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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 caracteristics ,
- 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
} *scientifical technical commissions*
FAO : Food and Agricultural Organization
FIL : Fédération Internationale de Laiterie = IDF : International Dairy Federation
ISO : International StandardizationOrganization
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 routin 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 butyromeric 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 routin 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 figures1 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. Figurs 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 routin methods for fat determination in cheese.