ARTICLE

DETERMINATION OF THE FAT ACIDITY (LIPOLYSIS) – PRINCIPLE AND CRITICAL POINTS OF THE STANDARDISED METHODS

The determination of the fat acidity (lipolysis) is important in the analysis of the product quality.

Two standardised methods for the determination of this quality criterion of fat exist at the international level:

- **ISO/TS 22113 | IDF/RM 204** for milk, cream and dried milk
- **ISO 1740 | IDF 6** for butter.

The analytical principle is the same for the both methods, that is an extraction of the fat content and an acidity titration of this extracted fat using Tetra n'Butyl Ammonium Hydroxyde in presence of thymol blue as coloured indicator.

In addition to the standardised requirements of the standardised methods, a significant attention must be paid to the following technical points in the implementation of the test:

- **Extraction (or separation for butter)**

  - *For milk, cream and dried milk:*

- The nature (principally type of phosphate) and the pH of the extraction solution (6.6) have a very significant impact on the final result. Because of its unavailability, the product initially expected, namely the sodium tetraphosphate, will be replace by the sodium hexaphosphate in the revision in process of the next standard.
- The « effective » passage to the water bath at 95 °C for a minimum of 15 minutes, because an insufficient passage impacts also the final results.
- The optional use of the centrifugation to complete the separation of the fat content enables the guarantee of a clear separation and the absence of non fat content in the fat phase during the titration, which could distort the results (input of acidity).

  - *For butter*

This operation is not really an extraction but rather a separation. The critical point is to have a « pure » fat content without inclusion of non-fat following a filtration problem.

- **Titration**

For this step, it is necessary to consider the following points:

- First, the titre of the titrant used for the final calculations. The Tetra n'Butyl Ammonium Hydroxyde (TBAH) solution has the tendency to tap $C0_2$ (atmospheric) and to see its titre progressed over time (even if this evolution is less consequent than the other alkaline titrants as soda or alcoholic postash, it is even so significant concerning the results) So, first it should be preserved this titrant of a such evolution (using a soda lime $C0_2$ absorber, for example) and tested a standard reference fat before each analytical set or realised a titration of this titrant in exchange for a standard solution of potassium hydrogen phthalate (which can be realised by the laboratory using pure dried product).
- The realisation of the titration under nitrogen is absolutely necessary so that not to have interferences with the ambient CO_2, which will acidify the contents of the titration vessel and then have the tendency to overestimate the volume of the titrant and the final result.
- The detection reproducibility of the final point. Even if the curve with the Bromo Thymol blue is very visible, this step is crucial to obtain reproducible results. The use of a « final point standard » during an analytical set is not possible for this type of method because of the ambient CO_2 interference, which will generate an evolution of the titration vessel colour. We can also note that automated systems of detection of the final point using an adjusted optical tube with a wavelength between 600 and 620 nm (as recommended in ISO 22113 standard) are totally adapted to ensure the quality and reproducibility of this step.

These technical elements are important control points for this type of methods and can significantly impact the accuracy of the determinations. They should be integrated in the consideration and the implementation of the method.