SUMMARY OF LA LETTRE DE CECALAIT, N°27 (OCTOBER 1998)

FREEZING POINT : STATE OF THE ART AND EVOLUTION IN STANDARDIZATION

(summary of the lecture held by L. Laloux (LCHA, Paris) at CECALAIT annual session)

Milk freezing point is a quality criteria which can be used for estimating the amount of extraneous water in milk. It is specified by the European regulations, especially directive 92/46 and regulation 2597/97. The last one specifies that milk should have a freezing point "near to the mean freezing point of the area where it is collected". It is determined by using standard IDF 108B:1991. The cryoscope reference method for the determination of the freezing point is based upon plateau seeking in the freezing curve. The studies carried out by IDF/AOAC/ISO E601 group showed that estimating this constant point was rather difficult.
For example, two different cryoscopes may have differences in software, using different cut off points, which will result in two different values for the freezing point (see Fig 1 in La Lettre de CECALAIT).
However, even after software harmonization, group E601 noticed that different cryoscopes gave different results. It seemed to depend on the dimensions and thermal properties of the thermistor shank. The most important points are :

- measurement duration : if it is too long, the room temperature may affect the measurement
- the material and the dimensions of the thermistor probe. See table 1 in La Lettre de CECALAIT, which shows the differences between the results given by two cryoscopes, one equipped with a stainless steel probe, the other with a PVC probe.

Following these studies, new definitions and new cryoscopes have been developed. The plateau is now reached when the temperature rise has been maximal 0.5 m°C over the last 20 s.

- The dimensions specified for the shank are :
  - internal diameter : 13,7 ± 0,3 mm;
  - external diameter : 16,0 ± 0,2 mm;
  - height : 50,5 ± 1,0 mm

- The thermistor should be of the glass probe type and its thermal properties should be
  - resistance between 500 and 25000Ω at 0°C
  - heat transfer from the shank into the sample lower than 2,5 mJ/s

These points will all appear in the new version of IDF standard 108 to be issued at the end of 1999.
At last the working group proposed a programme for further works :
- to undertake an interlaboratory study with the above described cryoscopes (this has already been done in October, with CECALAIT preparing and dispatching the samples and treating the results)
- to define new routine methods
- to study more carefully the influence of heat treatments of milk on the freezing point. See table 2 in La Lettre de CECALAIT.

In conclusion, the evolution of standardization and cryoscopes and how it will affect laboratory practice is still a major topic whereas "The mean freezing point of the area where milk is collected" remains a delicate question.

MILK AND DAIRY PRODUCTS ANALYSIS : MIR OR NIR, ADVANTAGES AND DISADVANTAGES

(summary of the lecture held by G. Mazerolles (INRA, Poligny) at CECALAIT annual session)

In the last 30 years, infrared spectrometry has been used more frequently in dairy laboratories. Mid infrared instruments (MIR) were the first able to give accurate and precise quantitative results in milk analysis. Since

then, the development of PCs and softwares made it possible to adapt near infrared (NIR) instruments or Fourier transform (FTIR) infrared instruments (MIR or NIR) to routine analysis.
Wavelengths from 2.5 to 50 µm belong to the MIR spectrum. In this region, analytes, but also solvants have strong absorption bands, affected by the environment of the chemical groups. Wavelengths from 0.7 to 2.5 µm belong to the NIR spectrum. They are more energetic, therefore penetration and diffusion of the wavelength are more important than in MIR spectrum. Spectral information is not as well described. On the other hand, the components of the optical systems in NIR are less expensive than in MIR.
A spectrophotometer is always composed by an infrared source, a dispersive or interferential system and a detector. They work either in transmission or in reflexion (see figures in La Lettre de CECALAIT).
Concerning calibrations, MIR instruments are ususally calibrated at well defined wavelengths. The same is possible in NIR, with concentrations related to absorbances by way of multilinear regression. Nevertheless, the most recent applications of NIR use rather a broader spectrum and need mathematical pretreatment of data and complex statistical techniques like principal component analysis or partial least squares. Actually, NIR analysis has mostly begin to develop since computers have been able to do these complex statistical calculations fast.
A survey of the litterature of the fifteen last years about dairy analysis shows, as expected, more MIR than NIR results. It confirms that MIR analysis remains the best technique for routine milk analysis. However, results obtained from the 80s show that NIR performs nearly as well as MIR and point out the interesting potential of NIR.
In milk, NIR signal does not seem to be affected by lipolysis, but the influence of a preservative agent is still unknown. Concerning cheese, the litterature survey showed interesting results and prospects for NIR. For instance, it seems that a single calibration could be used regardless of the maturation of the cheese.
Other interesting prospects concern milk analysis in farm, study of milk coagulation and on line process control, where NIR is coupled to optical fibers. This last application is already working in some production units.