

Application News

Energy Dispersive X-Ray Fluorescence Spectroscopy

SSI-XRAY-001

Measuring Sulfur Content in Carbon Black using Shimadzu EDX-720 Energy Dispersive X-ray Fluorescence Spectrophotometer

Introduction

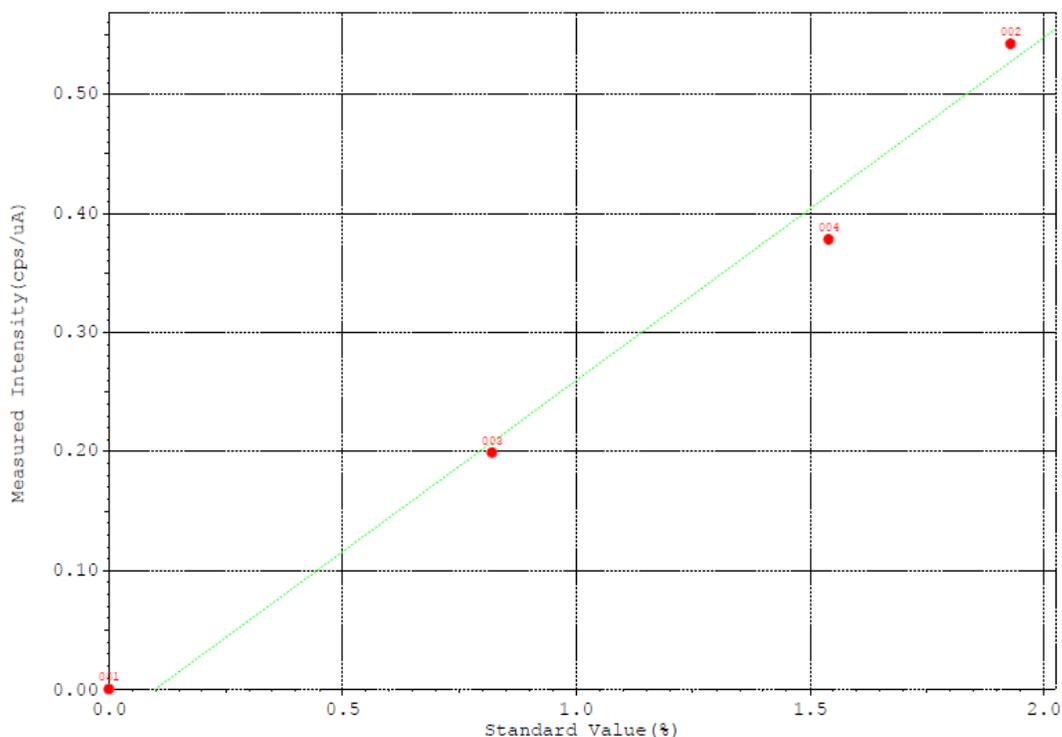
Carbon black is a material produced by the incomplete combustion or thermal decomposition of gaseous or liquid hydrocarbons. It is a form of amorphous carbon that characteristically has a high surface-area-to-volume ratio. The main use for carbon black is in the production of rubber products, especially tires, where it is used as a pigment and a filler to reinforce the rubber in the tire and add strength. It is also used in many plastic products and toners and inks used in printers.

With increasing environmental concern, it is critical for manufacturers of carbon black to monitor the amount of sulfur in their product in order to minimize the amount emitted into the atmosphere and to also ensure the carbon black meets the customer's specifications. In order to effectively measure the sulfur released to the atmosphere, the sulfur content of the starting material (typically petroleum or natural gas) is determined, then the sulfur content of the carbon black is measured and the difference is the amount that was released into the atmosphere.

Many carbon black manufacturers are now using energy dispersive x-ray fluorescence (EDXRF) methodology to quickly and accurately measure the sulfur content. ASTM method D1619-11, **Standard Test Methods for Carbon Black – Sulfur Content**, describes two different methods for measuring the sulfur content in carbon black. These methods are 1) High temperature combustion with infrared absorption detection, and 2) X-ray fluorescence. This study will focus on the X-ray fluorescence method using the Shimadzu EDX-720 Energy Dispersive X-Ray Fluorescence Spectrophotometer and carbon black sulfur standards from Laboratory Standards and Technologies in Borger, Texas.

Calibration

The Carbon Black Sulfur Standards were analyzed using the Shimadzu EDX-720 and used to create a calibration curve. The Standards were measured in a Helium atmosphere using a 10mm collimator.



No.	Flag	Standard Sample	Std.Value %	Calc.Value	Dev.	--- Int. (cps/uA) --- Net	BG
1	GC	Std A	0.0000	0.0984	-0.0984	0.000147	0.005109
2	GC	Std C	1.9300	1.9805	-0.0505	0.541986	0.017200
3	GC	Std D	0.8200	0.7890	+0.0310	0.198961	0.011959
4	GC	Std B (new)	1.5400	1.4102	+0.1298	0.377806	0.014338

Sample Measurement

After calibration, the four carbon black sulfur standards were analyzed as samples to verify that the EDX-720 is capable of accurately measuring the sulfur content in the samples. Below is a list of the actual sulfur content in the standards as well as the sulfur content measured by the instrument.

Standard	Actual % sulfur	Measured % sulfur
A	0.00	0.101
B	1.54	1.330
C	1.93	1.803
D	0.82	0.736

Conclusion

The Shimadzu EDX-720 Energy Dispersive X-Ray Fluorescence Spectrophotometer proves to be a reliable and accurate instrument for measuring the sulfur content in carbon black samples. It requires no sample prep, no chemicals, and is non-destructive to samples. Furthermore, the instrument does not require consumable parts like catalysts or combustion tubes, has few moving parts to maintain, and does not have a high-temperature furnace, making it the ideal instrument for quick and reliable measurements of sulfur in carbon black.