

N/Protein Determination in Milk Powder according to the Dumas combustion method

Reference: **UNI EN ISO 14891**, **FIL-IDF 185**, 2002 "Milk and milk products - Determination of nitrogen content - Routine method using combustion according to the Dumas principle"

Tested with **VELP Scientifica NDA 701 Dumas Nitrogen Analyzer** (Code F30800070)



Introduction

Powdered milk or dried milk is a manufactured dairy product made by removing almost all the water. This product has a far longer shelf life than liquid milk and does not need to be refrigerated, due to its low moisture content.

It can be reconstituted by adding water in order to obtain a ready-to-use liquid milk: on the water addition depends the fat content, so its high nutritional quality, combined with its versatile and multi-functional properties, makes it attractive for many food applications.

Dried milk is widely used as food ingredient because it is suitable for several applications: in confectionery, such as chocolate and caramel candy, manufacture of infant formula and recipes for bakery.

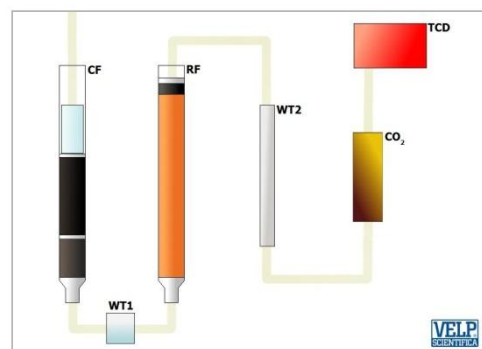
Protein Determination in Milk powder

The Dumas method starts with a combustion furnace (CF) to burn the sample, obtaining elemental compounds.

Water is removed by a first physical trap (WT1 - **DriStep™**), placed after the combustion, and a second chemical one (WT2). Between the two, the elemental substances passed through a reduction furnace (RF).

The auto-regenerative CO₂ adsorbers (CO₂) let pass only the elemental nitrogen that is detected by the **LoGas™** innovative Thermal Conductivity Detector (TCD) with no requirement for a reference gas.

The NDA 701 is controlled via PC through the intuitive **DUMASoft™**.



NDA 701 Preliminary Operations (daily)

Follow the operating manual to start the NDA 701 and check that the following parameters are set:

Temperature Combustion reactor (Code A00000158): 1030 °C

Temperature Reduction reactor (Code A00000226): 650 °C

Flow rate MFC1 He: 190 ml/min

Flow rate MFC2 He: 200 ml/min

Condition the system by testing 2 EDTA standard (Code A00000149) and 3 to 5 empty tin foils (Code A00000153) as Check up.

Verify the calibration curve with one or more tests as Standard by testing the same standard used for the curve creation.

Sample Preparation

Milk powder sample doesn't need to be homogenized.

Using a spatula, put the sample directly into the tin foil.

Close the tin foil, obtaining a capsule.

Load the capsule into the autosampler.

Analysis Procedure


Fill the following fields in the database: **Sample name, Weight, Method, Sample type, Calibration number**

The dedicated method "MILK POWDER" shows the following parameters:

Protein factor: 6.38

O₂ flow rate: 400 ml/min

O₂ factor: 1.6 ml/mg

Press  to start the analysis.

Analysis time: from 3 minutes for one run.

Typical Results on Milk powder

Sample quantity (mg)	Nitrogen %	Protein %
46.80	2.380	15.185
51.90	2.353	15.010
49.70	2.336	14.905
52.60	2.335	14.894
49.30	2.335	14.895
54.50	2.357	15.039
53.40	2.363	15.075
48.80	2.339	14.923
46.20	2.378	15.170
50.70	2.369	15.115
Average ± SD%	2.355 ± 0.018	15.021 ± 0.114
RSD% *	0.754	0.757

Protein Expected Value: 15 %

Protein Factor: 6.38

* RSD% = (Standard Deviation * 100) / Average

Conclusion

The obtained results are reliable and in accordance with the expected value.

Results have been obtained with the following calibration curve: in a range of 0 - 5 mg N with 5 measurements of EDTA standard (%N = 9.57) (Code A00000149).

Benefits of Dumas combustion method are:

- High productivity. non-stop performance
- Time saving. few minutes required
- Moderate running costs
- Totally unsupervised. fully automated
- Omission of harsh and toxic chemicals
- Eco-friendly. low amount of residues and wastes

Several organizations working with standardization and recommendation of chemical methods have approved combustion methods for the determination of nitrogen.

Thanks to development in sophisticated instrumentation, the Dumas principle became a practical alternative for the determination of nitrogen in milk products.