THE PREPARATION OF DRIED MILK AND DRIED DAIRY PRODUCTS FOR ANALYSIS OF THEIR COMPOSITION PRINCIPLES AND CRITICAL POINTS

Dried milk and dried dairy products are subject to composition criteria, in particular, moisture and fat contents.

To ensure compliance with these criteria and also monitor the production operations, these products are regularly analysed using chemical or instrumental analytical methods.

For these methods, a preparation of the sample is required. This step is generally described in the standardised analytical methods (AFNOR and/or ISO in particular):

- **ISO 5537: Dried milk – Determination of moisture content**
- **ISO 5543: Caseins and caseinates - Determination of fat content**
- **ISO 21543: Milk products – Guidelines for the application of near infrared spectrometry**

As with all the other dairy products, the steps of sampling and samples preparation of the dried products are essential to achieving results, accurate and representative of the initial sample.
The sub-sampling step is often not necessary for these types of products, because it is not necessary to carry out a mass reduction of the samples received in this context (except for caseins and caseinates in some cases)

You will find below a description of these different steps, their objectives, the operating conditions to set up and critical points to control.

Sampling

As with the most analytical standards, the sampling part is not included in the documents concerning the composition determination of these products. In this case, reference may be made to the ISO 707 | IDF 50 standard which specifies the sampling methods to be applied in a production area.

Once the sample has been taken, it should be packed in an airtight and waterproof container and then stored at room temperature. As these types of products are very hygroscopic, special attention should be paid to the watertight of the container to ensure the stability of the product during storage before analysis: presence of cap with airtight seal in the case of vials, nature of the material and characteristic of its water permeability in the case of bags, etc.

Moreover, the choice of the container (type and volume, and time of storage before analysis) should be carried out taking into account many elements of the preparation step.

The main critical points of this step are:

- **The representativeness of the sample taken in relation to the product to be characterised**
- **Compliance with the choice conditions of the container adapted to the use.**

Preparation

The preparation recommended in the standards seems to be simple (except for caseins and caseinates which will be developed at the end of this chapter), but this step can nevertheless have a significant influence on the later analytical determinations.

Its general principle is to mix the sample by shaking and rotating in a container **with a capacity of about twice the volume of the sample** in order to be able to carry out test samples representative of the sample received.
In fact, during storage, a sedimentation of the grains according to their size and a gas exchange between the external layer of the product and the air in the headspace can be observed, leading to a potential non-homogeneity in the sample received.

From a practical point of view, the initial volume of the container vs the quantity of the sample will therefore determine whether the laboratory should transfer the sample to a container with a larger volume or if shaking is possible in the initial container. In the initial reflection on this point, it will also be necessary to take into account that any transfer involves a risk of moisture absorption.
The choice should therefore be considered beforehand by integrating the analytical methods (criteria, time before analysis, etc.) and the objectives of the test.

A specific method for caseins and caseinates

For caseins and caseinates, after shaking such as that described above, a grinding may be necessary if the particle size of the product is greater than 500 µm (using a specific sieve). This point is specified in the analytical methods and is very important for the accuracy of the results obtained.

Test sample

Once the sample is homogeneous, the laboratory should carry out its test samples for all the determinations in a short time interval so as not to expose the product to ambient air and thus see an evolution of its moisture content.
In the course of these operations, the laboratory has to ensure that the weighing is carried out as quickly as possible and also that the container is closed between each test sample.

If the laboratory wishes to keep the sample for a duplicate analysis, for example, the storage methods must be defined and validated to guarantee that the product does not change over time.

The main critical points of this step are:

- **The choice of the container according to the analysis methods and the objectives**
- **Compliance with the shaking methods as described above, which could cause, if they were not applied, a non-representative and non-homogenous test sample**
- **The shortest possible exposure to the air of the product to be analysed**
- **A quick realisation of the sample tests.**

Conclusion

As you understand, compliance with good practices during these steps is the only solution to ensure the quality of the determinations that are carried out in the laboratories. Indeed, only this respect will ensure the representativeness of the initial sample throughout the analytical process and also the quality of the associated analytical determinations.

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