APPLICATION NOTE

No. AN41663

Analysis of refractories

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

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

Introduction

Refractories are, as defined by ASTM C7, non-metallic materials having chemical and physical properties allowing for applications in structures or as components of system that are exposed to environmental conditions above 1000 F (538 C). These materials are mainly used as linings in furnaces, kilns, incinerators and reactors

To meet the requirement of extreme heat exposure, refractories are normally compositions of the oxide formed aluminum (alumina), silicon (silica), magnesium (magnesia) and zirconium (zirconia).

The main elements must be monitored in the production of the refractories to insure proper composition for the desired application. Many minor and trace elements must also be monitored in the refractories to insure optimal stability and reduce potential contamination issues for their customer.

Refractories are highly stable oxide composition, making it very difficult to dissolve the samples for standard wet chemical or flame AA/ ICP analysis. It is for this reason that wavelength dispersive X-ray fluorescence (WDXRF) is an ideal instrument for daily routine process quality assurance and control along with advance research and development.

WDXRF analysis is a stable fast analytical technique which requires very little sample preparation. 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.

Calibration

Analyzing the refractory 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.

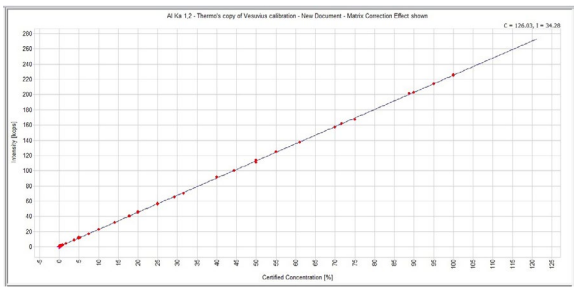

Figure 1: Al Regression Plot

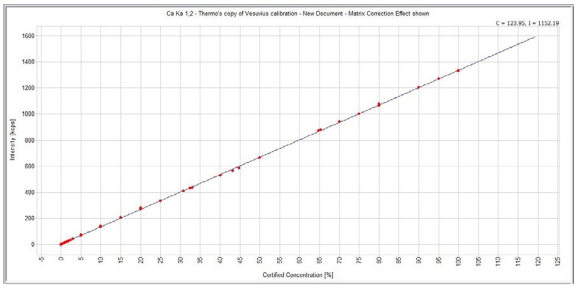

Figure 2: Ca Regression Plot

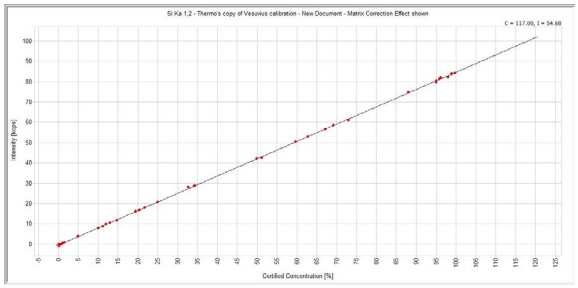

Figure 3: Si Regression Plot

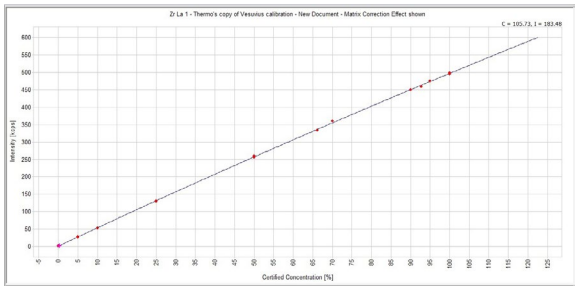

Figure 4: Zr Regression Plot

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.

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.

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 Lß	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 La	1.0-5.0	0.0005	0.531	0.0010
Y Kα	0.5-1.0	0.0001	0.099	0.0001
Zr La	0.03-100	0.0005	0.306	0.0018

Table 1: Typical results for refractories

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Conclusion

The results show that refractory 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 newest and most advance state-of-the-art Thermo Scientific OXSAS WDXRF software which operates with the latest Microsoft Windows® 10 packages.

To see our full X-ray product portfolio, please visit
thermofisher.com/xray

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