Application Note

Measurement of Sulfur in Rubber

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1 Introduction

Sulphur is added in the natural rubber to produce plastic materials. However, it is important to measure the sulphur contained in the rubber to define the chemical and mechanical properties of the final product.

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 con-

Figure 1: EMIA 820V

tamination and different phases or forms of carbon and sulfur.

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.

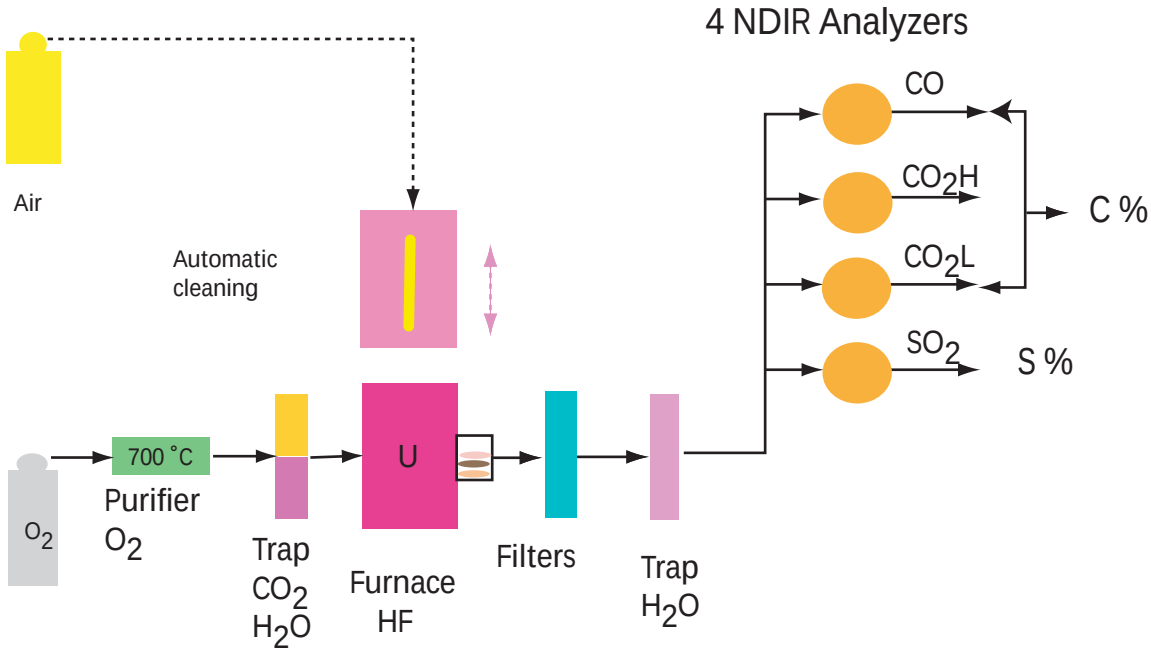

Figure 2: Operating principle

3 Sample preparation

The sample was in the form of a powder.

1. Weight 0.05 g of sample into a ceramics crucible

2. Weight 0.5 g of pure iron, 1.5g of Tungsten and 0.3 g Tin as accelerator, And cover the sample with each metal.

4. Set the ceramic 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	30

	Carbon	Sulfur
Purge time		15 sec
Integration wait time		5 sec
Integration time		100 sec
Comparator level		2.0 %
Comparator wait time		40 sec

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. Enter 1.0g as sample weight for blank analysis. Repeat measurement 3 times at minimum.

4. Weight 1.0g of JSS 243-4 (S:0.346mass%) into a ceramics crucible. 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 Rubber sample is different. (As for the details, refer to the content of the blank shift of the instruction manual.)

7. Weight 0.5g of Pure Iron, 1.5g of Tungsten and 0.3g of Tin in the crucible. Enter 0.05g as sample weight for blank analysis. Repeat measurement 3 times at minimum.

6 Results on rubber carbide

Table 2: Rubber

Weight (g)	Carbon (mass%)	Sulfur (mass%)
0.05102		3.159
0.05052		3.122
0.05087		3.127
0.05112		3.174
0.05092		3.170
Average		3.150
Standard Deviation		0.024
RSD(%)		0.77
Range		0.052

7 Summary

Instrument: EMIA-820V C/S Determinator

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

Sample: Rubber
Type: Chip
Weight: 0.05 g

Accelerator: Pure iron (P/N 905.110.300.001) 0.5g
Tungsten (P/N 905.110.140.001) 1.5g
Tin (P/N 905.202.200.001)0.3g

Crucible: Ceramic (P/N 905.202.200.001)

8 Conclusion

Sulfur measurement in rubber 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.

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