

Measurement of Carbon and Sulfur in Cement

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1 Introduction

Carbon and Sulfur determination is very important for all kinds of cement samples. For exemplae, the sulfur content will affect the solidification point. This application note describes the method and conditions to use with the EMIA 820V C/S analyzer to measure cement.

2 Instrumentation

2.1 Principle

The test was performed on the model EMIA 820V. The measurement principle is shown in Figure 2.

The sample is placed in a ceramic crucible in a high frequency induction furnace. The sample is

heated at a programmable temperature. Gases produced during the combustion are then analyzed using four Infrared detectors, after dust and moisture removal. The analysis of SO_2 determines sulfur concentration. The analysis of low and high CO2 and CO determine carbon concentration.

2.2 Unique Features

2.2.1 - Programmable Temperature Curves

The high frequency or induction furnace is equipped with a plate current control function. This allows users to easily optimize the temperature according to the samples. Some customized temperature curves can be created in order to observe various phenomena such as surface contamination and different phases or forms of carbon and sulfur.



Figure 1: EMIA 820V





2.2.2 - Direct gas analysis without conversion

Four Infrared analyzers (NDIR) are are used to directly analyze CO, $\rm CO_2$ and $\rm SO_2$ over the full range of concentrations. No converter is used nor cellulose filter to trap $\rm SO_3$ generated in the converter.

2.2.3 - Computer System

All EMIA Series Analyzers are operated by a separate computer system. The software is compatible with Windows 95/98/2000/NT/XP. It includes several functions such as maintenance, diagnostis, statistical studies, curve and data traceability, etc.

2.2.4 - Automatic Cleaning

The double Auto Cleaner option features two brushes to simultaneously clean the combustion tube and the cylindrical dust filter after each measurement. The dust is removed to the dust box by a difference in pressure, which avoids the need for an external vacuum cleaner.

2.2.5 - Automation

It is possible to add standard modules for partial to full automation for 24/7 operation. For more detail see EA.TN 26: Options for Partial and Complete Automation.

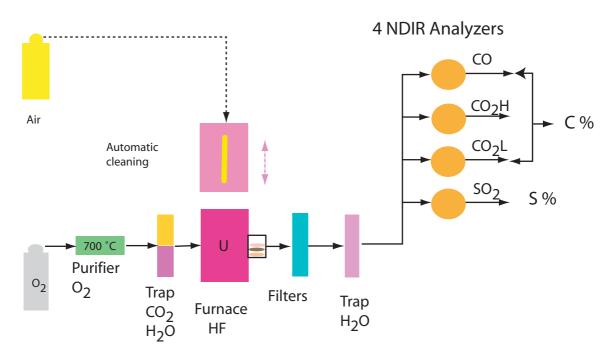


Figure 2: Operating principle

3 Cement preparation

The sample was in the form of a powder.

- 1. Weigh 0.1g of sample into a preburned ceramic crucible.
- 2. Weigh 0.5g of Pure Iron, 1.5g of Tungsten and 0.3g of Tin as accelerators into the crucible.
- 3. Set the ceramic crucible with sample on the crucible stand, and press the [START] button to begin analysis.





4 Conditions of analysis

Table 1: Operating conditions

	Start power (mA)	End power (mA)	Time from start to end power (sec)
Step 1	0	175	5
Step 2	175	175	35

	Carbon	Sulfur
Purge time	15 sec	15 sec
Integration wait time	5 sec	5 sec
Integration time	60 sec	70 sec
Comparator level	1.0 %	2.0 %
Comparator wait time	15 sec	25 sec

5 Calibration

- 1. Set up the system to the analytical conditions for the steel in the operator's instruction manual.
- 2. Calibrate the system following the procedure in the operator's instruction manual.
- 3. Weigh 1.5g of Tungsten and 0.3g of Tin as blank into a ceramic preburned crucible. Enter 1.0g as sample weight for blank analysis. Repeat the measurement a minimum of 3 times.
- 4. Weigh 1.0g of JSS 150-14 (C: 0.48mass%) into a ceramic preburned crucible. And cover the sample with 1.5g of Tungsten and 0.3g of Tin. Repeat the measurement a minimum of 3 times.
- 5. Weigh 1.0g of JSS 243-4 (S: 0.346mass%) into a ceramic preburned crucible and cover the sample with 1.5g of Tungsten and 0.3g of Tin. Repeat the measurement a minimum of 3 times.

- 6. Set the instrument using the operating conditions in Table 1.
- 7. Compensate the blank signal because the analytical conditions for the steel standard sample and the cement sample are different. (For details, refer to the content of the blank shift in the instruction manual.)
- 8. Weigh 0.5g of Pure Iron, 1.5g of Tungsten and 0.3g of Tin as blank in a ceramic preburned crucible. Enter 0.1g as sample weight for blank analysis. Repeat the measurement a minimum 3 times.

6 Results on cement

Table 2: Cement results

Weight (g)	Carbon (mass%)	Sulfur (mass%)
0.1001	0.193	0.834
0.1011	0.191	0.819
0.1008	0.192	0.820
0.1021	0.190	0.824
0.1007	0.191	0.823
Average	0.191	0.824
Standard Deviati	ion 0.001	0.006
RSD(%)	0.60	0.72
Range	0.003	0.015





7 Summary

Instrument: EMIA-820V C/S Determinator

Calibration: JSS 150-14 (C: 0.48 mass%)1.0g

JSS 243-4 (S: 0.346 mass%)1.0g

Sample: Cement

> Type: Powder Weight: 0.1g

Pure Iron (P/N 905.110.300.001) 0.5g Accelerator:

> Tungsten (P/N 905.110.140.001) 1.5g Tin (P/N 905.201.340.001) 0.3g

Crucible: Ceramic (P/N 905.202.200.001)

Crucible Preburned at 1000 °C

8 Conclusion

Carbon and Sulfur measurement in cement samples is compatible with the EMIA 820 V Series equipped with a high frequency furnace. The extraction is complete and efficient in all cases, and the results are repeatable.

This technical note is adapted from an Horiba technical note.





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