CECALAIT-S NEWSLETTER

1st quarter 2009, N° 68

CENTRE D'EXPERTISE ET DE CONTROLE DES ANALYSES LAITIERES

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ARTICLE

NEW PROFICIENCY TEST PROPOSED BY CECALAIT: THE DETECTION OF INHIBITORS IN RAW MILK

The detection of inhibitors in raw milk is an important control in the dairy industry on two counts: firstly, in cheese technology due to their inhibitory effect on lactic acid bacteria which can cause poor acidification, and secondly for their undesirable effects on consumer health. European regulations have fixed a maximum permissible dose for each molecule, the MRL (Maximal Residues Limit).
To make sure the methods implemented are reliable, laboratories need to test their performances objectively, so participation in proficiency tests is a pertinent way to reach this objective.
The responses to a survey conducted in 2007 with French private laboratories permitted Cecalait to develop samples, and to propose two proficiency tests on inhibitors in milk in 2008.

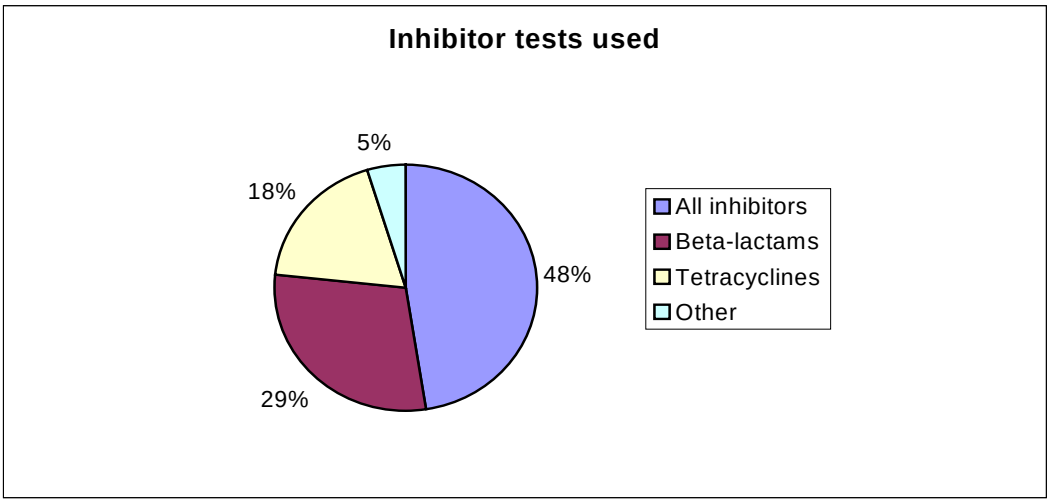
DEVELOPMENT OF THE PROFICIENCY TEST:

1- Results of the needs survey conducted with industrial laboratories

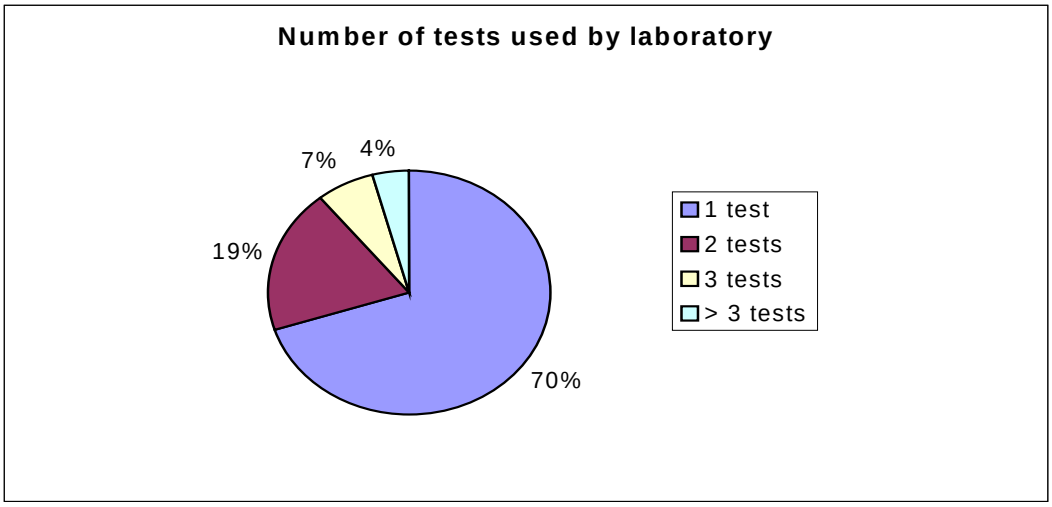
This survey was sent to 682 potential clients in 2007. Globally, we received a favourable response. Indeed 74 / 154 laboratories that replied (which represents more than half) were interested in a proficiency test on inhibitors being set up.

This survey contained questions concerning the methods used by the laboratories and the implementation of the proficiency test.

1-1 Specificity of the methods used



This survey underlined that about half of the methods permit the detection of all the inhibitors, and the other half concerned specific methods. The majority of the latter were methods for detecting ß-lactams, followed by tetracyclines. The detection of other inhibitors represented only 5% of the methods used.



This survey also underlined that about 1/3 of the laboratories used several methods.

According to these results, the proficiency test proposed also had to be able to evaluate the methods

implemented for the detection of all the inhibitors – acidification type microbiological methods – as well as the specific methods for the detection of ß-lactams and tetracyclines – immunological and/or enzymatic tests.

1-2 Implementation of the proficiency test:

The interested laboratories desired:

- a frequency of 2 tests per year (46% of laboratories),
- 5 samples per test (55%) or 10 (41%): we chose the higher number, 10 per proficiency test,
- a sample volume of 10 ml (48%), corresponding to the smallest volume proposed. For technical reasons, we fixed the sample volume at 5 ml. The inoculations of the detection tests are very small (from 50 to 500 µl); one sample can thus be used to test several methods.

2- Development of the samples

On the basis of the specifications, tests were carried out to refine the preparation of the samples:

2-1 Stability and homogeneity tests

The organisation of a proficiency test implies a delay between the preparation of the samples and their analysis, including transport, from 1 to several days for the foreign laboratories. Many modalities of preservation can be used but we have chosen to freeze-dry the samples.

The stability and homogeneity were studied on a first batch of samples using the official method of detection by acidification, followed by a confirmation by diffusion on agar with *Bacillus stearothermophilus*. This confirmation method enables the concentration in ß-lactams and tetracyclines to be estimated by measuring the inhibition diameter and comparing it with a control sample.

2-2 Preliminary proficiency test

The homogeneity and stability studies were satisfactory and a pre-test concerning 6 participants was set up in December 2007. Thanks to this test we were able to optimise the composition of the samples so that all the commercial kits can be used.

PRESENTATION AND INTERPRETATION OF THE RESULTS

This first proficiency test also permitted the presentation of the results to be considered, for which the following information had to appear:

- The presence or not of an inhibitory molecule,
- If it belongs to the family of ß-lactams or tetracyclines,
- Its concentration,
- Its MRL.

The detection tests can generate doubtful results, which are difficult to interpret. Moreover, in the case of inhibitors, the result will firstly depend on the technical performances of the detection test used, particularly its specificity, and its detection threshold for each inhibitory molecule.

Therefore each result cannot be compared with a reference result, as in the qualitative proficiency tests on pathogens with "Absence / Presence", resulting in a simple interpretation "True / False". Each participant must thus compare its own results in relation to presence, concentration and inhibitor family, taking into account the performances of the method used. The indication of the MRL can help with this interpretation.

Thanks to all these considerations, the statistical analysis sent to each participant contains:

1- A table of methods: recapitulating all the methods used by the participating laboratories, with a classification in 3 categories according to the inhibitor type detected: all inhibitors, ß-lactams or tetracyclines.

2- A table of results: positive (+), negative (-) or doubtful (+/-) results of all the participants in the 3 test categories described above. Below this table, the list of the 10 samples with for each the name of the inhibitor added or not and if need be its family, its concentration and its MRL are presented.

3- Individual results: of each participant with 3 tables corresponding to the 3 categories of detection method. Each table presents, for each sample, the laboratory's result, the name of the molecule added or not and its concentration. The list of the samples is again indicated below the tables.

CONCLUSION:

About 70 laboratories participated in both proficiency tests on detection of inhibitors proposed in 2008, which underlines the interest of the industrial laboratories for this type of intercomparison test. This proficiency test enables the laboratories' performances to be evaluated and can provide better knowledge of the laboratories' staff, on the specificity and the detection threshold of the tests they use.

Patricia ROLLIER

Thanks to the laboratories who participated in the preliminary test in 2007

STANDARDS, DRAFT STANDARDS

Classification in alphabetic order by theme

ISO published standards

MILK FAT FROM ENRICHED DAIRY PRODUCTS		
MILK FAT / FATTY ACID CONTENT	ISO 23065:2009 (IDF 211) March 2009	MILK FAT FROM ENRICHED DAIRY PRODUCTS Determination of omega-3 and omega-6 fatty acid content by gas-liquid chromatography

NEW EU REGULATIONS

Classification is established in alphabetical order of the first keyword

COLOURS

O.J.E.U. L 6, 10th January 2009 – Commission Directive 2008/128/EC of 22 December 2008 laying down specific purity criteria concerning colours for use in foodstuffs
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2009:006:0020:0063:EN:PDF

O.J.E.U. L 44, 14th February 2009 – Commission Directive 2009/10/EC of 13 February 2009 amending Directive 2008/84/EC laying down specific purity criteria on food additives other than colours and sweeteners
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2009:044:0062:0078:EN:PDF

FLAVOURING SUBSTANCES

O.J.E.U. L 55, 27th February 2009 – Commission Decision of 26 February 2009 amending Decision 1999/217/EC as regards the register of flavouring substances used in or on foodstuffs
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2009:055:0041:0043:EN:PDF

PROTECTED DESIGNATIONS OF ORIGIN

O.J.E.U. L 8, 13th January 2009 – Commission Decision of 19 December 2008 rejecting an application for entry in the register of protected designations of origin and protected geographical indications provided for in Council Regulation (EC) n° 510/2006 [Dziugas (PGI) (cheese)]
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2009:008:0024:0024:EN:PDF

O.J.E.U. L 8, 13th January 2009 – Commission Decision of 19 December 2008 rejecting an application for entry in the register of protected designations of origin and protected geographical indications provided for in Council Regulation (EC) n° 510/2006 [Germantas (PGI) (cheese)]
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2009:008:0025:0025:EN:PDF

O.J.E.U. L 34, 4th February 2009 – Commission Regulation (EC) n° 104/2009 of 3 February 2009 approving non-minor amendments to the specification for a name entered in the register of protected designations of origin and protected geographical indications [Gorgonzola (PDO) (cheese)]
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2009:034:0016:0016:EN:PDF

O.J.E.U. L 38, 7th February 2009 – Commission Regulation (EC) n° 115/2009 of 6 February 2009 approving minor amendments to the specification for a name entered in the register of protected designations of origin and protected geographical indications [Bleu de Causses (PDO) (cheese)]
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2009:038:0028:0032:EN:PDF

O.J.E.U. L 53, 26th February 2009 – Commission Regulation (EC) n° 159/2009 of 25 February 2009 approving minor amendments to the specification for a name entered in the register of protected designations of origin and protected geographical indications [Chabichou du Poitou (PDO) (cheese)]
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2009:053:0008:0012:EN:PDF

BOOKSHOP: LATEST PUBLICATIONS

The classification in alphabetic order of the first keyword allows you to consult the references according to your interests. The web site allows you to know more, or to order the book.

QUALITY ASSURANCE

KONIECZKA P.; NAMIESNIK J. **– Quality assurance and quality control in the analytical chemical laboratory: a practical approach** – CRC Press Editions – February 2009 – ISBN 9781420082708 – 304 pages

http://www.crcpress.com

This publication highlights the importance of quality assurance and quality control in the analytical laboratories and insists on the utility to use reference materials and validation methods.

HACCP

ARVANITOYANNIS I.S. **– HACCP and ISO 22000: Application to foods of animal origin** –Wiley Editions – February 2009 – ISBN 978-1-4051-5366-9 – 560 pages

http://eu.wiley.com

This book presents and compares the HACCP and ISO 22000/2005 food safety management systems. Numerous practical examples illustrating the application of ISO 22000 to the manufacture of food products of animal origin (dairy products, meat, eggs, poultry).

FORTHCOMING EVENTS

Classified in chronological order

18-22 May 2009 Sochi, Russia	IDF / ISO Analytical week	http://2009.lactose.ru
22-25 June 2009 Paris, France	14th International Congress of Metrology	www.metrologie2009.com

IN THE PRESS – ON THE WEB

Classification in alphabetical order of keywords

ESCHERICHIA COLI

Lab M broth detects *E. coli* in milk
http://www.laboratorytalk.com/news/lbm/lbm137.html

Oxoid optimises *E. coli* / coliform selective agar
http://www.laboratorytalk.com/news/oxo/oxo380.html

► These two articles present new possibilities to detect *Escherichia coli* or coliforms in milk.

MELAMINE

Compact IC detects melamine in food
http://www.laboratorytalk.com/news/mea/mea815.html

Delta develops melamine screening method
http://www.laboratorytalk.com/news/qcl/qcl203.html

► These two articles present methods to detect melamine in milk powder and raw milk.

La Lettre de CECALAIT est éditée par ACTILAIT / CECALAIT, B.P. 70129, 39802 POLIGNY CEDEX
ACTILAIT : association. Président : Patrick RAMET ; Directeur : Vincent OVERNEY
Directeur de la publication : Patrick RAMET
Créatrice : Annette BAPTISTE
Maquette : A. BAPTISTE, I. BECAR
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Rédaction achevée le 14 avril 2009 – Traduction achevée le 20 avril 2009
Impression : ACTILAIT / CECALAIT, B.P. 70129, 39802 POLIGNY CEDEX
Tél. : 33.(0)3.84.73.63.20 - Fax : 33.(0)3.84.73.63.29
1er trimestre 2009
Dépôt légal : à parution
ISSN 1298-6976