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APPLICATION NOTE 42478

Elemental Analysis: N/Protein by single reactor and sulfur determination of food and animal feed by combustion method

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Keywords

Animal feed, Combustion, Food, Nitrogen, Protein, Sulfur

Goal

To assess the performance of the elemental analyzer for the N/Protein determination using a single reactor in alternative to the Kjeldahl method. Report the performance also for sulfur determination.

Introduction

The nutritional composition of food plays a very important role in food industries for research and quality control purposes. New regulations regarding processed food and most raw foods include a series of tests aimed at determining food contents and their contribution to a healthy diet. One of the tests used in the production process is the determination of protein content of food and animal feed, through the determination of the nitrogen. Protein content is fundamental for the nutritional quality of animal feed and for the safety of final food products for human consumption. Official regulations establish the protein content and labeling requirements, which enable consumers to define price and quality comparisons based on % protein declarations.

Sulfur is an essential component of living matter. The lack of sulfur can affect negatively the synthetization of proteins. For cows, sulfur is a key indicator of the Cation-Anion Balance of Ration: a very important parameter enabling to measure the regeneration of the breast in later lactation. For horses, the right sulfur amount affects the beauty of the mantle.

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For these reasons, there is a need of accurate quantitation of protein and sulfur in animal feed. The use of a simple and automated technique allowing fast analysis with excellent reproducibility, avoiding the risk of handling toxic chemicals is required. Regarding protein content, the alternative to the classical Kjeldahl method, is the Dumas (combustion) method which is approved by different associations (AOAC, AACC, AOCS, ASBC, IDF, ISO and IFFO).

The Thermo Scientific™ Flash*Smart*™ Elemental Analyzer (Figure 1), based on the dynamic combustion method (modified Dumas method), provides rapid and automated nitrogen and sulfur determination without use of hazardous chemicals and offers advantages in precision over traditional methods.

Figure 1. Thermo Scientific Flash*Smart* EA.

This note presents N/Protein and sulfur data of several food and animal feed samples in a wide range of types and concentrations, measured with the Flash*Smart* EA. Results are shown to demonstrate its repeatability, accuracy and precision.

Figure 2. MultiValve Control (MVC) Module.

Methods

The standard configuration for N/Protein determination is based on a double reactors system: first reactor for combustion and catalytic oxidation of the combustion gases, the second is used to reduce nitrous oxides as N_2. The superior performance of the Flash*Smart* EA allows the reduction of the amount of oxidation catalyst needed for nitrogen analysis using a single combustion/reduction reactor (25 mm diameter). The reactor filled with less amount of oxidation catalyst and copper ensures the complete conversion of gases produced by the combustion. It also offers advantages such as the capability of higher number of analyses before the maintenance and the possibility to install two analytical circuits which are used alternatively (for example nitrogen in the left furnace and sulfur in the right furnace, or nitrogen in the left furnace and sulfur by FPD Detector in the right furnace) in one analyzer. Adding two autosamplers enables to reduce to only few seconds the switching time from one analytical circuit to the second, removing the need for tools or mechanical modification. The proprietary MVC Module also ensures very low helium consumption by switching from helium to nitrogen or argon gas, when the instrument is in Stand-By Mode. The cost of analysis is significantly reduced.

The Elemental Analyzer operates according to the dynamic flash combustion of the sample. Samples are weighed in tin containers and introduced into the combustion reactor via the Thermo Scientific MAS Plus Autosampler together with oxygen. For N/Protein determination (left reactor), after combustion, the produced gases are carried by a helium flow to a layer of copper, then swept through CO_2 and H_2O traps, a GC column and finally detected by a Thermal Conductivity Detector (TCD) (Figure 3). For sulfur determination (right reactor), after combustion the resulted gases are carried by a helium flow to a layer filled with copper, then swept through a water trap and a GC column which provides the separation of the combustion gases, and finally, detected by a Thermal Conductivity Detector (TCD). (Figure 3).

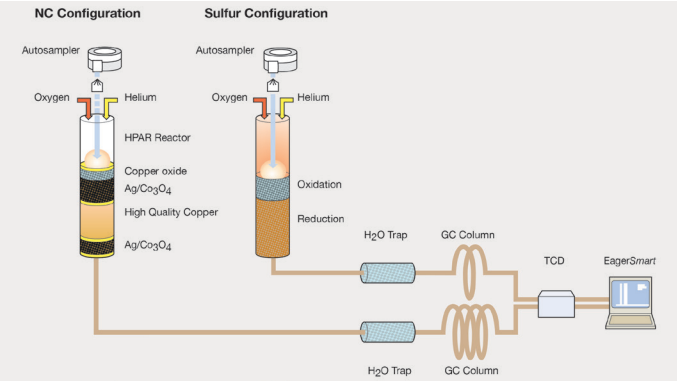

Figure 3. N-Protein / S Configuration

A complete report is automatically generated by the Thermo Scientific™ Eager*Smart*™ Data Handling Software and displayed at the end of the analysis. The dedicated software converts automatically the nitrogen % in protein content using a specific protein factor.

Both pneumatic circuits for N/Protein and sulfur determination are preset in the system in order to switch automatically from one to the other through the MVC Module controlled by the dedicated EagerSmart Data Handling Software without any additional manual intervention. The Eager*Smart* Data Handling Software page which control the MVC Module, Figure 4, shows how to switch from Left to Right furnace, passing from nitrogen determination to sulfur analysis. Indications on how to switch from helium carrier gas to nitrogen or argon gas when the instrument is in Stand-By Mode are also provided.

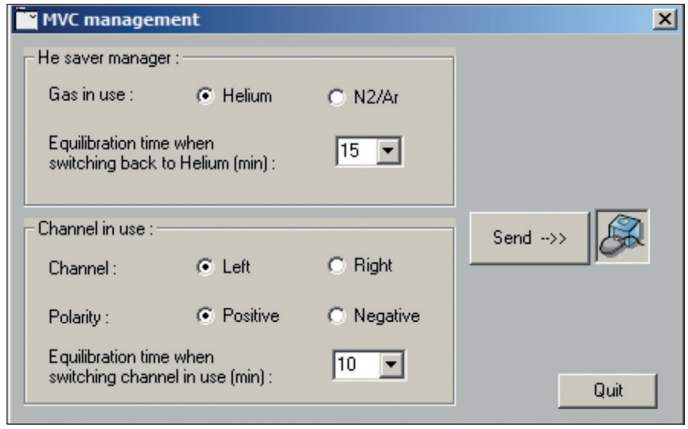

Figure 4. Eager*Smart* Data Handling Software window for the MVC Module.

Results

Thermo Scientific Pasta Reference Material (2.227 N% ±0.097, 0.135 S% ±0.004) was analyzed. For N/Protein determination, the calibration was performed with 50-70 mg of aspartic acid (10.52 N%) standard and the sample weight was 130-150 mg. The protein factor 6.25 was used to calculate the protein content. For sulfur analysis, the calibration was performed with 2-3 mg of BBOT (2,5-Bis (5-tert-butyl-benzoxazol-2-yl) thiophene) and sample was weighed at 3-4 mg. Table 1 shows the experimental data obtained.

Table 1. Repeatability of N/Protein and sulfur data of Thermo Scientific Pasta Reference Material.

N%	RSD%	Protein %	RSD%	S%	RSD%
0.21 0.20 0.20	0.26	13.84 13.77 13.74	0.37	0.135 0.137 0.135	0.85

Table 2 shows the Nitrogen/Protein and sulfur data of different food samples analyzed in triplicate including the range of weight used for analysis. The protein factor used to calculate the protein content was 6.25 for most of the samples while 6.38 for milk sample.

Table 2. Repeatability of N/Protein and Sulfur data of food simples.

Sample name	N/Protein determination					Sulfur determination		
	W (mg)	N%	RSD%	Protein %	RSD%	W (mg)	S%	RSD%
Milk (liquid)	150 – 180	0.51 0.51 0.51	0	3.20 3.20 3.19	0.18	3 – 4	0.0487 0.0471 0.0478	1.68
Biscuits	130 – 150	1.20 1.20 1.18	0.97	7.51 7.48 7.38	0.91	3 – 4	0.0687 0.0692 0.0674	1.36
Whole corn flakes	130 – 150	1.49 1.48 1.50	0.67	9.34 9.22 9.35	0.78	3 – 4	0.101 0.099 0.099	1.16
Honey cereals	130 – 140	1.27 1.26 1.25	0.79	7.97 7.85 7.80	1.11	3 – 4	0.0607 0.0597 0.0603	0.84
Corn cereals	140 – 150	0.83 0.84 0.84	0.69	5.18 5.22 5.25	0.67	3 – 4	0.0641 0.0655 0.0644	1.08
Chocolate cereals	130 – 140	1.31 1.28 1.31	1.33	8.20 7.98 8.18	1.50	3 – 4	0.0790 0.0795 0.0806	1.03
Soy bean	130 – 150	5.62 5.62 5.62	0	35.12 35.10 35.14	0.06	3 – 4	0.250 0.255 0.246	1.80
Soy meal	130 – 150	8.19 8.19 8.18	0.07	51.21 51.22 51.10	0.13	3 – 4	0.371 0.379 0.372	1.17
Maize	130 – 150	1.23 1.26 1.24	1.22	7.68 7.86 7.75	1.17	3 – 4	0.0875 0.0881 0.0883	0.47

Table 3 shows the Nitrogen/Protein and Sulfur data of two animal feed samples for horses analyzed in triplicate. The weight used for N/Protein determination was 80-100 mg and the protein factor used to calculate the protein content was 6.25. The sample was weighed for sulfur analysis at 3-4 mg.

Table 3. Repeatability of N/Protein and Sulfur data of animal feed for horses.

Sample name	N/Protein determination				Sulfur determination	
	N%	RSD%	Prot. %	RSD%	S%	RSD%
1	3.66 3.64 3.66	0.32	22.87 22.74 22.85	0.32	12.00 12.20 12.20	0.95
2	7.43 7.51 7.48	0.54	46.44 46.94 46.75	0.54	2.04 2.00 1.98	1.52

In addition, flour and orange juice samples were analyzed. The sulfur content was determined by FPD Detector. Table 4 shows the N/Protein and sulfur data obtained. The flour sample was analyzed 5 times while the orange juice was analyzed 10 times.

Table 4. Repeatability of N/Protein (TCD) and trace Sulfur (FPD) data.

Sample name	N%	RSD%	Prot. %	RSD%	S ppm	RSD%
Flour	1.73 1.72 1.73 1.71 1.72	0.49	10.81 10.75 10.81 10.69 10.75	0.49	92 93 91 92 93	0.91
Orange juice	0.0501 0.0503 0.0493 0.0495 0.0503 0.0504 0.0495 0.0516 0.0498 0.0512	1.48	0.313 0.314 0.308 0.309 0.314 0.315 0.309 0.322 0.311 0.320	1.48	32 32 29 33 30 33 30 30 29 33	5.35

Conclusions

Using one large reactor for N/Protein determination instead of the classical two reactors configuration, extended the analytical capabilities of the Thermo Scientific Flash*Smart* Analyzer. All data were obtained with an acceptable repeatability and no matrix effect was observed when changing the configuration, indicating the complete combustion of the sample.

In addition, the dual analytical configuration capability using the MVC Module allows to perform the following functions:

- Analysis of N/Protein on the left reactor and sulfur on the right reactor
- Automated control of two MAS Plus Autosamplers.
- Automated switch from the left channel to the right channel, or vice versa, increasing laboratory productivity.
- Reduced helium consumption by switching from helium to nitrogen or argon when the system is in Stand-By Mode.
- Auto-Ready: return automatically to helium carrier gas from Stand-By Mode and prepare for analysis.
- Fully control the workflow by the Eager*Smart* Data Handling Software.

Acknowledgements

We would like to thank Salma Abd El Hadi, IIS Curie-Sraffa student (Milan, Italy) for his collaboration in the analysis of the samples during his internship in Thermo Scientific OEA Demo Lab (Milan, Italy).

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AN42478-EN 0719