Elemental analysis

Quantitative elemental analysis in food cycle supported by an automated Elemental Analyzer

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Abstract

The food cycle includes a variety of processes and materials that are carefully regulated by federal authorities and various international organizations to ensure product quality and consumer and environment safety. One of the main tools for quality control of materials in the food cycle is elemental characterization.

Elemental analysis utilizes carbon, hydrogen, nitrogen, sulfur and oxygen, which help determine the structure of an unknown compound, as well as to evaluate the structure and purity of a synthesized compound. This poster presents data of different samples in the food cycle in a large concentration range to evaluate the performance of the Thermo Scientific™ Flash*Smart*™ Elemental Analyzer for the determination of nitrogen, carbon, hydrogen, sulfur and oxygen.

Introduction

The globalization of the food and feed market, and the associated production and recycling processes, demand accurate and reliable control of product characteristics for the protection of commercial value, but mainly to safeguard consumer health and manufacturer reputation. The Flash*Smart* Elemental Analyzer (Figure 1) enables quantitative elemental determinations at high and low levels of concentrations for solid and liquid samples in one single system, adapting to your needs and covering a wide spectra of analysis in the food cycle. The elemental analysis performed is based on the combustion (Dumas) method, allowing an easy, fast, cost-effective and environmentally friendly way to analyze your samples. The Analyzer copes effortlessly with modern laboratory requirements such as accuracy, day to day reproducibility and high sample throughput.

Figure 1. Thermo Scientific Flash*Smart* Elemental Analyzer

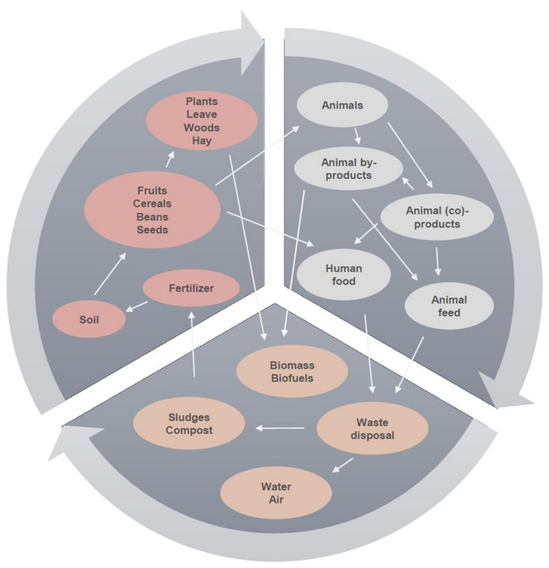
Elemental analysis is mainly useful in food cycle quality control for (Figure 2):

1. Determination of protein content through the nitrogen content in food and animal feed is one of the tests in Official Methods for processed and raw products. Official regulations establish the protein content and labeling requirements of the nutritional features, which enable consumers to perform price and quality comparisons based on % protein declarations.

2. Elemental characterization of soils, leaves, plant, crops and fertilizers to set agronomy management plans and to get information on the deficiency or excess of nutritional elements in soils and plants.

3. Production of compost as one of the most efficient ways of recycling waste. Elemental analysis is a key factor to ensure the quality of the final product that can be used as fertilizers in agronomy.

Figure 2. The food cycle schematics



Method

The Flash*Smart* EA can be configured differently according to the sample matrix, element for analysis, and laboratory requests. The main configurations shown are CHNS/O and N/Protein.

For CHNS determination the Flash*Smart* EA operates with the dynamic flash combustion of the sample. Samples are weighed in tin containers and introduced into the combustion reactor (left furnace) via the Thermo Scientific™ MAS Plus Autosampler with the proper amount of oxygen. After combustion, the resultant 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). For oxygen determination, the system operates in pyrolysis mode. Samples are weighed in silver containers and introduced into the pyrolysis chamber (right furnace) via the MAS Plus Autosampler. The reactor contains nickel coated carbon maintained at 1060 ° C. The oxygen present in the sample, combined with the carbon, forms carbon monoxide which is then gas chromatographically separated from other products and detected by a TCD (Figure 3).

For N/Protein, the Flash*Smart* EA operates according to the dynamic flash combustion of the sample. The sample is weighed in tin containers and introduced into the combustion reactor via the MAS Plus Autosampler with oxygen. After combustion, the produced gases are carried by 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 TCD (Figure 4).

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 automatically converts the nitrogen content in the protein % by using a specific protein factor.

Figure 3. CHNS/O Configuration

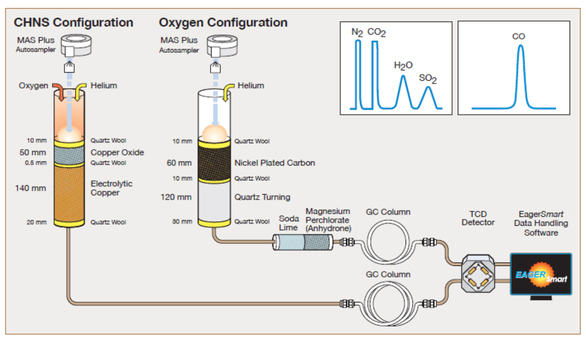
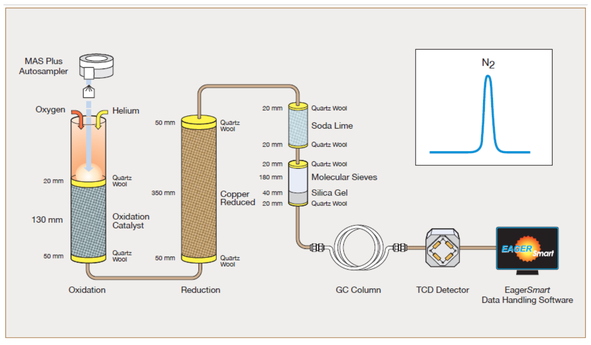


Figure 4. N/Protein configuration



Results

Several sample matrices in a large range of concentrations were analyzed to demonstrate the performance of the Flash*Smart* EA for Food Cycle control. Most of the solid samples were homogenized by a ball mill.

The accuracy and precision of the Flash*Smart* Analyzer for N/Protein determination was evaluated by the analysis of BIPEA (Bureau InterProfessionnel d'Etudes Analytiques, France) Reference Materials. The results obtained were compared with the average and range values indicated in the Reference Materials Certificates (Table 1 and Table 2).

Table 1. BIPEA sample information

BIPEA Ref. Mat.	Kjeldahl Protein		Combustion Protein	
	Av. %	Tolerance	Av. %	Tolerance
Feed for Sow	16.0	0.6	16.2	0.6
Dehydrated Alfalfa	14.8	0.6	15.1	0.6
Hyperproteic Powder	85.4	3.4	86.4	3.5

Table 2. N/Protein data of BIPEA Reference Materials by Flash*Smart* Analyzer

BIPEA Ref. Mat.	Feed for Sow		Dehydrated Alfalfa		Hyperproteic Powder	
	N%	Protein %	N%	Protein %	N%	Protein %
	2.61	16.31	2.43	15.21	13.74	85.90
	2.61	16.34	2.42	15.13	13.72	85.70
Average %	2.61	16.32	2.42	15.17	13.73	85.84
RSD%	0.00	0.13	0.29	0.37	0.10	0.10

Two tests were performed to show accuracy and repeatability with low and high nitrogen content. For low nitrogen determination, starch and starch slurry were selected.

Table 3. N/Protein repeatability at low nitrogen content

Sample	N%	RSD%	Protein %	RSD%
Starch 1	0.0484 0.0486 0.0483	0.31	0.3024 0.3040 0.3016	0.40
Starch 2	0.0360 0.0367 0.0362	0.99	0.2250 0.2294 0.2262	1.00
Starch Slurry	0.0166 0.0165 0.0159	2.32	0.1038 0.1029 0.0994	2.28

Table 3 shows the N/protein data. The calibration was performed with 50-100 mg Aspartic acid standard using K factor as the calibration method. The sample weight was 200-300 mg for starch and 300-320 mg for starch slurry that was adsorbed on Chromosorb®.

For high nitrogen determination, a variety of samples was analyzed (Table 4).

Table 4. N/Protein repeatability at high nitrogen content

Sample	N%	RSD%	Protein %	RSD%
Soy	7.89 7.87 7.90	0.19	49.29 49.20 49.39	0.19
Soybean Meal	7.88 7.87 7.88	0.07	49.22 49.21 49.24	0.03
Soy Protein Concentrate	10.53 10.50 10.51	0.15	65.81 65.63 65.72	0.14
Gluten	9.83 9.81 9.85	0.20	61.44 61.33 61.56	0.19
Corn Gluten	10.05 10.04 10.05	0.03	62.81 62.79 62.82	0.03
Meat Meal	8.99 8.95 8.97	0.22	56.19 55.94 56.06	0.22
Fish Meal	11.42 11.40 11.45	0.22	71.37 71.25 71.56	0.22
Milk Protein Concentrate	12.25 12.29 12.26	0.12	76.56 75.75 76.62	0.13

Table 4 shows the N/Protein data analyzed in triplicate. The calibration was performed with 70-100 mg Nicotinamide standard using K factor as the calibration method; the samples weight was 80-300 mg.

Table 5. N/Protein data of food and animal feed compared to Kjeldahl method

Sample	Flash*Smart* EA		Kjeldahl	
	N%	Protein %	N%	Protein %
Soya	6.27	39.20	6.27	39.18
Lentils	4.35	27.17	4.35	27.19
Rice	1.13	7.08	1.12	7.00
Wheat	1.75	10.91	1.74	10.89
Beans	3.74	23.35	3.74	23.38
UHT milk	0.53	3.38	0.53	3.37
Crude milk	0.47	3.03	0.47	3.02
Pasteurized milk	0.50	3.21	0.50	3.19
Milk powder	4.32	27.56	4.30	27.43
Yoghurt	0.080	0.51	0.078	0.50
Mascarpone cheese	0.635	4.05	0.638	4.07
Biscuits	1.40	8.80	1.39	8.72
Flour	1.34	8.40	1.32	8.24
Fish meal	10.46	65.4	10.45	65.3

Table 5 shows the N/Protein data of food and animal feed analyzed in triplicate. The protein factor 6.25 was used to calculate the protein content. The instrument calibration was performed with aspartic acid (10.52 N%) or nicotinamide standard (22.94 N%); the samples weight was 200 – 300 mg. The data obtained were comparable to the Kjeldahl method.

Table 6. NCS data of soils and plants

Sample	N%	RSD%	C%	RSD%	S%	RSD%
Soil 1	1.05	0.67	13.45	0.78	0.527	0.37
Soil 2	0.0583	0.26	0.872	0.24	0.0102	1.39
Soil 3	0.427	0.79	7.051	0.14	0.0378	0.76
Plant 1	3.49	0.44	35.96	0.15	1.39	0.83
Plant 2	1.19	0.80	46.89	0.13	0.245	1.24
Plant 3	1.14	0.95	49.08	0.19	0.0634	1.31
Fertilizer 1	10.56	0.23	4.58	1.05	0.21	1.22
Fertilizer 2	25.52	0.09	0.044	1.90	0.085	0.71
Fertilizer 3	20.67	0.31	0.939	0.41	23.96	0.12

Table 6 shows the NCS data of soils, plants and fertilizers analyzed in triplicate. The instrument calibration was performed with BBOT standard; the sample weight was 10-20 mg for soils, 3-6 mg for plants and fertilizers.

Table 7. CHNS/O data of sludges, compost and biomass

Sample	N%	RSD%	C%	RSD%	H%	RSD%	S%	RSD%	O%	RSD%
Sludge 1	3.84	0.8	26.58	0.35	4.53	0.16	1.05	0.97	22.97	0.24
Sludge 2	0.702	0.23	59.47	0.3	4.87	0.09	0.292	0.65	25.67	0.46
Sludge 3	1.24	0.75	32.47	0.74	2.61	1.13	0.864	1.02	8.91	0.23
Compost 1	1.74	0.33	21.56	0.32	2.58	39	0.149	1.69	15.09	0.08
Compost 2	2.14	0.047	22.18	0.38	2.66	0.38	1.46	0.68	23.1	0.09
Compost 3	2.16	0.54	24.46	0.21	2.64	0.38	0.883	0.75	24.51	0.18
Biomass 1	5.72	3.13	47.29	0.39	5.93	1.65	0.273	2.65	34.22	0.44
Biomass 2	3.59	2.82	46.52	0.15	5.92	0.61	0.181	2.78	37.26	0.36

Table 7 shows the CHNS/O data of sludges, compost and biomass. The instrument calibration for CHNS was performed with BBOT standard; the sample weight was 3 – 4 mg. For oxygen determination, benzoic acid was used for calibration; the sample weight was about 1-2 mg.

Table 8. Heat values and CO_2 emission factor of compost and biomass

Sample	GHV (Kcal/kg)	RSD%	NHV (Kcal/kg)	RSD%	CO_2 E.T.	RSD%
Compost 1	2548.1	0.02	2397.3	0.02	97.2	0.02
Compost 2	1777.3	0.06	1640.8	0.06	118.3	0.06
Compost 3	1882.1	0.1	1746.6	0.11	122.6	0.11
Biomass 1	4454.3	0.15	4149	0.17	99.46	0.16
Biomass 2	4251.7	0.14	3948.3	0.14	103.15	0.14

Table 8 shows the relative heat values and CO2 emission factor for the compost and biomass samples calculated automatically by the Eager*Smart* Data Handling Software.

Conclusions

- The Thermo Scientific Flash*Smart* Elemental Analyzer is the optimal solution for the analysis of CHNS/O and N/Protein for all types of sample matrices in diverse application fields, delivering accuracy, reproducibility and speed of analysis
- The Flash*Smart* Elemental Analyzer, based on the combustion method (Dumas), offers advantages over the Kjeldahl Method for the Nitrogen/Protein determination in terms of automation, ease of use and cost per sample.
- Protein data, heat values and CO_2 emission can be calculated automatically by the Eager*Smart* Data Handling Software.
- The Dumas Combustion Method has been approved and adopted by Official Organizations such as AOAC, ASBC, AACC, AOCS, IFFO and ISO.

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