique and detection method				
UNCERTAINTY /	ISO / TS 19036: 2006	MICROBIOLOGY OF FOOD AND ANIMAL FEEDING STUFFS				
QUANTITATIVE DETERMINATIONS	February 2006	Guidelines for the estimation of measurement uncertainty for quantitative determinations				
MILK		1				
MILK /	ISO 9874: 2006	MILK				
PHOSPHORUS	(IDF 42)	Determination of total phosphorus content – Method using				
CONTENT	February 2006	molecular absorption spectrometry				
MILK / DAIRY PRODUC	CTS					
		MILK AND DAIRY PRODUCTS				
	ISO 11866-1: 2005	Enumeration of presumptive Escherichia coli				
PRODUCTS	ISO 11866-2: 2005	Part 1: Most probable number technique using 4-				
E. COLI	(IDF 170-1 and 170-2)	methylumbelliferyl-β-D-glucuronide (MUG)				
	December 2005	Part 2: Colony-count technique at 44 degrees C using membranes				

SAKAZAKII

MILK AND DAIRY PRODUCTS

Detection of Enterobacter sakazakii

# **NEW EU STANDARDS AND REGULATIONS**

#### Classification is established in alphabetical order of the first keyword

#### **CONTAMINANTS / DIOXINS / PCBs**

**O.J.E.U. L 32, 4<sup>th</sup> February 2006 -** Commission Regulation (EC) No 199/2006 of 3 February 2006 amending Regulation (EC) No 466/2001 setting maximum levels for certain contaminants in foodstuffs as regards dioxins and dioxin-like PCBs

http://europa.eu.int/eur-lex/lex/LexUriServ/site/en/oj/2006/1\_032/1\_03220060204en00340038.pdf

**DIOXINS** / FOODSTUFFS

**O.J.E.U. L 42, 14<sup>th</sup> February 2006** - Commission Recommendation of 6 February 2006 on the reduction of the presence of dioxins, furans and PCBs in feedingstuffs and foodstuffs

http://europa.eu.int/eur-lex/lex/LexUriServ/site/en/oj/2006/1\_042/1\_04220060214en00260028.pdf

HYGIENE / CONTROL / FOODSTUFFS

**O.J.E.U. L 338, 22<sup>nd</sup> December 2005 -** Commission Regulation (EC) No 2076/2005 of 5 December 2005 laying down transitional arrangements for the implementation of Regulations (EC) No 853/2004, (EC) No 854/2004 and (EC) No 882/2004 of the European Parliament and of the Council and amending Regulations (EC) No 853/2004 and (EC) No 854/2004

http://europa.eu.int/eur-lex/lex/LexUriServ/site/en/oj/2005/1\_338/1\_33820051222en00830088.pdf

MICROBIOLOGY / FOODSTUFFS

**O.J.E.U. L 338, 22<sup>nd</sup> December 2005 -** Commission Regulation (EC) No 2073/2005 of 15 November 2005 on microbiological criteria for foodstuffs

http://europa.eu.int/eur-lex/lex/LexUriServ/site/en/oj/2005/1\_338/1\_33820051222en00010026.pdf

MYCOTOXINS / FOODSTUFFS

**O.J.E.U. L 70, 9<sup>th</sup> March 2006 -** Commission Regulation (EC) No 401/2006 of 23 February 2006 laying down the methods of sampling and analysis for the official control of the levels of mycotoxins in foodstuffs http://europa.eu.int/eur-lex/lex/Lex/UriServ/site/en/oj/2006/1\_070/1\_07020060309en00120034.pdf

**PESTICIDE /** RESIDUES / MAXIMUM LEVELS

**O.J.E.U. L 29, 2<sup>nd</sup> February 2006** - Commission Regulation (EC) No 178/2006 of 1 February 2006 amending Regulation (EC) No 396/2005 of the European Parliament and of the Council to establish Annex I listing the food and feed products to which maximum levels for pesticide residues apply http://europa.eu.int/eur-lex/lex/LexUriServ/site/en/oj/2006/1\_029/1\_02920060202en00030025.pdf

**O.J.E.U. L 75, 14<sup>th</sup> March 2006 -** Commission Directive 2006/30/EC of 13 March 2006 amending the Annexes to Council Directives 86/362/EEC, 86/363/EEC and 90/642/EEC as regards maximum residue levels for the benomyl group

http://europa.eu.int/eur-lex/lex/LexUriServ/site/en/oj/2006/1\_075/1\_07520060314en00070016.pdf

#### **VETERINARY MEDICINAL PRODUCTS** / RESIDUE / FOODSTUFFS

**O.J.E.U. L 34, 7<sup>th</sup> February 2006** - Commission Regulation (EC) No 205/2006 of 6 February 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 toltrazuril, diethylene glycol monoethyl ether and polyoxyethylene sorbitan monooleate <u>http://europa.eu.int/eur-lex/Lex/Lex/LexUriServ/site/en/oj/2006/1 034/1 03420060207en00210023.pdf</u>

# **IN THE PRESS – ON THE WEB**

Classification in alphabetical order of keywords

#### CAMPYLOBACTER JEJUNI

New PCR method to detect Campylobacter jejuni

http://www.ap-foodtechnology.com/news/ng.asp?n=65264 c-jejuni-food-safety ► A new PCR based method to detect *Campylobacter jejuni* has been developped by researchers in China.

# FORTHCOMING EVENTS

Classified in chronological order

#### SALMONELLA

10 – 12 May 2006 Saint-Malo, France 4<sup>th</sup> international symposium on salmonella and salmonellosis

http://www.zoopole.com/ispaia/i3s2006

### **STANDARDISATION**

29 May – 2 June 2006 Vilnius, Lithuania

IDF / ISO analytical week

http://www.fil-idf.org

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