Nitrogen and Carbon Determination in Soils and Plants by the Thermo Scientific FLASH 2000 Analyzer Using Argon as Carrier Gas

Application Note 42219

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Key Words

Argon, Carrier Gas, Combustion, Elemental Analysis, NC, Plants, Soils

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 2000 Analyzer with argon as carrier gas

Introduction

Nitrogen and carbon determination in soils is important for the evaluation of the organic matters and for the determination of fertilizers needed. They also provide significant information on the nutritional elements of plants, which are key factors for their future growth. The determination of nitrogen content allows to control the quality of different types of crop, used for feeding and processing, as well as for N-cycle and N-fixation monitoring in agricultural and environmental research.

The importance of soil 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 which allows fast analysis with an excellent reproducibility is the key for efficient nitrogen and carbon determination.

Figure 1. Thermo Scientific FLASH 2000 NC Soils Analyzer.

The Thermo Scientific™ FLASH™ 2000 Analyzer (Figure 1), using helium gas carrier and operating with the dynamic flash combustion of the sample, meets the laboratory requirements, such as accuracy, reproducibility and high sample throughput. However, the potential shortage and rise in the cost of helium has increased the need for alternative gas, such as argon.

This application note reports data on nitrogen and carbon determination in soils and plants reference materials in different concentrations and it shows the performance of the FLASH 2000 Analyzer using Argon as carrier gas and the reproducibility of its results.

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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™ 200R Autosampler with the proper amount of oxygen. After the 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 and finally detected by a Thermal Conductivity Detector (TCD). The analytical configuration as well as the TCD detector are the same as those used with helium as carrier gas (Figure 2).

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

Results

Soils and plants reference materials with different nitrogen and carbon concentrations were analyzed in order to compare the results of the FLASH 2000 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 weighed 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.

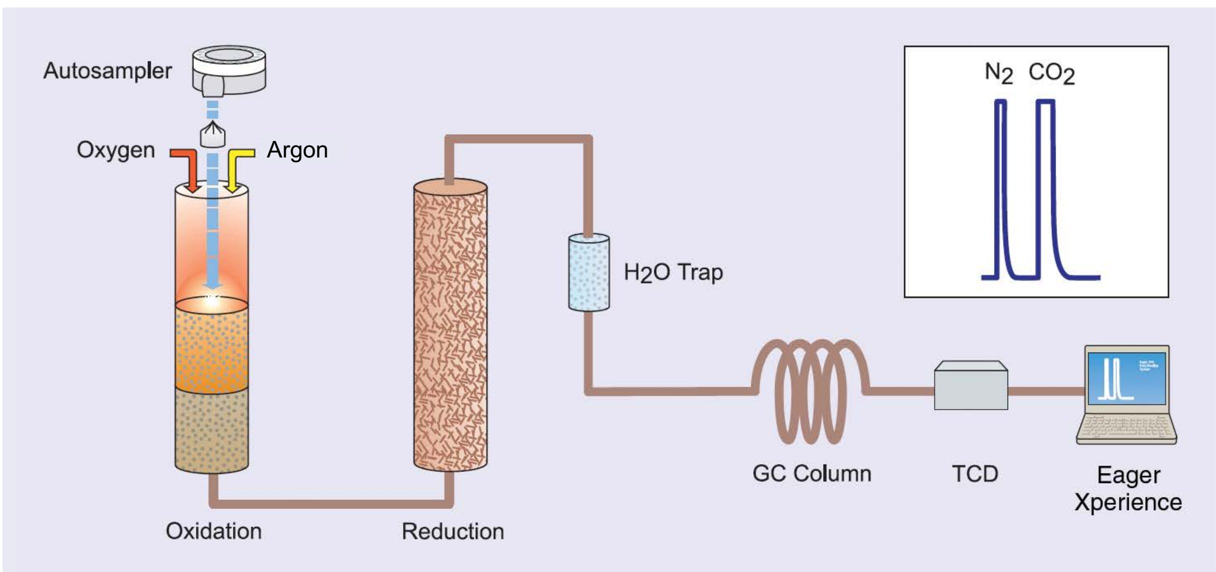

Figure 2. Thermo Scientific FLASH 2000 Analyzer nitrogen/carbon configuration.

Analytical Conditions	
Combustion Furnace Temperature:	950 °C
Reduction Furnace Temperature:	840 °C
Oven Temperature:	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 (%)
		0.132		1.64			0.124		1.55	
Low Organic Content Soil Ref. Mat.	150–200	0.133	0.435	1.61	1.54	90–100	0.122	2.024	1.55	0.74
		0.133		1.66			0.127		1.57	
		0.28		3.22			0.27		3.16	
Medium Organic Content Soil Ref. Mat.	100–150	0.28	0	3.19	0.65	90–100	0.27	0	3.15	0.18
		0.28		3.18			0.27		3.15	
		0.28		2.76			0.26		2.70	
Loamy Soil Ref. Mat.	100–150	0.28	0	2.70	1.53	90–100	0.26	0	2.72	0.56
		0.28		2.68			0.26		2.69	
		0.38		5.33			0.38		5.34	
Chalky Soil Ref. Mat.	80–90	0.38	1.51	5.45	1.16	90–100	0.37	1.53	5.35	0.11
		0.39		5.36			0.38		5.35	

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 weighed 150–250 mg, while 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. Results were very satisfactory.

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		0.8566		0.0662		0.8162	
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	1.5085	0.8455	1.7822	0.0664	1.0824	0.8154	1.0234
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	

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 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

Conclusion

Good repeatability, accuracy and precision were obtained with the Thermo Scientific FLASH 2000 NC Soils Analyzer using argon as carrier gas. Outcomes from the analysis using argon as carrier gas have confirmed the excellent compatibility with the results obtained using helium, while reducing overall operational costs.

No memory effect was observed when changing the sample, meaning the total combustion and detection of the elements.

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