Nitrogen, Carbon and Sulfur Determination in Paper by Flash Combustion

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Overview

Purpose: To show the characterization of paper samples by Organic Elemental Analysis (OEA).

Methods: Paper samples were analyzed using an elemental analyzer with an automatic autosampler.

Results: Data collected of nitrogen, carbon and sulfur from different paper samples are discussed to assess the performance of the OEA analyzer.

Introduction

In the production process of paper, elemental composition is periodically monitored and tested for the characterization of raw and final products. Nitrogen and carbon are the most important parameters in quality control whilst the sulfur content is an indication of impurities present in the materials.

The Thermo Scientific™ FLASH 2000 analyzer (Figure 1) permits the fast, quantitative determination of elements in paper materials without any sample pre-treatment. The system, which is based on the dynamic combustion of the sample, provides automatic and simultaneous nitrogen, carbon and sulfur determination in a single analysis run.

FIGURE 1. FLASH 2000 Elemental Analyzer

Method

The sample is weighed in a tin capsule and introduced into the combustion reactor via the Thermo Scientific™ MAS™ 200R autosampler together with the proper amount of pure oxygen. In the NCS configuration, the gases produced after combustion of the sample are carried by a helium flow through a copper layer, and then swept through a water trap and a GC column that separates the combustion gases which are finally detected by a Thermal Conductivity Detector. Total run time is 10 minutes (Figure 2).

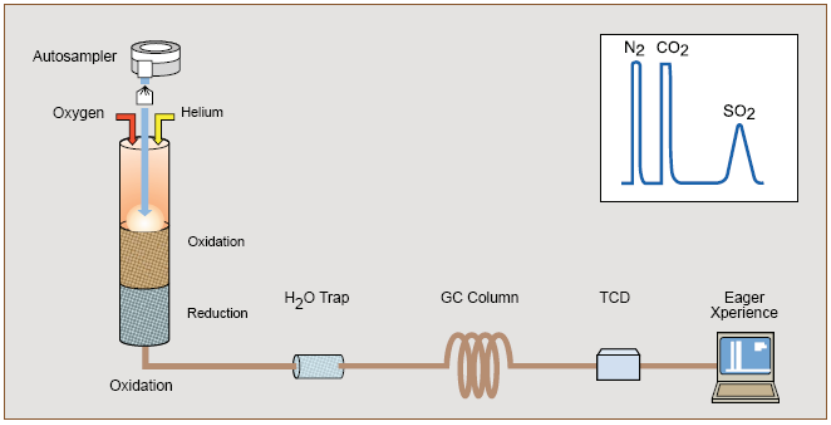
For nitrogen only determination, the gases produced after combustion are carried by a helium flow to a second reactor filled with copper, then swept through CO_2 and H_2O traps, then through a GC column and finally sensed by a Thermal Conductivity Detector. Total run time is less than 5 minutes (Figure 3).

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

Analytical Conditions – NCS determination

Reactor Temperature:	950 °C
Oven Temperature:	65 °C
Helium Carrier Flow:	140 ml/min
Helium Reference Flow:	100 ml/min
Oxygen Flow:	250 ml/min
Oxygen Injection End:	5 sec
Sampling Delay Time:	12 sec
Run Time:	600 sec
Standard:	2 - 3 mg BBOT
Sample Weight:	3 - 4 mg

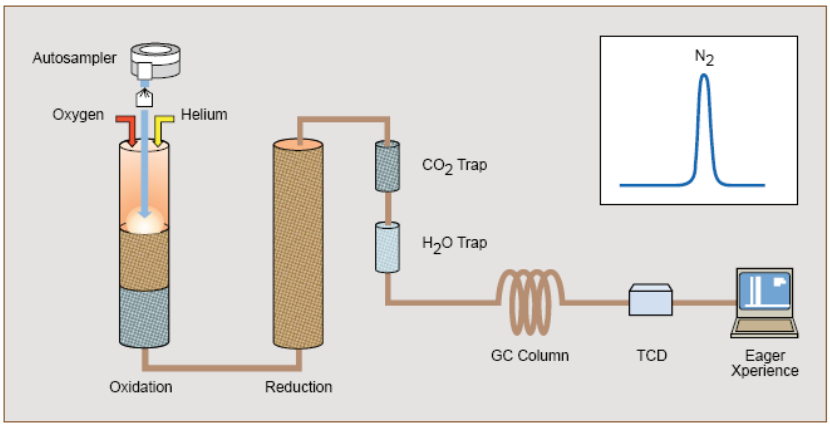
FIGURE 2. NCS configuration.



Analytical Conditions – NCS determination

Combustion Reactor Temperature:	950 °C
Reduction Reactor Temperature:	840 °C
Oven Temperature:	50 °C
Helium Carrier Flow:	140 ml/min
Helium Reference Flow:	100 ml/min
Oxygen Flow:	300 ml/min
Oxygen Injection End:	20 sec
Sampling Delay Time:	10 sec
Run Time:	360 sec
Standard:	40 – 50 mg Aspartic Acid (10.52 %N)
Sample weight:	90 – 100 mg

FIGURE 3. Nitrogen determination



Results

Different paper samples, cut into small pieces were chosen to assess the system. Samples were analyzed several times to evaluate the reproducibility of the method.

Table 1 shows the NCS data of paper samples. Whilst in the NCS configuration, the system was calibrated with 3 – 4 mg of BBOT* standard using K factor as calibration method. The sample weight of paper was 3 - 4 mg. Samples were analyzed with the addition of Vanadium Pentoxide for a complete conversion of sulfur. Figure 4 shows a typical NCS chromatogram.

**BBOT: 2,5 bis(5-tert-butyl-benzoxazol-2-yl)thiophene:* 6.51 %N, 72.53 %C, 7.44 %S

TABLE 1. NCS determination.

Sample	N %	RSD %	C %	RSD %	S %	RSD %
Paper A	1.8279 1.8310 1.8690	1.2421	42.0787 42.1599 42.1559	0.1086	0.0437 0.0439 0.0431	0.9556
Paper B	0.0282 0.0298 0.0284	3.0270	42.3373 42.3655 42.3274	0.0467	0.0394 0.0422 0.0390	4.3372
Paper C	0.9866 1.0042 0.9835	1.1261	42.4505 42.4331 42.4767	0.0517	0.0422 0.0425 0.0417	0.9592
Paper D	1.2053 1.2293 1.2119	1.0200	42.3358 42.3647 42.3141	0.0600	0.0360 0.0356 0.0358	0.5587
Paper E	1.2529 1.2521 1.2550	0.1195	42.1291 42.2987 42.3493	0.2729	0.0374 0.0363 0.0372	1.5851

FIGURE 4. Typical NCS chromatogram

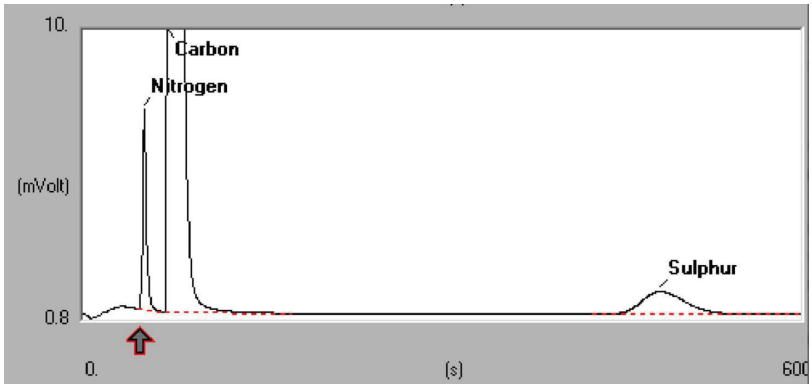


Table 2 shows the data of nitrogen determination of the electrostatic paper E in order to compare the results obtained previously by the NCS configuration. The instrument was calibrated with Aspartic acid (10.52 %N) as standard using K factor as calibration method. Table 3 shows the relative comparison, demonstrating that there are no significant differences in nitrogen percentages using different weight ranges. This confirms the complete conversion of the element during combustion.

TABLE 2. Nitrogen data of Electrostatic Paper E.

Weight (mg)	N %	Average N %	RSD %
99.8 107.2 106.0 102.1 107.4	1.25 1.23 1.26 1.24 1.26	1.25	1.04

TABLE 3. Nitrogen comparison data of Electrostatic Paper E by NCS and N determination.

NCS Configuration				Nitrogen configuration			
Weight (mg)	N %	Average N %	RSD %	Weight (mg)	N %	Average N %	RSD %
				99.80	1.25		
3.958	1.2329			107.20	1.23		
4.025	1.2521	1.2533	0.1195	106.00	1.26	1.25	1.04
3.969	1.2550			102.10	1.24		
				107.40	1.26		

Figure 5 shows the curve calibration obtained for nitrogen only determination.

FIGURE 5. Nitrogen Curve Calibration.

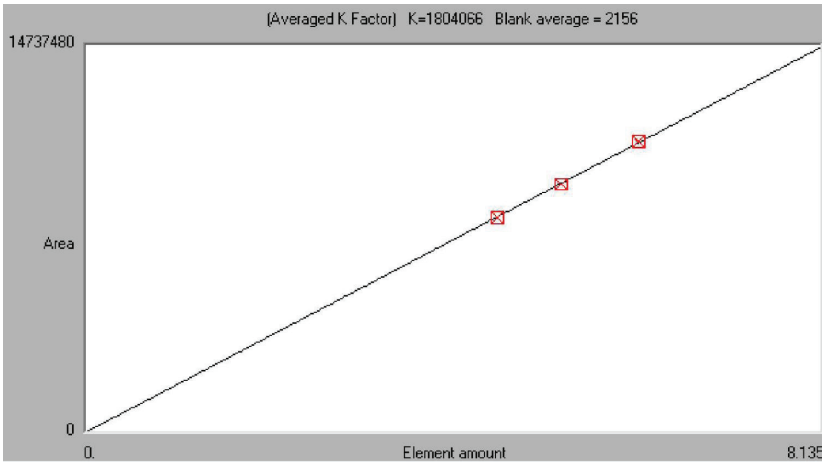


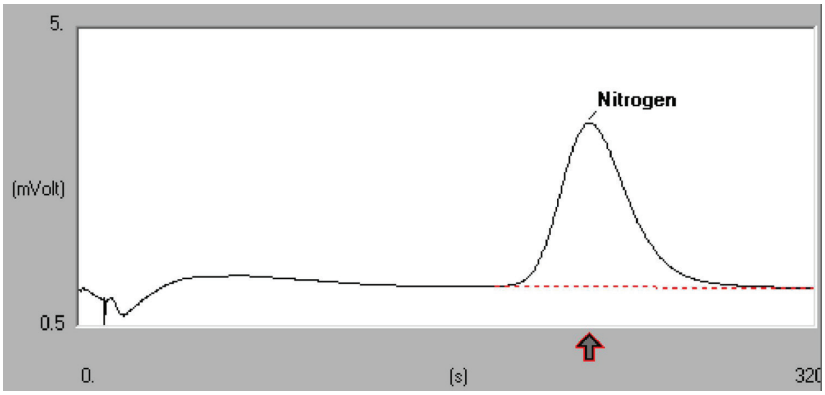
Table 4 shows the data of nitrogen determination of other paper samples. Whilst in the nitrogen configuration, the system was calibrated with 50 - 100 mg of Aspartic Acid (10.52 %N) and the paper sample weight was 70 - 100 mg.

TABLE 4. Nitrogen Determination.

Sample	N %	RSD %
Paper 1	0.3282 0.3368 0.3326	1.2932
Paper 2	0.3334 0.3372 0.3344	0.5880
Paper 3	0.2513 0.2501 0.2469	0.9119
Paper 4	0.2959 0.3003 0.2963	0.8179
Paper 5	0.2880 0.2930 0.2888	0.9263

Figure 6 shows a typical chromatogram of Nitrogen only determination

FIGURE 6. Typical Nitrogen chromatogram.



Conclusion

All data were obtained with a good reproducibility and no matrix effect was observed when changing the sample.

We demonstrate that the advantage of the FLASH 2000 analyzer lies in its ability to perform NCS determination in a single run, then, by changing the configuration and increasing the sample weight, it is possible to perform nitrogen only determination.

Using the elemental analyzer, it is also possible to characterize the different industrial paper applications according to the chemical concentration of the elements. This makes it possible to choose the most suitable paper recycling system, and perform a Life Cycle Assessment (LCA) on the environmental impact of the products from their production to their disposal.

This poster demonstrates that the FLASH 2000 OEA copes with all the demanding requirements of modern laboratories such as flexibility, accuracy, reproducibility, sensitivity, and automation.

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