

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.