Nitrogen/Protein determination of infant food by the Thermo Scientific Flash*Smart* Elemental Analyzer using helium or argon as carrier gases

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Goal

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

Introduction

The nutritional composition of infant food plays a very important role in food industries for research and quality control purposes. Food market globalization requires accurate control of product characteristics to protect commercial value, to safeguard consumer health, and to maintain manufacturer reputation. The selection of infant food products available in the market is very wide, and consumers' decisions depend on a set of information that includes quality valuation. New regulations regarding all processed food and most raw foods include a series of tests aimed at determining food contents and their contribution to a healthy diet for infants and children. Official regulations establish protein content and labeling requirements which enable consumers to make quality comparisons.

One of the tests used in the production process is the determination of protein content through nitrogen analysis. It is very important to have an accurate and precise technique for periodically performing this test, and an automated technique that provides fast analysis, excellent reproducibility, and avoids the use of toxic chemicals is ideal. An alternative to the classical Kjeldahl method, based on the Dumas (combustion) method, has been developed and is approved by different organizations.

The Thermo Scientific™ Flash*Smart*™ Elemental Analyzer (Figure 1), based on the dynamic combustion method (Dumas method), provides rapid and automated nitrogen determination without use of hazardous chemicals and offers advantages in precision over traditional methods. The Flash*Smart* Elemental Analyzer allows runs at both high and low nitrogen levels with no need to change configurations and without matrix effects.

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

However, due to a possible worldwide shortage and increase in the cost of helium, the Flash*Smart* Analyzer can work with an alternative gas, argon which is readily available.

Methods

The Elemental Analyzer operates according to the dynamic flash combustion (modified Dumas method) of the sample. Powdered samples were weighed in tin containers and liquid samples were adsorbed on the inert material Chromosorb, then weighed in tin containers. Both were introduced into the combustion reactor with oxygen via the Thermo Scientific™ MAS Plus Autosampler. After combustion, the produced gases were carried by a helium or argon flow to a second reactor filled with copper. Then they were swept through CO_2 and H_2O traps, a GC column, and were finally detected by a thermal conductivity detector (Figure 2). A complete report is automatically generated by the Thermo Scientific™ Eager*Smart*™ Data Handling Software and displayed at the end of the analysis. Using the nitrogen data obtained and a protein factor, the software automatically calculates the protein content.

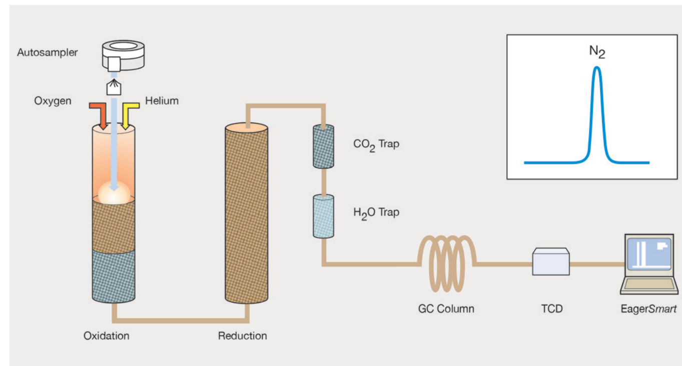

Figure 2. Flash*Smart* Elemental Analyzer nitrogen configuration.

Table 1 shows the analytical conditons using helium and argon as carrier gases.

Table 1. Analytical conditions

Analytical Conditions		
Parameter	**Helium Carrier Gas**	**Argon Carrier Gas**
Combustion Reactor Temperature	950 °C	950 °C
Reduction Reactor Temperature	840 °C	840 °C
Oven Temperature	50 °C	50 °C
Carrier Gas Flow	140 mL/min	60 mL/min
Reference Gas Flow	100 mL/min	60 mL/min
Total Run Time	400 sec	600 sec
Sample Delay	10 sec	10 sec
Oxygen Flow	300 mL/min	300 mL/min
OxyTune Category	@ 30 sec: aspartic acid, Cat. A: samples 1, 3, 4, 5, 6, 7, 8, 9, 10 and 11 Cat. C: urea/water solution and liquid samples 12 and 13 Category E (d = 1.4) for sample 2	@ 30 sec: aspartic acid, @ 30 sec: all powder samples Cat. C: urea/water solution and liquid samples 12 and 13
GC Column	0.5 m, installed outside the oven	1 m, installed in the oven

Note: The Eager*Smart* Data Handling Software provides a new option AGO (Argon Gas Option) that allows the user to manage the flow of argon gas during the run.

Results

To evaluate the performance of the system using helium and argon as carrier gases, several infant food samples (powders and liquids) with different nitrogen concentrations were chosen.

Table 2 shows the sample information and Table 3 shows the standards used for calibration and the weight of each sample. K factor was used as the calibration method in all cases.

Table 2. Sample information.

Ref. No.	Matrix Type	Consuming Age
1	Milk based powder, sucrose free	1–3 years
2	Milk based powder	From birth onward
3	Milk based powder	1–3 years
4	Milk based powder	1–3 years
5	Milk based powder	3 years onward
6	Milk based powder	1–8 years
7	Milk based powder	6–12 months
8	Milk based powder, sucrose free	0–6 months
9	Milk based powder	1–10 years
10	Milk based powder	From birth onward
11	Milk based powder, sucrose free	From birth onward
12	Milk based powder	From birth onward
13	Milk based powder	From birth onward (premature babies)

Table 3. Standard and sample weight.

Helium Carrier Gas			Argon Carrier Gas		
Standard	**Sample**	**Weight (mg)**	**Standard**	**Sample**	**Weight (mg)**
Aspartic acid (10.52 N%) 50-100 mg	1	200–220	Aspartic acid (10.52 N%) 50-70 mg	1	110–115
	2	210–220		2	110–115
	3	220–230		3	110–115
	4	200–220		4	110–115
	5	170–180		5	110–115
	6	200–220		6	110–115
	7	190–200		7	110–115
	8	200–210		8	110–115
	9	200–220		9	110–115
	10	200–240		10	110–115
	11	200–220		11	110–115
Urea/water solution (0.46 N%) 160-200 mg	12	180–220	Urea/water solution (0.46 N%) 260-270 mg	12	220–270
	13	180–220		13	220–270

Table 4 shows the nitrogen/protein data obtained from samples listed in Table 3 by using helium and argon as carrier gases. Each sample was analyzed in triplicate. The protein factor used to calculate the protein content through the nitrogen value was 6.38.

Table 5 presents a summary of the protein averages comparing data from helium gas, argon gas, and from the Kjeldahl analysis. The protein % determined by Kjeldahl analysis was performed at least 3–4 times for every sample, and includes the acceptable range for results. The data comparison demonstrates the performance of the instrument using both gases.

Table 4. Nitrogen/protein data using helium and argon as carrier gas.

Sample Ref. No.	Helium as carrier gas			Argon as carrier gas		
	N%	Protein %	RSD%	N%	Protein %	RSD%
1	2.34 2.33 2.34	14.90 14.89 14.92	0.11	2.31 2.30 2.34	14.76 14.68 14.93	0.85
2	2.23 2.23 2.23	14.26 14.22 14.26	0.16	2.14 2.16 2.13	13.66 13.78 13.57	0.76
3	2.79 2.80 2.79	17.78 17.88 17.80	0.32	2.75 2.77 2.74	17.54 17.65 17.50	0.43
4	2.44 2.42 2.43	15.54 15.47 15.51	0.21	2.42 2.41 2.40	15.45 15.41 15.34	0.34
5	1.61 1.61 1.61	10.25 10.26 10.28	0.16	1.57 1.57 1.59	10.03 10.00 10.12	0.60
6	4.95 4.97 4.94	31.59 31.72 31.54	0.30	4.87 4.86 4.85	31.06 30.99 30.91	0.23
7	2.50 2.50 2.51	15.97 15.96 16.00	0.13	2.52 2.50 2.48	16.06 15.97 15.84	0.71
8	1.69 1.69 1.69	10.78 10.79 10.81	0.14	1.67 1.65 1.64	10.68 10.53 10.49	0.90
9	2.48 2.49 2.48	15.83 15.91 15.81	0.31	2.46 2.48 2.44	15.67 15.81 15.59	0.72
10	2.09 2.09 2.07	13.32 13.35 13.21	0.54	2.06 2.04 2.07	13.15 13.04 13.18	0.55
11	1.69 1.69 1.69	10.78 10.80 10.77	0.13	1.65 1.66 1.65	10.56 10.59 10.55	0.18
12	0.199 0.201 0.195	1.27 1.28 1.24	1.54	0.202 0.204 0.196	1.29 1.30 1.25	2.08
13	0.449 0.461 0.450	2.87 2.94 2.87	1.42	0.475 0.477 0.461	3.03 3.04 2.94	1.86

Table 5. Summary of protein results and comparison with Kjeldahl method.

Ref. No.	Kjeldahl values			FlashSmart EA using helium carrier gas		FlashSmart EA using argon carrier gas	
	N%	Protein %	Acceptable range	N%	Protein %	N%	Protein %
1	2.35	15.0	14.25–15.75	2.34	14.91	2.32	14.79
2	2.19	14.0	13.30–14.70	2.23	14.24	2.14	13.67
3	2.66	17.0	16.15–17.85	2.79	17.82	2.75	17.57
4	2.35	15.0	14.25–15.75	2.43	15.50	2.41	15.40
5	1.57	10.0	9.50–10.50	1.61	10.26	1.58	10.05
6	4.80	30.6	29.07–32.13	1.61	31.62	4.86	30.99
7	2.51	16.0	15.20–16.80	2.50	15.98	2.50	15.96
8	1.72	11.0	10.45–11.55	1.69	10.79	1.66	10.57
9	2.48	15.8	15.00–16.59	2.48	15.85	2.46	15.69
10	2.01	12.8	12.16–13.44	2.08	13.30	2.06	13.12
11	1.60	10.2	9.69–10.71	1.69	10.78	1.66	10.57
12	0.19	1.24	1.178–1.302	0.198	1.27	0.201	1.28
13	0.45	2.88	2.736–3.024	0.453	2.89	0.471	3.00

Conclusions

All data shown were obtained with excellent repeatability, and no matrix effect was observed when changing the sample. Good repeatability was obtained with the Flash*Smart* EA Nitrogen Analyzer using argon as carrier gas, and data from argon and helium were comparable. The data is also comparable with data obtained by the Kjeldahl method.

As a complete automatic system, the Flash*Smart* Elemental Analyzer can analyze nitrogen in a wide content range, in solid and liquid samples, and without the use of sample digestion or the toxic chemicals used in traditional methods.

The Dumas combustion method has been approved and adopted by official organizations such as ASBC, AOAC, AACC, AOCS, ISO, and IDF (International Dairy Federation) Standard.

The Flash*Smart* Elemental Analyzer meets all stringent requirements of modern laboratories such as flexibility, repeatability, sensitivity, automation, speed, and low cost per analysis.

Acknowledgement

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