



Background

The freezing point depression (FPD) test is used to measure extraneous water in milk. Interpretation of FPD results in England and Wales was based on a major notional survey conducted in 1977 jointly by Public Analysts and the (then) National Institute for Research in Dairying at Shinfield. Samples were tested using the Hortvet method (see Part 1 of BS3095). A key conclusion from this work was that it is likely that milks having an FPD of 529m°H and less, contain extraneous water.

As a consequence, in recent years the UK milk industry and enforcement officers considered milk to be genuine if it had an FPD of 530m°H or more. It is also known that "true" freezing points as measured by the old IDF Standard differ from those determined by the Hortvet method and milk of 530°mH reads 512m°C.

Freezing point depressions can be converted from Hortvet (T_H) to true Celsius (T_C) by using the following formula (BS3095: Sections 1.1 and 1.2):

$$T_C = 0.9656T_H$$

Consequently, 530 TH = 511.8Tc (IDF method 108B)

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, including Advanced/Fiske, whose products are widely used throughout the UK and Western Europe. An extensive study of basic cryoscope design was undertaken, resulting in certain recommended modifications. The modifications included replacing the stainless steel probe with one made from plastic, and upgrading the cryoscope software in order to redefine the plateau temperature.

Performance of instruments

Modified instruments 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 precision data was in-line with previously reported data viz.

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.

Furthermore, all new or modified instruments performing the reference method may affect the interpretation of the presence or absence of extraneous water in milk.

In order to address these issues, a new international reference method, ISO5764/IDF1 08 (2009) was agreed that requires new or modified instruments for compliance.

The UK dairy industry chose to adopt the new standard ISO5764/IDF108 (2009) as soon as possible. QCL support the industry's implementation in a number of ways.

Instrument Upgrades

The following Advanced/Fiske instrumentation which can be upgraded as previously described:

Model: Advanced 4D3

Serial number suffix: G or later

Model: Fiske Mk2

Serial number suffix: B or later

Fitting costs are available from the QCL Service department (01342 820822). This cost can be minimised if carried out at the same time that the instrument is serviced. Discounts are available if more than one system is upgraded at the same site at the same time.

Older models including Advanced 4D2, 4L2 and 4C2 as well as the Fiske Model MS2 4400, do not have software systems capable of being upgraded. All upgraded new instruments are capable of running routine and reference methods, user selectable.

Calibration Standards

QCL calibration standards retain the same part number due to the very minor changes in salt concentration required by the standard. However, they have been relabelled in accordance with the directives of ISO5764/IDF108 (2009), with attention to the preferred use of the centigrade scale and shelf-life considerations. QCL calibration standards are traceable to prime standards as defined in ISO5764/IDF108 (2009) and employ a shelf-life tested preservative in a concentration that does not change the published accuracy of the standards. Instrument calibration protocol remains unchanged.

A new range of QCL cryoscope tubes (part number BOR025, pack 12) made from borosilicate glass was introduced for the reference and for all routine methods.

Plastic thermistor probes conforming to ISO5764/IDF108 (2009) and routine methods can be purchased as part number 4D3102.

The new software employed to detect plateau temperature in ISO5764/IDF108 (2009) requires strict adherence to specified dimensions of probes, stirrers and tubes, and their relative positioning adjacent to the mandrel. Users are advised to consult fitting instructions carefully and if in any doubt consult a QCL engineer.

Use of older cryoscopes*

Whilst older cryoscopes cannot be upgraded to comply with the new reference method, they can be kept in use for routine methods. However, in order to obtain on average agreement with the new reference method, a correction factor should be established according to Annex B of International Standard ISO5764/IDF108(2009). This requires establishment of an average agreement between results from the new reference method (ISO5764/IDF 108(2009)) and a routine method for the milk samples typically tested in that laboratory).

The following information, some of it historical, is taken from recent publications on this subject, and further observations on the international standard ISO5764/IDF1 08 (2009), of which you are advised to purchase a copy.

Standards organisations

With the adoption of this new standard, both ISO5764:1987 and IDF Standard 108B Part 1 will be superseded, as will the current British Standard for measuring freezing point by the thermistor cryoscope method. The new ISO Standard 5764:2009 has been adopted as British Standard BS EN ISO5764.

The new ISO/IDF International Standard is available from:

IDF Secretariat
Diamant Building 80
Boulevard Auguste Reyers
1030 Brussels
Belgium

Also available from Central Secretariat (Geneva) and BSI (Gunnersbury).

Relationship between methods for determining FPD*

The new International Standard for determining freezing points of milk ISO5764/IDF108 (2009), gives results slightly different from the previous method (IDF108B). The September 1999 ring trial indicated that this new method gives results on milk differing on average from the earlier method by up to 3m°C. Early indications from laboratories using the new method indicated lower differences between old and new methods. Differences in results between old and new methods will however vary depending upon locality of the milk.

In converting to the new method, the opportunity has also been taken to carry out a further conversion from results as measured in m°H to values in "true" freezing points in m°C.

Annex C of the new method recommends that each National Reference Laboratory check the results obtained using the old method against the new method and advise of any changes which might be required in the target freezing point values set for genuine milk (see enforcement).

Enforcement*

Target value for genuine milk

Using International Standard ISO5764/IDF108 (2009), it has been decided that the UK Milk Industry and enforcement officers should consider milk to be genuine if it has an FPD of 509m°C or more.

The calculated correction factor (see Annex B of the new standard), is only valid for the specific combination of type of test samples and routine method chosen. Where action is to be taken on a freezing point test result, it is recommended that the reference method given in ISO5764/IDF108 (2009) is used.

**QCL has interpreted this information from publications received within the UK dairy industry and from ISO5764/IDF108 (2009) itself. However, it remains the sole responsibility of the user to decide how best to implement the new method within the users' laboratory.*