

Application Note # XE-2015-3290

Quantitative analysis of Si in rubber samples

EDXRF Analyzer X-Calibur

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All samples were analyzed using the Xenemetrix X-Calibur Bench-top system. This report demonstrates the capability of the Xenemetrix X-Calibur SDD to analyse Si in rubber samples using regression method. Si in oil certified reference standard from VHG Lab was used to calibrate the instrument and the measurements on the unknown samples were carried out. Using at least one "type standard" is required to improve the method.

1. Introduction

X-Calibur is an excellent bench-top XRF analyzer for qualitative and quantitative analysis of elements. It offers an accurate, fast, precise, simple and non-destructive analysis technique. Well suited for elemental quantitative analysis of different types of samples. The X-Calibur employs state-of-the-art closed coupled optics, which enable a very powerful and count efficient analysis. The high count rate is translated into excellent precision and low detection levels of all elements.

Objective

To show the capability and ease of use of Xenometrix X-Calibur SDD analyzer to quantify the Si content in rubber samples.

2. Experimental

Equipment

All measurements were conducted using an X-Calibur SDD EDXRF analyzer connected to a vacuum pump to optimize the Si detection. The acquisition time was 30 sec per sample.

Samples

Five rubber samples were received and analyzed “as is”, without any sample preparation. No type samples were received for calibration the X-Calibur. Thus, 1000 ppm Si in oil certified reference standard from VHG Lab was used to calibrate the analyzer.

Measurement Parameters

All measurement parameters are easily controlled through the software. Operators simply choose the correct method from the analysis screen (there may be more than one method stored). The results can be reported using a variety of different options: results are reported on the display screen; result report can be sent to an external printer; results can be saved in the database history within the analyzer.

Instrument Configuration: X-Calibur

Tube Excitation: Max 50W, Air-cooled X-ray tube, Rh anode

Detection: Silicon Drift Detector (SDD)

Acquisition time: 30 sec

Atmosphere: Vacuum

3. Results

Qualitative analysis

A typical spectrum of one of the samples is shown in Figure 1 below.

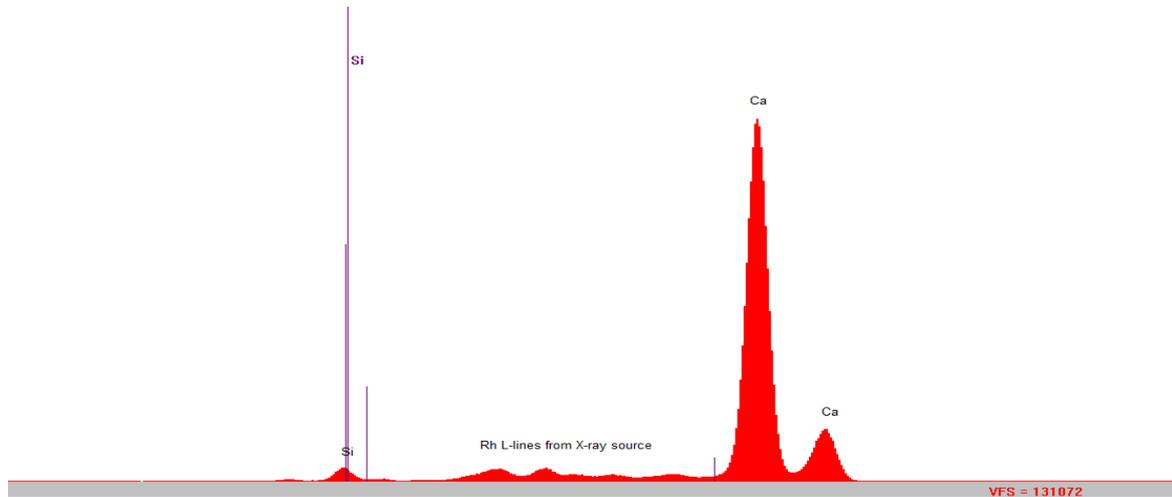


Figure 1: Typical spectrum of sample 1

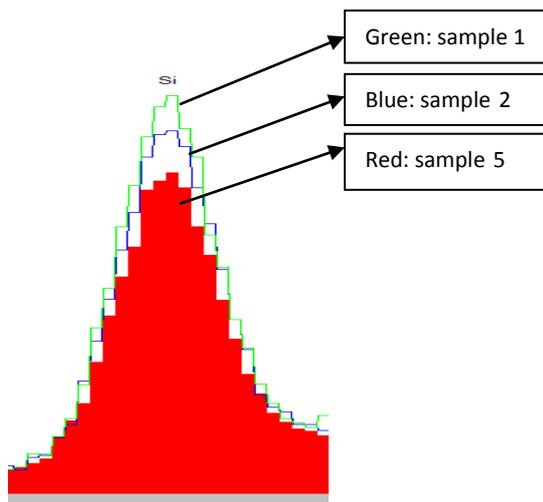


Figure 2: Si peak comparison between samples 1, 2 and 5.

Quantitative analysis

Two pieces of each sample were supplied. Thus, the results shown in table 1 for each sample are the average concentration on two pieces.

Table 1: Quantitative analysis of unknown rubber samples

Sample ID	Si Conc. [ppm]
1	2925
2	2807
3	2700
4	2636
5	2449

4. Conclusion

This application report shows the great potential of EDXRF technique and Xenemetrix X-Calibur analyzer to perform qualitative and quantitative elemental analysis on rubber materials in short time and without sample preparation at all.

For calibration the X-Calibur, one certified reference standard of Si in oil was used. However, in order to perform more accurate and optimal quantitative analysis, the following points are strongly recommended:

1. Using type standards "standards with known concentration and similar matrix as the unknown samples".
Type standard can be easily achieved by sending one of the unknown samples to wet chemistry analysis and then use it as calibration standard.
2. Using at least three type standards to calibrate the analyzer with concentration range suitable for the "target expected concentration".



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