



Lab Report CS/ONH 22

G4 ICARUS Series 2

- Fast and reliable Carbon and Sulfur Determination in Cast Iron

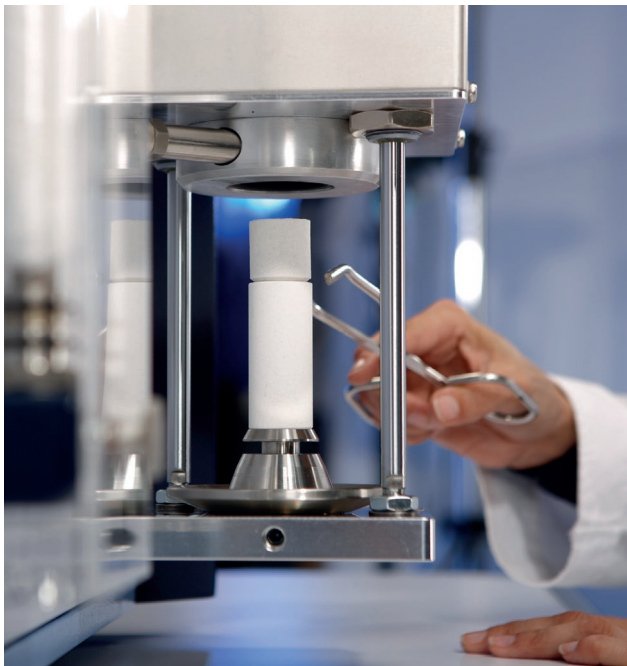
Introduction

The importance of the element carbon in iron and steel making is well known. Properties like hardness, hardenability, ductility and brittleness depend to a large extent on the content of carbon in the material. That makes carbon the main alloying element in cast iron with typical levels greater than 2%. In the iron matrix, carbon can be present in different forms, e.g. dissolved in the matrix, bound to other elements as carbides, or elementary as free graphite.

Sulfur in contrast is typically an unwanted, contaminant element in cast iron. It can e.g. prevent the graphite formation in cast iron and thus impact the mechanical properties and its workability. Quality- and Process-Control require the fast and accurate analysis of the carbon and sulfur content in cast iron and casted products. 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 SO₂ and carbon to CO₂. 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- and pressure-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 cast iron and related materials with high precision in typically below one minute. Even in difficult matrices like grey cast iron, where carbon is unevenly distributed in the form of graphite flakes, combustion analysis delivers accurate results because it is a volumetric method, analyzing the entire sample mass.

Sample Preparation

Please refer to DIN EN ISO 14284 or ASTM E1806 for a detailed description of the standard practice for proper sampling of cast iron, steel and iron.

Note: Due to ASTM E1806 samples taken during cast iron production and pig iron samples should not be washed with organic solvents to avoid the risk to alter the distribution of metal and graphite.

Method Parameters

- Purge time: 5 s
- Start delay: 1 s
- Baseline check before analysis: 2 s
- Analysis time 1: 25 s (power Level: 4)
- Analysis time 2: 20 s (power level: 0)
- Baseline check after analysis: 2 s
- Crucible cool time: 10 s

- Sample mass: ~0.2 - 0.3 g (weighed to 0.1 mg)
- Accelerators¹⁾:
 - Tungsten
 - High purity iron chips



Calibration

The calibration of the analyzer is performed by means of cast iron reference material, e.g.: EZRM B 481-1, EZRM B 483-1 or other suitable reference material.



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 preheated²⁾ 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 preheated²⁾ 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 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 an appropriate amount of sample into a preheated²⁾ 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⁾ One scoop (~1.5 g) of tungsten and one scoop (~0.7 g) of iron chip is recommended for general purpose analysis of cast iron. Use two spoons (~3 g) of tungsten if sulfur contents below 50 ppm shall be determined with highest precision. For best precision, the accelerator shall be weighed to the nearest 0.1 g and the sample to accelerator ratio should stay constant.

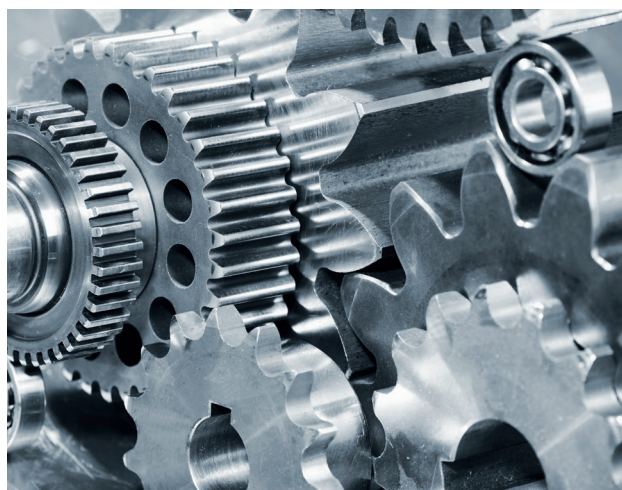
²⁾ For optimal precision, ceramic crucibles should be pre-heated in a muffle furnace at ≥ 1250 °C for a minimum of 15 min or ≥ 1000 °C for a minimum of 2 hours. To avoid contamination, crucibles must be handled with clean tongs and transferred to a desiccator for storage.

Results

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

EZRM B 481-1		
	certified values: C: 3.907 (\pm 0.009)% S: 0.0040 (\pm 0.0004)%	
Mass / g	Carbon / %	Sulfur / %
0.3033	3.901	0.039
0.3008	3.899	0.038
0.3059	3.911	0.042
0.3010	3.902	0.036
0.2993	3.912	0.041
Mean ³⁾	3.900	0.0039
STD ³⁾	0.006	0.0002

EZRM B 483-1		
	certified values: C: 2.463 (\pm 0.012)% S: 0.103 (\pm 0.002)%	
Mass / g	Carbon / %	Sulfur / %
0.2668	2.459	0.1032
0.3025	2.451	0.1030
0.3026	2.465	0.1039
0.3044	2.452	0.1039
0.2993	2.474	0.1053
Mean	2.461	0.104
STD	0.009	0.001



³⁾ Mean = arithmetic average; STD = absolute standard deviation (1σ)

Pig Iron (Ch11/3)		
Mass / g	Carbon / %	Sulfur / %
0.2045	4.50	0.0317
0.2025	4.49	0.0319
0.1990	4.51	0.0321
0.2072	4.47	0.0318
0.2031	4.50	0.0318
Mean	4.49	0.0319
STD	0.02	0.0002

Cast Iron 1		
Mass / g	Carbon / %	Sulfur / %
0.3028	1.369	0.0143
0.3055	1.368	0.0146
0.3093	1.372	0.0149
0.3014	1.380	0.0147
0.2967	1.372	0.0146
Mean	1.37	0.0146
STD	< 0.01	0.0002

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 and supports a complete and clean combustion. The analytical performance in combination with the speed of analysis and its ease of use makes the G4 ICARUS Series 2 the ideal choice for the quality control of cast iron, pig iron and even related foundry products like mold powder.



Cast Iron 2		
Mass / g	Carbon / %	Sulfur / %
0.3067	4.42	0.0040
0.3042	4.44	0.0040
0.3029	4.40	0.0040
0.3073	4.40	0.0039
0.3046	4.41	0.0040
Mean	4.41	0.0040
STD	< 0.02	< 0.0001

Cast Iron 3		
Mass / g	Carbon / %	Sulfur / %
0.3023	2.75	0.141
0.3071	2.74	0.138
0.3090	2.75	0.141
0.3060	2.72	0.140
0.3027	2.71	0.140
Mean	2.73	0.140
STD	< 0.02	< 0.002

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Bruker AXS GmbH
info.baxs@bruker.com

Worldwide offices
bruker.com/baxs-offices

Online information
www.bruker.com/csonh

www.bruker.com

