



Challenge

Determination of Ca, Fe, Mg, Na, K, Al, and Ti in mullite and fiber cement

Solution

Simple and robust routine analysis using flame AAS with the novAA 800 F

Intended audience

Industrial quality control labs analyzing building materials in process and quality control

Determination of Major Elements in Building Materials Using Flame AAS

Introduction

Building materials play an important role in today's industry. Their composition has a big influence on material properties and fields of application, as well as on their behavior in ecosystems and recyclability. Tightly monitoring the elemental composition of building materials, such as cement, is key for ensuring their quality, and this composition is thus routinely measured in industrial quality control labs.

The following application note describes a method for determining the composition of mullite and fiber cement as conducted by an on-site wet-chemical process lab of a producer for building materials.

The novAA 800 F is a robust atomic absorption spectrometer equipped with accessories that simplify routine analysis. An autosampler with integrated dilution function makes time-consuming wet-chemical sample preparation redundant, thus enabling high sample throughput even for high-matrix samples. The Segmented Flow Star (SFS 6.0) is an injection switch for segmented sample introduction and continuous rinsing that reduces carry-over effects and contributes to a smooth operation, thus ensuring reliable results.

An automatic burner head cleaner, or Scraper, guarantees stable measuring conditions when using the C_2H_2/N_2O flame. Using this tool leads to an automatic removal of the inevitable carbon deposits on the burner slit. The latter flame conditions are beneficial for the analysis of refractory elements and analytes interfered by the sample matrix.

Material and Methods

Samples and reagents

- Mullite
- Fiber cement

Sample preparation

Approx. 1 g of the mullite sample and 0.5 g of fiber cement were dried, weighed, and digested using the microwave digestion system TOPwave (vessel type PM60) with a mixture of HF, HCl, and HNO₃. A subsequent complexation using H₃BO₃ was performed to avoid analyte loss due to formation of insoluble compounds of Ca and Al with HF. For determination of Ti this step was omitted. It was found that Ti is more stable in the presence, HF than in H₃BO₃ treated solutions.

The final solutions were transferred into graduated vessels (plastic vessels for HF containing solutions) and filled to a volume of 50 mL with deionized water.

Each sample was prepared twice and the mean value of both measured values was formed for each element.

For measurement the sample solutions were prediluted manually. If the concentration still exceeded the calibration range, this solution was further diluted by the autosampler. The solutions were diluted with 1.5% HNO₃ and 0.1% CsCl/LaCl₃ except for Ti which was diluted with 10% HCl and 0.1% CsCl/LaCl₃.

In order to observe signal depressing matrix effects, the sample solution was spiked with a defined analyte concentration (QC spike) and recovery rates recorded.

Calibration

A standard calibration has been applied using 0.1% CsCl/LaCl₃ and 1.5% HNO₃ or 10% HCl for Ti, respectively. Calibration curves are shown below.

Table 1: Concentration of calibration standards

Standard	Concentration [mg/L]								
	Ca ¹	Ca ²	Fe ¹	Fe ²	Mg	Na	K	Al	Ti
Cal. 0	0	0	0	0	0	0	0	0	0
Cal. Std. 1	1	0.05	1	0.2	0.02	0.02	0.2	5	10
Cal. Std. 2	2	0.1	2	0.5	0.05	0.05	0.4	10	20
Cal. Std. 3	4	0.2	3	1.0	0.1	0.1	0.8	15	40
Cal. Std. 4	6	0.5	4	1.5	0.15	0.2	1.2	20	80
Cal. Std. 5	-	1.0	-	2.0	0.2	-	-	25	-

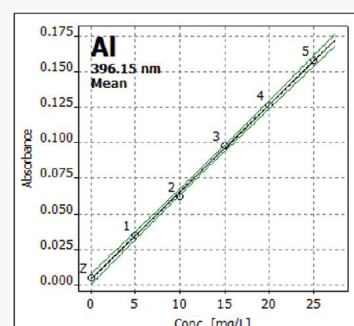
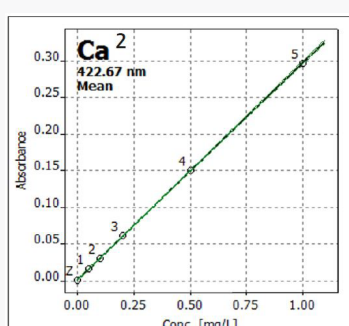
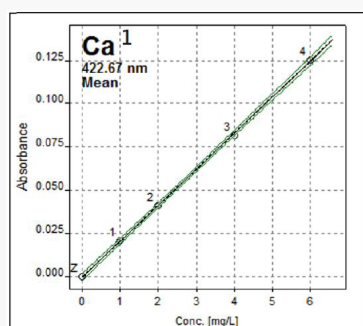


Figure 1-3: Exemplary calibration curves, ¹ Calibration curve for fiber cement; ² calibration curve for mullite

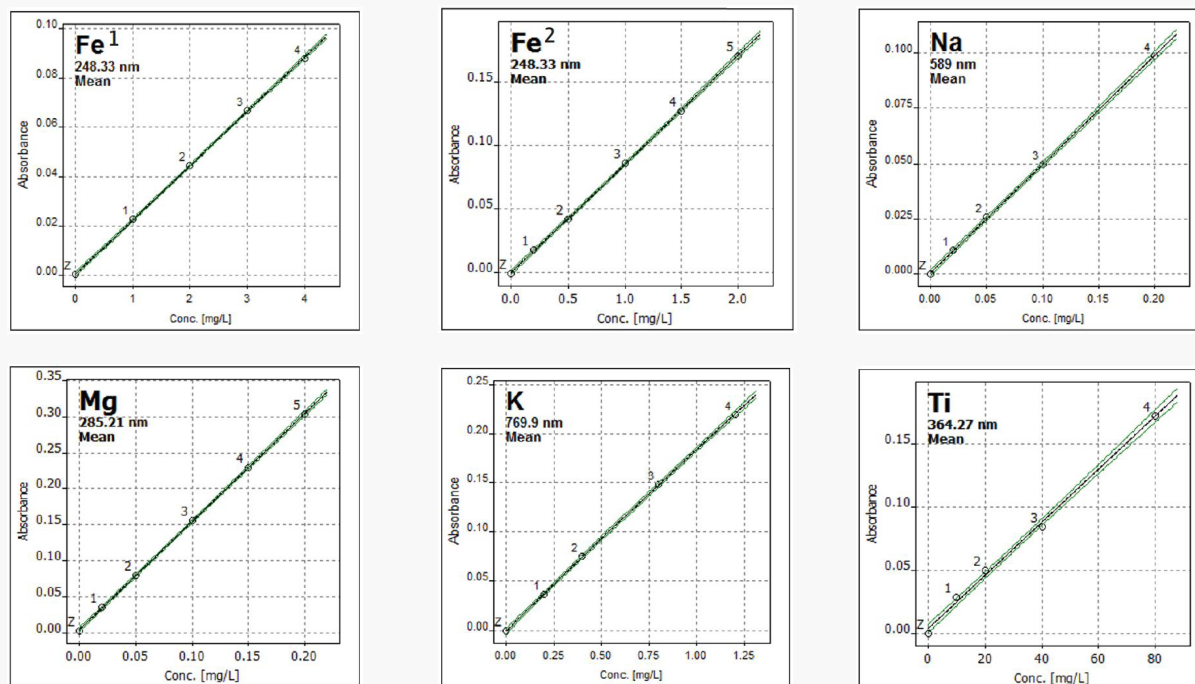


Figure 4-9: Exemplary calibration curves, ¹ Calibration curve for fiber cement; ² calibration curve for mullite

Instrumentation

The measurements were performed using the flame AAS with novAA 800 F. The system was equipped with a 50 mm burner head, the injection switch SFS 6.0, the automatic burner head cleaner Scraper, and an autosampler with automatic dilution function.

Table 2: Instrument settings and method parameters

Element	Wavelength [nm]	Slit [nm]	Lamp current [mA]	Burner height [mm]	Flame type	Fuel gas flow [L/h]
Ca	422.7	1.2	3.0	6	C ₂ H ₂ /N ₂ O ¹	195
Fe	248.3	0.5	6.0	5	C ₂ H ₂ /N ₂ O ² C ₂ H ₂ /air	190 60
Mg	285.2	1.2	1.5	6	C ₂ H ₂ /air	50
Na	589.0	0.8	3.0	5	C ₂ H ₂ /air	50
K	769.9	1.2	4.0	8	C ₂ H ₂ /air	60
Al	396.2	1.2	5.0	6	C ₂ H ₂ /N ₂ O ¹	220
Ti	364.3	0.5	7.0	7	C ₂ H ₂ /N ₂ O ¹	250

¹ Refractory metals require higher atomization temperatures, hence a C₂H₂/N₂O gas mixture may enhance the analysis

² Fe determination: using the C₂H₂/N₂O flame, better recovery rates were achieved for fiber cement (less interferences)

Results and Discussion

Table 3 shows the measurement results for the samples and recovery rates for the QC spike.

Table 3: Measurement results and QC recovery

Sample	Element	DF	Concentration \pm SD [g/kg]	RSD [%]	QC * recovery rate [%]
Fibre cement	Ca	1000	382 \pm 9.24	0.9	101
	Fe	50	19.2 \pm 0.31	0.7	108
	Mg	1000	13.1 \pm 0.17	1.0	105
	Na	50	0.87 \pm 0.02	0.9	102
	Al	25	21.8 \pm 0.57	0.8	107
	K	50	1.89 \pm 0.07	0.9	98.9
Mullite	Ca	100	2.85 \pm 0.02	1.0	104
	Fe	50	6.66 \pm 0.12	0.5	94.0
	Mg	20	0.17 \pm 0.01	0.3	98.2
	Al	200	106 \pm 5.19	1.6	98.8
	Ti	5	6.09 \pm 0.40	0.8	108

DF: dilution factor; if the calibration range was exceeded, further dilution was performed by the autosampler

* QC recovery rate of a defined analyte concentration, with which the fiber cement and mullite samples were spiked (typically in the range of Cal. Std. 2)

Summary

The novAA 800 F provides a robust and simple solution for measuring various elements in building materials in process and quality control of small sample amounts. This is suitable for routine analysis in industrial QC labs with moderate sample loads. The achieved RSD values and recovery rates for mullite and fiber cement samples prove the reliability and precision of the method applied.

When handling high amounts of samples, an autosampler considerably increases the sample throughput. Furthermore, both the autosampler with integrated dilution function and the Segmented Flow Star, SFS 6.0, enable simple handling of samples with high matrix load.



Figure 10: novAA 800 F

This document is true and correct at the time of publication; the information within is subject to change. Other documents may supersede this document, including technical modifications and corrections.

Headquarters

Analytik Jena GmbH+Co. KG
Konrad-Zuse-Strasse 1
07745 Jena · Germany

Phone +49 3641 77 70
Fax +49 3641 77 9279

info@analytik-jena.com
www.analytik-jena.com

Version 2.0 · Author: Anju
en · 07/2023

© Analytik Jena | Pictures ©: Pixabay/byrev (p. 1)

Recommended device configuration

Table 4: Overview of devices and accessories.

Article	Article number	Description
AAS - novAA 800F	812-08000-2	AA spectrometer
Burner head 50 mm	810-60057-0	For acetylene/nitrous-oxide or acetylene/air flame
Scraper	810-60127-0	For automatic burner head cleaning
Segmented Flow Star SFS 6.0	810-60129-0	Switching valve
Air compressor, 50 Hz	810-60055-0	To supply AAS with water- and oil- free compressed air
AS-FD	810-60501-0	Autosampler with auto-dilution for flame
HCL package	810-60082-C	Package with 8 coded lamps

This document is true and correct at the time of publication; the information within is subject to change. Other documents may supersede this document, including technical modifications and corrections.

Headquarters

Analytik Jena GmbH+Co. KG
Konrad-Zuse-Strasse 1
07745 Jena · Germany

Phone +49 3641 77 70
Fax +49 3641 77 9279

info@analytik-jena.com
www.analytik-jena.com

Version 2.0 · Author: Anju
en · 07/2023

© Analytik Jena | Pictures ©: Pixabay/byrev (p. 1)