Application Note

Measurement of Carbon and Sulfur in Copper

Alain Salaville, Jérôme Barraqué, Application Laboratory, Longjumeau, FRANCE

Keywords: copper, carbon, sulfur, EMIA 820V

1 Introduction

Copper in reddish metal, it is malleable, ductile and an extremely good conductor of both heat and electricity. Copper metal is widely used for electrical wiring, water piping, and corrosion resistant parts, either pure or in alloy such as brass and bronze. Carbon and Sulphur are considered as impurities, so it is important to measure and check the Copper purity.

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 CO_2 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.

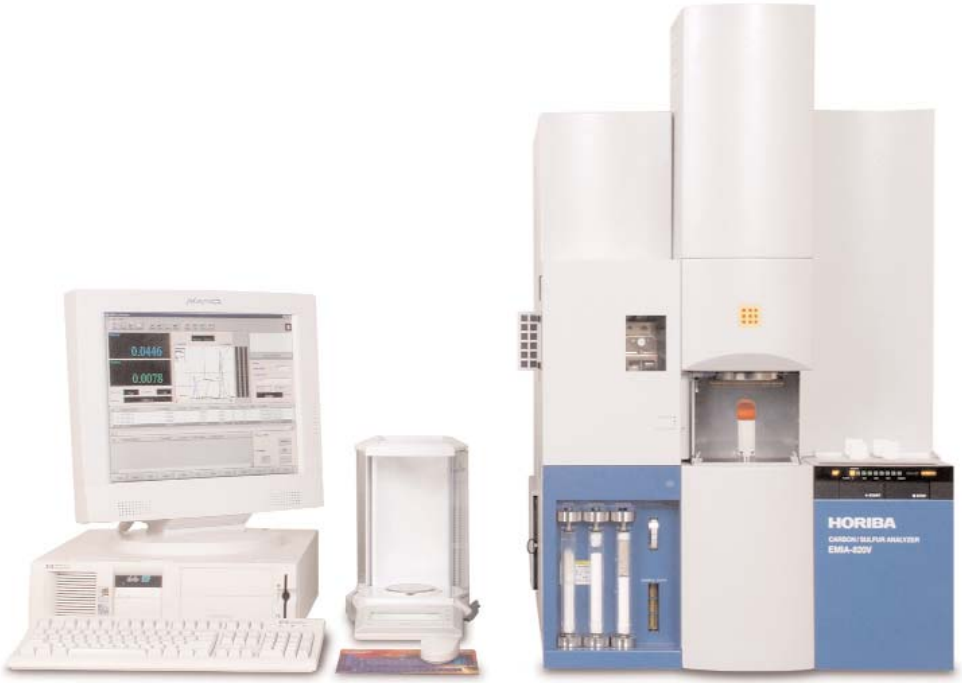

Figure 1: EMIA 820V

2.2.2 - Direct gas analysis without conversion
Four Infrared analyzers (NDIR) are used to directly analyze CO, CO_2 and SO_2 over the full range of concentrations. No converter is used nor cellulose filter to trap 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.

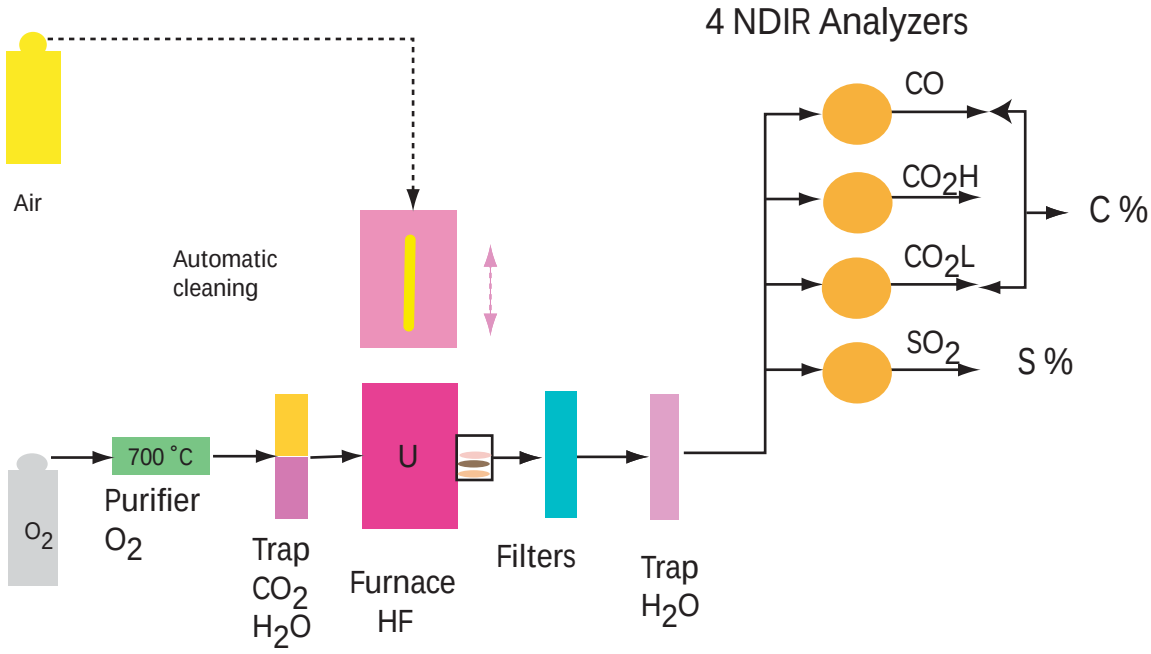

Figure 2: Operating principle

3 Sample preparation

The sample was in the form of a powder.
1. Rinse the sample with following reagents to remove the oil on the surface.
2. Rinse the sample in 30% nitric acid for one minute, wash the sample in deionized water three times.
3. Rinse the sample with fresh guaranteed Ethanol, repeat the three times.
4. Dry in the stream of warm air using heir dryer after washing the sample.
5. Weight 1.0g of sample into a ceramic crucible baked previously by another furnace.
6. Do not use accelerator, the contaminant from accelerator make blank value unstable.
7. Set the crucible with sample on the crucible stand, and press the [START] button to start 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	30 sec	50 sec
Comparator level	0.0 %	0.0 %
Comparator wait time		

5 Calibration

1. Set up the system to the analytical condition for the steel in the operator's instruction manual.

2. Calibrate the system following the procedure in the operator's instruction manual.

3. Weight 1.5g of Tungsten and 0.3g of Tin as blank into a ceramics crucible baked previously by another furnace. Enter 1.0g as sample weight for blank analysis. Repeat measurement 3 times at minimum.

4. Weight 1.0g of JSS 155-12 (C: 0.041 mass%, S 0.0060 mass %) into a ceramics crucible baked previously by another furnace. And cover the sample with 1.5g of Tungsten and 0.3g of Tin. Repeat measurement 3 times at minimum.

5. Change sample analysis condition to the above table condition.

6. Compensate the blank signal because analytical condition to steel standard sample and an unknown sample is different. (As for the details, refer to the content of the blank shift of the instruction manual.)

7. Measure the only ceramic crucible baked previously by another furnace as blank without accelerator. Enter 1.0g as sample weight for blank analysis. Repeat measurement 3 times at minimum.

6 Results on Copper

Table 2: Copper

Weight (g)	Carbon (mass%)	Sulfur (mass%)
1.005	2.53	8.50
1.009	2.89	9.18
1.006	2.68	9.87
1.002	2.79	10.44
1.008	2.85	9.68
Average	2.75	9.53
Standard Deviation	0.15	0.73
RSD(%)	5.3	7.7
Range	0.36	1.94

7 Summary

Instrument: EMIA-820V C/S Determinator

Calibration: JSS 150-12 (C: 0.041 mass% 1.0 g, S: 0.0060 mass%) 1.0g

Sample: Copper
Type: Wire (1 dia*10mm)
Weight: 1 g

Accelerator: None

Crucible: Ceramic (P/N 905.202.200.001)

Crucible Preburning: Crucible Preburning unit (FK-10)

8 Conclusion

Carbon and Sulfur measurement in Copper 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.

In the USA:
HORIBA Jobin Yvon Inc.
3880 Park Avenue
Edison, NJ 08820
Tel: 1-732-494-8660
Fax: 1-732-494-8796
E-mail: info@jobinyvon.com

In France:
HORIBA Jobin Yvon S.A.S.
16-18, rue du Canal
91165 Longjumeau Cedex
Tel: (33) 1/64 54 13 00
Fax: (33) 1/69 09 90 88

Germany: (49) 89/46 23 17-0
Italy: (39) 2/57 60 56 90
U.K.: (44) 20/82 04 81 42

In Japan:
HORIBA Ltd.
2 Miyanohigashi, Kisshoin
Minami-ku, Kyoto 601-8510
TEL: (81) 75 313 8121
FAX: (81) 75 321 5725
www.jyhoriba.jp

China: (86) 10/6849 22 16
Spain: (34) 91/724 16 57

Other Countries: Contact HJY S.A.S.

www.jobinyvon.com