APPLICATION NOTE

F&F-D-002-2012/A3

N/Protein Determination in Starch

according to the Dumas combustion method

Reference: **AOAC 992.23** Crude Protein in Cereal Grains and Oilseeds

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

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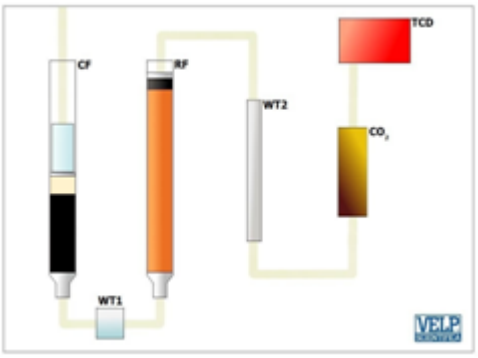
N/PROTEIN DETERMINATION IN STARCH
DUMAS COMBUSTION METHOD

Introduction

Starch is the most common carbohydrate in the human diet and is contained in large amounts in such staple foods as potatoes, wheat, maize (corn), rice, and cassava. Pure starch is a white, tasteless and odorless powder that is insoluble in cold water or alcohol. Starch has a lot of application in food industry as food additive, modified starches and sugars. Its protein content is an important parameter for the starch refining process, but also from a healthy point of view for people who has allergies and intolerance.

Protein Determination in Starch



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 CO_2 adsorbers (CO_2) 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: 220 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

Grind the test samples using a grinder to suitable fineness (particle size ≤ 0.5 mm) to obtain ≤ 2,0 % relative standard deviation (RSD) for 10 successive nitrogen determinations.
Weigh around 290 mg of starch with an accuracy of 0,01 mg directly in the tin foil placed on the plate of the balance.
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 STARCH method shows the following parameters:
Protein factor: 6.25
O_2 flow rate: 400 ml/min
O_2 factor: 1.6 ml/mg
Press to start the analysis.

Analysis time: from 3 minutes for one run.

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Typical Results on Starch

Sample quantity (mg)	Nitrogen %	Protein %
296.90	0.038	0.238
297.30	0.038	0.238
288.10	0.037	0.232
298.70	0.037	0.233
293.10	0.036	0.227
290.20	0.037	0.231
290.50	0.038	0.238
292.60	0.037	0.230
290.80	0.038	0.237
293.00	0.038	0.240
296.90	0.038	0.238
Average ± SD%	**0.037 ± 0.001**	**0.234 ± 0.004**
RSD% *	**1.870**	**1.820**
Protein Labeled Value: 0.01 - 0.1%		
Protein Factor: 6.25		

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

Conclusion

The obtained results are reliable and in accordance with the labeled value.
The combustion method, relying on the Dumas principle, for the determination of total nitrogen in starch , has been included as an official alternative to the Kjeldahl method.

Results have been obtained with the following calibration curve: in a range of 0,0097 – 0,2765 mg N with 5 measurements (from 5,2 to 148,2 mg) of Glycine standard solution 1% (1 g of Glycine in 100 ml of distilled water, so nitrogen concentration is 0.1866%).

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 and popular alternative for the determination of nitrogen in organic matrices.

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