Nitrogen and Carbon Determination of Soils and Plants by the Thermo Scientific FLASH 2000 Analyzer Using a Single Reactor

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Overview

Purpose: Demonstrate the nitrogen and carbon determination by Organic Elemental Analysis (OEA) using a single combustion/reduction reactor.

Methods: Soils and plants reference materials were analyzed through an elemental analyzer with automated autosampler using a single reactor.

Results: Data collected of nitrogen and carbon from different soils and plants reference material are discussed to assess the performance of the OEA analyzer using a single reactor without matrix effect.

Introduction

Nitrogen and carbon determination by combustion analysis is very commonly used for soils, plants, leaves, sediments, deposit on filters, animal tissues analysis and marine sciences analysis because these elements provides important information for their characterization in agricultural and environmental research.

The importance of soil and plant testing has increased in the last 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.

The Thermo Scientific™ FLASH™ 2000 Analyzer (Figure 1), operating with the dynamic flash combustion of the sample, meets the laboratory requirements, such as accuracy, reproducibility and high sample throughput. The standard configuration is based on a double reactors system: first reactor for combustion and catalytic oxidation of the combustion gases, the second is used to reduce nitrous oxides as N_2.
The superior performance of the Thermo Scientific FLASH 2000 Analyzer allows reducing the amount of oxidation catalyst needed for NC analysis using a single reaction tube. The reactor filled with less amount of oxidation catalyst and copper ensures the complete conversion of gases produced by the combustion and offers advantages as large numbers of analysis before the maintenance, shorter analytical circuit allowing faster analysis, and the possibility to install two analytical circuits which are used alternatively (for example NC in the left furnace and CHNS/NCS in the right furnace, or NC in the left furnace and Sulfur by FPD detector in the right furnace) in a single analyzer. Using two autosamplers, the switching time from one analytical circuit to the second is only of few seconds, without the need for tools or mechanical modification.

FIGURE 1. Thermo Scientific FLASH 2000 NC Analyzer.

Methods

The FLASH 2000 Analyzer operates according to the dynamic flash combustion method. Samples are weighed in tin containers and introduced into the combustion reactor via the Thermo Scientific™ MAS 200R Autosampler together with oxygen. For NC *single reactor configuration*, after combustion, the resultant gases are carried by a helium flow to oxidation catalyst and to reduced copper, and finally pass through a halogen and sulfur absorber located inside the reaction tube.

After the reaction tube, water is trapped; a GC column separate gases and finally gases are detected by a sensitive and stable thermal conductivity detector (TCD) (Figure 2). A complete report is automatically generated by the dedicated Thermo Scientific™ Eager Xperience Data Handling Software and displayed at the end of the analysis.

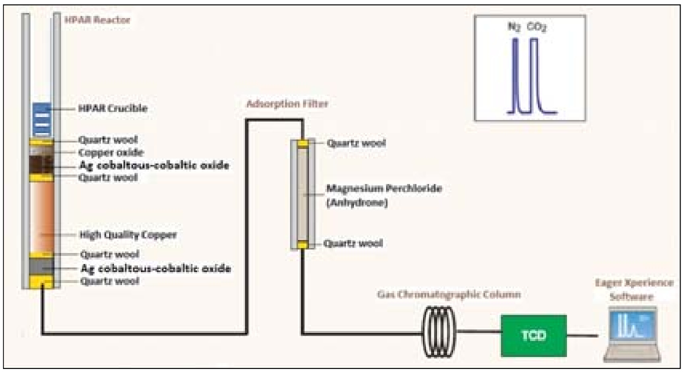

FIGURE 2. FLASH 2000 NC configuration.

To allow a large range of amount of nitrogen and carbon detected with a high quality of separation of these two elements, a three-meter GC column installed in the oven is used. Figure 3 shows a typical NC chromatogram.

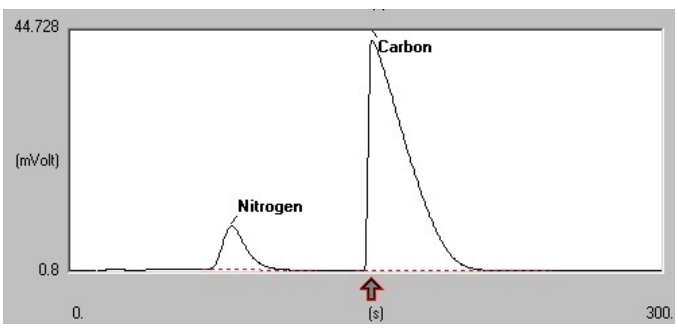

FIGURE 3. Typical NC chromatogram.

Results

Soils and plants reference materials with different nitrogen and carbon concentrations were analyzed in order to evaluate the performance of the single reactor. Table 1 shows the certified N % and C % and the relative uncertainty.

Reference Materials Description	Specification			
	N %	Uncertainty (±)	C %	Uncertainty (±)
Thermo Scientific Soil	0.21	0.01	2.29	0.07
Low Organic Content Soil	0.133	0.023	1.61	0.09
Medium Organic Content Soil	0.27	0.02	3.19	0.07
Loamy Soil	0.27	0.02	2.75	0.12
Chalky Soil	0.35	0.02	5.39	0.09
Sandy Soil	0.07	0.01	0.83	0.05
Birch Leaves	2.12	0.06	48.09	0.51
Orchard Leaves	2.28	0.04	50.40	0.40
Alfalfa	3.01	0.20	na	-

TABLE 1. Certified Nitrogen and Carbon of Reference Materials.

Table 2 shows the calibration sequence for soil analyses while Table 3 shows the weight of Soils Reference Materials analyzed and the experimental results obtained using Quadratic Fit or Linear Fit as calibration method. All results are obtained with a good repeatability and the averages values are inside the specification of the reference materials certificates.

Run	Standard	Type	Weight	Theoretical Value	
			mg	N %	C %
1	Benzoic acid	STD	5.974	-	68.85
2	Benzoic acid	STD	6.455	-	68.85
3	Aspartic acid	STD	6.548	10.52	36.09
4	Aspartic acid	STD	5.799	10.52	36.09
5	Aspartic acid	STD	2.528	10.52	36.09
6	Aspartic acid	STD	2.092	10.52	36.09
7	TF Soil ref. mat	STD	6.288	0.21	2.29

TABLE 2. Calibration sequence for soils analysis.

Sample Information		Quadratic Fit Calibration Method				Linear Fit Calibration Method			
Reference Material	W (mg)	N%	RSD%	C%	RSD %	N%	RSD %	C%	RSD%
Thermo Scientific Soil	54.879 62.212 59.210	0.21 0.20 0.20	2.84	2.26 2.23 2.25	0.68	0.20 0.20 0.20	0	2.27 2.25 2.27	0.51
Low Organic Content Soil	52.310 59.495 56.449	0.12 0.12 0.13	4.68	1.57 1.56 1.59	0.97	0.12 0.12 0.13	4.68	1.58 1.56 1.59	0.97
Medium Organic Content Soil	57.837 66.428 70.375	0.27 0.27 0.27	0	3.15 3.15 3.16	0.18	0.27 0.27 0.26	2.17	3.17 3.16 3.18	0.32
Loamy Soil	66.068 56.356 65.322	0.26 0.26 0.26	0	2.67 2.69 2.68	0.37	0.25 0.25 0.26	2.28	2.68 2.71 2.70	0.57
Chalky Soil	53.043 59.841 67.876	0.37 0.37 0.37	0	5.32 5.34 5.37	0.47	0.36 0.36 0.37	1.59	5.34 5.35 5.37	0.28
Sandy Soil	240.058 255.114 229.087	0.065 0.066 0.066	0.88	0.813 0.817 0.818	0.32	0.064 0.065 0.065	0.89	0.818 0.821 0.823	0.31

TABLE 3. Experimental Nitrogen and Carbon data of Soils Reference Materials.

Table 4 shows the calibration sequence for plants analyses while Table 5 shows the weight of Plant Reference Materials and the experimental results obtained using Quadratic Fit or Linear Fit as calibration method. All results are obtained with a good repeatability and the values of the reference materials are inside the specification.

Run	Standard	Type	Weight	Theoretical Values	
			mg	N %	C %
1	Benzoic acid	STD	6.442	-	68.85
2	Benzoic acid	STD	6.749	-	68.85
3	Aspartic acid	STD	7.796	10.52	36.09
4	Aspartic acid	STD	7.03	10.52	36.09
5	Aspartic acid	STD	2.342	10.52	36.09
6	Aspartic acid	STD	2.21	10.52	36.09
7	Acetanilide	STD	11.756	10.36	71.09
8	Acetanilide	STD	12.772	10.36	71.09
9	Acetanilide	STD	5.233	10.36	71.09

TABLE 4. Calibration sequence for plant analysis.

Sample Information		Quadratic Fit Calibration Method				Linear Fit Calibration Method			
Reference Material	W (mg)	N%	RSD%	C%	RSD%	N%	RSD%	C%	RSD%
Birch Leaves	16.185 15.952 16.746	2.17 2.16 2.16	0.27	48.45 48.37 48.41	0.08	2.19 2.19 2.20	0.26	48.47 48.39 48.44	0.08
Orchard Leaves	16.777 15.214 15.878	2.28 2.29 2.29	0.25	50.40 50.34 50.19	0.22	2.30 2.32 2.31	0.43	50.43 50.36 50.21	0.22
Alfalfa	18.079 20.441 20.585	3.03 3.03 3.02	0.19	43.06 43.06 43.15	0.12	3.01 3.01 3.00	0.19	43.07 43.06 43.15	0.11

TABLE 5. Experimental Nitrogen and Carbon data of Plants Reference Materials.

Table 6 shows the NC data of other matrices analyzed to evaluate the repeatability. No memory effect was observed when changing the sample matrix indicating that the combustion is complete at this higher weight of sample.

Sample	W (mg)	N%	RSD%	C%	RSD%
France Maize	19.393 19.908 20.273	1.21 1.21 1.21	0	42.02 41.96 41.87	0.18
Russian Maize	19.580 18.960 19.001	1.55 1.55 1.55	0	43.14 43.12 43.03	0.14
Soja	19.626 19.036 19.226	7.93 7.95 7.95	0.15	41.82 41.93 41.43	0.77
Alfalfa	19.858 19.767 19.742	2.53 2.56 2.56	0.68	44.00 43.48 43.74	0.59
Sunflower	19.222 19.765 20.313	5.81 5.77 5.82	0.46	43.68 43.61 43.69	0.10
Oatmeal	20.354 20.067 20.152	1.87 1.83 1.82	1.43	42.56 42.54 42.39	0.22
Soja Bean	20.333 20.656 20.179	7.59 7.56 7.54	0.33	43.20 43.20 43.20	0
Barley flour	20.188 20.349 20.119	1.83 1.81 1.85	1.09	41.56 41.55 41.60	0.06
Wheat Flour	20.441 20.513 19.914	1.37 1.38 1.37	0.42	40.00 40.03 40.06	0.07
Rice Flour	20.162 20.267 20.201	1.35 1.33 1.38	1.26	40.74 40.70 40.75	0.13

TABLE 6. Experimental Nitrogen and Carbon data of other matrices.

Conclusion

Using one large reaction tube coupled with the 3 m length GC column, instead of the classical two reactors configuration, extended the analytical capabilities of the Thermo Scientific FLASH 2000 Elemental Analyzer:

- High quality of results
- Faster NC analysis
- Larger sample weight for vegetal
- Easier maintenance
- High productivity: possibility to use a single reactor in the left furnace and another reactor in the right furnace for NC/NC or NC/Sulfur determinations and others.

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