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APPLICATION NOTE 42231

Elemental Analysis: NCS characterization of paper

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Keywords

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Goal

This application note shows NCS determination of paper with the Flash*Smart* EA, for material quality control.

Introduction

Industrial paper is made of cellulose fiber and animal or synthetic adhesives. Often, powders are added to the cellulose and adhesives to obtain different final paper products. By adding kaolin, calcium carbonate, talc or other powders, glazed paper, with bright cellulose fiber, (less porous and highly compacted) is obtained.

In the production process of pulp and industrial paper, the elemental composition is periodically monitored and tested for the characterization of raw and final products. Nitrogen and carbon are the most important parameters for quality control, as nitrogen content gives information on the temperature stability of the paper. Sulfur also is determined as it provides the level of impurities of the paper samples.

As the demand for material characterization testing has grown in recent years, the classical analytical methods showed to be no longer suitable, for their time-consuming sample preparation and for their use of hazardous reagents. As the elements are frequently present at trace levels, laboratories need low limit of detection. For this reason a simple and automated technique is the requirement for modern laboratories dealing with routine analysis.

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The Thermo Scientific™ Flash*Smart*™ Elemental Analyzer (Figure 1) meets laboratory requirements such as accuracy, day to day reproducibility and high sample throughput. NCS analysis are performed simultaneously in a single analysis run. The modularity of the system allows the configuration of the FlashSmart EA for nitrogen determination or for trace sulfur analysis when coupled to the Flame Photometric Detector (FPD). 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.

This note presents data on NCS determination of paper to show the performance of the Flash*Smart* Elemental Analyzer.

Methods

For NCS determination the Flash*Smart* Elemental Analysis operates with dynamic flash combustion of the sample. Samples are weighed in tin containers and introduced into the combustion reactor via the Thermo Scientific™ MAS Plus Autosampler with oxygen. After combustion the resulted gases are carried by a helium flow to a layer containing copper, they are swept through a water trap, and then through a GC column, which provides the separation of the combustion gases. Finally, they are detected by a Thermal Conductivity Detector (TCD). Total run time is less than 10 minutes. (Figure 2).

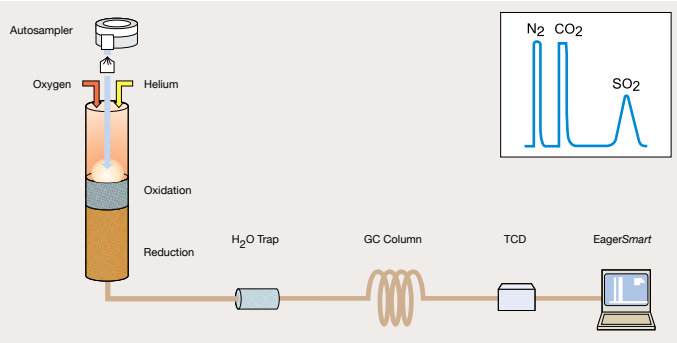

Figure 2. NCS configuration.

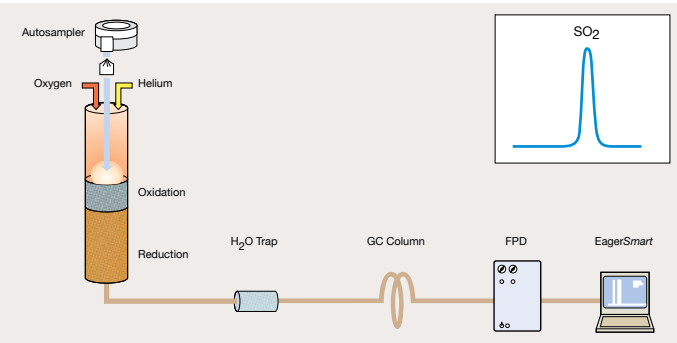

Figure 3. Sulfur configuration by FPD detector.

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

For trace sulfur determination, the gases produced by combustion are carried by a helium flow to a layer containing copper, then they are swept through a water trap, a short GC column and finally the sulfur is measured by the Flame Photometric Detector (FPD). Total run time is less than 5 minutes. (Figure 3).

For nitrogen determination only, the Elemental Analyzer operates with dynamic flash combustion of the sample using helium or argon as carrier gas. Samples are weighed in tin containers and introduced into the combustion reactor via the MAS Plus Autosampler with oxygen. After combustion, the produced gases are carried by a helium flow to a second reactor containing copper, and then swept through CO_2 and H_2O traps, and a GC column. They are finally detected by the Thermal Conductivity Detector (TCD). Total run time is 5 minutes (Figure 4).

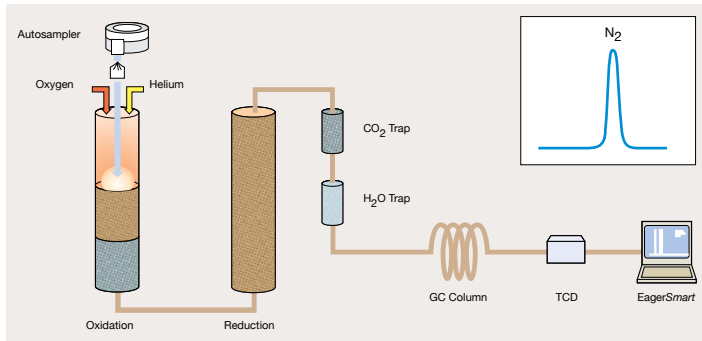

Figure 4. Nitrogen configuration.

A report is generated by the Thermo Scientific™ Eager*Smart*™ Data Handling Software and displayed at the end of the analysis.

Results

Different sample matrices were analyzed several times to demonstrate the repeatability of the Flash*Smart*™ Elemental Analyzer. The wood and cellulose samples were dried and homogenized with a ball mill and paper samples were cut into smaller pieces.

Table 1 shows the NCS data of wood and paper samples. To calibrate the instrument, BBOT* standard was analyzed weighing 2–3 mg using K factor as calibration method. Samples were weighed at 3–4 mg.

As paper samples B and C contain traces of nitrogen (less than 100 ppm), the analyses of these samples were repeated using only nitrogen configuration (Table 3).

Table 2 shows low level of sulfur content in wood, cellulose and paper samples, by using the Flash*Smart*™ Elemental Analyzer coupled with the FPD Detector. Thermo Scientific Soil Reference Material was analyzed to calibrate the system using quadratic fit as calibration method. Samples were weighed at 1–4 mg, depending of sample nature.

Figures 5 and 6 show a typical NCS and sulfur chromatograms.

**BBOT: 2,5-Bis (5-tert-butyl-benzoxazol-2-yl) thiophene*

Table 1. NCS data of wood and paper samples.

Sample	N%	RSD%	C%	RSD%	S%	RSD%
Wood	0.4023 0.3826 0.3838	2.8349	52.0534 52.1236 52.2494	0.1905	0.0320 0.0292 0.0299	4.7986
Paper A	0.2360 0.2357 0.2198	4.0207	43.8517 43.6566 43.1214	0.5319	0.0772 0.0772 0.0768	0.2997
Paper B	< 0.01	—	3.6829 3.6699 3.6907	0.2854	0.1736 0.1726 0.1736	0.3332
Paper C	< 0.01	—	3.6073 3.6620 3.6139	0.8231	0.1846 0.1868 0.1819	1.3307

Table 2. Sulfur data of wood, cellulose and paper samples by FPD detector.

Sample	S ppm	RSD%
Wood 1	36 35 37 37 34	3.64
Wood 2	166 179 167	4.24
Cellulose	13 13 12	4.68
Paper	566 575 570	0.79

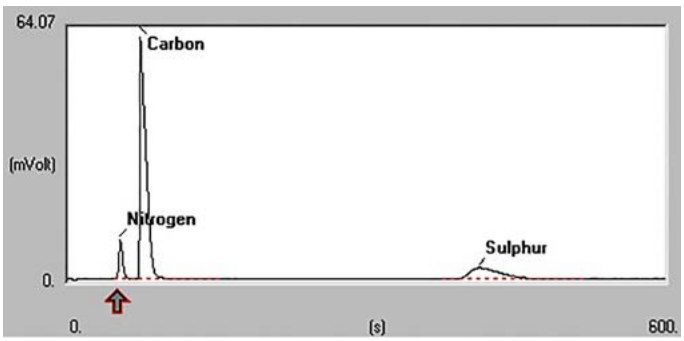

Figure 5. Typical NCS chromatogram.

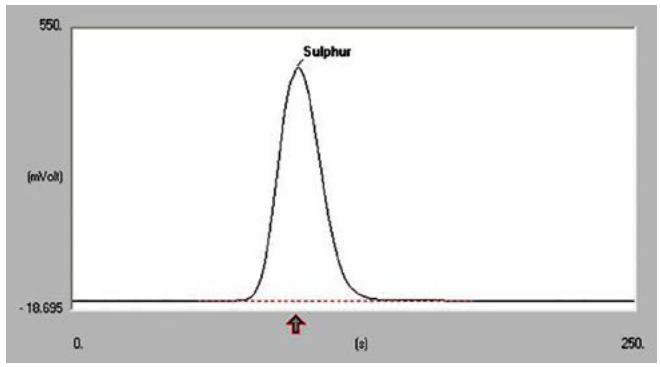

Figure 6. Sulfur chromatogram.

Table 3 shows the nitrogen data of wood and paper samples. The standard used to calibrate the system for wood and electrostatic paper was 50–100 mg of aspartic acid and the sample was weighed at 100–150 mg.

For paper samples B and C, 7–8 mg atropine standard was used to calibrate the instrument and the samples were weighed at 200–250 mg.

Figure 7 shows a typical nitrogen chromatogram.

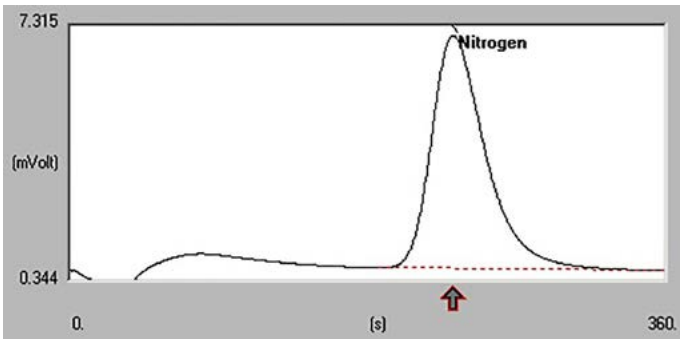

Figure 7. Typical Nitrogen chromatogram.

Different paper samples were analyzed to compare results of analyses carried out using helium and argon as carrier gas. In both cases, the calibration was performed with 30–40 mg of aspartic acid using K factor as calibration method and the sample was weighed at 50–80 mg. Table 4 shows the comparison of the data.

Table 3. Nitrogen data of wood and paper samples.

Sample	N%	RSD%
Wood powder 1	0.2270 0.2330 0.2250	1.823
Wood powder 2	0.1700 0.1710 0.1750	1.538
Electrostatic paper 1	1.2198 1.2360 1.2747 1.2264 1.2364	1.721
Electrostatic paper 2	1.4424 1.4345 1.3912 1.4644 1.4525	1.945
Electrostatic paper 3	0.4695 0.4298 0.4616 0.4867 0.4170	4.596
Paper B	0.0037 0.0040 0.0038	3.985
Paper C	0.0039 0.0038 0.0043	6.614
Paper D	0.0351 0.0331 0.0339	2.958
Paper E	0.3334 0.3372 0.3344	0.588
Paper F	0.2513 0.2501 0.2469	0.912
Paper G	0.2959 0.3003 0.2963	0.818
Paper H	0.2880 0.2930 0.2880	0.926

Table 4. Nitrogen data comparison of paper samples using Helium and Argon as carrier gas.

Paper Sample	Helium as carrier gas		Argon as carrier gas	
	N%	RSD%	N%	RSD%
1	1.7291 1.7271 1.7406	0.4206	1.7817 1.7671 1.7573	0.694
2	0.9802 0.9732 0.9727	0.4222	0.9549 0.9494 0.9431	0.6221
3	1.1196 1.1251 1.1262	0.3147	1.1160 1.0995 1.1035	0.7780
4	1.1963 1.1943 1.1948	0.0871	1.2042 1.2019 1.2064	0.1869
5	1.2400 1.2338 1.2487	0.6032	1.2439 1.2386 1.2582	0.8131

Conclusion

The Thermo Scientific Flash*Smart* Elemental Analyzer enables to perform accurate and reproducible NCS determination in paper samples.

It enables also the characterization of different industrial paper applications according to the chemical concentration of the elements. This allows industries and consumers to choose the most suitable paper recycling system. The Life Cycle Assesment (LCA) can also be measured. Therefore, the influence final products have on the environment, from their production to their disposal, can be assessed.

The Flash*Smart* Elemental Analyzer meets laboratory requirements in terms of automation and high sample throughput. The modularity of the system allows to be flexible and to perform different elemental characterization, with no need to change the hardware.

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