Trace Sulfur Determination by the Thermo Scientific FLASH 2000 elemental analyzer coupled with FPD detector for Material Characterization

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Application Note 42189

Key Words
Trace sulfur analysis, Catalysts, Carbon fibers, Carbon Nanotubes, Paper, Polymers, Polyethylene films

Introduction

The development and production of materials requires vigorous quality control of raw materials, additives, stabilizers, intermediate and finished products. The chemical composition of these is tightly connected with their specific characteristics and their chemical/physical/mechanical properties. Monitoring key elements at lower concentrations is routinely applied to exploration programs, research and environmental projects and is strictly controlled to meet national and international regulations to avoid potential danger due to hazardous and toxic substances.

The importance of sulfur testing has grown in recent years and many of the classical methods are now no longer suitable for routine analysis. Analytical instruments based on the combustion of samples improve the reliability of the data available to industry, without the use of hazardous chemicals.

Trace total sulfur content can be accurately determined by using the Thermo Scientific™ FLASH 2000 analyzer coupled with a Flame Photometric Detector (FPD) (Figure 1). The method combines the advantages of an elemental analyzer with the sensitivity, selectivity and robustness of a FPD. The coupling is simple and allows the determination of total sulfur at high and low concentrations (5 – 10 ppm) in the same instrument without matrix effect.

Methods

The elemental analyzer operates according to the dynamic flash combustion of the sample. Samples are weighed in a tin capsule and introduced into the combustion reactor via the Thermo Scientific™ MAS™ 200R autosampler together with a proper amount of oxygen. After combustion the resultant gases are carried by a helium flow to a layer filled with copper, then to a trap filled with anhydrone and swept through a GC column that allows separation of the combustion gases; finally being detected by the Flame Photometric detector (FPD) (Figure 2). Total run time is 5-6 minutes. A complete report is automatically generated by the Thermo Scientific™ Eager Xperience dedicated data handling software and displayed at the end of the analysis.

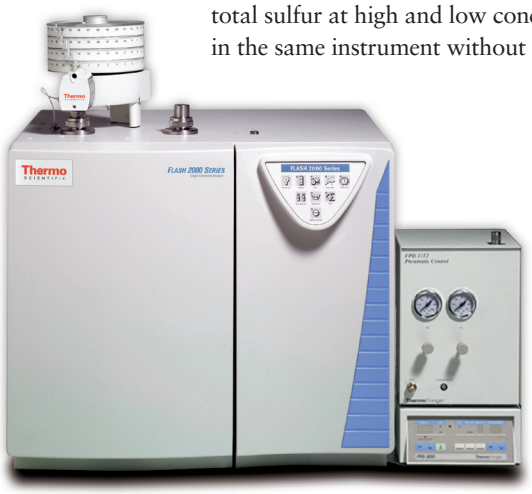

Figure 1. FLASH 2000 elemental analyzer coupled with Flame Photometric detector (FPD)

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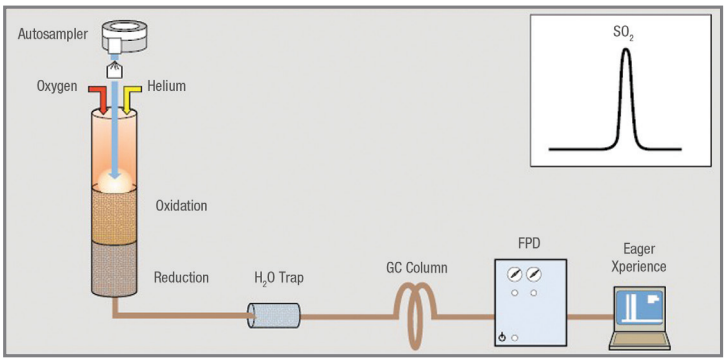

Figure 2. FPD sulfur determination layout

Results

Different materials such carbon fibers, carbon nanotubes, catalysts, paper, polymers and polyethylene films were chosen to demonstrate the performance of the system analyzing different matrices and to show the reproducibility obtained with the system in a large range of trace sulfur content.

The system was calibrated with Thermo Scientific Pasta and Soil Reference Materials with 0.135% S and 0.032%S respectively using Quadratic Fit as calibration method.

Table 1 shows the sulfur data of a polyethylene film sample while Table 2 shows the sulfur data obtained of polymers. The samples were cut into small pieces and analyzed without additives. The weight of sample was 0.5–3 mg.

Table 1. Reproducibility of sulfur determination of polyethylene film sample

S ppm	RSD%
571 573 540 542 558	2.7922

Table 2. Reproducibility of sulfur determination in polymers

Sample	S ppm	Av. S ppm	RSD %
A	495 466 467 456 447	466	3.87
B	876 902 820 879	869	4.01
C	1369 1442 1445	1419	3.03
D	1866 1856 1813 1931	1867	2.61
E	2969 2772 3016 2821	2894	4.02
F	350 328 317 321 320 336	329	3.80
G	178 186 182	182	2.20
H	59 59 52 61 56	57	6.11
I	41 40 42 36 38	39	6.11
J	25 32 29 23	27	14.79

Table 3 shows the sulfur data obtained of different materials (catalysts, carbon fiber, carbon nanotubes and paper). Catalysts were homogenized by a ball mill while paper was cut in small pieces. Samples were analyzed with the addition of approximately 5 mg of Vanadium Pentoxide as additive and the sample weight was about 0.5 – 5 mg. No memory effect was observed changing the matrix or sulfur content.

Table 3. Reproducibility of sulfur determination of different materials

Sample Nature	Samples Code	S ppm	Average S ppm	RSD%
Carbon Nanotubes	1	609 641	625	3.620
	2	652 629	641	2.539
	3	3391 3221 3413	3342	3.144
	4	1263 1301	1282	2.096
Carbon Fiber	1	704 712 659 672 705 726 722 715 706 715	704	3.06
Catalyst	1	12 14 15	13.66	11.1770
	2	13 11 11	11.66	9.8974
	3	73 76 71	73.33	3.4317
	4	14 16	15	9.7502
Paper	1	566 575 570	570	0.791

Conclusion

The Thermo Scientific FLASH 2000 analyzer coupled with a FPD detector is the ideal solution for the analysis of low and high concentrations of sulfur for material characterization in terms of stability, accuracy, reproducibility, sensitivity and automation without matrix effect.