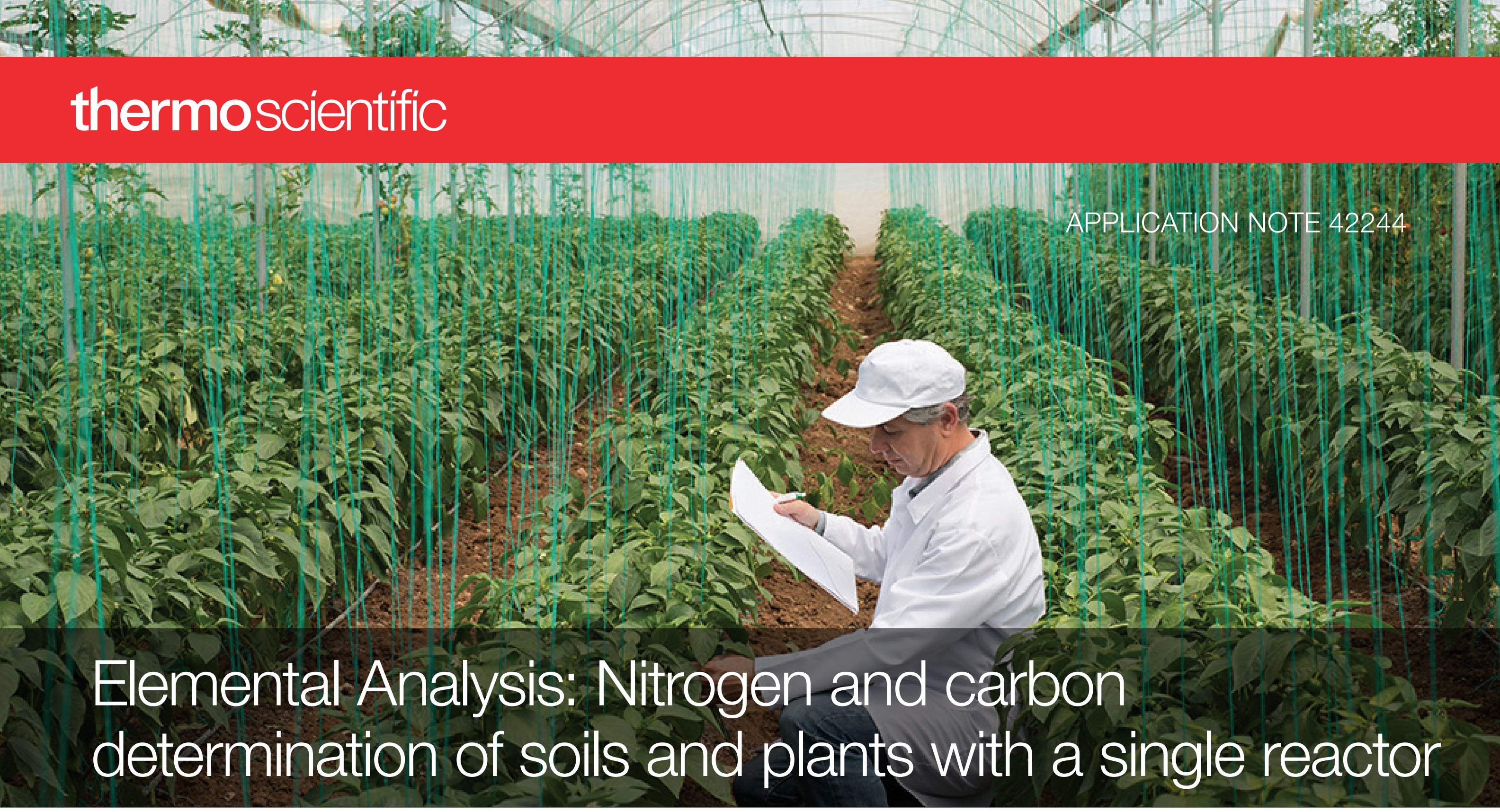

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APPLICATION NOTE 42244

Elemental Analysis: Nitrogen and carbon determination of soils and plants with a single reactor

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Keywords

Carbon, Combustion, Nitrogen, Plants, Soils, Vegetals

Goal

This application note shows nitrogen and carbon determination for soils and plants using the Flash*Smart* EA in single combustion/reduction reactor configuration.

Introduction

Nitrogen and carbon determination by combustion analysis is very common for soils, plants, leaves, sediments, filtered material and animal tissues. Nitrogen and carbon provide important information for 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, for their time-consuming sample preparation and for the required use of 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*Smart*™ Analyzer (Figure 1), operating with the dynamic flash combustion of the sample, meets modern laboratory requirements. The standard configuration is based on a double reactor system: the first reactor is used for combustion and catalytic oxidation of the combustion gases, the second is used to reduce nitrous oxides to N_2. The Flash*Smart* EA Analyzer allows the reduction of the amount of

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oxidation catalyst needed for NC analysis using a single combustion/reduction reactor tube (25 mm diameter). The reactor filled with less amount of oxidation catalyst and copper ensures the complete conversion of gases produced by the combustion. This application note shows the performance of the Flash*Smart* EA for nitrogen and carbon determination of soils and plants by using a single reactor configuration.

Methods

The Flash*Smart* Elemental 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 Plus Autosampler with a proper amount of oxygen. For NC in the single-reactor configuration, after combustion the resultant gases are carried by a helium flow to oxidation catalyst and to reduced copper. Finally, it passes through a halogen and sulfur absorber located inside the reaction tube. After the reaction tube, water is trapped. A GC column separates gases by a Thermal Conductivity Detector (TCD) (Figure 2). A comprehensive report is generated by the dedicated Thermo Scientific™ Eager*Smart*™ Data Handling Software.

Analytical Conditions

For the detection of a large amount of nitrogen and carbon be detected, a 3-meter GC column is used. Figure 3 shows a typical NC chromatogram.

Three tests show the performance of the single-reactor configuration of soils and plants samples in different weights. In Test A and B the sample weight is similar to the one used in the configuration with double reactors but using the conditions of Test B the analysis is faster. Test C was developed for higher sample weight of vegetals. The analytical parameters are as shown in Table 1.

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

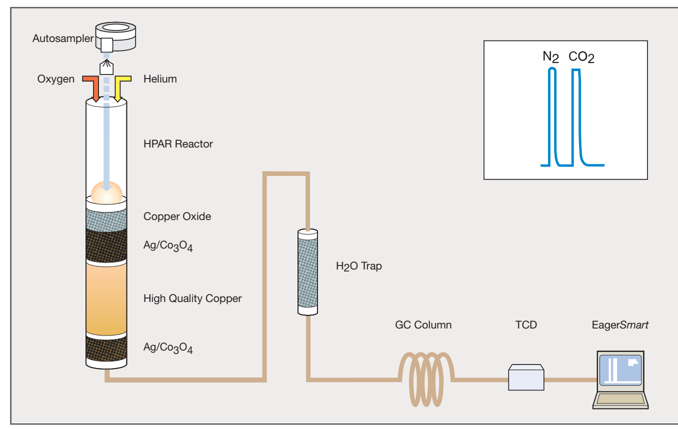

Figure 2. NC determination.

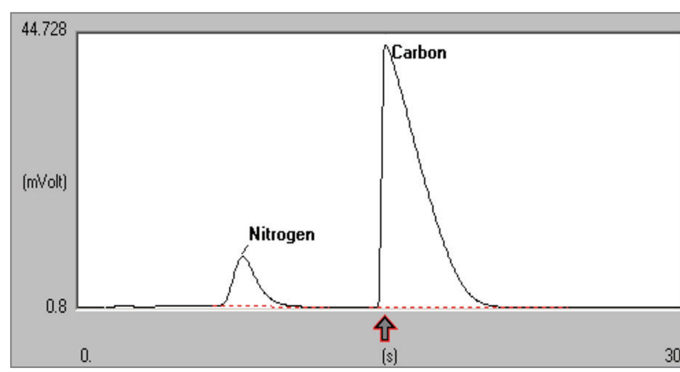

Figure 3. Typical NC chromatogram.

Table 1. Analytical parameters.

Test	Test A	Test B "Fast"	Test C
Sample Weight	50–70 mg soils, 250–300 mg sand, 5–7 mg plants	50–70 mg soils, 250–300 mg sand, 5–7 mg plants	About 20 mg of vegetals
Furnace Temperature	950 °C	95 °C	950 °C
Oven Temperature	50 °C	65 °C	50 °C
Helium Carrier Flow	200 mL/min	200 mL/min	170 mlL/min
Helium Reference Flow	40 mL/min	40 mL/min	40 mL/min
Oxygen Flow	160 mL/min	160 mL/min	300 mL/min
Analysis Time	330 sec	270 sec	420 sec
Oxygen Injection Time	12 sec	12 sec	9 sec
Sampling Delay	20 sec	20 sec	17 sec

Table 2. Certified nitrogen and carbon of reference materials.

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	na
Oatmeal	1.90	0.10	45.51	0.17

Results

Soil and plant reference materials with different nitrogen and carbon concentrations were analyzed in order to evaluate the performance of the single reactor following A, B and C test conditions. Table 2 shows the certified N% and C% and the relative uncertainty.

Test A

The sequence of analyses for the calibration is showed in Table 3. Table 4 shows the weight of reference materials analyzed and the experimental results obtained using quadratic fit or linear fit as the calibration method.

Table 3. Calibration sequence for Test A.

Run	Standard	Type	Weight (mg)	Theoretical Values	
				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	Soil ref. material	STD	6.288	0.21	2.29
8	Soil ref. material	STD	8.467	0.21	2.29

Table 4. Experimental nitrogen and carbon data of reference materials.

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
Birch Leaves	6.788 7.249 6.065	2.14 2.17 2.12	1.17	47.93 47.92 48.03	0.13	2.11 2.13 2.10	0.72	48.04 47.99 48.19	0.22
Orchard Leaves	6.445 6.615 5.809	2.30 2.31 2.31	0.25	50.00 50.21 50.24	0.26	2.27 2.27 2.28	0.25	50.12 50.31 50.41	0.29
Alfalfa	6.210 7.124 6.133	2.92 3.02 2.97	1.68	43.01 42.93 42.88	0.15	2.87 2.97 2.92	1.71	43.18 43.06 43.06	0.16
Oatmeal	6.001 6.428 6.567	2.02 1.99 1.98	1.04	45.44 45.38 45.69	0.36	1.99 1.97 1.95	1.02	45.61 45.53 45.84	0.35

Test B

The sequence of analyses for the calibration is showed in Table 5. Table 6 shows the weight of reference materials analyzed and the experimental results obtained using quadratic fit or linear fit as calibration method. All results are obtained with good repeatability and the values fall within the specification of the reference materials certificates. Table 7 shows the NC data of other matrices to demonstrate the repeatability.

Table 5. Calibration sequence for Test B.

Run	Standard	Type	Weight (mg)	Theoretical Values	
				N%	C%
1	Benzoic acid	STD	5.977	0	68.85
2	Benzoic acid	STD	6.554	0	68.85
3	Aspartic acid	STD	7.222	10.52	36.09
4	Aspartic acid	STD	6.617	10.52	36.09
5	Aspartic acid	STD	2.799	10.52	36.09
6	Aspartic acid	STD	2.108	10.52	36.09
7	Soil ref. material	STD	6.688	0.21	2.29
8	Soil ref. material	STD	6.894	0.21	2.29

Table 6. Experimental nitrogen and carbon data of reference materials.

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	54.371 59.476 52.999	0.21 0.21 0.20	2.79	2.24 2.25 2.27	0.68	0.20 0.21 0.20	2.84	2.25 2.26 2.28	0.67
Low Organic Content Soil	67.572 54.559 55.945	0.12 0.13 0.12	4.68	1.60 1.611 .58	0.96	0.12 0.13 0.13	4.56	1.61 1.61 1.59	0.72
Medium Organic Content Soil	54.040 60.170 57.114	0.27 0.27 0.26	2.17	3.16 3.18 3.13	0.8	0.26 0.26 0.26	0	3.17 3.19 3.15	0.63
Loamy Soil	50.217 57.960 67.524	0.25 0.25 0.26	2.28	2.68 2.65 2.66	0.57	0.25 0.25 0.25	0	2.69 2.66 2.68	0.57
Chalky Soil	62.091 61.176 62.267	0.36 0.36 0.36	0	5.32 5.33 5.34	0.19	0.36 0.36 0.36	0	5.35 5.35 5.37	0.22
Sandy Soil	216.902 231.937 244.618	0.065 0.066 0.065	0.88	0.823 0.826 0.817	0.56	0.065 0.065 0.064	0.89	0.827 0.829 0.820	0.57
Birch Leaves	5.749 6.634 5.854	2.10 2.13 2.12	0.72	48.11 48.02 47.90	0.22	2.08 2.10 2.10	0.55	48.25 48.11 48.04	0.22
Orchard Leaves	5.845 5.849 5.823	2.28 2.28 2.27	0.25	50.63 50.59 50.39	0.25	2.26 2.26 2.25	0.26	50.72 50.72 50.52	0.25
Alfalfa	6.467 6.120 7.337	2.92 2.97 3.00	1.36	42.94 43.04 42.92	0.15	2.88 2.93 2.95	1.24	43.07 43.18 43.01	0.20
Oatmeal	5.987 6.154 6.544	1.98 1.98 2.01	0.87	45.41 45.32 45.62	0.34	1.97 1.96 1.99	0.77	45.65 45.57 45.86	0.33

Table 7. Experimental nitrogen and carbon data of other matrices.

Sample	W (mg)	N%	RSD%	C%	RSD%
Soyabean	5.884 5.775 5.917	7.50 7.52 7.40	0.86	43.07 42.97 42.98	0.12
Barley Flour	6.126 5.900 5.877	1.83 1.81 1.81	0.63	41.35 41.37 41.33	0.04
Wheat Flour	6.069 5.759 5.808	1.38 1.38 1.37	0.42	39.78 39.80 39.77	0.04
Rice Flour	6.013 5.814 5.910	1.43 1.40 1.41	1.08	40.23 40.26 40.26	0.05

Test C

The sequence of analyses for the calibration is showed in Table 8. Table 9 shows the weight of reference materials and the experimental results obtained using quadratic fit or linear fit as the calibration method. Table 10 shows the NC data of other matrices to demonstrate the repeatability of the results.

Table 8. Calibration sequence for Test C.

Run	Standard	Type	Weight (mg)	Theoretical Values	
				N%	C%
1	Benzoic acid	STD	6.442	0	68.85
2	Benzoic acid	STD	6.749	0	68.85
3	Aspartic acid	STD	7.796	10.52	36.09
4	Aspartic acid	STD	7.031	10.52	36.09
5	Aspartic acid	STD	2.342	10.52	36.09
6	Aspartic acid	STD	2.223	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 9. Experimental nitrogen and carbon data of reference materials.

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 10. Experimental nitrogen and carbon data of other matrices.

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

Conclusions

The Flash*Smart* Elemental Analyzer offers advantages over traditional methods for the nitrogen and carbon determination of soils and plants.

Higher-weight samples of organic matter (such as soils and plants) can be analyzed and the Flash*Smart* EA provides accurate results, which fall within the specifications of the systems.

Thanks to the double-reactor configuration the Flash*Smart* EA can perform NC/NC or NC/S determination in a single system, with no need for hardware changes.

Modern laboratories can perform NC/NC or NC/S efficiently while meeting the requirements of lower operational costs and reduced cost per sample.

Find out more at **thermofisher.com/OEA**

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