Lab Report CS/ONH 20

G4 ICARUS Series 2

- Fast and Reliable Carbon and Sulfur Determination in Cement

Introduction

Cement is a hydraulic binder and the main ingredient of many basic building materials, such as concrete and mortar. Quality- and Process-Control require the fast and accurate analysis of the elemental and phase composition. Combustion analysis is an easy and fast method to control the limestone and gypsum / anhydrite addition in cements by measuring carbon and sulfur. This Lab Report shows the simplicity, speed and reliability of carbon and sulfur determination by the G4 ICARUS Series 2 using a high-frequency (HF) induction furnace.

Measuring Principle

The combustion takes place under a flow of oxygen within a sealed HF induction furnace. While the sample, with addition of a metallic accelerator, is heated by induction, it combusts at temperatures exceeding 2000 °C. Sulfur compounds are oxidized to SO_2 and carbon to CO_2. Total carbon and sulfur are calculated relative to the amount of these combustion gases, which are quantified by selective detection systems.

Combustion without compromises: G4 ICARUS!

With its powerful HF-furnace equipped with ZoneProtect™, its unique vacuum-free automatic cleaning system, HighSense™ detectors and electronic flow control, the G4 ICARUS Series 2 is a smart addition for every industrial user who depends on a reliable instrument even under harsh conditions. The G4 ICARUS Series 2 is capable to measure both carbon and sulfur in cements and related materials with high precision in typically less than one minute.

Sample Preparation

For optimal precision, the sample should be grinded or crushed to a uniform powder prior to analysis. If necessary, it should to be dried for at least 1 h at 110°C. No further sample preparation is necessary.

Method Parameters

- Purge time: 5 s
- Start delay: 5 s
- Baseline check before analysis: 5 s
- Analysis time 1: 35 s (power Level: 4)
- Analysis time 2: 25 s (power level: 0)
- Baseline check after analysis: 5 s
- Crucible cool time: 10 s

- Sample mass: ~0.2 g (weighed to 0.1 mg)
- Accelerators:
  - Tungsten/Tin: ~1.5 g
  - High purity iron chips: ~0.7 g

Calibration

The calibration of the analyzer is performed by means of reference materials with certified concentrations of carbon and sulfur. The analysis software supports single point calibration with one CRM and multi point calibration with multiple CRMs.

Procedure

I. Determination of the blank value

Run a minimum of 3 analysis of the blank value by adding the described amount of accelerators into a preheated[1)] crucible and analyze.

II. Measuring reference materials

- Choose CRMs for calibration and define them in the analysis software with designation and the certified concentrations.
- Weigh in an appropriate amount of reference material into a preheated[1)] crucible, and transfer the exact mass into the analysis software. Cover the material with the described amount of accelerator and analyze.
- Repeat step 2 a minimum of three times for each reference material (or sample weight) used.

Calibrate the analyzer with the blank values recorded under I. and the results obtained with reference materials II. (for more details refer to the user manual).

III. Sample measurement

- Weigh ~0.2 g of sample into a preheated[1)] crucible and transfer the exact sample mass to the analysis software. Cover the sample with the described amount of accelerator and analyze.
- Repeat step 1 until an appropriate number of repetitions is obtained.

[1)] For optimal precision, ceramic crucibles shall be pre-heated in a muffle furnace at ≥1250 °C for a minimum of 15 min or ≥1000 °C for a minimum of 2 hours. To avoid contamination, crucibles must be handled with clean tongs and transferred to a desiccator for storage.

Calibration Example

The typical concentration levels for total carbon and total sulfur in cement fall in the range of:

Carbon: 0.3 – 3% (1 – 11 $\%CO_2$)
and Sulfur: 0.005 - 1.5% (0.01 – 4 $\%SO_3$).

In the cement industry the concentration level is often expressed in form of $\%CO_2$ for carbon and $\%SO_3$ for sulfur. The analysis software is capable of converting and reporting the result expressed in these units on the fly, but reference material may only be specified in %C and %S. The conversion factors are: $\%C \rightarrow \%CO_2$: 3.6641 and $\%S \rightarrow \%SO_3$: 2.4969.

The typical concentration range is useful information to consider when selecting appropriate reference material for calibration. Although all detectors are linearized and an extrapolation of the calibration curve is possible, it is good laboratory practice that all samples fall within the calibrated range. In this example, the reference materials shown in Table 1 have been used:

Standard (lot#)	Material	Total carbon / %	Total sulfur / %
AR 4006 (411B)	ore	4.19	4.07
AR 4007 (1212E)	ore	7.27	3.26
AR 4012 (52199)	limestone	11.97	0.044
EZRM B 483-1	cast iron	2.46	0.103
Ag_2SO_4 (p.a.)	pure substance	n.a.	10.12 – 10.28 (98.5 - 100% purity)

Table 1: Reference materials used for calibration

The resulting calibration curves for carbon and sulfur (Fig. 1) with R^2 = 0.9999 prove the excellent linearity, accuracy and reproducibility of the G4 ICARUS Series 2 detection system and the entire method.

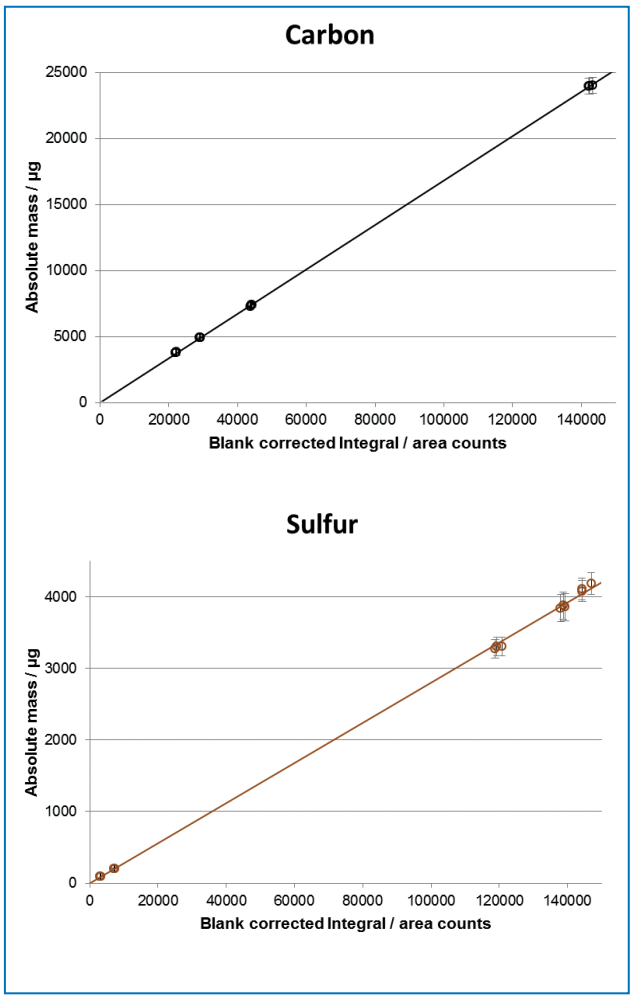

Figure 1: Calibration curve for carbon (top) and sulfur (bottom)

Results

The reproducibility of the G4 ICARUS Series 2 and the method outlined is demonstrated by a series of 10 repetitive measurements of real cement production samples.

Cement Sample 1

Mass / g	%CO_2	%SO_3
0.2022	8.504	2.964
0.2022	8.446	2.969
0.2026	8.468	2.976
0.2016	8.471	2.961
0.2029	8.435	2.991
0.2039	8.453	2.941
0.2018	8.501	2.984
0.2017	8.464	2.951
0.2028	8.416	2.951
0.2003	8.449	2.979
Mean	**8.46**	**2.97**
STD	0.03	0.02
%RSD	0.3	0.5

Cement Sample 2

Mass / g	%CO_2	%SO_3
0.2001	2.213	3.213
0.2018	2.224	3.211
0.1997	2.253	3.213
0.2026	2.246	3.196
0.2016	2.261	3.223
0.2010	2.220	3.228
0.2001	2.217	3.171
0.2011	2.257	3.241
0.2010	2.246	3.218
0.2035	2.239	3.196
Mean	**2.24**	**3.21**
STD	0.02	0.02
%RSD	0.8	0.6

Cement Sample 3

Mass / g	%CO_2	%SO_3
0.2037	9.248	1.750
0.1998	9.208	1.723
0.2003	9.245	1.753
0.2001	9.215	1.730
0.2038	9.281	1.725
0.2030	9.278	1.725
0.2028	9.186	1.710
0.2020	9.230	1.735
0.2012	9.230	1.718
0.2004	9.179	1.725
Mean	**9.23**	**1.73**
STD	0.03	0.01
%RSD	0.4	0.8

Summary

The combination of HighSense™ detection systems with precise electronic flow control in the G4 ICARUS Series 2 delivers excellent and long-time stable analytical performance. The powerful HF-furnace equipped with the industry-leading ZoneProtect™ and its unique automatic cleaner ensures lowest cost of ownership, high availability with little maintenance. The sample preparation for this method is straightforward and does not require any expert knowledge. The analytical performance in combination with the speed of analysis and its ease of use makes the G4 ICARUS Series 2 the ideal addition to XRF analysis for the quality control of cement and related products.

Bruker AXS GmbH

info.baxs@bruker.com

Worldwide offices

bruker.com/baxs-offices

Online information

www.bruker.com/csonh

www.bruker.com