

ASOMA® PHOENIX II

Determination of Chlorine in Rubber and Windshield Wiper Blades



Summary

This report demonstrates the suitability of the PHOENIX II XRF analyzer for determining the concentrations of chlorine in rubber and in finished windshield wiper blades. It will show that use of this instrument will deliver improvements in product quality by accurately and rapidly determining the chlorine content, which affects the rigidity of the rubber and the wiping smoothness of wiper blades.

The advantages can be seen in easily and quickly measuring the chlorine. Namely, ensuring proper chlorine levels to control product quality and minimize waste of over use of chemicals. Thus, the PHOENIX II can be used to minimize costs and maximize profitability.

Introduction

The PHOENIX II is an excellent benchtop XRF analyzer for at-line QC analysis or the laboratory alike. The PHOENIX II offers a fast, precise, simple and non-destructive analysis technique well suited for the determination of chlorine in bulk rubber stock and finished rubber products. Monitoring the chlorine is important in several applications. For example, the chlorine content helps determine the stiffness or rigidity of the rubber. In the finished windshield wiper blades, a proper chlorine level helps minimize wear and the blade squeaking against the window.

The PHOENIX II employs state-of-the-art optics. Polarization excitation offers unique benefits because it eliminates most of the background scatter emerging from the X-ray tube before it arrives at the sample. This results in a dramatic improvement in peak-to-background signal, especially in highly scattering materials such as rubber. This translates to vastly improved precision and lower detection limits than traditional direct excitation XRF systems can achieve.

The PHOENIX II uses an onboard PC computer with a simple touch screen interface. Thus, an external computer is not required. Data handling and results storage can be obtained on a thermal paper print out and are also stored in the hard drive of the PHOENIX II. The data can be readily transferred to a USB thumb-drive or a network Ethernet connection.

Calibrations are readily carried out using assayed standards, usually of flat rubber sample panels. This ensures easy traceability of results for quality purposes. This initial calibration process is a “once



only" procedure. Subsequently, the curve can be restandardized, if required, by the touch of a button on the main analysis screen.

The PHOENIX II offers power, versatility and performance all in a small, compact, easy-to-use design.

Experimental Portion

Equipment

All measurements were conducted using a PHOENIX II XRF analyzer. Performance is shown for using a total measurement time of 200 seconds.

Sample Preparation

Sample preparation is minimal. For bulk rubber samples simply cut a coupon approximately 2X2 inches square and place it over the aperture in the analysis chamber. For wiper blade samples, lay the section of blade in the specially designed sample holder for windshield wiper blades and screw down the sample holder top. This ensures the blade is kept flat for analysis. Once the sample is in place simply press the green ANALYZE button.

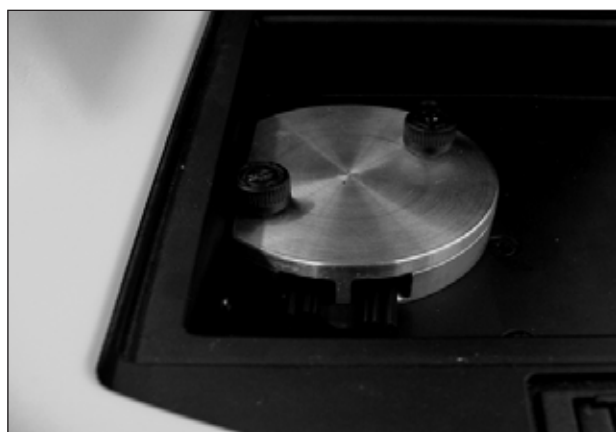
Flat Sample Preparation

White paper sample shown for better viewing



Windshield Wiper Blade Preparation

Unique SPECTRO sample holder



Measurement Parameters

All measurement parameters are easily controlled through the touch screen on the display panel. Operators simply choose the correct method from the analysis screen (there may be more than one Method stored, e.g. to measure bulk rubber samples or windshield wiper blades) and then press the green ANALYZE button.

The results can be reported using a variety of different options: results are reported on the display screen; on a thermal paper printout; on an optional external printer; and in the database history within the analyzer.

Instrument Configuration

ASOMA® PHOENIX II

Excitation: 48 kV 50 W Air-cooled X-ray Tube

Detection: Gas-filled Proportional Counter

Analytes: Cl

Optimization: X-ray voltage, current and X-ray filters

Atmosphere: Air

Options: HOPG Target, Detector filters *; Polypropylene 4 µm film

* If sulfur is also present in the rubber, filters are used to spectrally separate the sulfur and chlorine X-ray peaks. The hardware configuration is optimized for the measurement of Cl and S.

Note: No helium purge is required.

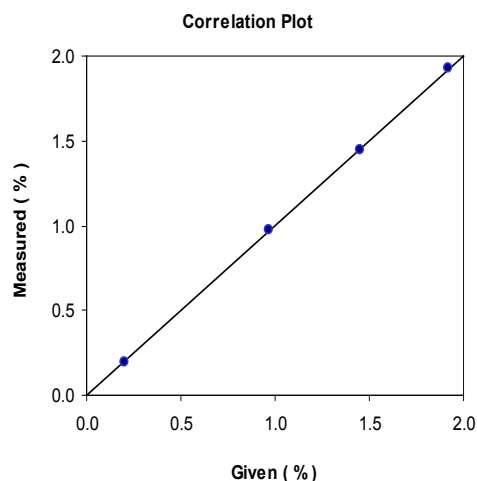
Results

Empirical calibrations are built using assayed standards supplied by customers. The most accurate results are obtained when using such “matrix-matched” calibration technique and calibration standards used are made using the rubber formulation of interest.

Chlorine in Rubber

Calibration for Chlorine in Rubber

Element: Cl		
Units: %		Std. Error of Estimate: 0.0077
Sample	Given	Measured
1	0.20	0.199
2	0.97	0.976
3	1.45	1.441
4	1.92	1.924



Precision for Chlorine in Rubber

10 repeat analyses at 200 seconds per measurement

Element: Cl				Units: %
Sample	Given	Mean	Std. Dev.	% Rel.
1	0.20	0.193	0.002	1.3
2	0.97	0.971	0.005	0.6
4	1.92	1.913	0.010	0.4

Determination of Chlorine in Rubber and Windshield Wiper Blades

Minimum Detection Limit (MDL)

Chlorine in Rubber

The Minimum Detection Limit (MDL) for an element is determined as three times the standard deviation of ten analyses of a blank sample. The following MDL was derived using this empirical method and applies to this matrix and concentration range.

Element	Count Time	MDL
Cl	200 sec	0.0026 %

Precision for Chlorine in Windshield Wiper Blades

10 repeat analyses at 200 seconds per measurement

Element: Cl			Units: %	
Sample	Given	Mean	Std. Dev.	% Rel.
1	0.5	0.494	0.007	1.4
4	2.0	1.998	0.014	0.7

Chlorine in Windshield Wiper Blades

Calibration for Chlorine in Windshield Wiper Blades

Element: Cl		
Units: % Std. Error of Estimate: 0.0045		
Sample	Given	Measured
1	0.5	0.50
2	1.0	1.01
3	1.5	1.49
4	2.0	2.01

Minimum Detection Limit (MDL)

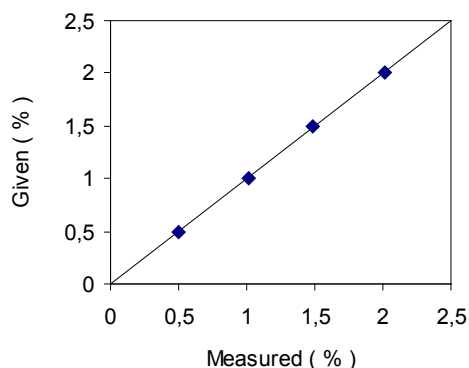
Chlorine in Wiper Blades

The Minimum Detection Limit (MDL) for an element is determined as three times the standard deviation of ten analyses of a blank sample.

The following MDL was derived using this empirical method and applies to this matrix and concentration range.

Element	Count Time	MDL
Cl	200 sec	0.020 %

Correlation Plot



Conclusion

As can be seen from the above data, the use of the PHOENIX II gives excellent performance when applied to the determination of chlorine in rubber and finished windshield wiper blades. Results are rapid, precise and analysis is easily carried out, even by non-laboratory personnel. Because no consumable chemicals are used, the relative "cost of ownership" is much lower than other analytical techniques.



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