TECHNICAL NOTE 42504

Elemental Analysis: high productivity of Thermo Scientific Flash*Smart* N/Protein – CHNS Analyzer with the MultiValve Control (MVC) Module

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Goal

To assess the performance of the Elemental Analyzer for micro and macro analysis in terms of automation and reproducibility.

Introduction

Carbon, nitrogen, hydrogen, sulfur by combustion analysis, is commonly used for the characterization of raw and final products in pharmaceutical, universities, agronomy, petrochemical and material industries for quality control and R&D purposes. The nitrogen content is fundamental for the nutritional quality of animal feed and for the safety of final food products intended for human consumption.

The nitrogen content allows the calculation of the protein amount. Official regulations establish the protein content and labeling requirements, which enable consumers to define price and quality comparisons based on % protein declarations.

The use of an accurate and automated analytical technique allows fast analysis with an excellent reproducibility whilst meeting laboratory demands for high productivity and low cost per analysis, avoiding the risk of handling toxic chemicals. 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 element determination without use of hazardous chemicals and offers advantages in precision over traditional methods. The Flash*Smart* Elemental Analyzer is equipped with two totally independent furnaces allowing the installation of two analytical circuits which are used alternatively and completely automatic through the Thermo Scientific™ MultiValve Control (MVC) Module (Figure 2). Each analytical circuit can receive is own autosampler. In this way the system copes effortlessly with the wide array of laboratory requirements such as accuracy, day to day reproducibility and high sample throughput. The Flash*Smart* EA allows runs at both high and low levels without matrix effects. Trace sulfur content can be accurately determined by using the Analyzer coupled with a Flame Photometric Detector (FPD). Sample protein content is calculated automatically using a conversion factor in the Thermo Scientific™ Eager*Smart*™ Data Handling Software.

Figure 1. Thermo Scientific Flash*Smart* Analyzer

Figure 2. 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 Thermo Scientific Flash*Smart*™ Elemental Analyzer allows the reduction of the amount of oxidation catalyst needed for nitrogen analysis using a single combustion/reduction reactor tube (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 CHNS or only 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. This feature is completely automatic through the MultiValve Control™ (MVC) Module (Figure 2). The 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. In this way 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 MAS Plus Autosampler 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 CHNS, after combustion the resulted gases are carried by a helium flow to a layer filled with copper, then swept through a GC column that provides the separation of the combustion gases, and finally, detected by a Thermal Conductivity Detector (TCD) (Figure 3). A complete report is automatically generated by the Eager*Smart* Data Handling Software and displayed at the end of the analysis. The dedicated software automatically converts the nitrogen % in protein content using a specific protein factor.

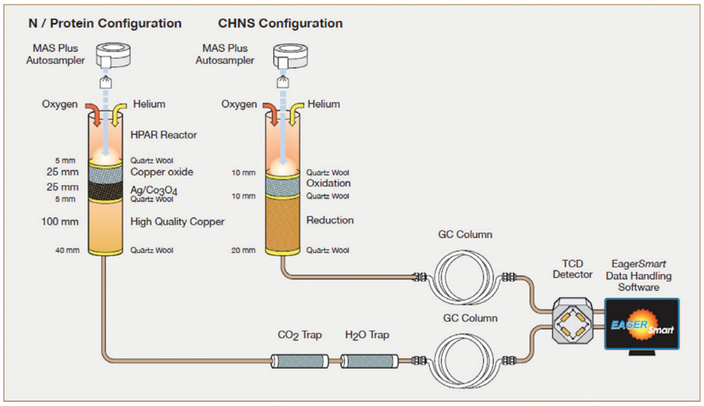

Figure 3. Flash*Smart* N/Protein – CHNS configuration

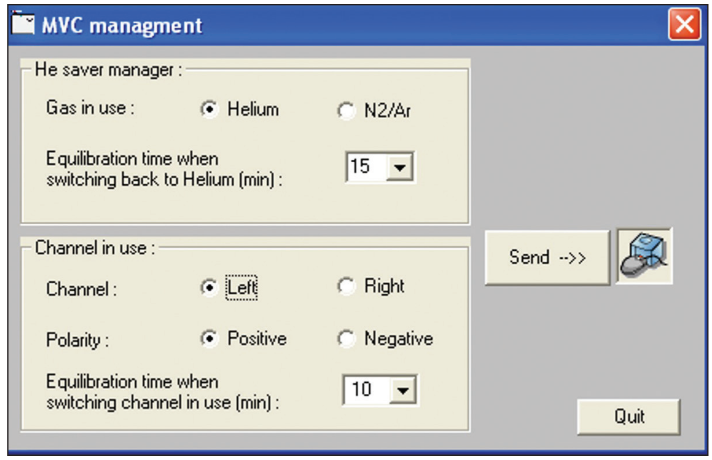

Figure 4. The MVC Management page on the Eager*Smart* Data Handling Software

Both pneumatic circuits are preset in the system in order to pass automatically from one to the other configuration through the MultiValve Control (MVC) Module controlled by the dedicated Eager*Smart* Data Handling Software without any operational action by the user.

The Eager*Smart* page which controls the MVC Module (Figure 4) shows in the lower part how to switch from Left to Right furnace to pass from N/Protein determination to CHNS analysis. In the upper section of the page, is indicated how to switch from helium carrier gas to nitrogen or argon gas when the instrument is not used for analysis.

Results

To evaluate the performance of the Flash*Smart* EA for N/Protein determination using the single reactor, Thermo Scientific Pasta Reference Material (2.227 N% ±0.097) was analyzed for 5 days. 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. Table 1 shows the repeatability obtained. The average of nitrogen content is inside the specification of the Analyzer.

Table 1. Day-by-day repeatability of N/Protein of Thermo Scientific Pasta Reference Material

Day	1		2		3		4		5	
Determination	N%	Prot. %	N%	Prot. %	N%	Prot. %	N%	Prot. %	N%	Prot. %
	2.17	13.57	2.20	13.75	2.18	13.63	2.18	13.63	2.18	13.63
	2.17	13.56	2.19	13.67	2.19	13.69	2.18	13.62	2.18	13.63
	2.17	13.56	2.21	13.79	2.18	13.63	2.19	13.68	2.17	13.56
	2.16	13.48	2.20	13.76	2.18	13.63	2.19	13.70	2.18	13.63
	2.17	13.57	2.20	13.75	2.18	13.63	2.20	13.74	2.19	13.69
	2.17	13.57	2.20	13.77	2.17	13.56	2.20	13.73	2.18	13.63
	2.18	13.61	2.20	13.72	2.17	13.56	2.19	13.70	2.18	13.63
	2.19	13.69	2.20	13.74	2.18	13.63	2.19	13.71	2.19	13.69
	2.19	13.68	2.19	13.70	2.18	13.63	2.21	13.79	2.18	13.63
	2.18	13.61	2.19	13.71	2.18	13.63	2.19	13.66	2.18	13.63
Average	2.18	13.61	2.20	13.74	2.18	13.63	2.19	13.70	2.18	13.63
RSD %	0.64	0.67	0.29	0.25	0.26	0.28	0.42	0.36	0.31	0.3

Table 2 shows the N/Protein data of different food and animal feed 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 – Part 1. Repeatability of N/Protein data of food and animal feed samples

Sample name	N/Protein determination				
	W (mg)	N%	RSD%	Prot. %	RSD%
Milk (liquid)	150–180	0.51 0.51 0.51	0	3.20 3.20 3.19	0.18
Biscuits	130–150	1.20 1.20 1.18	0.97	7.51 7.48 7.38	0.91
Whole corn flakes	130–150	1.49 1.48 1.50	0.67	9.34 9.22 9.35	0.78
Honey cereals	130–140	1.27 1.26 1.25	0.79	7.97 7.85 7.80	1.11
Corn cereals	140–150	0.83 0.84 0.84	0.69	5.18 5.22 5.25	0.67
Chocolate cereals	130–140	1.31 1.28 1.31	1.33	8.20 7.98 8.18	1.50
Soy bean	130–150	5.62 5.62 5.62	0	35.12 35.10 35.14	0.06
Soy meal	130–150	8.19 8.19 8.18	0.07	51.21 51.22 51.10	0.13
Mais	130–150	1.23 1.26 1.24	1.22	7.68 7.86 7.75	1.17
Cheese	100–140	4.94 4.96 4.97	0.31	30.88 31.91 31.09	0.34
Milk powder	80–90	1.67 1.67 1.67	0	10.41 10.44 10.41	0.17
Corn gluten	80–90	3.77 3.73 3.71	0.82	23.54 23.31 23.20	0.74
Feather meal	80–100	12.46 12.53 12.54	0.35	77.87 78.33 78.35	0.35
Sheeps food	130–140	2.94 2.96 2.96	0.39	18.36 18.51 18.35	0.49
Yeast	80–100	5.96 5.99 5.96	0.29	37.23 37.41 37.28	0.25

Table 2 – Part 2. Repeatability of N/Protein data of food and animal feed samples

Sample name	N/Protein determination				
	W (mg)	N%	RSD%	Prot. %	RSD%
Alfalfa	130–140	2.44 2.47 2.46	0.62	15.22 15.45 15.35	0.75
Poultry meal	80–100	8.13 8.11 8.06	0.45	50.79 50.71 50.39	0.42
Feed for sow	130–140	2.75 2.77 2.74	0.55	17.21 17.31 17.14	0.50

To evaluate the performance of the Flash*Smart* EA for CHNS determination, sulfanilamide pure organic standard was analyzed for 5 days. The calibration was performed with 2–3 mg of methionine standard. Table 3 shows the repeatability obtained. The data obtained fall within the technical specification of the system of sulfanilamide: 16.27 N% (±0.16), 41.84 C% (±0.30), 4.68 H% (±0.07) and 18.62 S% (±0.20).

Table 3. Day-by-day CHNS repeatability of sulfanilamide

Element	Day 1		Day 2		Day 3		Day 4		Day 5	
	%	RSD%	%	RSD%	%	RSD%	%	RSD%	%	RSD%
Nitrogen	16.34 16.36 16.32	0.12	16.38 16.31 16.32	0.23	16.25 16.26 16.28	0.09	16.30 16.28 16.31	0.09	16.37 16.36 16.37	0.04
Carbon	41.67 41.65 41.68	0.04	41.65 41.66 41.68	0.04	41.65 41.62 61.65	0.04	41.71 41.76 41.71	0.06	41.69 41.73 41.72	0.05
Hydrogen	4.67 4.67 4.67	0	4.66 4.67 4.67	0.12	4.69 4.68 4.67	0.21	4.67 4.68 4.67	0.12	4.67 4.69 4.69	0.25
Sulfur	18.68 18.66 18.60	0.22	18.64 18.66 18.69	0.13	18.71 18.71 18.74	0.08	18.71 18.62 18.71	0.28	18.72 18.73 18.75	0.08

Table 4 shows the CHNS data of different matrices from different application field analyzed in triplicate, including the range of weight used for analysis.

Table 4. Repeatability of CHNS data of several matrices

Application field	Sample	W (mg)	N%	RSD%	C%	RSD%	H%	RSD%	S%	RSD%
Agronomy	Broccoli plant	3–4	3.51 3.49 3.48	0.44	35.94 35.93 36.03	0.15	5.23 5.21 5.24	0.29	1.38 1.38 1.40	0.83
	Inorganic fertilizer	3–4	34.27 34.31 34.49	0.34	0.237 0.234 0.238	0.88	5.20 5.20 5.21	0.11	0.767 0.760 0.758	0.62
Environmental	Sewage sludge	3–4	0.704 0.700 0.702	0.23	59.45 59.66 59.30	0.30	4.89 4.88 4.89	0.09	0.294 0.293 0.290	0.65
	Compost	3–4	2.14 2.15 2.13	0.47	22.17 22.24 22.13	0.38	2.65 2.67 2.66	0.38	1.47 1.46 1.45	0.68
Food	Supplement	3–4	0.330 0.333 0.329	0.50	40.61 40.39 40.63	0.32	6.27 6.33 6.32	0.49	0.366 0.368 0.357	1.58
	Gelatine	3–4	15.80 15.83 15.84	0.15	44.61 44.65 44.62	0.04	6.62 6.66 6.62	0.31	0.531 0.536 0.537	0.60
	Indian tea	3–4	3.67 3.67 3.69	0.31	46.87 47.04 46.97	0.18	5.69 5.68 5.66	0.27	0.200 0.201 0.203	0.76
Geological	Rock 1	10–20	0.0062 0.0065 0.0064	2.40	0.151 0.156 0.158	2.20	0.164 0.177 0.169	3.90	0.179 0.173 0.180	2.05
	Rock 2	10–20	0.0033 0.0035 0.0031	6.06	0.0997 0.0982 0.0975	1.14	0.368 0.368 0.368	0.10	0.0269 0.0255 0.0260	2.68
Material characterization	Heavy fuel oil	2–3	0.547 0.549 0.555	0.76	86.58 86.68 86.51	0.10	10.55 10.59 10.56	0.20	2.28 2.30 2.29	0.40
	Carbon fiber	2–3	3.79 3.77 3.84	1.04	93.63 93.24 93.60	0.23	0.313 0.329 0.312	2.92	0.0324 0.0359 0.0339	5.15
	Rubber	2–3	0.626 0.628 0.622	0.47	81.96 81.87 82.36	0.32	11.22 11.25 11.27	0.24	1.56 1.55 1.57	0.61
Petrochemical	Black coal	2–3	1.34 1.33 1.33	0.27	79.86 79.67 79.68	0.14	4.56 4.57 4.56	0.11	0.345 0.340 0.351	1.59
	Bio-fuel	3–4	0.503 0.499 0.500	0.42	45.19 45.18 44.95	0.30	5.67 5.64 5.59	0.66	0.0324 0.0317 0.0327	1.59
	Woodchips	3–4	1.62 1.63 1.62	0.36	36.34 36.43 36.44	0.15	4.70 4.69 4.71	0.21	0.604 0.606 0.611	0.59

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Conclusion

The Flash*Smart* Analyzer is the optimal solution for the analysis of N/Protein and CHNS in terms of accuracy, reproducibility, automation, speed of analysis and cost per analysis. All data showed were obtained with an acceptable repeatability and no matrix effect was observed when changing the configuration.

The MultiValve Control module (MVC) is a device used for performing the following functions:

- 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.

Double analytical configuration capability reduce the timing to pass from one to another configuration increasing the productivity of a laboratory, reduce the possibilities to have leaks in the system, the maintenance is very easy considering only one reactor for CHNS and one reactor for N/Protein, and always is completely controlled by the software Eager*Smart* Data handling Software.

Same system, hardware, autosamplers and software can be used for other combinations such CHN/O, CHN/S, CHNS/O, CHN/CHN, NC/S, N-Protein / S, etc., using simple upgrade Kits of consumables in which combustion and pyrolysis for oxygen determination can be perform.

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