

CENTRE D'EXPERTISE ET DE CONTROLE
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CECALAIT NEWSLETTER

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May 27th 2003 CECALAIT Annual General Meeting

In order for you to organise your timetable, we would like to bring to your attention that CECALAIT's annual general meeting will take place on Tuesday 27th of May in Paris at the Maison du Lait, in the morning. As usual, the afternoon will be devoted to presenting technical subjects. Of course, you will receive, in due time a full program and the exact time of this event.

Analyser B 2000 ® Evaluation on Freezing Point Depression

Analyser B 2000 ® has already been evaluated by CECALAIT for milk composition parameters : fat, protein and lactose. The results were presented in CECALAIT's Newsletter n°34. The actual evaluation is devoted to determining the freezing point equivalent. Repeatability evaluation indicates that absolute standard deviation of repeatability is smaller than the thermistor cryoscope reference method limit. Repeatability and accuracy performances are in accordance with the manufacturer (BENTLEY) specifications.

APPARATUS

B 2000 ® is an automatic analyser manufactured and marketed by BENTLEY Instruments for analysis of fat, protein, and lactose in milk. CECALAIT evaluated these criteria and presented them in Newsletter n°34. B 2000 ® can also measure a freezing point equivalent.

It works using a filter mono beam infrared system combined with a conductivity cell. The apparatus is coupled with a micro-computer which deals with running and signal processing

Experimental

CECALAIT's physico-chemistry laboratory conducted the reference and infrared analyses for FPD (Freezing Point Depression) from August to October 2002.

The following characteristics were evaluated :

- repeatability
- accuracy

1-Repeatability

Repeatability was evaluated by automatic analysis of 104 herd milks, from Franche-Comté, preserved with 0,02% bronopol. The sampling and analyses was realised over three days with a first set of 54 samples, a second set of 40 samples and a third set of 10 samples. Due to the low amplitude of the results obtained with the second set, the 10 milks from the third set were artificially watered at 4% and 8%.

Measures were analysed automatically and each set of ten milks was duplicated in a non consecutive way.

The evaluation was carried out with samples covering a range of rates from 480 to 534 (m°C x-1).

Results

Repeatability was estimated by using the standard deviation of repeatability.

N	M (m°C x -1)	Sx (m°C x -1)	Sr (m°C)	Sr (%)	r (m°C)
54	518	6.9	0.8	0.15	2.1
40	519	4.7	1.1	0.22	3.1
10	498	11.9	0.9	0.17	2.4

N : number of samples

M : mean results

Sx : standard deviation of the results

Sr et Sr% : relative and absolute standard deviation of repeatability

r : maximal difference between duplicates in 95 % of occurrences

Conclusion

The standard deviation of repeatability *Sr* is smaller than the limit of the Thermistor Cryoscope Reference Method (ISO 5764:2002), *r* = 4 m°C and *Sr* = 1.4 m°C). The results (*r* mean = 2.5 m°C) comply with the manufacturer BENTLEY Instruments (*r* < à 2.8 m°C).

2- Accuracy

The apparatus was evaluated with the same milk samples as the ones used for the repeatability evaluation (sets 2 and 3). Immediately after the instrumental analyses, the samples were cooled to 20 °C, then analysed using the reference method (plateau seeking).

Infrared analyses

Infrared analyses were carried out in duplicate in a non consecutive way. In order to check the stability of the apparatus,

a control milk was inserted every 20 samples. The mean value of the duplicate was calculated and used for determining results.

The evaluation concerned the values obtained by the calibration performed by the BENTLEY company, which was not adjusted with local milks.

Reference analyses

Determination of freezing point was performed using thermistor cryoscope method following ISO standard 5764 : 2002.

Routine analyses were performed and checked in case of important residual matters.

Statistical analyses

Accuracy is evaluated by using mean results of deviations, standards deviations of differences, residual standard deviations and assessed linear regressions equations, taking the reference method for explained variable Y, and B 2000 for the explicative variable X.

Compared to set 2, set 3 of 10 milks showed a systematic bias due to the zero adjustment. The results from samples of this set were corrected from the value of this bias (-14 m°C) before calculation of these parameters (except for residual standard deviations). For the calculation of residual standard deviation, each set was submitted to a specific linear regression. Adjusted results according to regression parameters were then gathered and the residual standard deviation calculated.

Results

n	50
min (m°C x -1)	466
max (m°C x -1)	528
Y (m°C x -1)	518.5
X (m°C x -1)	511.7
Sy (m°C)	13.8
d (m°C x -1)	-6.8
Sd (m°C)	4.4
Sy,x (m°C)	3.3
b	0.859
a	79

n, min, max : number of samples, minimum and maximum value

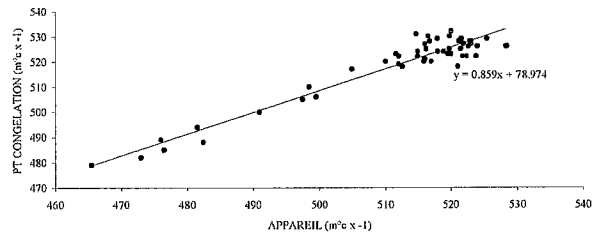
Y,X : mean results with reference and instrumental methods

Sy : standard deviation of results with the reference method

d : mean of the deviations

Sd : standard deviation of the differences

Sy,x : residual standard deviation for the linear regression



b,a : slope and bias of the linear regression

The table and figure above give the results and the linear regression obtained.

The slope and bias of the linear regression ($b= 0.859$ $a=79$) are significantly different from 1 and 0 ($P=1\%$). The residual standard deviation of the regression obtained is 3.3 m°C.

Conclusion

The standard deviation being 3.3 m°C allows for an accuracy in estimation of +/- 6.6 m°C. The results comply with BENTLEY specifications ($Sy,x < 4$ m°C).

CONCLUSION

Results of repeatability and accuracy comply with BENTLEY's specifications. The mean deviations observed between instrumental and reference values may be explained by the fact that evaluation concerned values obtained from the calibration performed by the manufacturer.

An adjustment based upon analysis of samples from the collection area is necessary, as required by IDF standard 128. It is also advisable to make more precise blanking in order to avoid any gaps.

Abbreviation

FPD : Freezing Point Depression

Bibliography

ISO 5764:2002 - Milk -- Determination of freezing point -- Thermistor cryoscope method (Reference method)

QUERVEL, X. et TROSSAT, P. **Rapport d'évaluation de l'analyseur B 2000, critère détection du point de congélation**, CECALAIT, 2003, Poligny, 3 p.

NEW EU REGULATION

BABY FOOD / EARLY CHILDHOOD /

Commission Directive 2003/13/EC of 10th February 2003 amending Directive 96/5/EC on processed cereal-based foods and baby foods for infants and young children (Text with EEA relevance)

Official Journal L 041 , 14/02/2003 P. 0033 - 0036

http://europa.eu.int/eur-lex/en/dat/2003/l_041/l_04120030214en00330036.pdf

BABY FOOD / EARLY CHILDHOOD / PREPARED FOODSTUFF /

Commission Directive 2003/14/EC of 10th February 2003 amending Directive 91/321/EEC on infant formulae and follow-on formulae (Text with EEA relevance)

Official Journal L 041 , 14/02/2003 P. 0037 - 0040

http://europa.eu.int/eur-lex/en/dat/2003/l_041/l_04120030214en00370040.pdf

CHLORAMPHENICOL / MILK / RESIDU / MRPLs : minimum required performance limits /

Commission Decision of 13th March 2003 amending Decision 2002/657/EC as regard to the setting of minimum required performance limits (MRPLs) for certain residues in food of animal origin (notified under document number C(2003) 764) (1) 17 2003/182/EC

http://europa.eu.int/eur-lex/en/dat/2003/l_071/l_07120030315en00170018.pdf

→For chloramphénicol in milk, MRPLs is 0,3 µg / kg.

MILK / BOVINE / SHEEP AND GOAT/ CONSUMER PROTECTION / VETERINARY INSPECTION /

Council Directive 2002/99/EC of 16th December 2002 laying down the animal health rules governing the production, processing, distribution and introduction of products of animal origin for human consumption

Official Journal L 018 , 23/01/2003 P. 0011 - 0020

http://europa.eu.int/eur-lex/pri/en/oj/dat/2003/l_018/l_01820030123en00110020.pdf

→ Addendum 3 : Milk and milk products (including cream) for human consumption :

"Effectiveness recognised" against food-and-mouth disease :

- (1) - of Ultra-high temperature (UHT = minimum treatment at 132°C for at least 1 second)
- (2) - if the milk has a pH of less than 7,0, simple high temperature - shorttime pasteurisation (HTST)
- (3) - if the milk has a pH of 7,0 or more, double HTST

"Effectiveness not recognised" against rinderpest and sheep and goat plague
for each of these treatments (1), (2), (3).

PACKAGING PRODUCT / PLASTICS / FOODSTUFF /

Corrigendum to Commission Directive 2002/72/EC of 6th August 2002 relating to plastic materials and articles intended to come into contact with foodstuffs (OJ L 220 of 15.8.2002)

Official Journal L 039 , 13/02/2003 P. 0001 - 0042

NEW ISO STANDARDS

ISO 14377 (Mars 2002)	CANNED EVAPORATED MILK - Determination of tin content - Method using graphite furnace atomic absorption spectrometry
ISO 14892 (Février 2002)	DRIED SKIMMED MILK - Determination of vitamin D content using high-performance liquid chromatography
ISO 6785 (Mai 2001)	MILK AND MILK PRODUCTS - Detection of <i>Salmonella</i> spp.
ISO 4833 5 (Février 2003)	MICROBIOLOGY OF FOOD - Horizontal method for counting of micro-organismes - Colony counting technique at 30°C

AFNOR VALIDATION RENEWAL

This alternative method was validated on 29th November 2002 and forwarded by AFNOR Certification to CECALAIT.

Name / Manufacturer	Date	Attestation number	Description
ACUPROBE	Validation 02.07.1995	BIO 12/4-02/95	Détection des <i>Listeria Monocytogenes</i> Tous produits d'alimentation humaine
Biomérieux Réf.39500 / Gen-probe Cat. N° 2920	Renewals 18.01.199 and 06.02.2003		<i>Listeria Monocytogenes</i> detection For all human foodstuffs
	End of validation 07.02.2007		

Texts of validation certificates as well as a three-monthly recapitulatory list are available at :
AFNOR Certification - 11 av. Francis de Pressensé - 93571 La Plaine St Denis cedex - Tél. : 01.41.62.80.00 – Fax : 01.49.17.90.00

ADDRESS BOOK

This address may be useful when dealing with British laboratories

BRC : British Retail Consortium / STANDARD / FOOD /

<http://www.brc.org.uk>

info@brc.org.uk

Second Floor
21 Darnmouth Street
London
SW1H 9BP
England

Tél. : + 020 7854 8900
Fax. : + 020 7854 8901

"The British Retail Consortium has actively been involved with the development and implementation of food related Standards since 1996. Standards currently available are :

1 - BRC Food Technical Standard :The BRC Technical Standard and Protocol for Companies Supplying Retailer Branded Food Products Issue 3

2 - The BRC/IOP Packaging Standard : The BRC/IOP Technical Standard and Protocol for Companies Manufacturing and Supplying Food Packaging Materials for Retailer Branded Products

3- BRC/FDF IP Standard : The BRC/FDF Technical Standard for the Supply of Identity Preserved Non-Genetically Modified Food Ingredients and Product (..)"

(Extract from the BRC web pages)

In REVIEWS - On the NET

MILK

FERMENTED MILK / NUTRITION / BLOOD PRESSURE /

► Solis B et al., The Effect of Fermented Milk on Interferon Production in Malnourished Children and in Anorexia Nervosa Patients Undergoing Nutritional Care, European Journal of Clinical Nutrition, 2002 ; 56 Suppl 4 : S 27-33

► SEPPÖ L. et al., A Fermented Milk High in Bioactive Peptides has a Blood-Pressure-Lowering Effect in Hyperactive Subjects, American Journal of Clinical Nutrition, février 2003, 77 (2) p. 326-330

PATHOGENES

E. COLI /

► "Scientists at a Scottish university have made a breakthrough in the drive to remove the *E. coli* 0157 bacterium from the food chain (...) The breakthrough came after the discovery of a different cell type lining the gastrointestinal tract of livestock (...) This new discovery will permit additional risk management procedures and should lead quite quickly to both direct interventions and more sophisticated controls, such as vaccines (..)"

E.coli breakthrough made, BBC 26/02/03
<http://news.bbc.co.uk/1/hi/scotland/2802301.stm>

SALMONELLA /

► Identification of genes activated during *Salmonella* infection. An important breakthrough in controlling diseases pathogen-borne diseases.

<http://www.ifr.bbsrc.ac.uk/media/NewsReleases/021218salmonella.html>, 18/12/2002,

BOOKSHOP

RHEOLOGY / CHEESE /

Sundaram Gunasekaran, M. Mehmet Ak. - **Cheese Rheology and Texture**, dec. 2002, 350 p. - (ISBN : 1587160218)

"(..)brings together the essential information on cheese properties with focus on relevant rheological techniques and presents data on a number of cheeses (..)" (extract from publisher's summary)

For information : www.CRCnetBase.com - To order : orders@crcpres.com

YOGHURT / MANUAL /

A.Y Tamime, R.K. Robinson - **Yoghurt : Science and Technology**, second edition, oct. 1999, 640 p. - (ISBN : 0849317851)

FORTHCOMING EVENTS

Events ordered according to chronology

ICE-CREAM /

14 - 16 May 2003
Thessaloniki, Greece

IDF Ice-Cream Symposium

Prof. Gregory . Zerfidis

Mail: gzerf@agro.auth.gr

ANALYTICAL TECHNIQUES /

19 - 23 May 2003
Holstebro, Denmark

**IDF / ISO / AOAC Analytical Week
and
Symposium on analytical techniques**

<http://www.fil-idf.org>

Caroline Brooks :
Cbrooks@fil-idf.org

TEXTURE / PROCESS / PERCEPTION /

11 - 13 June 2003
Papendal, Netherlands

**3rd NIZO Dairy Conference
Dynamics of Texture, Process & Perception**

<http://www.nizo.com>

info@nizo.nl

DAIRY PRODUCTS / NUTRITION / HEALTH /

24 - 27 August 2003
Melbourne, Australia

**IDF 2nd World Symposium on Dairy Products in Human Health and
Nutrition**

2003 Foods for Life Conference and Exhibition

Pamela Tyers
pamela.tyers@2003foodsforlife.com

www.2003foodsforlife.com

DAIRY PRODUCTS /

8 - 12 September 2003
Bruges, Belgium

**IDF World Dairy Summit and Centenary
Dairy Nature's Challenge**

<http://www.fil-idf.org>

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