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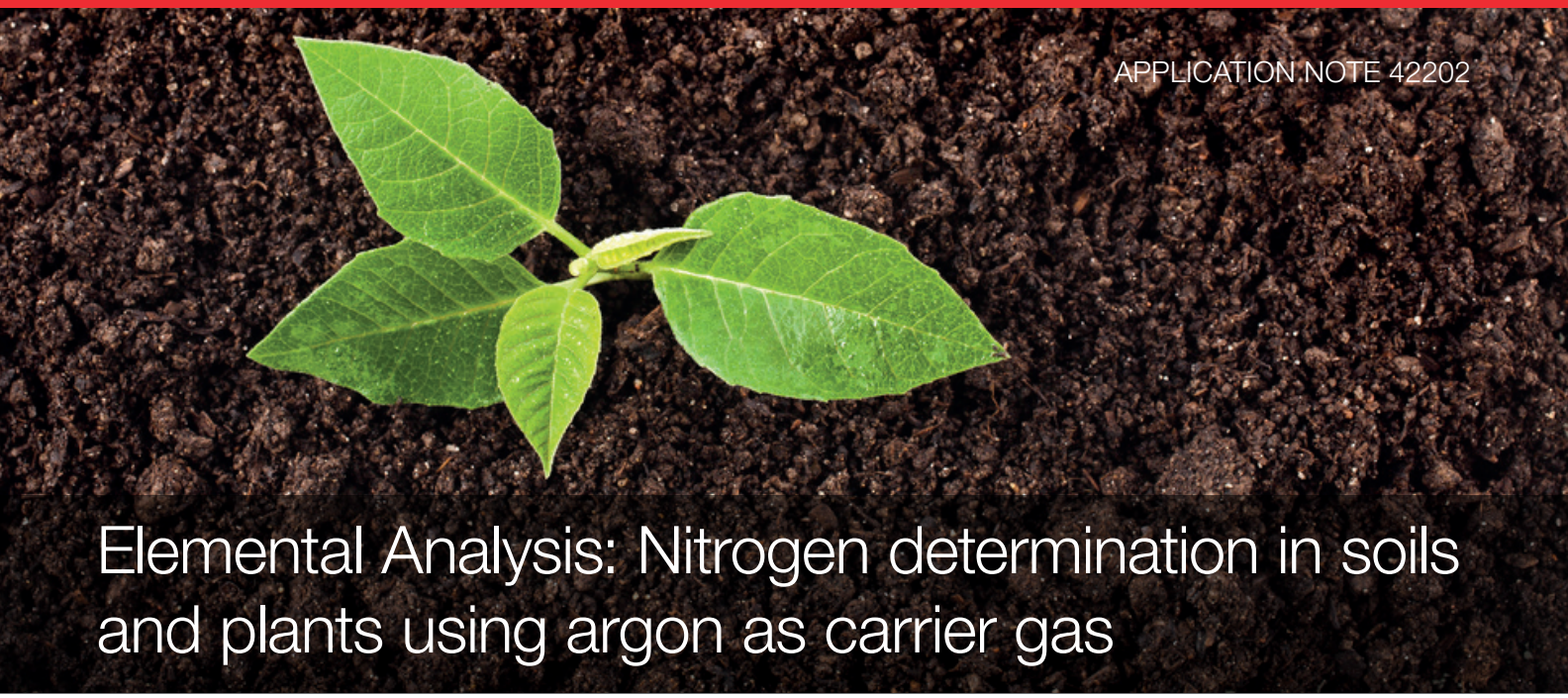

APPLICATION NOTE 42202

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

Authors

Dr. Liliana Krotz, Walter Galotta, and Dr. Guido Giazzi, OEA Thermo Fisher Scientific, Milan, Italy

Keywords

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

Goal

This application note presents data on nitrogen determination in soil and plant reference materials with different nitrogen concentrations to demonstrate the performance of the Flash*Smart* EA using argon as an alternative carrier gas and show the reproducibility of the results obtained.

Introduction

Nitrogen determination in soils is important for the evaluation of the organic matter and the calculation of the amount of fertilizer needed. The determination of nitrogen content provides information regarding the deficiency or excess of nutritional elements important for plant growth. Nitrogen content is important for determining the quality of various types of crops for feeding and processing, as well as for N-cycle and N-fixation monitoring in agricultural and environmental research.

As the demand for improved sample throughput and reduction of operational costs is increasing, it is essential to have an automated technique, which allows fast analysis with excellent reproducibility.

The Thermo Scientific™ Flash*Smart*™ Elemental Analyzer (Figure 1), copes with modern laboratory requirements such as accuracy, day to day reproducibility and high sample throughput. It operates according to the dynamic flash combustion of the sample and typically uses helium as carrier gas.

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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.

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

Methods

Samples are weighed in tin containers and introduced into the combustion reactor via the Thermo Scientific™ MAS Plus Autosampler with oxygen. After combustion, the resultant gases are carried by an argon flow to a second reactor filled with copper, then swept through CO_2 and H_2O traps, a GC column and are 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 the carrier gas (see Figure 2).

A complete report is automatically generated by the Thermo Scientific™ Eager*Smart*™ Data Handling Software. The Eager*Smart* Data Handling Software provides the option AGO (Argon Gas Option), enabling to modify the argon carrier flow during the run.

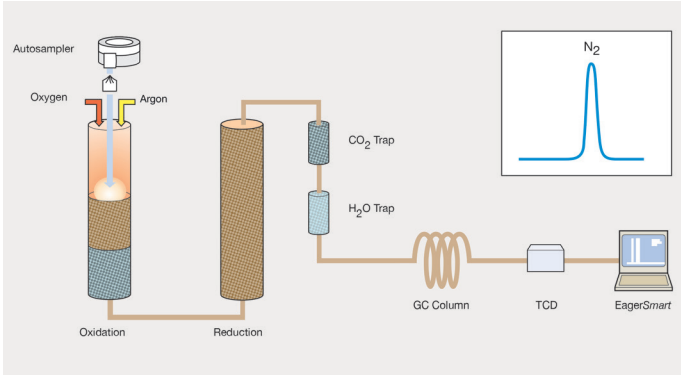

Figure 2. Nitrogen configuration.

Analytical Conditions	
Combustion Furnace Temperature	950 °C
Reduction Furnace Temperature	840 °C
Oven Temperature	50 °C (GC column inside the oven)
Argon Carrier Flow	60 ml/min
Argon Reference Flow	60 ml/min
Oxygen Flow	300 ml/min
Sample Delay	10 sec
Run Time	10 minutes

Note: The oxygen amount necessary for the complete combustion of samples is calculated automatically by the dedicated feature (OxyTune Function) of the Eager*Smart* Data Handling Software:

Results

To evaluate the performance of the system for soils and plants analysis, relative reference materials with different nitrogen concentrations were chosen. Instrument calibration was performed with approximately 50 – 70 mg of aspartic acid (10.52 N%) standard, using K factor as the calibration method.

Table 1 shows the nitrogen data obtained of the analysis of Thermo Scientific Soil Reference Material. The sample was weighed at 250 – 350 mg. The certified N% is 0.21 and the accepted range according to the technical specification is 0.19 – 0.23 N%. The average of the 12 runs is 0.19 N% with an RSD% of 2.55.

Table 1. Nitrogen data of Soil Reference Material.

Weight (mg)	N%
250.224	0.20
260.746	0.20
270.494	0.19
300.714	0.19
310.893	0.19
320.423	0.19
270.794	0.19
280.101	0.19
290.134	0.19
300.844	0.19
320.242	0.20
350.896	0.20

Four Soil Reference Materials from 0.13 to 0.35 N% were chosen to correlate the experimental results to the expected values. Table 2 shows the certified N% and the uncertainty factor. Table 3 shows the weight of samples analyzed and the experimental N% obtained.

Table 2. Expected N% of Soil Reference Materials.

Sample Description	Specification	
	N%	Uncertainty (±)
Low Organic Content Soil Reference Material	0.133	0.023
Medium Organic Content Soil Reference Material	0.27	0.02
Loamy Soil Reference Material	0.27	0.02
Chalky Soil Reference Material	0.35	0.02

Table 3. Experimental nitrogen data of Soil Reference Materials.

Sample Description	Weight	Experimental	RSD
	(mg)	N%	%
Low Organic Content Soil Reference Material	351.931 307.521 340.210	0.117 0.113 0.112	2.321
Medium Organic Content Soil Reference Material	285.120 336.816 322.010	0.264 0.258 0.265	1.443
Loamy Soil Reference Material	322.020 326.020 340.567	0.265 0.271 0.268	0.119
Chalky Soil Reference Material	254.416 326.987 350.471	0.337 0.365 0.372	5.173

A Sandy Soil Reference Material at 0.07 N% (700 ppm N) was chosen to evaluate the method at trace level. Table 4 shows the weight of samples analyzed, the experimental N% obtained, the certified N% and the uncertainty factor.

Table 4. Nitrogen data of Sandy Soil Reference Material.

Sample	Flash*Smart* EA		Specification	
Weight (mg)	Experimental N%	RSD%	N%	Uncertainty (±)
408.698	0.0700			
402.849	0.0711			
398.636	0.0703			
409.065	0.0707			
408.650	0.0691	1.8848	0.07	0.01
412.241	0.0718			
403.313	0.0694			
401.651	0.0692			
400.316	0.0718			
400.420	0.0732			

Four Plant Reference Materials from 0.15 to 3.01 N% were chosen to correlate the experimental results to the expected values. Table 5 shows the certified N% and the uncertainty.

Table 5. Expected nitrogen values of Plant Reference Materials.

Sample Description	Specification	
	N%	Uncertainty (±)
Birch Leaves	2.12	0.06
Orchard Leaves	2.28	0.04
Alfalfa	3.01	0.2

Table 6 shows the experimental N% obtained for birch and orchard leaves.

The nitrogen average data of the ten runs fall within the uncertainty range.

Table 6. Experimental nitrogen data.

Sample	Birch Leaves		Orchard Leaves		Alfalfa	
	Weight (mg)	N%	Weight (mg)	N%	Weight (mg)	N%
	203.77	2.11	100.46	2.24	120.889	3.06
	162.55	2.12	120.53	2.24	121.957	3.07
	162.34	2.11	120.21	2.26	119.196	3.09
	165.10	2.13	140.23	2.30	122.920	3.04
	165.08	2.12	140.32	2.25	118.133	3.03
	169.53	2.14	140.34	2.28	110.219	2.99
	170.05	2.13	150.60	2.31	105.062	2.97
	150.80	2.11	140.39	2.26	120.706	3.08
	153.99	2.11	140.20	2.30	119.268	3.09
	160.78	2.11	139.63	2.29	118.429	3.05
Average N%		2.12		2.27		3.05
RSD%		0.52		1.16		1.34

Conclusions

Good repeatability, accuracy and precision were obtained with the Flash*Smart* Elemental Analyzer using argon as carrier gas.

No memory effect was observed when changing the type of sample, indicating complete combustion and detection of the element.

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