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 on the novAA 800 F.

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 Spetrometer (AAS) 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 carryover and contributes to a smooth operation, thus ensuring reliable results.
An automatic burner head cleaner, or Scraper, guarantees stable measuring conditions and reproducible results by removing deposits from the burner slit, especially when working with the C_2H_2/N_2O flame. The latter flame conditions are beneficial for the analysis of refractory elements.

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Materials 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 weighed and digested using the microwave digestion system TOPwave (vessel type PM60) with a mixture of HF, HCl and HNO_3. A subsequent complexation using H_3BO_3 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 H_3BO_3 treated solutions.
The final solution was transferred into a graduated vessel 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 % HNO_3 and 0.1 % $CsCl/LaCl_3$ except for Ti which was diluted with 10 % HCl and 0.1% $CsCl/LaCl_3$.

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

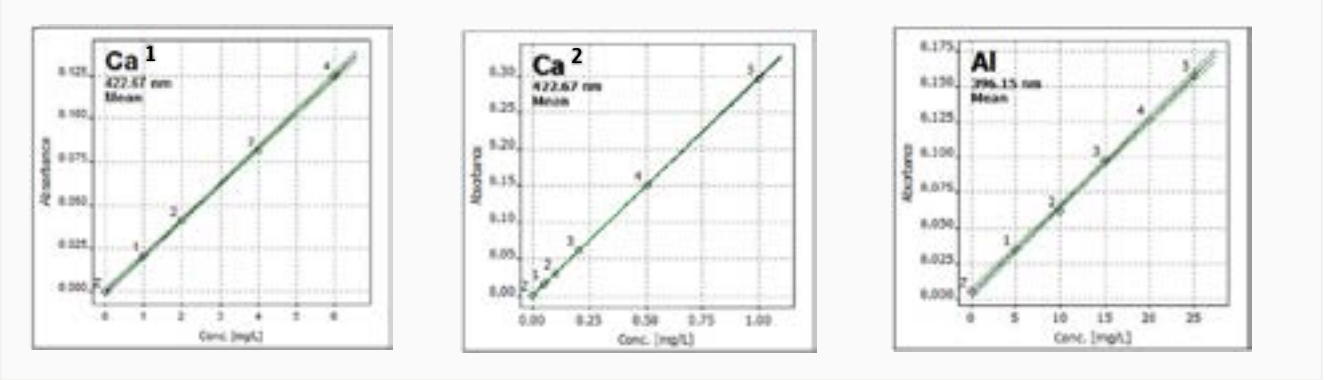
Calibration

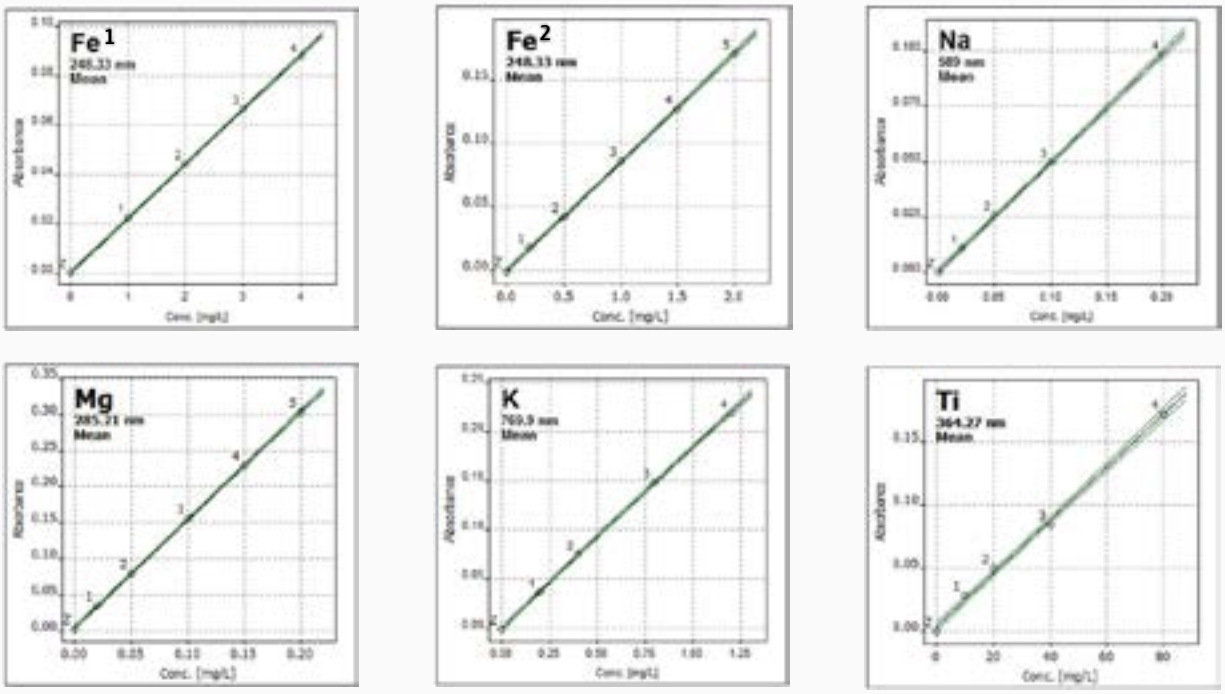
A standard calibration has been applied and standards were prepared manually using 0.1 % $CsCl/LaCl_3$ and 1.5 % HNO_3 or 10 % HCl for Ti, respectively.

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	0.1	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	-	2	0.2	-	-	25	-

Calibration curves





[1] Calibration curve for fiber cement; [2] calibration curve for mullite

Instrumentation

The measurements were performed using the flame AAS 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.

Instrument Settings and Method Parameters

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_2H_2/ N_2O [1,2]	195
Fe	248.3	0.5	6.0	5	C_2H_2/N_2O [1,3] C_2H_2/air	190 60
Mg	285.2	1.2	1.5	6	C_2H_2/air	50
Na	589.0	0.8	3.0	5	C_2H_2/air	50
K	769.9	1.2	4.0	8	C_2H_2/air	60
Al	396.2	1.2	5.0	6	C_2H_2/N_2O [1]	220
Ti	364.3	0.5	7.0	7	C_2H_2/N_2O [1]	250

1 Refractory metals require higher atomization temperatures, hence a C_2H_2/N_2O gas mixture may benefit the analysis
2 Ca determination in fiber cement: a 90° burner head rotation may benefit the sensitivity of the method and avoid an extremely high dilution factor
3 Fe determination: using the C_2H_2/N_2O 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 [g/kg]	RSD [%]	QC * recovery rate [%]
Fibre cement	Ca	1000	382 ± 9.24	0.9	101
	Fe	50	19.2 ± 0.31	0.7	108
	Mg	1000	13.1 ± 0.17	1.0	105
	Na	50	0.87 ± 0.02	0.9	102
	Al	25	21.8 ± 0.57	0.8	107
	K	50	1.89 ± 0.07	0.9	98.9
Mullite	Ca	100	2.85 ± 0.02	1.0	104
	Fe	50	6.66 ± 0.12	0.5	94.0
	Mg	20	0.17 ± 0.01	0.3	98.2
	Al	200	106 ± 5.19	1.6	98.8
	Ti	5	6.09 ± 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)

Conclusion

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 that 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 high-matrix samples.

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