

Application Note

The Measurement of Fe, Al, Ca and Mg in FeSi and FeSiMg Samples by Flame Atomic Absorption

Introduction

It is often difficult to obtain a high degree of analytical accuracy in the analysis of silicates due to the high concentration acid digest usually required to dissolve the silicate. Therefore we present this method as an example of how to overcome these difficulties in the analysis of Iron, Aluminium, Calcium and Magnesium.

Experimental

Instrumentation

A GBC Avanta Σ atomic absorption spectrometer, equipped with the unique Ultra-Pulse background Correction was used for all measurements. The AAS software for Windows[®] 95 offers complete control over the entire system, from rapid method development to complete instrument diagnostics and customized reports with true 32 bit pre-emptive multi-tasking. The instrumental conditions for the determination of Fe, Al, Ca, and Mg are shown in

Table 1. GBC hollow Cathode lamps were used for the determination of these elements.

Sample and Standard Preparation

Approximately 0.1g of the FeSi or FeSiMg samples were reacted for 2hrs in 10ml of conc. HCl, then 5ml of conc. Nitric acid was added and allowed to react for a further 2hrs with heating. Following this 6 drops of HF (48%) was then added to the mixture with continued heating until all the solid was dissolved. 0.1g of boric acid was then added to react with excess HF, and this mixture was transferred to a plastic volumetric flask and made up to 100mL with deionised water. For Ca and Mg determination a further 1 in 10 dilution was required. These dilutions and all standards were also 10% HCl, 5% Nitric, contained an equivalent of 6 drops HF and 0.1g boric acid per 100ml and were made up with deionised water. The working standards prepared were made to be a combination of all four

Element	Background Correction	Wavelength (nm)	Slit Width (nm)	Lamp Current (mA)
Fe	On	392.0	0.2	7.0
Al	On	396.2	0.5	10
Ca	On	422.7	0.5	10
Mg	On	202.6	1.0	3.0

Table 1: Instrumental conditions for the determination of Fe, Al, Ca and Mg.

elements and made to 250ml, Table 2 shows the respective concentrations of each standard.

Element	Bulk Standard	Working Standard Conc. (ppm)
Fe	1000 ppm BDH Standard	300, 400, 500, 600
Al	1000 ppm BDH Standard	2, 5, 7, 10
Ca	1000 ppm BDH Standard	5, 10, 15, 20
Mg	1000 ppm BDH Standard	5, 10, 15, 20

Table 2. Note: In some cases, an intermediate standard was required.

Results

Normal calibration curves were used to determine all four elements. Table 3 shows a summary of the results obtained.

Sample	Fe (%)	Al (%)	Ca (%)	Mg (%)
FeSi	18.2 ±0.4	0.20 ±0.01	1.87 ±0.09	0.52 ±0.01
FeSiMg 1	46.3 ±1.4	0.26 ±0.02	2.18 ±0.10	5.92 ±0.40
FeSiMg 2	40.0 ±1.5	0.62 ±0.02	2.52 ±0.03	7.33 ±0.40
FeSiMg 3	36.7 ±1.4	0.28 ±0.03	3.84 ±0.03	10.27 ±0.07
FeSiMg 4	33.2 ±0.6	0.25 ±0.02	4.64 ±0.06	11.31 ±0.50

Table 3: Percentage concentrations for each element.

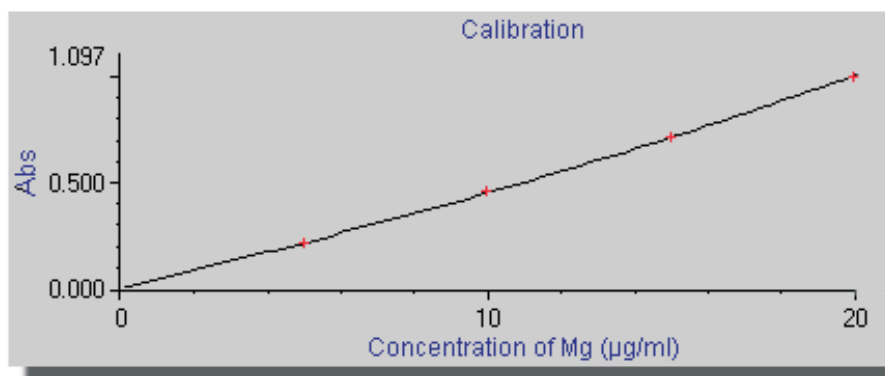


Figure 1: Example of a calibration curve for Magnesium.

Conclusion

The method used for analysing Iron, Aluminium, Calcium, and Magnesium in Iron Silicates proved successful, with good verification of the method used by spike recoveries. The method was conveniently automated by the use of the GBC Avanta Σ with Windows® 95 based software.

Storage of gasbox parameters allowed easy transition between the elements while still having the instrument individually optimized for each element. The use of the GBC high performance spray chamber and nitrous oxide burner allowed the use of low gas flow rates and maximum sensitivity. The lower flow rates provide lower running costs and reduce carbon formation on the burner jaw edges.