APPLICATION NOTE

F&F-D-005-2014/A1

N/Protein Determination in Pasta

according to the Dumas method (He/Ar as Carrier)

Reference: **AOAC 992.23** Crude Protein in Cereal Grains and Oilseeds; **AACC 46-30** Crude Protein Combustion Method; **ICC 167** Determination of crude protein in grain and grain products for food and feed by the Dumas Combustion Principle

Tested with **VELP Scientifica NDA 702 Dual Carrier Gas Dumas Nitrogen Analyzer** (Code F30800080)

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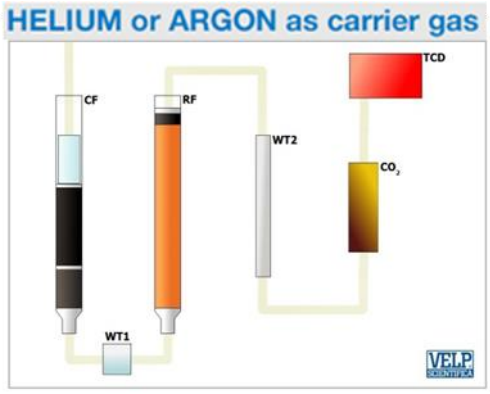
N/PROTEIN DETERMINATION IN PASTA
DUMAS COMBUSTION METHOD

Introduction

Pasta is a staple food of traditional Italian cuisine, with the first reference dating to 1154 in Sicily. Typically it is made from an unleavened dough of a durum wheat flour mixed with water and formed into sheets or various shapes, then cooked and served in any number of dishes. Pastas may be divided into two broad categories, dried (pasta secca) and fresh (pasta fresca).

The amount of protein in pasta depends on the type of flour used to manufacture it. If it is made from durum wheat, the pasta contains protein and gluten. Pasta is considered to be a good source of nutrition for vegetarians because it contains protein comprising six of the nine essential amino acids.

Protein Determination in Pasta



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 702 is controlled through the intuitive **DUMASoft™**.

NDA 702 Preliminary Operations (daily)

Follow the operating manual to start the NDA 702 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 Carrier gas (He/Ar): 190 ml/min
Flow rate MFC2 Carrier gas (He/Ar): 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

Collect 100 g of pasta for soup from durum wheat flour, into a beaker and mix it with a spoon by hand.
Grind finely it using a grinder to suitable fineness to obtain $\leq$ 2,0 % relative standard deviation (RSD) for 10 successive nitrogen determinations as indicated by the official methods.
Using a spatula, put ~ 100 mg of sample directly into the tin foil.
Close the tin foil, obtaining a capsule and load the capsule into the autosampler.

Analysis Procedure

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

The obtained results are in accordance with the expected value. Results have been obtained with the following calibration curve: in a range of 0 - 5.9 mg N with 5 measurements of EDTA standard (N% = 9.57) (Code A00000149).
The data obtained are included in the tolerance admitted by the EDTA certificate.

HELIUM as Carrier Gas		ARGON as Carrier Gas	
Sample quantity (mg)	**Protein %**	**Sample quantity (mg)**	**Protein %**
102.02	12.719	100.03	12.956
103.34	12.663	102.99	13.088
102.12	12.713	102.64	13.094
106.04	12.825	105.06	13.019
103.87	12.731	104.98	12.881
102.41	12.713	103.22	12.856
102.74	12.750	103.17	13.113
105.90	12.700	102.95	12.913
104.42	12.694	103.24	12.806
100.61	12.688	103.76	12.881
Average ± SD	**12.719 ± 0.044**		**12.961 ± 0.111**
RSD% *	**0.348**		**0.853**
Protein Expected Value: 12.8 %			
Protein Factor: 6.25			

***** RSD% = (Standard Deviation * 100) / Average
** Generally 6.25 is the protein factor for nutritional table of pasta, but also 5.70 can be used.

Conclusion

Results are extremely reliable and reproducible, as demonstrated by the RSD, by using helium or argon as carrier gas, with the same conditions (method and sample weight) since the goal is to obtain < 2.0% relative standard deviation, as requested by official methods.
Helium remains the best choice for premium accuracy but its shortages and interruptions are affecting any related product or instrument, including elemental analyzers. Argon, the best alternative available, has demonstrated to be a valid substitute, ensuring optimal results. VELP Scientifica NDA 702 Dual Carrier Gas Dumas Nitrogen Analyzer is the perfect response to simple, fast and precise nitrogen/protein determination, both with Helium and Argon as carrier gas.

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