APPLICATION NOTE

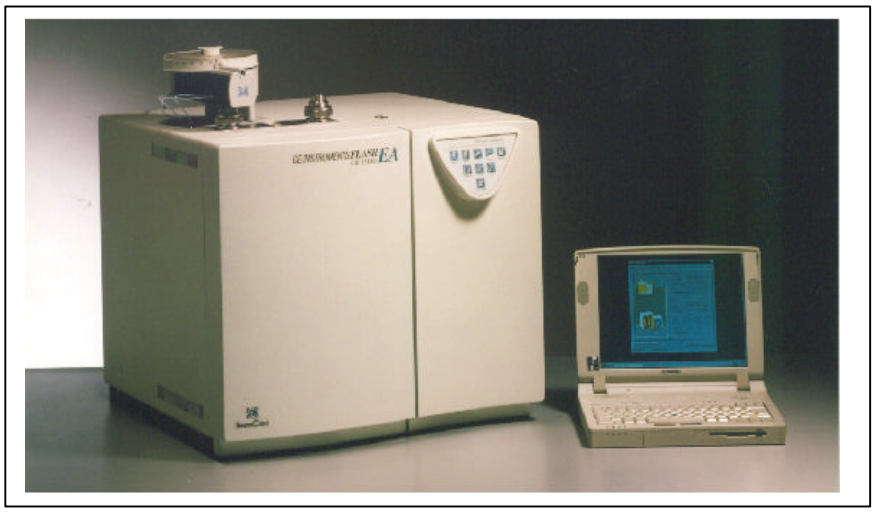
ThermoQuest

GC and GC/MS Division
Milan, Italy

AN 632A
Flash 2000 Protein Analyzer for Cereals and Beans

Liliana Krotz, Luigi Ragaglia and Guido Giazzi

- Approved by AOAC, method 990.03
- Adopted by AACC, method 46-30
- Large sample weight
- Fast analysis: less than 5 min



INTRODUCTION

Cereals and beans have engaged most of the crops grown on all cultivated land in every country because they have been the main component of the man's diet and the principal part of feeding stock for domestic animals.

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

For this reason, modern advances in instrumentation have greatly improved the capabilities of the combustion method making it faster, safer and more reliable than the traditional Kjeldahl method.

As a direct consequence of these advantages the combustion method was approved and adopted by: Association of official Analytical Chemists (AOAC method 990.03) and American Association of Cereal Chemists (AACC method 46-30).

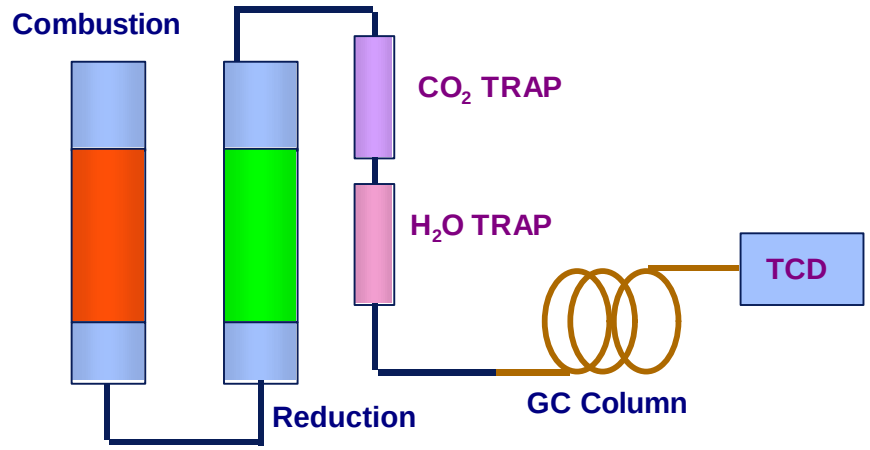
The Flash 2000 Protein Analyzer cope with a wide array of additional important requirements of modern laboratories such as accuracy, high sample throughput and low cost per analysis.

DESCRIPTION OF THE ANALYTICAL METHOD

The Analyzer is based on the dynamic flash combustion of the sample. The sample is weighed in a tin capsule and introduced into the combustion reactor via the MAS 200R Autosampler together with a proper amount of oxygen determined by OxyTune™ function.

After combustion, the produced gases are carried by a helium flow to a second reactor filled with copper, then swept through CO_2 and H_2O traps, a GC column and finally detected by a Thermal Conductivity Detector. A complete report is automatically generated by the Eager 300 dedicated software.

Analytical Layout of Flash 2000 Protein Analyzer



ANALYTICAL CONDITIONS

Combustion temperature: 900°C
Reduction temperature: 680°C
Oven temperature: 50 °C
Helium pressure: 250kPa
Helim flow rate:
Measurement: 140 ml/min
Reference: 100 ml/min
Oxygen flow rate: 300 ml/min (pressure 300 kPa)
Total run time: less than 5 minutes
Nominal sample weight: 200-300 mg
Standard: 50-100 mg Aspartic acid
Calibration Method: K factor

RESULTS

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

The reproducibility obtained analyzing a soia sample is reported in Table 1.

Figure 1 shows the statistical plot of these data. The excellent fluctuation obtained demonstrates the stability of the system.

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

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

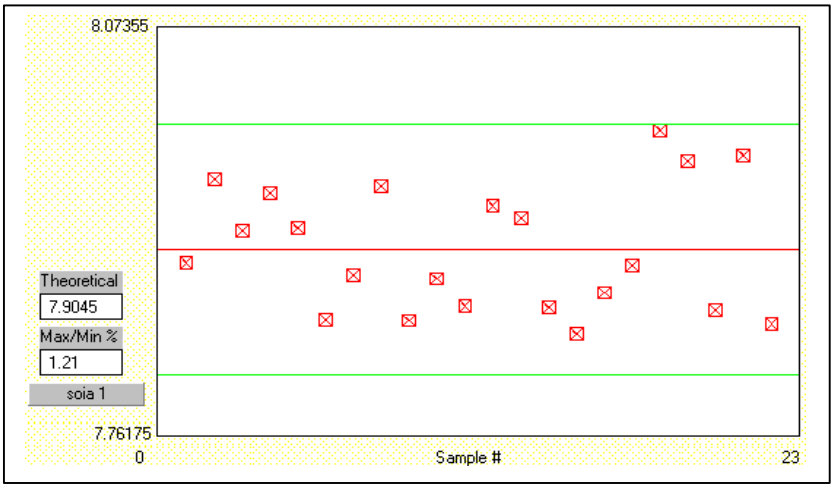


Table 2 shows the Nitrogen and Protein determination in various cereal and bean samples. The data show an excellent reproducibility.

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 type of sample, indicating the complete detection of the nitrogen present in the sample.

Table 2 – Nitrogen / Protein determination in cereals and beans

Sample	W (mg)	N %	Prot. %	RSD %	Sample	W (mg)	N %	Prot. %	RSD %
Corn	276.7	1.38	8.62	1.35	Lentils	296.0	3.99	24.96	0.43
	240.8	1.40	8.77			300.7	4.00	25.01	
	247.5	1.39	8.71			307.5	3.98	24.89	
	272.2	1.43	8.93			297.4	3.96	24.75	
	285.7	1.38	8.61			310.7	4.01	25.08	
	272.9	1.37	8.57			309.1	3.97	24.82	
	247.5	1.37	8.57			310.5	3.99	24.94	
	256.8	1.37	8.58			265.1	3.98	24.90	
	241.3	1.37	8.58			270.0	3.97	24.84	
	259.2	1.38	8.60			327.9	4.01	25.08	
Wheat HRS	259.2	2.99	18.70	0.40	Green Peas	297.0	3.92	24.49	0.53
	255.7	2.99	18.68			301.0	3.88	24.25	
	258.2	3.01	18.82			323.0	3.91	24.45	
Wheat CPS-W	213.9	2.24	14.00	0.44	Brown Peas	320.0	4.48	28.00	0.45
	250.9	2.26	14.12			312.4	4.45	27.81	
	250.2	2.25	14.09			284.9	4.48	28.03	
Wheat SWS	254.4	2.28	14.23	0.49		315.0	4.46	27.89	
	233.4	2.29	14.28			290.5	4.44	27.73	
	230.9	2.30	14.37						

A comparison of results of different cereals obtained by Flash 2000 Protein Analyzer and the Kjeldahl method is reported in Table 3.

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

and that the Flash 2000 Protein Analyzer represents the best alternative to the traditional wet method.

Table 3 - FlashEA™ 1112 Protein Analyzer vs. Kjeldahl method

Sample	Kjeldahl Method Protein %	Flash 2000 Protein 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