ASSESSMENT OF THE AFSSA / CECALAIT CRYOSCOPY TESTS
With the collaboration of SYNDILAIT (provision of consumer milk), RADIOMETER and FUNKE GERBER (loan of apparatus).

The new standard ISO 5764 concerning the freezing point of milk was published in 2002. In comparison with the previous text, this document clearly defines the reference method as being a plateau finding method (with a new definition of the plateau which is considered as having been reached when the temperature increase has not exceeded 0.5m°C over the last 20 seconds). The fixed time methods are considered to be routine methods and need to be tied to the reference method.

Following the application of the new method, certain countries have observed an interval of approximately 3m°C on samples analysed by means of both an apparatus complying with the new standard and an apparatus complying with the previous text.

Following the presentation of the results to a group of expert chemists (DG AGRI CEE), the president H. GLAESER expressly asked the other member states to voice their conclusions on this analytical comparison.

It seemed therefore essential for us to conduct a comparative study, at the French level, on raw milk and consumer milk, to determine if a significant interval exists between the two versions of the method, or not.

The study consisted in a comparison, in two laboratories, of results obtained between apparatus functioning according to the 1987 version and the 2002 version:
Laboratory 1: one apparatus complying with the standard ISO 5764 : 1987 and one complying with standard ISO 5764: 2002
Laboratory 2: one apparatus complying with the standard ISO 5764: 1987 and two complying with standard ISO 5764: 2002
The study was carried out on 26 raw milk samples conserved with Bronopol (0.02% final), corresponding to bulk milk from different regions in France, and on 35 consumer milk samples, representative of different technological treatments.

The results are as follows:

BY COMPARISON USING RAW MILK (VALUES in m°C x–1)

	ISO 5764 :1987 App 1	ISO 5764:1987 App 2	ISO 5764 :2002 App 3	ISO 5764 :2002 App 4	ISO 5764:2002 App 5
N	26	26	26	26	26
Mean	531,0	530,8	533,0	529,0	532,0
Difference ISO 5764 :1987/ISO 5764 : 2002*	-	-	+2,0	-1,8	+1,2
Standard deviation of the difference ISO 5764:1987 / ISO 5764 : 2002*	-	-	2,0	1,6	2,4

** The deviations are intra laboratory differences, that is:*
Lab 1: ISO 5764: 1987 Apparatus 1 and ISO 5764: 2002 Apparatus 3
Lab 2: ISO 5764: 1987 Apparatus 2 and ISO 5764: 2002 Apparatus 4 and 5

BY COMPARISON USING CONSUMER MILK (VALUES IN m°C x–1)

	ISO 5764 :1987 App 1	ISO 5764 :1987 App 2	ISO 5764 :2002 App 3	ISO 5764 :2002 App 4	ISO 5764:2002 App 5
N	35	35	35	35	35
Mean	519,0	519,1	521,7	517,3	520,9
Difference ISO 5764: 1987 / ISO 5764: 2002*	-	-	+2,7	-1,8	+1,8
Standard deviation of the difference ISO 5764: 1987 / ISO 5764: 2002	-	-	2,2	1,4	3,2

** The deviations are intra laboratory differences, that is:*
Lab 1: ISO 5764: 1987 Apparatus 1 and ISO 5764: 2002 Apparatus 3
Lab 2: ISO 5764: 1987 Apparatus 2 and ISO 5764: 2002 Apparatus 4 and 5

Firstly, it can be observed that the results obtained with raw milk and consumer milk, using the instruments complying with standard ISO 5764: 1987 were equivalent for the two apparatus tested (mean results equivalent).
Then, the presence of an effect "instrument" can be noted for the test results obtained with the samples analysed with the instruments complying with standard ISO 5764: 2002. In effect, the apparatus 3 and 5 present appreciably equivalent results (mean of determinations respectively 533.0 and 532.0 m°C x-1 with raw milk, and 521.7 and 520.9 m°C x-1 with consumer milk). These values are out of line with the results obtained with apparatus 4, presenting the same configuration (mean of determinations respectively 529.0 and 517.3 m°C x–1 with raw milk and consumer milk).
Observation of the analytical test means between the apparatus (including variability "version" and variability "instrument"), show an amplitude of 529 to 533 m°C for raw milk and 517.3 to 521.7 m°C for consumer milk.
There seems to exist a tendency relative to the instrument (all versions included), but, it is necessary to note that the deviations observed can be completely put down to the technical characteristics of the cryoscopic method, such as they are defined in the normative document ISO FIL 5764: a maximum deviation between duplicates (r) = 4 m°C and a reproducibility limit (R) = 6 m°C.
The results obtained in this French study have been transmitted to the group of expert chemists in Brussels, where they will question themselves on the results obtained throughout Europe.
A statistical analysis will be carried out on all the results and will serve as a basis of a general technical reflection on this precise point.