Lab Report CS/ONH 23

G4 ICARUS Series 2

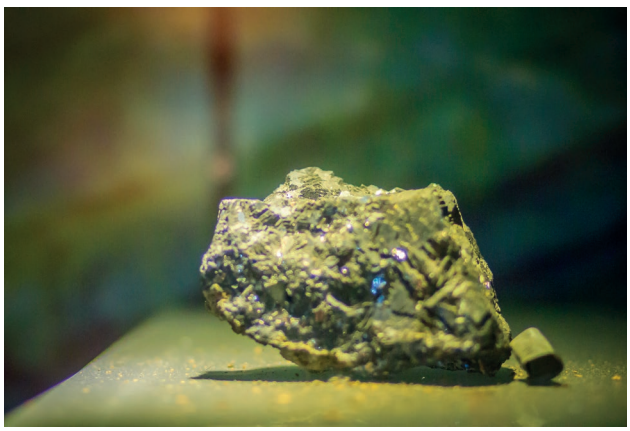
- Fast and reliable Carbon and Sulfur Determination in Ore Concentrates, Metal-Bearing Ores and related materials

Many metals occur naturally in form of sulfide minerals like Pyrite (FeS_2), Chalcocite (Cu_2S), Argentite (Ag_2S), Galena (PbS), Sphalerite (ZnS) and many more. Since the amount of economically important metal relative to the ore is small, a first step in the metal extraction process is the separation of target minerals from unwanted rock, gangue and waste minerals. The result, called ore concentrate, is a mixture of the target metal sulfide ores, other sulfide minerals and some gangue.

The sulfur level of the concentrate provides an estimation of the metal-in-concentrate and is used as a quality control measure, determining the efficiency of the concentration process.

In the metal recovery process, determining the carbon and sulfur level in metal-bearing ores is a necessary step for the mine operation to control the metallurgical process kinetics and to assess environmental aspects of the process waste. This is possible due to the fact that most metal-bearing

ores are a mixture of sulfide and carbonate minerals of known stoichiometry. Please refer to ASTM E 1915 for further process relevant information like acid-base characteristics. On the other end of the production process, pure products like MoS_2 are used as high performance additives to lubricants. 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. Especially its LED-powered HighSense™ SO_2 detector with extended high range makes it a valuable addition to any mining operations laboratory.



Molybdenite rock specimen from mining and quarrying industries. Molybdenite is a mineral of molybdenum disulfide, MoS_2.

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 Zone-Protect™, 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 equipped with the optional high range for the HighSense™ detector is capable to measure samples containing 50% sulfur with a high sample mass while still providing trace detection capabilities. A reasonable sample mass of ~0.1 g or more is essential for sulfidic sample material to keep the weighing error small and allow easy operation.

Note: Installation of an exhaust line at the outlet connector of the G4 ICARUS, running into a fume hood or a well ventilated area, is recommended.

Sample Preparation

For optimal precision, the sample should be grinded or crushed to a uniform powder prior to analysis and dried at 110 °C to constant weight. No further sample preparation is necessary.

Method Parameters

- Purge time: 10 s
- Start delay: 1 s
- Baseline check before analysis: 2 s
- Analysis time 1: 35 s (power level: 4)
- Analysis time 2: 35-45 s[1] (power level: 0)
- Baseline check after analysis: 2 s
- Crucible cool time: 10 s

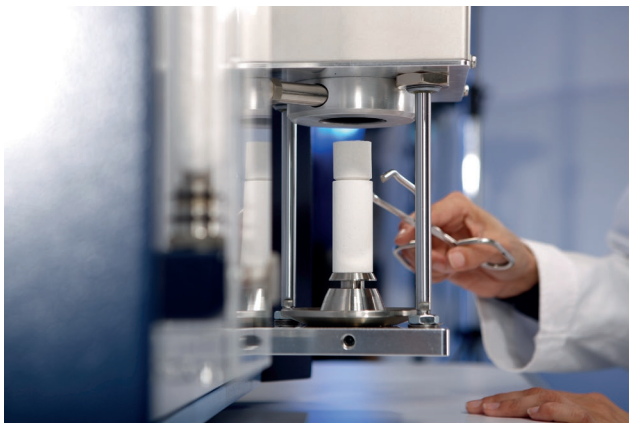
- Sample mass: ~0.1-0.2 g (weighed to 0.1 mg)
- Accelerators[2]:
  - Tungsten/Tin
  - High purity iron chips

Calibration

The calibration of the analyzer is performed by NIST, EZRM, Geostats (Australia) or other suitable ore concentrate reference material. Alternatively, pure substances like ZnS_2 or MoS_2 with suitable sulfur concentration can be used when a certificate of analysis and purity is provided.

1 Depending on sample mass and concentration. Start with higher time and reduce when detector signal returns to baseline level in-time.

2 One scoop (~1.5 g) of tungsten/tin and one scoop (~0.7 g) of iron chip is recommended for analysis of ores. Alternatively one scoop of Cu accelerator (~0.8 g) and one scoop (~0.7 g) of iron chip can be used for samples with high sulfur concentrations. Never use pure iron chip alone, as this will generate exorbitant splattering. For best precision, the accelerator shall be weighed to the nearest 0.1 g and the sample to accelerator ratio should stay constant.



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 pre-heated[3] crucible and analyze.

II. Measuring reference materials

1. Choose CRMs for calibration and define them in the analysis software with designation and the certified concentrations.
2. Weigh in an appropriate amount of reference material into a preheated[3] crucible, and transfer the exact mass into the analysis software. Cover the material with the described amount of accelerator and analyze.
3. 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.

IV. Sample measurement

1. Weigh an appropriate amount of sample into a preheated[3] crucible and transfer the exact sample mass to the analysis software. Cover the sample with the described amount of accelerator and analyze.
2. Repeat step 1 until an appropriate number of repetitions is obtained.
3. 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.

3 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.

Typical 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 ore samples.

CCU-1e (CCRMP)	certified values:	C: 0.100 (± 0.008)% S: 35.52 (± 0.22)%
Mass / g	**Carbon / %**	**Sulfur / %**
0.2003	0.105	35.61
0.2101	0.108	35.32
0.2017	0.102	35.47
0.1004	0.094	35.53
0.0997	0.101	35.69
0.1508	0.096	35.36
Mean[4]	**0.101**	**35.50**
STD[4]	0.005	0.14

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

GS309-4 (Cu, Pb, Zn, Ag ore)	certified values:	C: 0.22 (± 0.03)% S: 34.1 (± 1.3)%
Mass / g	**Carbon / %**	**Sulfur / %**
0.2010	0.21	33.7
0.2004	0.23	34.0
0.1998	0.20	33.3
0.1987	0.21	33.5
0.2013	0.23	33.7
Mean	**0.22**	**33.6**
STD	0.01	0.3

Iron concentrate, iron ore pellets and iron ore

Pyrite ore		
Mass / g	**Carbon / %**	**Sulfur / %**
0.1030	0.0717	53.5
0.1066	0.0721	53.3
0.0997	0.0728	53.6
0.1009	0.0726	53.3
0.1023	0.0712	53.6
Mean	**0.0721**	**53.5**
STD	<0.001	0.15

SnS_2 + C (lubricant)		
Mass / g	**Carbon / %**	**Sulfur / %**
0.1108	7.084	32.01
0.1054	7.061	32.16
0.1010	7.077	32.00
0.1998	7.066	31.96
0.2003	7.045	32.02
Mean	**7.07**	**32.03**
STD	0.02	0.08

MoS_2 (raw material)		
Mass / g	**Carbon / %**	**Sulfur / %**
0.0994	1.23	40.32
0.1002	1.18	40.22
0.1013	1.19	39.92
0.0512	1.22	39.96
0.0506	1.21	40.23
Mean	**1.21**	**40.13**
STD	0.02	0.18

Summary

The combination of HighSense™ detection 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. This makes the G4 ICARUS Series 2 an ideal choice for every mining operations laboratory.

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