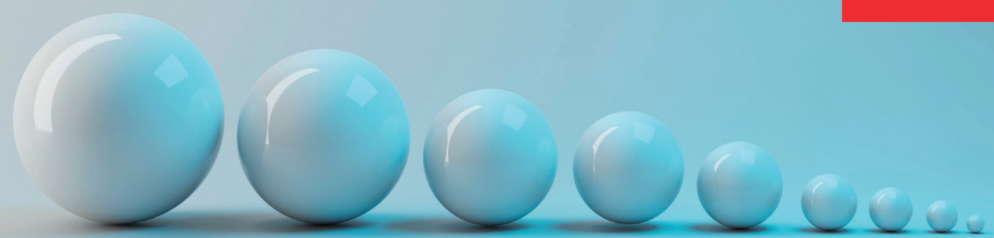
Technical note | 002251

Elemental analysis



Micro vs. macro analysis using the Flash*Smart* Elemental Analyzer

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Keywords

micro, macro, elemental analysis, precision, repeatability

Goal

To assess data precision and repeatability using the Flash*Smart* Elemental Analyzer for micro and macro analysis.

Introduction

Carbon, nitrogen, hydrogen, sulfur determination by combustion analysis, and oxygen determination by pyrolysis are commonly used for the characterization of raw and final products in diverse applications. Many materials are often inhomogeneous which can present a challenge for obtaining high quality data. Thermo Scientific™ Flash*Smart*™ Elemental Analyzer allows for optimization of the sample weight according to the element content for obtaining accurate and precise data using different configurations.

Sample homogenization is critical for data quality. Performing elemental analysis on high sample amounts does not ensure excellent reproducibility as this strongly depends on the matrix that is analyzed. Additionally, weighing more sample means increasing the consumption of consumables and gases with a considerable increase in the cost per analysis. The reproducibility of results is independent of the sample size but strongly dependent of the sample homogeneity. Here were demonstrate how the Flash*Smart* Elemental Analyzer can perform micro analysis with high precision and repeatability, allowing for optimized analytical workflows and reduced cost per sample.

Methods

The Flash*Smart* Elemental Analyzer (EA) is equipped with two fully independent furnaces allowing the installation of one or two analytical circuits that can be used sequentially. Each analytical circuit can be equipped with an autosampler that is fully controlled by the Thermo Scientific™ Eager*Smart*™ Data Handling Software. Optionally, the system can be used with a combination of a liquid and solid autosampler.

Operation of the Flash*Smart* EA is based on dynamic flash combustion of the sample. For CHNS analysis the sample is weighed in tin containers and introduced into the combustion reactor via the Thermo Scientific™ MAS Plus Autosampler alongside a pulse of oxygen. Produced gases are separated in a GC column before detection by a Thermal Conductivity Detector (TCD) (Figure 1).

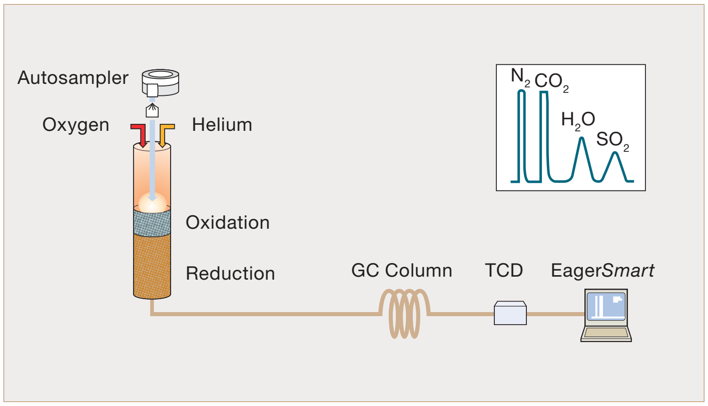

Figure 1. Flash*Smart* EA CHNS workflow

For NC determination, after the combustion, the produced gases are carried by helium flow to a second reactor filled with copper before being swept through an H_2O trap, a GC column and detected by TCD (Figure 2).

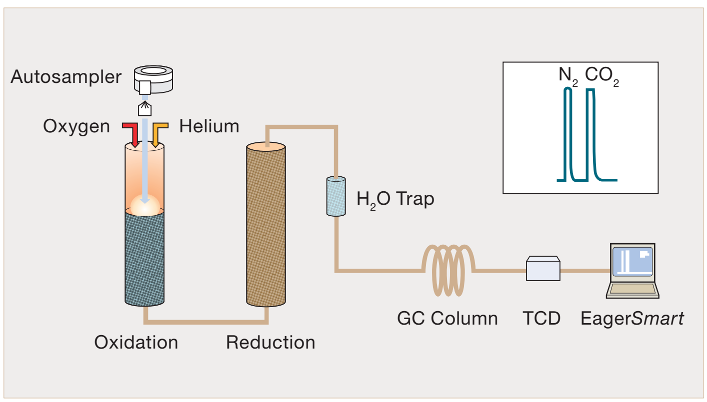

Figure 2. Flash*Smart* EA NC workflow

For nitrogen determination, after combustion, the produced gases are carried by a 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 TCD (Figure 3).

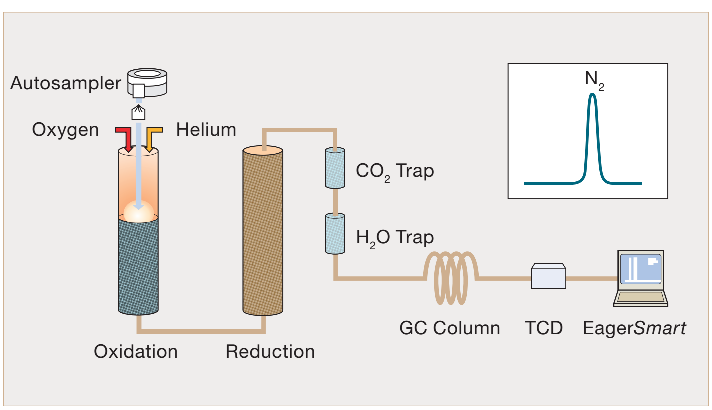

Figure 3. N/Protein configuration

A complete report is automatically generated by the Eager*Smart* Data Handling Software and displayed at the end of the analysis. Eager*Smart* Data Handling Software controls all analytical parameters of the instrument including the oxygen flow and the timing of oxygen injection. The software automatically calculates the amount of oxygen, relative to the sample matrix and sample weight, using patented Thermo Scientific™ OxyTune™ Function to ensure the complete combustion of the sample. This approach also decreases the cost per analysis by not wasting oxygen or consuming the copper unnecessarily.

Results

Different reference materials and samples were analyzed to evaluate analytical performance of the Flash*Smart* EA in micro and macro analysis mode.

Thermo Scientific Soil Reference Material in CHNS and NC Soil configurations

For CHNS determination, the system was calibrated with 2–3 mg BBOT standard, and the sample weight was 10–20 mg. For NC determination, the system was calibrated with 4–5 mg aspartic acid standard, and the sample weight was about 100 mg. The Soil Reference Material (0.21 N% ±0.01, 2.29 C% ±0.07, 0.0230 S% ±0.0040) was analyzed in five repetitions. Table 1 shows that data obtained in both micro (CHNS configuration) and macro (NC Soil configuration) workflow is comparable to reference values. By using the micro analysis workflow, user gains additional information about H% and S% in a single run.

Table 1. Comparison of Soil Reference Material data obtained using micro (CHNS configuration) and macro (NC Soil configuration) workflows (n=5)

	CHNS configuration	NC Soil configuration
Sample weight (mg)	10-20	100
N% RSD%	0.21 0.77	0.21 0.72
C% RSD%	2.27 0.57	2.26 0.52
H% RSD%	0.93 0.26	– –
S% RSD%	0.0213 1.59	– –

Soils, plants and algae reference materials in NCS and NC Soil configurations

Reference Materials with certified NCS% (Table 2) were analyzed using NCS and NC Soil configurations. For NCS determination, the system was calibrated with 2–3 mg BBOT standard, and the sample weight was 10–20 mg for soils and 3–4 mg for plants and algae. For NC determination, the system was calibrated with 4–5 mg aspartic acid standard, and the sample weight was about 100 mg for soils and 5–6 mg for plants and algae. Samples were analyzed in triplicate.

Table 3 shows that data obtained in both micro (CNS configuration) and macro (NC configuration) workflow is comparable to reference values reported in Table 2. Additionally, the CNS configuration allows for sequential NCS weight % analysis in a single run.

Table 2. Certified values of Reference Materials for soils, plants and algae

Reference material	Specification					
	N%	Uncertainty (±)	C%	Uncertainty (±)	S%	Uncertainty (±)
Loamy soil	0.27	0.02	2.75	0.12	0.04	n.a.
Low organic soil	0.13	0.023	1.61	0.09	0.01	n.a.
Sandy soil	0.07	0.01	0.83	0.05	0.01	n.a.
Orchard leaves	2.28	0.04	50.40	0.40	0.16	0.01
Alfalfa plant	3.01	0.20	n.a.	n.a.	0.27	0.04
Birch leaves	2.12	0.06	48.09	0.51	0.17	0.03
Bladderwrack algae	1.25	0.02	33.67	0.29	2.29	n.a.
Spirulina algae	10.81	0.06	47.21	0.39	0.60	0.03

Table 3. Comparison of different soils, plants and algae Reference Materials using micro (NCS configuration) and macro (NC configuration) workflows (n=3)

Reference material	NCS configuration						NC configuration			
	N%	RSD%	C%	RSD%	S%	RSD%	N%	RSD%	C%	RSD%
Loamy soil	0.27	0.74	2.74	0.20	0.0426	1.31	0.28	0.91	2.73	0.57
Low organic soil	0.13	1.05	1.61	0.32	0.0132	2.27	0.13	0.77	1.60	0.31
Sandy soil	0.0714	1.13	0.85	0.56	0.0155	1.97	0.0670	0.99	0.83	0.48
Orchard leaves	2.29	0.15	50.27	0.14	0.15	0.99	2.30	0.22	50.14	0.08
Alfalfa plant	3.06	0.38	43.74	0.10	0.27	1.84	3.05	0.55	43.77	0.12
Birch leaves	2.13	0.10	48.22	0.21	0.17	0.92	2.14	0.14	48.24	0.11
Bladderwrack algae	1.25	0.17	33.63	0.25	2.28	0.22	1.25	0.57	33.66	0.22
Spirulina algae	10.85	0.37	47.26	0.07	0.59	0.52	10.82	0.33	47.28	0.09

Rock analysis in CHNS configuration at nominal and high sample weight

A rock sample was analyzed by CHNS configuration using nominal weight (10–15 mg) and high weight (100–106 mg). For both tests, the calibration was performed with 2–3 mg BBOT standard, the number of replicates was 5 for nominal weight and 10 for high weight. Using lower sample weight allows for higher number of analysis before changing the reactor and requires less maintenance, resulting in lower cost per analysis. Table 4 shows that the data obtained in micro analysis mode is comparable to data obtained in macro analysis mode, with excellent repeatability.

Table 4. Comparison of rock sample CHNS weight % data at nominal and high weight

	CHNS at nominal sample weight	CHNS at high sample weight
Sample weight (mg)	10–15 mg	100–106 mg
N%	0.0031	0.0030
RSD%	1.79	2.08
C%	0.22	0.0547
RSD%	1.49	1.78
H%	0.22	0.22
RSD%	1.50	1.56
S%	0.0561	0.0551
RSD%	1.49	1.24

Food and feed in N/Protein and CHNS configurations

Two reference materials, Thermo Scientific Pasta (2.10 N% ±0.05) and Spirulina algae (10.81 N% ±0.06), and different food and feed samples were analyzed by CHNS and N/Protein configuration. Table 5 shows the standard used for calibration and the sample weight. Table 6 shows that N/Protein data obtained in micro (CHNS configuration) workflow for the two reference materials is in agreement with certified values, as is the N/Protein data obtained in macro (N/Protein configuration) workflow. The micro workflow allows users to additionally also gain weight % data for C, H and S while maintaining high N/Protein data quality and lower cost per sample.

Table 5. List of food and feed samples with standards and sample weight

Sample	CHNS configuration		N/Protein configuration	
	Standard	Sample weight (mg)	Standard	Sample weight (mg)
Thermo Scientific pasta Ref. Mat.	2–3 mg BBOT	3–4	60–70 mg aspartic acid	200–300
Spirulina algae Ref. Mat.	2–3 mg BBOT	3–4	90–100 mg nicotinamide	220–250
Crispy silkworms	2–3 mg BBOT	3–4	50–100 mg nicotinamide	200–230
Barley pests	2–3 mg BBOT	3–4	50–100 mg nicotinamide	220–240
Cricket flour	2–3 mg BBOT	3–4	50–100 mg nicotinamide	210–240
Indian tea (North)	2–3 mg BBOT	3–4	50–70 mg aspartic acid	150–200
Indian tea (Assam)	2–3 mg BBOT	3–4	50–70 mg aspartic acid	150–200

Table 6. Comparison of food and feed data obtained in micro (CHNS configuration) and macro (N/Protein configuration) workflow

Sample	Repetitions	CHNS configuration								N/Protein configuration	
		N%	RSD%	C%	RSD%	H%	RSD%	S%	RSD%	N%	RSD%
Thermo Scientific pasta	5	2.15	0.33	42.02	0.08	6.52	0.22	0.14	0.75	2.13	0.22
Spirulina algae	10	10.81	0.13	47.23	0.10	6.90	0.29	0.61	0.69	10.82	0.24
Crispy silkworms	5	7.93	0.17	55.72	0.16	8.17	0.16	0.48	0.12	7.98	0.27
Barley pests	5	7.94	0.14	55.64	0.14	8.36	0.16	0.20	0.27	7.96	0.39
Cricket flour	5	11.06	0.14	55.10	0.14	8.03	0.14	0.46	0.18	11.04	0.27
Indian tea (North)	3	3.68	0.31	46.96	0.18	5.68	0.27	0.20	0.76	3.67	0.27
Indian tea (Assam)	3	3.72	0.31	47.28	0.53	5.69	0.79	0.20	1.05	3.70	0.27

Conclusions

The Flash*Smart* Elemental Analyzer offers optimized micro workflows for the characterization of raw and final products in diverse applications, ensuring accuracy, reproducibility, automation, and low cost per analysis.

The all-in-one Flash*Smart* Elemental Analyzer design offers flexibility to serve varying sample matrix, from soil, plants and algae to rocks, food or feed. All data presented were obtained with an excellent repeatability and no matrix effect was observed when changing the sample type.

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