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Elemental Analysis: NC determination in soils and plants using argon as carrier gas

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Goal

This application note reports data on nitrogen and carbon determination in soils and plants reference materials in different concentrations and shows the performance of the Thermo Scientific Flash*Smart* EA with argon as carrier gas.

Introduction

The determination of nitrogen and carbon in soils enables to evaluate the organic matter and the amount of fertilizers needed for agronomy purposes. The nitrogen and carbon determination also provides significant information on the nutritional elements of plants, which affect their future growth. The determination of nitrogen content allows to control the quality of different types of crop, used for cattle feeding, as well as for N-cycle and N-fixation monitoring in agricultural and environmental research.

The importance of soils and plant testing has increased in the last few years, as many of the traditional methods are no longer suitable for routine analysis, due to their time consuming preparation and the required use of environmentally hazardous reagents. For this reason the need for an efficient analytical technique has become critical. As the demand for improved sample throughput, reduction of operational costs and minimization of human errors has increased dramatically, a simple and automated technique, allowing fast analysis with excellent reproducibility is key for efficient nitrogen and carbon determination.

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The Thermo Scientific™ Flash*Smart*™ Elemental Analyzer (Figure 1), using helium gas carrier and operating with the dynamic flash combustion of the sample, meets modern laboratory requirements. Considering the need for cost efficiencies and the likely increase in helium gas cost, an alternative gas to be used as carrier gas is needed. Argon can be used as alternative to helium in the Flash*Smart* EA.

Methods

The Elemental Analyzer operates with 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 with oxygen. After combustion, the produced gases are carried by a gas flow to a second reactor containing copper, then they are swept through a H_2O trap, a GC column. At the end they are detected by a Thermal Conductivity Detector (TCD). The analytical configuration, TCD Detector included are the same when using helium as well as when using argon as carrier gas. (Figure 2).

The Thermo Scientific™ Eager*Smart*™ Data Handling Software generates a complete report, displayed at the end of the analysis. The Eager*Smart* Data Handling Software features the option AGO (Argon Gas Option), which allows the user to manage the flow of argon gas during the run.

Figure 1. Flash*Smart* Elemental Analyzer.

Results

Soils and plants reference materials with different nitrogen and carbon concentrations were analyzed in order to compare the results of the Flash*Smart* Elemental Analyzer, when using argon or helium as carrier gas.

Table 1 shows the nitrogen and carbon results of the analysis of Thermo Scientific Soil Reference Material. The calibration was performed with 15–16 mg aspartic acid using K factor as calibration method. The sample was weighed at 240–250 mg. The certified N% is 0.21 and C% is 2.29, the accepted range according to the technical specification is 0.19–0.23 for nitrogen and 2.27–2.31 for carbon. The average of the 10 runs is 0.191 N% and 2.299 C% with a RSD% 2.146 for nitrogen and 1.213 for carbon.

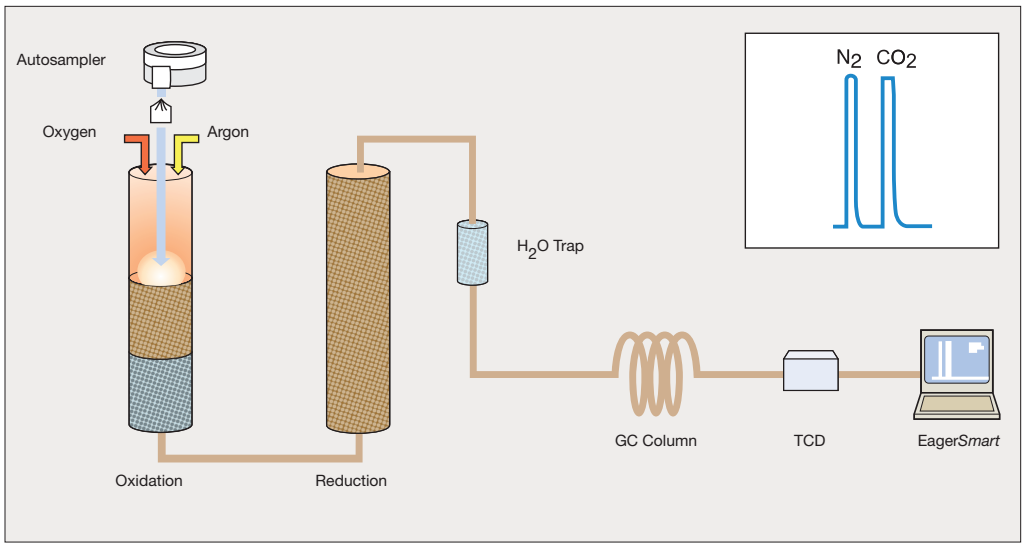

Figure 2. Nitrogen/carbon configuration.

Analytical Conditions	
Combustion furnace temp.:	950°C
Reduction furnace temp.:	840 °C
Oven temp.:	95°C (GC column inside the oven)
Argon carrier flow:	60 mL/min
Argon reference flow:	60 mL/min
Oxygen flow:	250 mL/min
Oxygen injection time:	15 sec for soils, 20 sec for plants
Sample delay:	12 sec
Run time:	10–14 min

Table 1. Nitrogen and carbon data of Thermo Scientific Soil Reference Material.

Weight (mg)	N%	C%
250.3	0.193	2.268
250.7	0.194	2.251
244.9	0.189	2.279
240.5	0.181	2.316
252.0	0.192	2.314
253.2	0.191	2.299
244.1	0.188	2.289
247.4	0.194	2.305
244.9	0.190	2.339
246.3	0.195	2.332

Four Soils Reference Materials were analyzed for the comparison of results. With argon as carrier gas, the calibration was performed with 50–200 mg of Thermo Scientific Soil Reference Material using Linear Fit as calibration method. Using helium as carrier gas, the calibration was performed with 4–5 mg of aspartic acid using K factor as calibration method. Table 2 shows the certified N% and C% and the relative uncertainty. Table 3 shows samples weights and the results with argon and helium as carrier gas.

Table 2. Certified nitrogen and carbon of Soil Reference Materials.

Sample Description	Specification			
	N%	Uncertainty (±)	C%	Uncertainty (±)
Low Organic Content Soil Reference Material	0.133	0.023	1.61	0.09
Medium Organic Content Soil Reference Material	0.27	0.02	3.19	0.07
Loamy Soil Reference Material	0.27	0.02	2.75	0.12
Chalky Soil Reference Material	0.35	0.02	5.39	0.09

Table 3. Experimental nitrogen and carbon data of Soils Reference Materials.

Sample	Argon Carrier Gas					Helium Carrier Gas				
	Weight (mg)	N%	RSD%	C%	RSD%	Weight (mg)	N%	RSD%	C%	RSD%
Low Organic Content Soil Ref. Mat.	150–200	0.132 0.133 0.133	0.435	1.64 1.61 1.66	1.54	90–100	0.124 0.122 0.127	2.024	1.55 1.55 1.57	0.74
Medium Organic Content Soil Ref. Mat.	100–150	0.28 0.28 0.28	0	3.22 3.19 3.18	0.65	90–100	0.27 0.27 0.27	0	3.16 3.15 3.15	0.18
Loamy Soil Ref. Mat.	100–150	0.28 0.28 0.28	0	2.76 2.70 2.68	1.53	90–100	0.26 0.26 0.26	0	2.70 2.72 2.69	0.56
Chalky Soil Ref. Mat.	80–90	0.38 0.38 0.39	1.51	5.33 5.45 5.36	1.16	90–100	0.38 0.37 0.38	1.53	5.34 5.35 5.35	0.11

A Sandy Soil Reference Material at 0.07 N% (700 ppm N, uncertainty ± 0.01) and 0.83 C% (uncertainty ± 0.05) was analyzed using argon and helium as carrier gas. With argon as carrier gas, the calibration was performed with 50–200 mg of Thermo Scientific Soil Reference Material using Linear Fit as calibration method and the sample was weighed at 150–250 mg. For the run with helium as carrier gas, the calibration was performed with 4–5 mg of aspartic acid using K factor as calibration method and the sample weighed 90–100 mg. Table 4 shows the results obtained with argon and helium as carrier gas.

Three Plants Reference Materials were analyzed for the comparison of performance and results. With argon as carrier gas, the calibration was performed with 14–15 mg of atropine, EDTA (EthyleneDiamineTetraAcetic acid) and aspartic acid using Linear Fit as calibration method while for helium gas the calibration was performed with 4–5 mg of aspartic acid using K factor as calibration method. Table 5 shows the certified N% and C% and the relative uncertainty. Table 6 shows the weights of samples used and the experimental results obtained with argon and helium as carrier gas. The average of the three runs falls in the uncertainty range value.

Table 4. Nitrogen and carbon data of Sandy Soil Reference Material.

Argon Carrier Gas				Helium Carrier Gas			
Exp. N%	RSD%	Exp. C%	RSD%	Exp. N%	RSD%	Exp. C%	RSD%
0.0711	1.5085	0.8566	1.7822	0.0662	1.0824	0.8162	1.0234
0.0683		0.8433		0.0652		0.8067	
0.0696		0.8452		0.0667		0.8301	
0.0710		0.8680		0.0673		0.8188	
0.0699		0.8455		0.0664		0.8154	
0.0704		0.8232		0.0662		0.8184	
0.0702		0.8419		0.0656		0.8171	
0.0704		0.8328		0.0656		0.8158	
0.0720		0.8207		0.0661		0.8301	
0.0716		0.8265		0.0649		0.8141	

Table 5. Expected nitrogen and carbon values of Plants Reference Materials.

Sample Description	Specification			
	N%	Uncertainty (±)	C%	Uncertainty (±)
Birch Leaves Ref. Mat.	2.12	0.06	48.09	0.51
Orchard Leaves Ref. Mat.	2.28	0.04	50.40	0.40
Alfalfa Ref. Mat	3.01	0.20	Not Available	—

Table 6. Analysis results of nitrogen and carbon determination of Plants Reference Materials.

Sample	Argon Carrier Gas					Helium Carrier Gas				
	Weight (mg)	N%	RSD%	C%	RSD%	Weight (mg)	N%	RSD%	C%	RSD%
Birch Leaves Ref. Mat.	15–16	2.16 2.13 2.16	0.81	48.32 48.55 48.59	0.24	4–5	2.09 2.09 2.10	0.27	48.06 48.13 48.25	0.20
Orchard Leaves Ref. Mat.	15–16	2.25 2.24 2.23	0.45	50.11 50.21 50.53	0.56	4–5	2.27 2.28 2.30	0.75	50.53 50.50 50.41	0.12
Alfalfa Ref. Mat.	15–16	2.98 2.94 2.96	0.68	43.60 43.59 43.92	0.42	4–5	2.91 2.89 2.88	0.53	43.65 43.57 43.68	0.13

Conclusions

Good repeatability and accuracy were obtained with the Thermo Scientific Flash*Smart* Elemental Analyzer using argon as carrier gas. Outcomes from the analyses using argon as carrier gas have confirmed the excellent compatibility with the results obtained when using helium.

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