



4TH INTERNATIONAL WORKSHOP ON WATER IN FOOD

"FREEZING POINT DEPRESSION OF MILK - PROBLEMS WITH IMPLEMENTATION OF THE NEW STANDARD IN THE UK"

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EU Regulation 2597/97 requires that drinking milk must have a freezing point close to the average freezing point of raw milk recorded in the area of origin of the drinking milk collected.

Standard Method Being Changed and Adopted by ISO IDF (IDF 108B to IDF 108C).



Set up Study “Determination of Freezing Point Depression of Milk from Cows, Goats, Ewes and Buffaloes”

The objectives of this study were:

1. to determine the freezing point depression in raw milk samples from cow, goat, ewe and buffalo samples representative of milk throughout England and Wales, and representative of winter feeding and summer grazing;



2. to determine the freezing point depression in raw milk samples from cow, goat, ewe and buffalo samples from tracking herds sampled regularly over a period from February to May
3. to undertake analysis using a cryoscope modified to comply with the requirements of the proposed IDF Standard 108C and provide comparative data on a minimum of 50 samples analysed using the existing standard IDF 108B.



In order to support UK negotiations on the implementation of Commission Regulation 2597/97 the UK requires contemporary data on the freezing point of raw milk from a variety of species; furthermore adequate provision needs to be made for the possible adoption of a modified IDF reference method for the measurement of milk freezing point.

The IDF proposal to change the standards applicable to cryoscopes had not been adopted at the start of the project, and remains subject to approval; contractor obtained a modified instrument from the supplier.



The mean and standard deviations obtained for bulk cow's milk were:

- 517.51 m°C and 5.5415 m°C (108C, and
- 520.01 m°C and 4.8518 m°C (108B).

Goat's milk were:

- 543.29 m°C and 6.815 m°C (108C), and
- 544.86 m°C and 6.662 m°C (108B).



Ewe's milk were:

- 550.62 m°C and 7.707 m°C (108C), and
- 552.21 m°C and 8.580m°C (108B).

Buffalo's milk were:

- 512.14 m°C and 21.300 m°C (108C), and
- 514.36 m°C and 21.862 m°C (108B).



Seasonal differences were noted for cow's milk samples, for the 108C:

the January/February mean was - 519.68 m°C.

the June mean was - 515.10 m°C.

A difference of 3.16 m°C, in the same direction, was observed with 108B results.

No significant regional difference (north vs. south) was observed using either instrument.



Differences (“fed inside” vs. “fed outside”) were noted for goats and ewes. The trend was the reverse of the seasonal difference observed for cow’s milk.

In total the samples analysed comprise, 412 bulk cow’s milk, 78 tracking cow’s milk, 84 goat’s milk, 35 ewe’s milk and 14 buffaloes milk, making a total of 623 samples.



Limits have been set for the freezing point of milk in the past. Council Directive 92/46 Article 5 .9 (3) stated that Member States must ensure that heat-treated milk must have a freezing point not higher than -0.520°C .

Thus where the FPD is greater than $-0.517\text{m}^{\circ}\text{C}$ the supply is assumed to be free of extraneous water.



Extract from FSA Recommendations to Analysts

The freezing point test is used to measure extraneous water in milk. In the late 1990's small but significant differences were found in the freezing point of samples tested using cryoscopes from two different instrument manufacturers. This, with the active participation of the two major manufacturers of cryoscopes, led to an extensive study of basic cryoscope design, resulting in recommended modifications. The modifications involved replacing the stainless steel probe with one made from delrin plastic and upgrading the cryoscope.



Extract from FSA Recommendations to Analysts

EVALUATION OF INSTRUMENT CHANGES

Modified instruments from both manufacturers were evaluated in a major international ring trial initiated in September 1999 and reported in early 2000.

19 laboratories participated in the trial and each tested 18 pairs of blind duplicate samples. The results of the trial showed closer agreement between the two makes of cryoscopes and precision data in-line with previously reported data viz.



Extract from FSA Recommendations to Analysts

Repeatability (r) (95% of results within) 4m °C.

Reproducibility (R) (95% of results within 6m °C)

Whilst the modifications to cryoscopes have led to a tighter standard they have also brought about significant practical changes to the test for extraneous water in milk.



Extract from FSA Recommendations to Analysts

Target value for genuine milk

Using International Standard ISO 5764/IDF 108 (2002) UK milk industry and enforcement officers should consider milk to be genuine if it has a FPD of 509 m⁰C or more.

This value is equivalent to the Hortvet value of 530 m⁰CH and 512 m⁰C as measured by IDF method 108B.



Extract from FSA Recommendations to Analysts

Where action is to be taken on a freezing point test result it is recommended that the reference method given in ISO 5764/IDF 108 (2002) is used.



But was that correct?

Recent anecdotal work does not confirm that!

So second study carried out in 2004/5.



A total of 155 raw milk samples were collected from farms located throughout England and Wales during October to December 2004. Farms were selected in order to ensure that a full range of freezing point depression values could be expected. A total of 50 samples of processed milk were collected from retail outlets in the Wolverhampton area during the same period. Retail samples included skimmed, semi-skimmed and whole pasteurised milk, Channel Island milk, UHT and sterilised milk.



Raw milk samples were taken from individual farm bulk tanks and transported to the laboratory within 12 hours of collection in refrigerated vans. The sampling and storage of the samples was consistent with that adopted for routine milk payment purposes. Raw milk samples received at the laboratory were stored in refrigerated conditions and analysed within 12 hours of receipt. Samples of heat treated milk (from supermarkets) were transported in cool boxes, stored in refrigerated conditions and analysed within 24 hours of purchase, and in all cases within the use by date.



Samples were analysed using seven cryoscopes. A single supplier (Advanced Instruments) provided five of the cryoscopes; two of these were configured to the old specifications and two to the new one. The fifth cryoscope was a model that had been used in 1977 to establish the original UK freezing point presumptive limit. A second supplier provided two further cryoscopes configured to the new specifications.



Conclusions from Study

The mean freezing point of farm milk supplies recorded in this study (524.9 m°C) was close to that reported in the UK 1977 study (523.3 m°C) from which the target value for genuine milk was derived. (However it is noted that the current study covers only milk collected in October and November whereas the 1977 study covered a full 12 month period).



The freezing point of commercially processed (retail) milk (-1.75°C) was close to that of farm milk samples showing that little extraneous water was present.

There was no evidence that the changes in specification of the equipment from IDF 108B to IDF 2002 had an effect on the sample results that would, on its own, lead to a change to the milk standards established by the 1977 survey.



Work in the project showed that, on the basis of a limited comparison of preserved and un-preserved salt solutions, there were statistically significant differences.



What Do Member States Do?

Region or area?

Initial information suggests a country wide standard (freezing point depression value).

Final Conclusion - don't rush into project work!