

## **APPENDIX 6 FREEZING POINT DETERMINATION**

### **1. Methods for determination.**

The milk analyzer determines the freezing point of each sample and the quantity of added water. The milk analyser does not measure the freezing point, but calculates it from the components it depends on. The basic components in the milk are water, solids, lactose, FAT, proteins, minerals (salts) and acids. The freezing point depends only on the diluted in the milk components and quantity of the solvent (in the milk it is water). The ultrasonic technology allows direct measurement of FAT, proteins, lactose + salts (the soluble components, only influencing the freezing point), and the quantity of the solvent in % is determined by  $100\% - \text{total solids \%}$ ,  $\text{total solids} = \text{lactose \%} + \text{FAT \%} + \text{proteins \%} + \text{salts \%} + \text{acids \%}$ .

Without understanding the meaning of the freezing point – determined or shown from the milk analyzer added water result easily may lead to a mistake for the value of this parameter.

### **2. The basic freezing point.**

Milk freezes at lower temperature than water. The average freezing point of the raw milk in the most regions is at about  $-0,540^{\circ}\text{C}$ . The average reading for your region is called “basic” freezing point.

The freezing point of milk is a “physiological constant”. This does not mean that it will not vary. In fact feed, breed, season, time of lactation, climate, whether the sample is taken at the beginning, middle or end of lactation – all these factors will have an effect on the freezing point of the individual sample. This means that there is an average value of all these numbers. The more samples used in obtaining this average, the more reliable it is as a base. Or the basic freezing point is an average of freezing points of milk, taken from many cows. When a laboratory checks a producer, it is only comparing the average of the producer’s cows against a larger area average.

The Health authorities establish the basic freezing point or agriculture departments in some regions, sometimes by universities, separate dairy producers, or their associations. Frequently, tolerances have been established on top of a basic freezing point to allow some variations in the milk as well as device or operator variations.

Without mentioning the basic freezing point, the Association of Official Analytical Chemists now recommends an upper limit freezing point at  $-0,525^{\circ}\text{C}$  (2,326 standard deviations above the most recently determined North American average of  $-0,5404^{\circ}\text{C}$ ), below which there will be at 95%

confidence that will show 99% of all freezing point determinations on unwatered milk:

“if the freezing point is  $-0,525^{\circ}\text{C}$  or below, milk may be presumed to be free of water or may be confirmed as water free by tests, specified below. If the freezing point is above  $-0,525^{\circ}\text{C}$ , milk will be designated as “presumptive added water” and will be confirmed as added water or added water free by tests specified below. Evaluate extreme daily fluctuations in the freezing point of herd, pooled herd, or processed milk for presence of added water”.

“Presumed added water”, as described above, must be “confirmed” by means of tests on authentic milk samples obtained as specified in the AOAC METHODS.

After determination the freezing point of your sample via the milk analyzer, the added water is calculated using the following formula:

$$\text{AddedWater} = \frac{\text{FrPoint}_{\text{Base}} - \text{FrPoint}_{\text{Calc}}}{\text{FrPoint}_{\text{Base}}} * 100[\%]$$

Where:

FrPointBase is the basic freezing point

FrPointCalc is measured freezing point

Note:

If the freezing point is not correctly determined, the result for the added water is not valid. In this case results for FrPoint and AddWater are not shown on the display and on the printout from the printer. If the density of the measured sample is 0, the result for AddWater is not valid and is also not shown on the display and the printouts.

Sample:

First variant

If you’ve entered for milk analyzer basic freezing point  $-0.520^{\circ}\text{C}$  (according article 5.9 of the EU Milk Hygiene Directive 92/46/EEC), measured freezing point  $-0.540^{\circ}\text{C}$ , using the above pointed formula you’ll receive  $-3,8\%$ . Because it is not possible the added water to be negative value, the milk analyzer indicates 0% added water. The reason for this is the tolerance in the basic freezing point, reasons for which are described below.

If in the same milk we add 3,8% water, and the basic freezing point is the same, the milk analyzer will measure freezing point  $-0.520^{\circ}\text{C}$ , and will indicate again 0% added water.

Second variant

If you've entered for the device basic freezing point  $-0.540^{\circ}\text{C}$ , measured freezing point  $-0.540^{\circ}\text{C}$ , the milk analyzer will indicate 0%. When you add 3,8% water, the device will indicate 3,8%-added water.

From the above mentioned follows that it is very important to enter correct basic freezing point in the device.

The device's results for added water may give information about doubt of added water in the milk and the exact value of this added water may be determined after a "cowshed sample" is taken and the result for the freezing point, measured by the milk analyzer of the "cowshed sample" is entered as basic freezing point in the formula for calculation of added water.

Then the result from this formula will give us the absolute value of the added water for the corresponding milk supplier.