Nitrogen Determination in Soils and Plants by Flash Combustion using Argon as Carrier Gas

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Overview

Purpose: To show nitrogen determination of soils and plants by Organic Elemental Analysis (OEA) using argon as the carrier gas.

Methods: Soils and plants were analyzed by an elemental analyzer with an automatic autosampler, using argon as the carrier gas.

Results: Data collected of nitrogen from different soils and plants are discussed to assess the performance of the elemental analyzer using argon as the carrier gas.

Introduction

Nitrogen in soils is important for the evaluation of organic matter and the calculation of the amount of fertilizer to be added by providing information regarding the deficiency or excess of nutritional elements important for plant growth. Nitrogen content is important in 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.

For this reason the use of an accurate instrumental analytical technique is required, as the demand for improved sample throughput, reduction of operational costs and minimization of human errors is becoming every day more notable, it is essential to have a simple and automatic technique which allows fast analysis with excellent reproducibility.

The Thermo Scientific™ FLASH 2000 analyzer (Figure 1), which typically uses helium as its carrier gas and is based on the dynamic flash combustion of the sample, copes effortlessly with the wide array of laboratory requirements such as accuracy, day to day reproducibility and high sample throughput. However as in recent years there have been worldwide shortages and large increases in the cost of helium, and it has been necessary to test an alternative gas, argon, which is readily available.

This paper presents data on nitrogen determination in soil and plant reference materials with varying nitrogen concentrations to show the performance of the system using argon as the carrier gas, and the reproducibility of the results obtained.

FIGURE 1. FLASH 2000 Elemental Analyzer

Methods

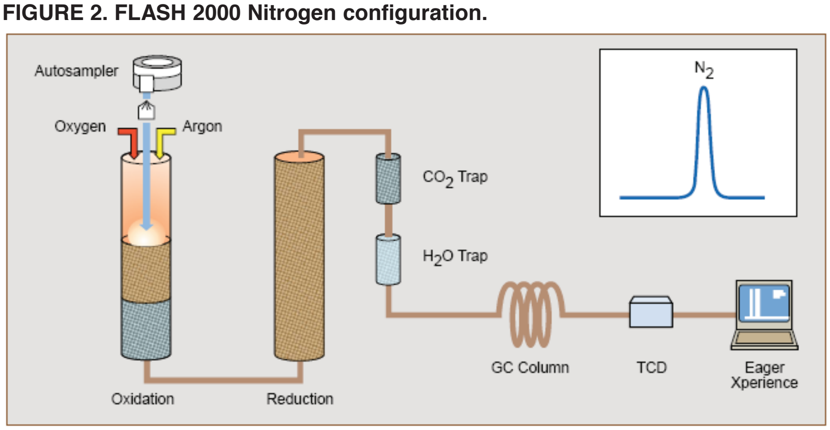
Samples are weighed in tin capsules and introduced into the combustion reactor via the Thermo Scientific™ MAS™ 200R autosampler together with the proper amount of oxygen.

After combustion, the produced 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, finally being 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 (Figure 2).

A complete report is automatically generated by the Thermo Scientific™ Eager Xperience data handling software and displayed at the end of the analysis.

The Eager Xperience software provides a new *AGO (Argon Gas Option)* function which allows the modification of the argon carrier flow during the run to optimize the analysis.

FIGURE 2. FLASH 2000 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 min

Note: The oxygen amount necessary for the complete combustion of samples is calculated automatically by the Thermo Scientific™ OxyTune™ function present in the Eager Xperience software.

Results

To evaluate the performance of the system for soil and plant 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 calibration method.

Table 1 shows the nitrogen data obtained of the analysis of Thermo Scientific™ Soil Reference Material. The sample weight was 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 a RSD % of 2.55.

TABLE 1. Nitrogen data of Thermo Scientific 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. Table 3 shows the weight of sample used and the experimental N % obtained which was very satisfactory.

TABLE 2. Expected N % of Soil Reference Materials.

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

TABLE 3. Experimental Nitrogen data of Soil Reference Materials.

Sample Description	Weight (mg)	Experimental N %	RSD %
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 sample used, the experimental N % obtained, the certified N % and the uncertainty.
The data obtained was very satisfactory.

TABLE 4. Nitrogen data of Sandy Soil Reference Material.

Sample	FLASH 2000		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			

Three 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 Material.

Sample Description	Specification	
	N %	Uncertainty (±)
Birch Leaves	2.12	0.06
Orchard Leaves	2.28	0.04
Alfalfa	3.01	0.20

Table 6 shows the experimental N% obtained which were very satisfactory.
The nitrogen average of the ten runs is inside the uncertainty.

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
	162.55	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
	153.99	2.11	139.63	2.29	118.429	3.05
Av. N %		**2.12**		**2.27**		**3.05**
RSD %		**0.519**		**1.156**		**1.34**

Figure 3 shows a typical calibration with Aspartic acid using K factor as calibration method

FIGURE 3. Typical calibration curve.

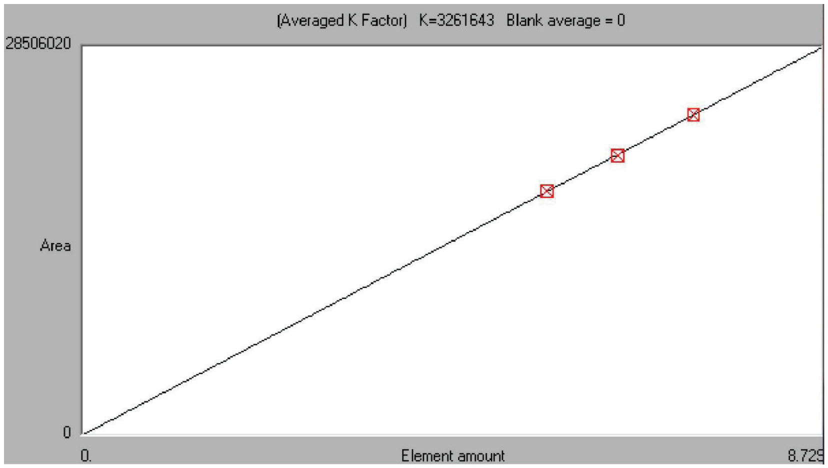
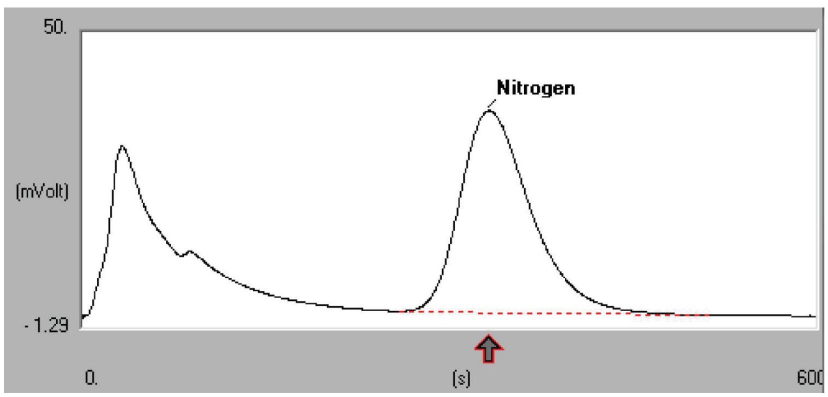
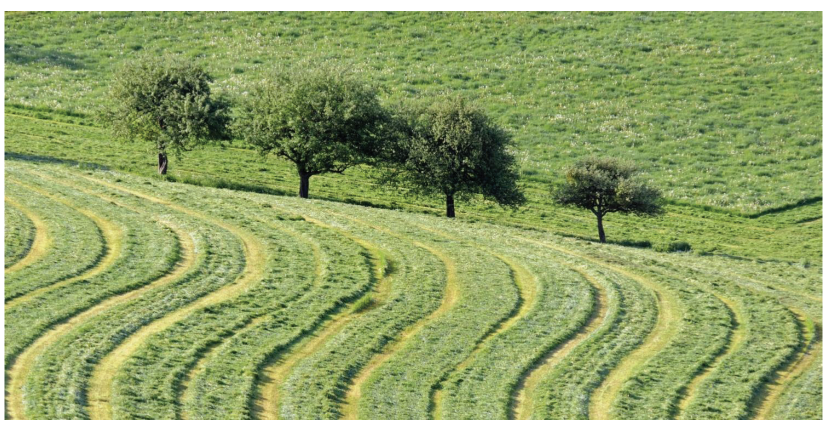


Figure 4 shows a typical nitrogen chromatogram

FIGURE 4. Typical Nitrogen chromatogram.





Conclusion

Good repeatability, accuracy and precision was obtained with the FLASH 2000 Nitrogen Analyzer using argon as the 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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