

SUMMARY OF LA LETTRE DE CECALAIT, N°28 (JANUARY 1999)

DAIRY INTERNATIONAL STANDARDIZATION : AIMS, PARTICIPANTS, EUROPEAN AND NATIONAL LEVEL

(summary of the lecture held by R. Grappin (INRA - SRTAL, Poligny) at CECALAIT annual session)

Codex Alimentarius is an international organization created in 1962 by the FAO and the WHO, with 161 states as members, nowadays. It has to elaborate definitions and criterions applicable to food, to contribute to international harmonization, to make international trade easier and to protect the consumer.

It is divided into 8 horizontal Codex Committees, one of them concerning methods, analysis and sampling –CCMAS-.

It is also divided into 15 vertical committees, each of them concerning a specific kind of product. Only six committees are actually working, one of them about milk and dairy products.

The tasks of Codex Alimentarius are :

- definition and elaboration of physico-chemical and microbiological methods of analysis,
- definition of the methodology, ie terminology to be used, principle of collaborative studies, analytical characteristics ,
- quality assurance,
- security, environmental care.

The Codex methods are graded into 4 types.

Type I concerns « definition » methods, such as the Kjeldahl method. Type II concerns reliable reference methods, validated by collaborative studies. Type III means alternative, indirect methods, which are however reliable and have been validated by collaborative studies. Type IV is for « provisional » methods, where analytical performances are not fully assessed .

Nevertheless, it should be remembered that Codex is not an expert Commission. The Committees members are **states**. Therefore, for studying methods or producing standards, Codex needs to appeal to international experts and standardizing bodies. They are listed in table 1 : organizations producing standards. (see Table 1, for this article, in La Lettre de CECALAIT).

Considering the participation of the standardizing bodies, a Codex standard must follow a three steps circuit illustrated in the diagram, placed under table 1 in La Lettre de CECALAIT

(NB : OMC = WTO : World Trade Organization).

When fully detailed, the production of a standard (part ② of the above mentioned diagram) is described by the diagram given at the end of this article in La Lettre de CECALAIT : Production of a standard.

Clearly, it is a long-lasting procedure. But standards issued by Codex Alimentarius have gained an increased importance since WTO creation in 1994. Indeed they are used as international arbitrations in case of national legislation contests, for instance.

List of abreviations (for this summary and for diagrams and table in this article in La Lettre de CECALAIT)

AFNOR : Association Française de Normalisation : French Standardizing body

AOAC : Association of Official Analytical Chemists

CCMAS : Codex Committee on Methods, Analysis and Sampling

CE = EC: European Community

CEN : Comité Européen de Normalisation : European Standardization Committee

CG d'UMA : Commission Générale d'Harmonisation des Méthodes d'analyse }
CST : Commission Scientifique et Technique } *scientific technical commissions*
FAO : Food and Agricultural Organization
FIL : Fédération Internationale de Laiterie = IDF : International Dairy Federation
ISO : International Standardization Organization
IUPAC : International Union of Pure and Applied Chemistry
JOCE : Journal Officiel des Communautés Européennes : EC Official Journal
OMC = WTO : World Trade Organization
WHO : World Health Organization

COMPARISON OF METHODS OF DETERMINATION OF FAT IN CHEESE.

The reference method for the determination of fat in cheese is the gravimetric SBR (Schmid-Bondzynski-Ratzlaff) method (IDF 5B :1986 or ISO 1735). It is based upon digestion of the sample with hydrochloric acid, addition of ethanol and subsequent fat extraction by a mixture of diethyl ether and light petroleum. But, like most gravimetric methods, it is rather long and difficult to perform. Therefore, for everyday analysis, most laboratories use routine methods, the most common among these being the butyrometric ones.

In France, the Van Gulik butyrometric method has been used since the early 50s. It was standardized in 1969 and after, in 1972 and is still applicable. It is based upon digestion with sulfuric acid, followed by centrifugation in a Van Gulik butyrometer in the presence of amyl alcohol.

However some studies showed the defects of the method. Particularly, in 1961, in Germany, E. Heiss compared different butyrometric methods with the SBR method and pointed out high differences between the results given by the Van Gulik method and those obtained by the reference method. He suggested then a new method, based upon digestion with a mixture of perchloric and acetic acid, at a temperature of 85°C instead of 65° in the former method, without amyl alcohol. Afterwards, this new method has been tested and adopted in a lot of laboratories and now it is quite as used as the Van Gulik method, though it is not standardized.

In the ringtests which have been organized for 3 to 5 years by CECALAIT on hard and soft cheese, participants may use either method. The whole results were put together and classified according to the method used in order to study the accuracy of each one versus the reference method.

In the ringtests, 6 different cheeses, ie 6 different fat contents, are used. For each fat content and each participant, the mean of the differences between routine results and reference results is calculated. The reference values were obtained in the same ringtests, with the same samples, analyzed with the SBR method. Thus, it was possible to sort out two populations of mean differences, one corresponding to users of the Heiss method, the other to users of the Van Gulik method. Outliers were eliminated and afterwards, accuracy was assessed by :

- the mean of mean differences also called mean bias, which corresponds to the systematic error of the method,
- the standard deviation of mean differences, which corresponds to the standard deviation between laboratories using the same method and highly contributes to its reproducibility.

The results are shown in figures 1 to 4 in the article in La Lettre de CECALAIT. Table 1 sums up the data and the results.

Whatever cheese, the accuracy biases dispersion of the Van Gulik method is clearly higher than the Heiss method. Figures 1 to 4, with their positive dissymmetry, show an overestimation tendency of that method. It is less « robust » than the Heiss method.

In conclusion, it appears that the Heiss method is fairly more accurate than the Van Gulik method. Of course, further studies with other types of cheese are necessary in order to assess clearly the accuracy of each method versus the SBR method. However, this study constitutes an important step

in the course of the revision of the standards concerning routine methods for fat determination in cheese.

INTERESTING RECENT EEC REGULATION

Regulations n° 2560/98 on 1998/11/27, **2686/98** on 1998/12/11, **2692/98** on 1998/12/14 and **2728/98** on 1998/12/17, of the Commission modifying annexes I to III of regulation n° 2377/90 of the Council concerning maximum residue limits of veterinary drugs in foods of animal origin. (JOCE L320 on 1998/11/28, L337 on 1998/12/12, L 338 on 1998/12/15, L 343 on 1998/12/18)

Regulation n° 2521/98 on 1998/11/24 modifying regulation n° 577/97 concerning the application of regulation n° 2991/94 of the Council establishing standards for spreadable fat and of regulation n° 1898/87 of the Council concerning milk and dairy products denomination in marketing. (JOCE L315 on 1998/11/25)

Directive 98/72/CE of the European Parliament and of the Council on 1998/10/15 modifying directive 95/2 concerning food additives other than colouring agents or sweeteners. (JO L295 on 1998/11/4)

Directive 98/82/CE of the Commission on 1998/10/27 modifying the annexes of directive(s) ...86/363/CE...of the Council concerning fixing of maximum residue limits for pesticides on and in food of animal origin.... (JO L290 on 1998/10/29)

↳ others texts

➤ **directive 98/83** on 1998/11/3 of the Council, indirectly concerning milk and dairy products. Indeed it specifies the quality criteria of water intended for human consumption, which include water used in food industry. (JOCE L330 on 1998/12/5)

➤ At last, for your information, the huge (more than 600 pages) directive 98/98 on 1998/12/15 [...] concerning the tentative harmonization of laws and regulations on classification, package and **labelling** of hazardous substances. (JOCE L 355 on 1998/12/30)

Official Journals of the European Communities of the last 45 days may be consulted on Internet <http://europa.eu.int/eur-lex>

Older texts may be ordered on Internet <http://www.eudor.com>

INTERESTING NEW STANDARDS

NF EN 12689 1998, september 1998 (ICS 07.080). BIOTECHNOLOGY. Guide for the evaluation of purity, biological activity and stability of microorganisms-based products

AFNOR VALIDATION

AFNOR (French standardization organization) validated recently the following alternative method : **RAPID L. mono**, a special culture medium which allows a fast detection of *Listeria monocytogenes*.

LIST OF BIBLIOGRAPHIC REFERENCES

It is the list of references that we noticed in our literature survey during the past months and that we decided to put into our data base on dairy analytical techniques. Should you be interested in any of these references, please contact us.

NB : we remind you that we can copy neither book nor standard.

FORTHCOMING EVENTS

➤ Reminder

**19-24 APRIL 1999 ANALYTICAL WEEK :
IDF/ISO/AOAC, IN OTTAWA (CANADA)**
& at the same time

19 APRIL 1999 : Symposium IDF/ISO/AOAC

"Laboratory accreditation and proficiency
testing"

For information, please contact

IDF

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➤ OTHER EVENTS

➤ **28 - 30 APRIL 1999** : IDF Symposium in
PENANG, MALAYSIA
"Recombined milk and milk products "

➤ **9 – 11 june 1999** : 5th International
symposium on authenticity of foods, in LA
BAULE (France)

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➤ **15 – 17 july 1999** : IDF Symposium in
SAINT MALO (France)

« Membrane processing and new products in
dairy industry. »

For information, please contact

Prof. J.L. MAUBOIS
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65, rue de Saint-Brieuc
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➤ **1 – 4 August 1999** : IAMFES ANNUAL
MEETING, in DEARBORN , Michigan,
ETATS-UNIS

IAMFES : International Association of Milk,
Food and Environmental Sanitarians

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➤ **18 – 21 october 1999** : METROLOGIE 99,
9th International Congress of metrology in
Bordeaux (France)

For information, please contact

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