APPLICATION NOTE

F&F-D-007-2013/A3

# N/Protein Determination in Cereals

## 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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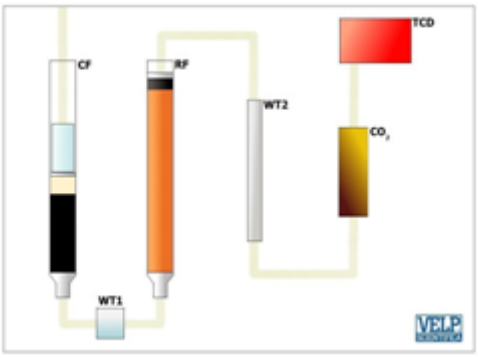
# N/PROTEIN DETERMINATION IN CEREALS
# DUMAS COMBUSTION METHOD

## Introduction

Cereal products are one of the most important staple foods of people and have been so for thousands of years.
About two billion tones of cereals are produced in the world annually. The cereal and grains products that most of us eat everyday will look very different to the grain grown in the field or paddy. Many different processing steps are taken in order to turn the coarse grain into the products on our supermarket shelves.
Cereals contain between 8-15% of different kinds of proteins such as albumins, globulins, prolamines, gliadins, glutelins and glutenins, but also provide carbohydrates, dietary fiber and vitamins.
Protein content in malted barley is an important criteria in evaluating the quality of beer: water-soluble barley proteins play a major role in the formation, stability, and texture of head foams.
Cereal production is not only destined for human consumption but also for animals.

## Protein Determination in Cereals




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 $CO_2$ absorbers ($CO_2$) 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 200 mg of the already milled cereal in a tin foil directly on 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 “CEREAL” 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 Cereals

| Sample | Sample quantity (mg) | Nitrogen % | Protein % |
|---|---|---|---|
| **Sorghum** | 197.40 | 1.904 | 11.903 |
| | 200.60 | 1.906 | 11.911 |
| | 199.70 | 1.920 | 12.001 |
| | 201.60 | 1.886 | 11.790 |
| | 200.40 | 1.895 | 11.841 |
| | **Average ± SD%** | **1.902 ± 0.013** | **11.889 ± 0.08** |
| | **RSD% *** | **0.670** | **0.669** |
| **Corn** | 201.30 | 1.311 | 8.191 |
| | 198.40 | 1.305 | 8.159 |
| | 197.80 | 1.307 | 8.166 |
| | 198.10 | 1.316 | 8.225 |
| | 198.00 | 1.286 | 8.037 |
| | **Average ± SD%** | **1.305 ± 0.011** | **8.156 ± 0.071** |
| | **RSD% *** | **0.875** | **0.873** |
| **Malt** | 194.20 | 1.663 | 10.394 |
| | 198.70 | 1.695 | 10.593 |
| | 199.30 | 1.663 | 10.393 |
| | 196.10 | 1.674 | 10.460 |
| | 196.50 | 1.638 | 10.240 |
| | **Average ± SD%** | **1.667± 0.021** | **10.416± 0.218** |
| | **RSD% *** | **1.239** | **1.226** |
| Expected Protein Value: for Sorghum P%= 11.9, for Corn P%= 8.0, for Malt P%=10.0 | | | |
| Protein Factor: 6.25 | | | |

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

## Conclusion

Despite of the low homogeneity of the cereal samples, the results are repeatable. The combustion method, relying on the Dumas principle, for the determination of total nitrogen in cereal samples, 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 - 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.
Of those related to brewing, the American Society of Brewing Chemists (ASBC) has approved combustion methods for nitrogen determination in brewing grains as well as in wort and beer.


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