APPLICATION NOTE

No. AN41672

Analysis of catalyst for NOx reduction

Thermo Scientific ARL PERFORM'X Series Advanced X-Ray Fluorescence Spectrometers

Key words
ARL PERFORM'X 4200 W, Catalyst, XRF, X-ray fluorescence

Introduction

Environmental regulations are becoming stricter in regards to toxic emissions. Companies must monitor and control the amount of NOx gas that is emitted from their factories. To control the emissions, the toxic gases must be passed through systems known as scrubbers, where the NOx gas is broken into harmless components such as N_2, O_2 and H_2O.

Scrubbers are selective catalytic reductions (SCR manufactured from a variety of ceramic materials and used as a carrier for active catalytic compounds. The catalyst materials can vary from base metals, zeolites or various precious metals.

The most commonly used material is zeolite for the catalysts. Zeolite catalysts are able to withstand extreme high temperature and are very durable. The catalysts are normally designed in one of three ways. Either as a plate, a honeycomb geometry or in a corrugated orientation. The honeycomb configuration is produced by an extruder process and is used as a ceramic carrier for low dust conditions. The plate types are coated using lower pressure drops or the coating is rolled onto the wire mesh.

The catalytically active materials used are compounds such as titanium dioxide, vanadium oxide and tungsten oxide. For routine process control, these and many other commonly found elements in catalytic materials can easily be analyzed using wavelength x-ray fluorescence (WDXRF).

WDXRF analysis is a stable fast analytical technique which requires very little sample preparation. Elemental analysis techniques such as ICP or AA require the samples to be digested and placed into an aqueous solution for analysis. In contrary, samples in WDXRF simply can be analyzed as loose powder, pressed pellet, lithium borate fusion or even in a liquid solution.

Instrument

Thermo Scientific ARL PERFORM'X series spectrometer used in this analysis was a 4200 watt system. This system is configured with 6 primary beam filters, 4 collimators, up to nine crystals, two detectors, helium purge and our 5GN+ Rh X-ray tube for best performance from ultra- light to heaviest elements thanks to its 50 micron Be window. This new X-ray tube fitted with a low current filament ensures an unequalled analytical stability month after month.

The ARL PERFORM'X offers the ultimate in performance and sample analysis safety. Its unique LoadSafe design includes a series of features that prevent any trouble during sample pumping and loading. Liquid cassette recognition prevents any liquid sample to be exposed to vacuum by mistake. Over exposure safety automatically ejects a liquid sample if X-ray exposure time is too long.

The Secutainer system protects the primary chamber by vacuum collecting any loose powders in a specially designed container, easily removed and cleaned by any operator. For spectral chamber protection, the ARL PERFORM'X uses a helium shutter designed for absolute protection of your goniometer during liquid analysis under helium operation. In the "LoadSafe Ultra" optional configuration, a special X-ray tube shield provides total protection against sample breakage or liquid cell rupture.

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Calibration

Analyzing the catalyst samples accurately and precisely requires not only the detection of low X-ray intensities at the trace elemental concentrations but also at very high concentrations. For optimal analytical analysis, the conditions and parameter used in this measurement were set to maximize the excitation of each element while keeping the background as low as possible.

A set of certified standards were used in the creation of elemental regression plots. Plots for Al, Ca, Si and Zr are shown in below graphs. These graphs are linear regression of the known concentration plotted against the measured intensities. The linearity of these curves depends upon the stability of the instrument, inter-elemental correction capabilities of the software, the accuracy of the standard and the quality of the sample preparation.

Results

The calibration ranges are the dynamic range within the calibration standards used for this report. These ranges can be increased by the addition of standards containing the newly desired concentration. The standard deviations (SD) and relative standard deviations (RSD) are the typical deviation from the standard values achieve from the linear regression at 60 seconds counting times.

Elements	Calibration Range (%)	SD (%)	RSD (%)	LoD (%)
Al Kα	0.08 - 100	0.0066	0.171	0.0051
Ca Kα	0.04 - 100	0.0156	0.027	0.0004
Co Kα	0.5 - 1.0	0.0001	0.333	0.0004
Cr Kα	0.004 - 12	0.0002	1.053	0.0006
Fe Kα	0.05 - 50	0.0018	0.036	0.0007
Hf Lβ	1.0 - 2.5	0.0004	0.432	0.0022
K Kα	0.001 - 15	0.0006	0.126	0.0006
Mg Kα	0.015 - 100	0.0061	0.252	0.0086
Mn Kα	0.05 - 10	0.0006	0.288	0.0006
Na Kα	0.01 - 8.0	0.0080	0.801	0.0196
P Kα	0.03 - 12	0.0004	0.450	0.0010
S Kα	0.4 - 3.0	0.0035	0.189	0.0030
Si Kα	0.4 - 100	0.0154	0.072	0.0006
Ti Kα	0.01 - 5.0	0.0005	0.468	0.0080
W Lα	1.0 - 5.0	0.0005	0.531	0.0010
Y Kα	0.5 - 1.0	0.0001	0.099	0.0001
Zr Lα	0.03 - 100	0.0005	0.306	0.0018

Table 1: Typical results for catalysts

Standardless analysis

One of the most useful developments in the analytical software programs for XRF has been the availability of “standardless” or semi-quantitative packages. These packages allow for quantitative data to be obtained for complete unknown samples.

Thermo Scientific UniQuant™ is a factory calibration based on 64 pure element standards that allows for concentration determination of unknown samples in any matrix by using complex mathematical algorithms for up to 79 elements. These algorithms correct for matrix effects as well as inter-elemental effects to provide a highly accurate and precise quantitative results.

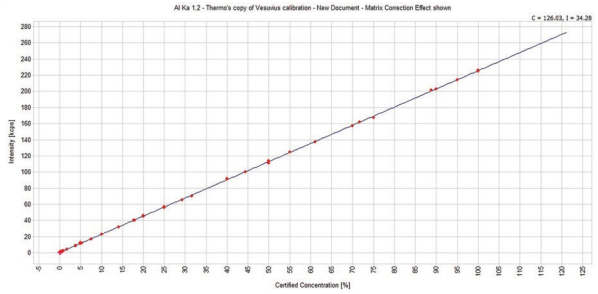
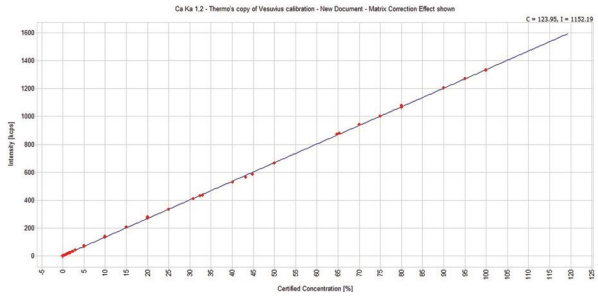


Figure 1: Al Regression Plot



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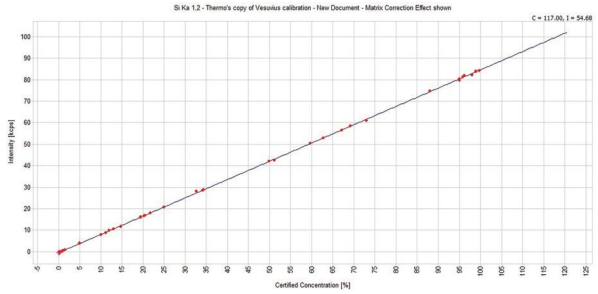

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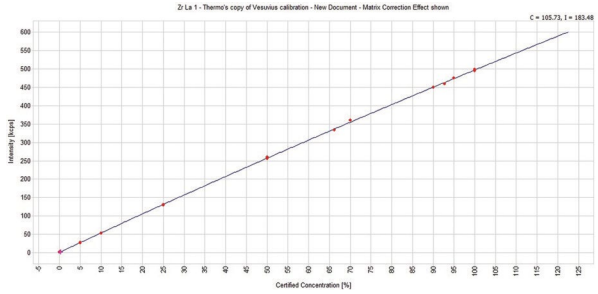

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Conclusion

The results show that catalyst analysis can easily be performed with the ARL PERFORM'X sequential XRF spectrometer. The precision and accuracy are shown to be incredibly high in this matrix type. Of course the accuracy and precision can easily be increased by extended the elemental counting times. This would allow for much better SD and %RSD at all concentration ranges.

Furthermore, operation is made easy through the advanced state-of-the-art Thermo Scientific OXSAS WDXRF software which operates with the latest Microsoft Windows® 10 packages.

Find out more at **thermofisher.com/xray**

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