Nitrogen/Protein Determination in Flours by the Thermo Scientific FLASH 2000 Analyzer using Argon as Carrier Gas

Application Note 42201

Dr. Liliana Krotz, OEA Product Specialist, Walter Galotta, OEA Product Support Engineer, and Dr. Guido Giazzi, OEA Product Manager, Thermo Fisher Scientific, Milan, Italy

Key Words

Argon, combustion, flour, nitrogen, protein

Goal

This application note presents data on nitrogen/protein determination in flour reference materials with different nitrogen concentrations to demonstrate the performance of the system using argon as an alternative carrier gas and show the reproducibility of the results obtained.

Introduction

Flour is a fine powder made by grinding cereal grains or other suitable edible plant matter which contains starch as polysaccharides and protein. It is most commonly made from wheat but also rye, barley, rice and corn (similar to "polenta"). Ground legumes and nuts, such as soy, peanuts, almonds, and other tree nuts are also called flours. It is the key ingredient of bread; a staple food in many countries, and its availability has often been a major economic and political issue.

Regulations in many countries require specific control of flour quality. One of the most important parameters is protein content, which can be monitored through the precise and accurate determination of the levels of nitrogen in the product to determine nutritional quality.

Figure 1. FLASH 2000 Elemental Analyzer

Helium supply has been beset with problems over the last few years, with world-wide shortages and subsequent price increases. It has therefore been necessary to test an alternative gas, argon, which is readily available.

The Thermo Scientific™ FLASH 2000 analyzer (Figure 1) copes effortlessly with a wide array of laboratory requirements such as accuracy, day by day reproducibility and high sample throughput. It operates according to the dynamic flash combustion of the sample and typically uses helium as the carrier gas.

Thermo
SCIENTIFIC

Methods

Samples are weighed in tin capsules and introduced into the combustion reactor via the Thermo Scientific™ MAS™ 200R autosampler together with the proper amount ofoxygen. After combustion, the resultant gases are carried by an argon flow to a second reactor filled with copper, then swept through CO_2 and H_2O traps, a GC column and are finally detected by a thermal conductivity detector (TCD). The analytical configuration as well as the TCD detector are as the same as those used with helium as the carrier gas (see Figure 2).

A complete report is automatically generated by the Thermo Scientific™ Eager Xperience dedicated data handling software and displayed at the end of the analysis. The Eager Xperience software provides a new option AGO (Argon Gas Option) which modifies the argon carrier flow during the run to optimize the analysis.

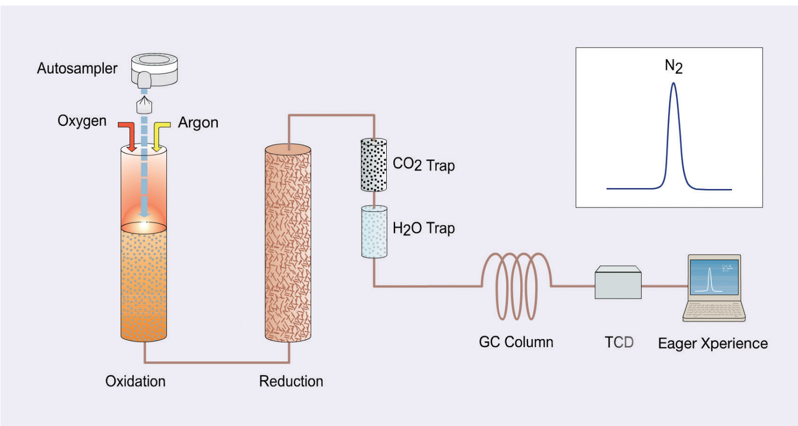

Figure 2 – FLASH 2000 Nitrogen configuration

Analytical conditions	
Combustion Furnace Temperature:	950 °C
Reduction Furnace Temperature:	840 °C
Oven Temperature:	50 °C (GC column inside the oven)
Argon Carrier Flow:	60 ml/min
Argon reference Flow:	60 ml/min
Oxygen Flow:	300 ml/min
Oxygen Injection End:	30 sec for standard, Category B (OxyTune) for flours and 30 sec for soy bean meal
Sample Delay:	10 sec
Run time:	10 mins

Official methods requirements for Protein determination by combustion

AOAC (Method 992.23) and AACC (Method 46-30, 1999) indicate that a suitable fineness of grind must be determined (for each different material analyzed) to achieve precision that gives RSD of ≤ 2% for 10 successive determinations of nitrogen.

Results

To evaluate the performance of the system for flour analysis, samples with different nitrogen content were chosen. Instrument calibration was performed with approximtely 50 - 70 mg of Aspartic acid standard (10.52 N %), using K factor as calibration method. To evaluate the calibration, Thermo Scientific™ Pasta Reference Material was analyzed. The protein factor 6.25 was used to calculate the protein content.

Table 1 shows the nitrogen/protein data obtained of the analysis of Pasta Reference Material. The sample weight was 150 – 160 mg. The certified N % is 2.227 and the uncertainty declared by the supplier is 0.097.

Table 1 – Nitrogen/Protein data of Pasta Reference Material

N %	RSD %	Protein %	RSD %
2.21		13.81	
2.23		13.94	
2.19		13.69	
2.20		13.75	
2.22	0.542	13.87	0.535
2.21		13.81	
2.20		13.75	
2.22		13.87	
2.21		13.81	
2.20		13.75	

Five Flour Reference Materials from 1.36 to 7.55 N % were chosen to correlate the experimental results to the expected values. Table 2 shows the certified N % and the uncertainty declared by the supplier.

Table 2 – Expected N % of Flour Reference Materials

Sample Description	Supplier Specification	
	N %	Uncertainty (±)
Wheat Flour Reference Material	1.36	0.25
Rice Flour Reference Material	1.38	0.05
Barley Flour Reference Material	1.9	0.04
Oatmeal Reference Material	1.9	0.10
Soy Bean Meal Reference Material	7.5	0.05

Table 3 shows the experimental N/Protein data obtained which were very satisfactory. The sample weight was 140 – 150 mg.

In all cases the RSD % was less than 2 % as requested in the Official Methods.

Table 3 – Experimental N % of Flours Reference Material

Sample	Wheat Flour		Rice Flour		Barley Flour		Oatmeal	
Determination	N %	Protein %	N %	Protein %	N %	Protein %	N %	Protein %
%	1.37	8.57	1.36	8.49	1.89	11.80	1.88	11.76
	1.36	8.51	1.37	8.59	1.91	11.94	1.88	11.73
	1.34	8.36	1.40	8.73	1.91	11.91	1.86	11.61
	1.35	8.41	1.40	8.74	1.91	11.95	1.90	11.90
	1.36	8.52	1.40	8.75	1.94	12.10	1.86	11.64
	1.36	8.49	1.38	8.65	1.91	11.91	1.85	11.56
	1.33	8.28	1.38	8.65	1.87	11.66	1.92	11.97
	1.36	8.51	1.38	8.62	1.89	11.80	1.87	11.67
	1.33	8.30	1.41	8.83	1.90	11.89	1.92	12.01
	1.36	8.52	1.39	8.69	1.92	11.97	1.89	11.79
Average %	1.35	8.45	1.39	8.67	1.90	11.89	1.88	11.76
RSD %	1.034	1.210	1.130	1.106	0.997	0.997	1.302	1.307

Table 5 – Experimental N % of Soy Bean Meal Reference Material (130 – 190 mg)

Weight (mg)	N %	Av. N %	RSD %	Protein %	Av. Protein %	RSD %
130.48 132.67 129.90	7.50 7.51 7.50	7.50	0.077	46.86 46.91 46.84	46.87	0.077
140.57 141.41 140.28	7.49 7.53 7.45	7.49	0.534	46.81 47.05 46.57	46.81	0.513
161.30 160.29 162.44	7.48 7.51 7.58	7.52	0.682	46.73 46.95 47.38	47.02	0.703
170.53 171.02 171.22	7.47 7.53 7.51	7.50	0.407	46.66 47.06 46.91	46.88	0.431
182.89 182.54 180.50 91.57	7.49 7.56 7.55 7.54	7.53	0.413	46.81 47.23 47.22 47.12	47.09	0.417

Conclusions

Good repeatability, accuracy and precision was obtained with the FLASH 2000 Nitrogen Analyzer using argon as the carrier gas. The RSD % obtained was less than 2% according to the AOAC and AACC Official Methods Performance Requirements.

No memory effect was observed when changing the type of sample, indicating complete combustion and detection of the element.

Africa +43 1 333 50 34 0
Australia +61 3 9757 4300
Austria +43 810 282 206
Belgium +32 53 73 42 41
Canada +1 800 530 8447
China 800 810 5118 (free call domestic) 400 650 5118
Denmark +45 70 23 62 60
Europe-Other +43 1 333 50 34 0
Finland +358 9 3291 0200
France +33 1 60 92 48 00
Germany +49 6103 408 1014
India +91 22 6742 9494
Italy +39 02 950 591
Japan +81 45 453 9100
Latin America +1 561 688 8700
Middle East +43 1 333 50 34 0
Netherlands +31 76 579 55 55
New Zealand +64 9 980 6700
Norway +46 8 556 468 00
Russia/CIS +43 1 333 50 34 0
Singapore +65 6289 1190
Spain +34 914 845 965
Sweden +46 8 556 468 00
Switzerland +41 61 716 77 00
UK +44 1442 233555
USA +1 800 532 4752

AN42201_E 01/14C