

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

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## 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.



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



## 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 anhydrous 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.

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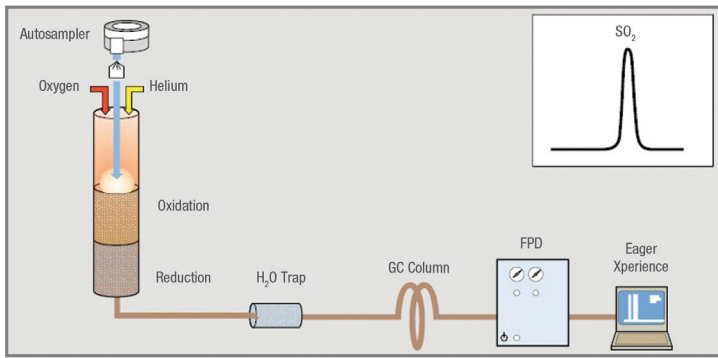


Figure 2. FPD sulfur determination layout

## 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	2.7922
573	
540	
542	
558	

Table 2. Reproducibility of sulfur determination in polymers

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

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

## 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.

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