Trace Sulfur determination in metals by the Thermo Scientific FLASH 2000 Elemental Analyzer coupled with FPD detector

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Key Words

Trace sulfur analysis, sensitivity, accuracy, easier sample handling, automated analysis, assured stability, difficult to destroy

Goal

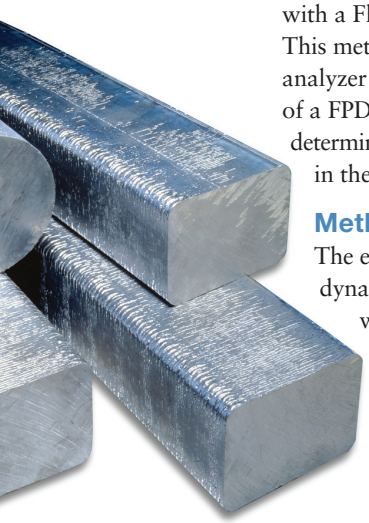
This application note demonstrates a method for the precise, sensitive and selective determination of low sulfur traces in metals using organic elemental analysis and flame photometric detector.

Introduction

In metallurgy, some elements are of great importance; nitrogen, carbon and sulfur are particularly so where steels are concerned. The addition of nitrogen and carbon improves the properties of the specific metal, although there are certain areas where their presence is undesirable and should be avoided if possible. Sulfur plays an important role in the mechanical properties of steel and alloys, where it is usually present within the range of 10 – 3000 ppm.

Automatic analyzers using dynamic flash combustion have been used for many years for total sulfur determination, but have been limited in many applications due to the detection limit. This limit has been sufficient for most analytical needs up until now, but today, trace sulfur analysis is becoming more demanding.

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). This method combines the advantages of the elemental analyzer with the sensitivity, selectivity and robustness of a FPD. The coupling is simple and allows total sulfur determination 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

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

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 separates 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.

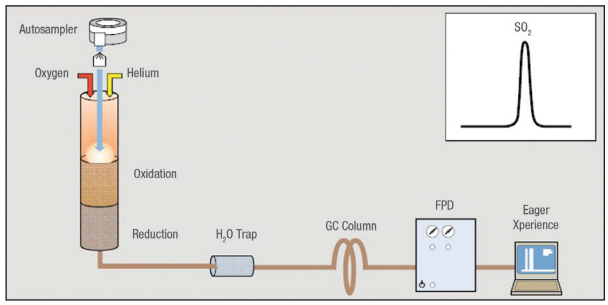


Figure 2. Layout of sulfur determination by FPD detector

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Results

Different samples were chosen to demonstrate the performance of the system analyzing different matrices and to show the reproducibility obtained within the system in a large range of trace sulfur content. Metal material - weight sample range 0.5 / 5mg - were cut into small pieces and analyzed without any pre-treatment.

Table 1 shows a typical sequence of analysis. Instrument calibration was performed with cast iron BCR* (0.1670% S) using Quadratic Fit as the calibration method (runs 3 to 6). Figure 3 shows the relative calibration curve obtained. The accuracy of the system was verified by analyzing the same cast iron sample ten times as unknown (runs 7 to 16) as shown in Table 2.

* BCR: Bureau Communautaire de Référence - Community Bureau of Reference

Table 1. Typical sequence

Run	Sample	Type	Weight (mg)
1	Cast Iron BCR	Bypass	
2	Cast Iron BCR	Bypass	
3	Cast Iron BCR	Standard	0.652
4	Cast Iron BCR	Standard	1.090
5	Cast Iron BCR	Standard	1.621
6	Cast Iron BCR	Standard	2.207
7	Cast Iron BCR	Unknown	1.445
8	Cast Iron BCR	Unknown	1.602
9	Cast Iron BCR	Unknown	1.291
10	Cast Iron BCR	Unknown	2.105
11	Cast Iron BCR	Unknown	1.165
12	Cast Iron BCR	Unknown	1.011
13	Cast Iron BCR	Unknown	1.827
14	Cast Iron BCR	Unknown	1.509
15	Cast Iron BCR	Unknown	1.658
16	Cast Iron BCR	Unknown	2.198

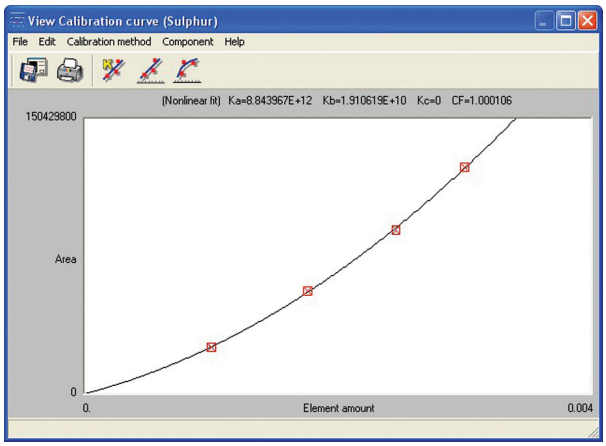

Figure 3. Typical curve calibration

Table 2. Test of accuracy

Cast Iron	%S	Average %S	RSD%
Run 7	0.1645		
Run 8	0.1642		
Run 9	0.1696		
Run 10	0.1703		
Run 11	0.1637	0.1675	1.8158
Run 12	0.1726		
Run 13	0.1653		
Run 14	0.1695		
Run 15	0.1685		
Run 16	0.1666		

Table 3 shows sulfur determination in various metal and alloy samples analyzed several times. All data were obtained with a good reproducibility and no matrix effect was observed when changing sample nature.

Table 3. Reproducibility of sulfur determination.

Sample	S ppm	Average S ppm	RDS %
Carbon steel	242 243	242.5	0.29
Bronze	158 153 160 153 162	157	2.60
Steel 1	205 193 204 190 205	199	3.66
Steel 2	52 54 52 55 53	53	2.45
CrMoNi alloy	72 71 67 66 66	68	4.21

Conclusion

The Thermo Scientific FLASH 2000 elemental analyzer coupled with a FPD detector is the ideal solution for the analysis of trace concentration of sulfur in metals in terms of stability, accuracy, reproducibility, sensitivity and automation without matrix effect.

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