thermo scientific

APPLICATION NOTE 42157

Nitrogen and Protein determination of cereals and beans with the Flash*Smart* Elemental Analyzer

Authors

Dr. Liliana Krotz, and
Dr. Guido Giazzi,
Thermo Fisher Scientific,
Milan, Italy

Keywords

AACC, AOAC, Beans, Cereals, Food Quality, Labeling, Protein, Soya

Goal

To demonstrate the performance of the Thermo Scientific Flash*Smart* Elemental Analyzer for food quality and labeling purposes for cereal and bean samples, while showing compliance to international standards requirements.

Introduction

Cereals and beans are some of the most widely grown crops globally as they are the main component of the human diet and the principal part of feeding stock for domestic animals.

One of the most important nutrients is protein and the monitoring of the amount of nitrogen must be accurate to determine the nutritional quality of these products. In addition to its dietary importance, protein content also has become a quality guideline for some cereals trade transactions.

The globalization of the food market requires accurate and reliable control of products characteristic for the protection of commercial value, but mainly to safeguard consumer health and manufacturer reputation. Official regulations establish the protein content and labeling requirements, which enable consumers to define price and quality comparisons based on % protein declarations. For this reason, the use of a simple and automated technique allowing fast analysis with excellent reproducibility, and that can avoid the risk of handling toxic chemicals is required. An alternative to the classical Kjeldahl method, based on Dumas (combustion) method, has been developed and approved by industry associations (AOAC, AACC, AOCS, ASBC, ISO, IFFO, IDF and others).

ThermoFisher SCIENTIFIC

The Thermo Scientific™ Flash*Smart*™ Elemental Analyzer (Figure 1), based on the dynamic combustion of the material (Dumas method), requires no sample digestion or toxic chemicals, while providing important advantages in terms of time, automation and quantitative determination of nitrogen in a large range of concentration.

Figure 1. Thermo Scientific Flash*Smart* Elemental Analyzer.

Method

The Flash*Smart* Elemental Analyzer is based on the dynamic flash combustion technique (modified Dumas method). The sample is weighed in tin containers and introduced into the combustion reactor from the Thermo Scientific™ MAS Plus Autosampler with oxygen determined by the Thermo Scientific OxyTune® function.

After combustion of the sample, the produced gases are carried by a helium flow to a second reactor filled with copper, then through CO_2 and H_2O traps, a GC column. Finally they are detected by a Thermal Conductivity Detector (TCD). A complete report is generated by the Thermo Scientific™ Eager*Smart*™ Data Handling Software.

Analytical Conditions	
Combustion Temperature	950 °C
Reduction Temperature	840 °C
Oven Temperature	50 °C
Helium Flow Rate	
Measurement	140 ml/min
Reference	100 ml/min
Oxygen Flow Rate	300 ml/min
Total Run Time	less than 5 minutes
Nominal Sample Weight	200-300 mg
Standard	50-100 mg Aspartic acid
Calibration Method	K factor

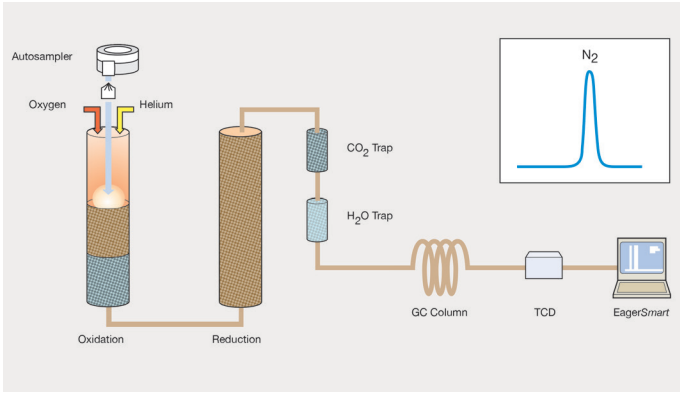

Figure 2. Flash*Smart* Elemental Analyzer nitrogen configuration.

Results

The samples were homogenized with a rotor speed mill (particle size 1 mm) and dried at 130 °C for 1 hour. The calibration was performed with aspartic acid using the K factor as calibration method. The protein content is calculated using the protein factor 6.25.

The reproducibility obtained analyzing a soya sample is reported in Table 1. No significant differences in nitrogen and protein values were observed when changing the weight of sample from 100 to 300 mg.

Table 1. Nitrogen/protein reproducibility of soya sample.

Weight (mg)	N%	Protein %
103.4	7.89	49.34
205.9	7.96	49.74
296.6	7.92	49.49
217.4	7.95	49.67
189.8	7.92	49.50
200.2	7.85	49.06
232.4	7.88	49.28
230.2	7.95	49.70
188.5	7.85	49.06
211.8	7.88	49.26
178.9	7.86	49.13
216.0	7.94	49.61
188.0	7.93	49.55
185.1	7.86	49.12
242.5	7.84	49.00
186.7	7.87	19.19
202.9	7.89	49.33
218.0	7.99	49.97
198.7	7.97	49.82
204.0	7.86	49.11
220.1	7.98	49.85
206.2	7.85	49.05

Figure 3 shows the statistical plot of the data. The excellent fluctuation obtained demonstrates the stability of the system and the reproducibility of the results.

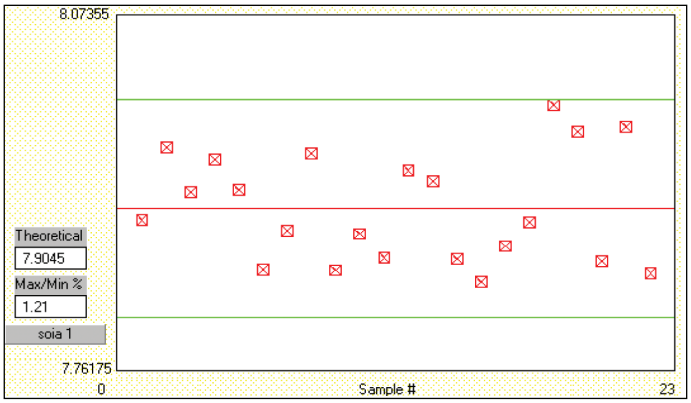

Figure 3. Statistical plot of nitrogen data.

Statistical Data	
Number of runs	22
Nitrogen	
Average %	7.90
Std. Dev	0.048
RSD%	0.610
Protein	
Average %	49.40
Std. Dev	0.301
RSD%	0.610

Table 2 shows the nitrogen and protein determination in various cereal and bean samples to validate the system at different content of nitrogen and protein. The data shows excellent reproducibility.

In all cases the relative standard deviation was less than 2%, according to the AOAC method 990.03 for animal feed.

No memory effect was observed when changing the sample, meaning the complete detection of the nitrogen in the sample.

Table 2. Nitrogen/protein determination in cereals and beans.

Sample	W (mg)	N%	Prot. %	RSD%
Corn	276.7 240.8 247.5 272.2 285.7 272.9 247.5 256.8 241.3 259.2	1.38 1.40 1.39 1.43 1.38 1.37 1.37 1.37 1.37 1.38	8.62 8.77 8.71 8.93 8.61 8.57 8.57 8.58 8.58 8.60	8.60
Wheat HRS	259.2 255.7 258.2	2.99 2.99 3.01	18.70 18.68 18.82	0.40
Wheat CPS-W	213.9 250.9 250.2	2.24 2.26 2.25	14.00 14.12 14.09	0.44
Wheat SWS	254.4 233.4 230.9	2.28 2.29 2.30	14.23 14.28 14.37	0.49 \
Lentils	296.0 300.7 307.5 297.4 310.7 309.1 310.5 265.1 270.0 327.9	3.99 4.00 3.98 3.96 4.01 3.97 3.99 3.98 3.97 4.01	24.96 25.01 24.89 24.75 25.08 24.82 24.94 24.90 24.84 25.08	0.43
Green Peas	297.0 301.0 323.0	3.92 3.88 3.91	24.49 24.25 24.45	0.53
Brown Peas	320.0 312.4 284.9 315.0 290.5	4.48 4.45 4.48 4.46 4.44	28.00 27.81 28.03 27.89 27.73	0.45

A comparison of results of different cereals obtained by the Flash*Smart* Elemental Analyzer and the Kjeldahl method is reported in Table 3.

The data shows that the two methods are perfectly comparable, demonstrating the validity of the combustion method for N/Protein analysis.

Table 3. Flash*Smart* Elemental Analyzer versus Kjeldahl method.

Sample	Kjeldahl Method Protein %	Flash*Smart* Elemental Analyzer Protein %
Soya	39.18	39.20
Lentils	27.19	27.17
Rice	7.00	7.08
Wheat	10.89	10.91
Beans	23.38	23.35

Conclusions

The Thermo Scientific Flash*Smart* EA, based on the combustion method, demonstrates to be an excellent solution for nitrogen/protein determination of samples with high protein content due to the superior repeatability obtained, with no memory effect observed when changing the type of sample. This indicates the complete and accurate detection of the nitrogen content.

The Dumas Combustion method has been approved and adopted by Official Organizations as ASBC, AOAC, AACC, AOCS, ISO, IFFO and IDF.

Find out more at **thermofisher.com/OEA**

ThermoFisher SCIENTIFIC