Application Note:
AN41650

Small Spot Analysis: Performance in Ferrous Base

Thermo Scientific ARL PERFORM'X 4200 WDXRF Spectrometer

Key Words

- ARL PERFORM'X
- Small spot analysis
- Ferrous base
- XRF
- X-ray fluorescence

Introduction

To widen the usual capabilities of X-ray fluorescence analysis, the ARL PERFORM'X spectrometer now offers the ability for small spot analysis. These analyses can be either qualified or quantified by simply measuring a chosen spot on the sample surface.

The small spot analysis size allows determination of embedded particles or inhomogeneous specimens and product failure investigations. With a choice of 1.5 mm or 0.5 mm, the ARL PERFORM'X spectrometer offers a perfect complement to bridge the gap between traditional bulk analysis and standard micro-analysis using microscopic techniques such as SEM. A camera is included for selecting the spots of interest.

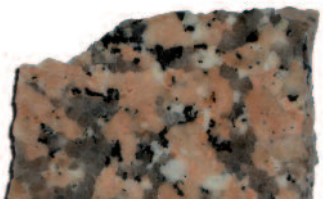

Figure 1: Example of an inhomogeneous sample

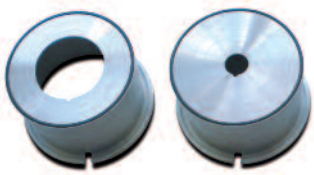

Figure 2: Cups for small spot analysis with 30 mm and 10 mm apertures

Instrument

The ARL PERFORM'X spectrometer used in this analysis was a 4200 watt system. This system is configured as standard with 6 primary beam filters, 4 collimators, up to nine crystals, two detectors, and our 5 GN Rh anode X-ray tube for best performance from ultra light to heaviest elements.

The ARL PERFORM'X analyzer also features small spot analysis allowing for 1.5 mm and 0.5 mm areas. All of the results presented in this application note used the 0.5 mm spot analysis configuration.

Iron base calibration at 0.5 mm

In order to assess the performance that can be reached when analyzing a spot of only 0.5 mm a calibration has been performed using ferrous base standard samples. The standards used in this small spot calibration can have diameters ranging from 11 mm to 52 mm; however they must be homogeneous in composition.

Typical examples of these calibration curves are shown in Figures 1, 2 and 3 (reverse side).

Table 1 gives a summary of limits of detection obtained using a set of international steel standards with the 0.5 mm spot. Results were obtained by using 100 seconds per element counting times and the best conditions and parameters in regards to crystal, detector, collimator and power.

Typical stability and precision tests

A stability test consisting of running a typical sample over 10 days was performed. Each element was analyzed using 100 seconds per element. The stability of an instrument reflects the precision that can be obtained. It should be noted that the accuracy of the instrument is dependent upon the accuracy of the standards used to calibrate the instrument. The stability for each element is given in Table 2 (reverse side) along with the certified and calculated concentrations.

ELEMENT	LINE	LOD (PPM) 100s
Al	Kα	840
Si	Kα	725
P	Kα	208
S	Kα	160
Ti	Kα	317
V	Kα	205
Cr	Kα	160
Mn	Kα	250
Co	Kα	390
Ni	Kα	340
Cu	Kα	220
Ta	Lβ	330
Zr	Kα	57
Nb	Kα	55
Mo	Kα	60
Sn	Kα	265

Table 1: Limits of detection in ferrous base for 0.5 mm spot size

Standard-less analysis for small spots

The most useful development in the analytical programs in XRF has been the availability of "standard-less" packages. These packages allow for quantitative data to be obtained for completely unknown samples.

As in many real life situations, obtaining any or enough standards to create a calibration is not always possible. This is certainly the case when analyzing defects or unknown contamination. In such situations, we can offer the most comprehensive standard-less software on the market: Thermo Scientific UniQuant package. It 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 highly accurate and precise quantitative results.

Conclusion

It is seen that analyses using small spots can easily be performed with the ARL PERFORM'X sequential XRF spectrometer. The precision and accuracy are excellent for such a small analyzed area in these matrix types for routine or R&D analyses. When an approximate analysis of an inclusion or a defect in a sample is sufficient, a standard-less program like UniQuant will provide good results without use of specific standard samples. Sometimes only a qualitative analysis using a fast scan method may also be sufficient.

Furthermore, operation is made easy through the new state-of-the-art OXSAS software which is able to operate with the latest Microsoft Windows® 7 system.

ELEMENT	LINE	CERT. CONC. (%)	ANALYZED CONC. (%)	STD DEV (%)
Al	Kα	0.24	0.21	0.047
Cr	Kα	1.31	1.32	0.015
Cu	Kα	0.1	0.10	0.010
Mn	Kα	1.5	1.47	0.026
Mo	Kα	0.03	<0.03	0.002
Nb	Kα	0.05	0.06	0.003
Ni	Kα	0.32	0.32	0.017
P	Kα	0.03	0.02	0.008
Si	Kα	0.74	0.63	0.041
Sn	Kα	0.1	0.10	0.009
Ta	Lβ	N.A.	0.07	0.015
Ti	Kα	0.05	0.04	0.012
V	Kα	0.31	0.29	0.012
Zr	Kα	0.05	0.07	0.003

Table 2: Accuracy and precision data in ferrous base for 0.5 mm spots

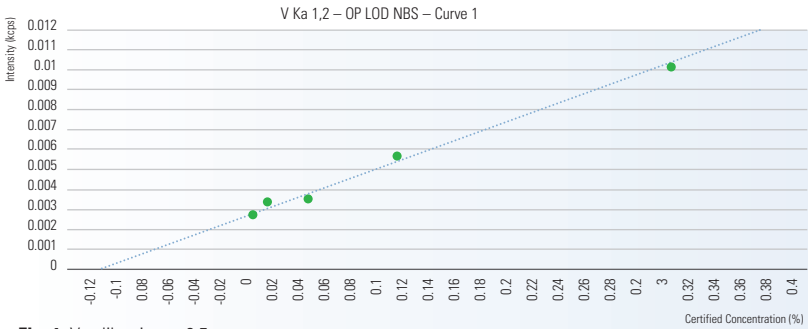

Fig. 1: V calibration at 0.5 mm

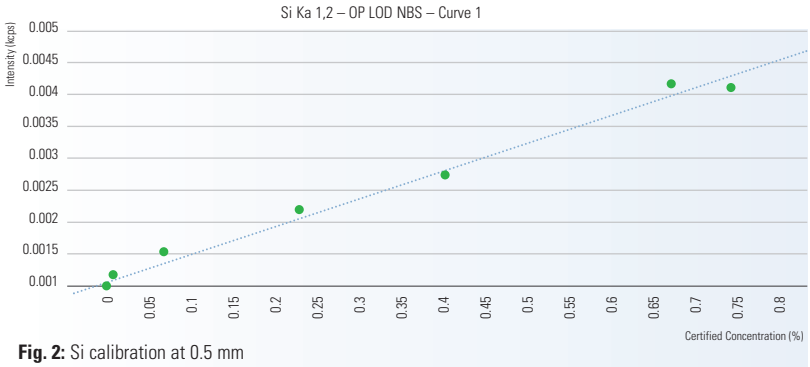

Fig. 2: Si calibration at 0.5 mm

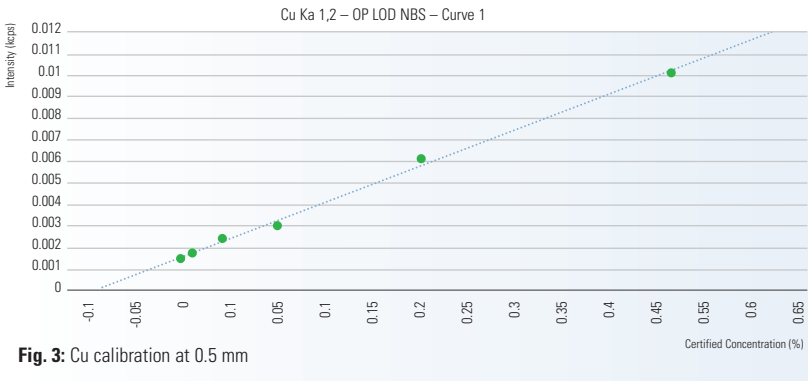

Fig. 3: Cu calibration at 0.5 mm

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