Application note | 41651

X-ray fluorescence

Inclusion analysis in glass with WDXRF Spectrometer

Keywords
Elemental Analysis, WDXRF Spectrometer, XRF Analysis, XRF Elemental Analysis, XRF Instrument, XRF Spectrometer, XRF Spectrometry, XRF Technique, XRF Technology

Introduction

To widen the usual capabilities of X-ray fluorescence analysis, the ARL PERFORM'X spectrometer 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.

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 our 5GN+ 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.

ARL PERFORM'X Series Advanced X-ray Fluorescence Spectrometer

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 contaminations. 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 interelemental effects to provide precise quantitative results.

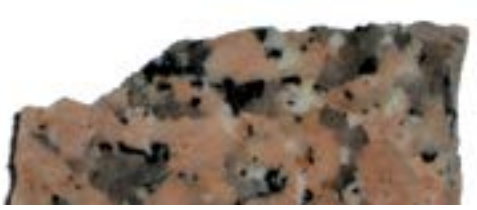

Figure 1. Example of an inhomogeneous sample.

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

Figure 3. Glass sample with shiny inclusion.

Inclusion in glass

Glass samples have been submitted for analysis. One of the samples shows a shiny inclusion that we cannot determine in the traditional way (Figure 3). This sample has been analyzed with UniQuant to obtain the global composition over a diameter of 29 mm. Pre-defined UniQuant counting times, analytical conditions and parameters with regard to crystal, detector, collimator and power were used.

As the sample is a glass the results are expressed as oxides (Table 1). The analysis over a 29 mm diameter does not permit to assess if the shiny inclusion affects the elemental determinations because all the detected components can be part of a glass composition.

In order to determine the nature of the inclusion, small spot analyses were performed for a few elements on the bulk surface and on the shiny inclusion (see Table 2). This analysis quickly provides the information that the shiny particle is made principally of copper while there is no copper in the glass sample itself. This means that the result for CuO in Table 1 can be removed as it does not belong to the glass matrix.

Element Si also appears in the spot analysis because the particle is smaller than 0.5 mm in diameter and therefore the area of analysis partly includes the glass matrix.

Conclusion

It is seen that analyses using small spots can easily be performed with the ARL PERFORM'X sequential XRF spectrometer. The UniQuant standard-less program can easily provide concentration data if required. This analysis permits to quickly determine the nature of an inclusion or a defect in an heterogeneous sample.

Furthermore, operation is made easy through the state-of- the-art OXSAS software which is able to operate with the Microsoft Windows® 10 system.

Oxide/Element	Conc. %	StdErr %
SiO_2	73.40	0.23
Na_2O	12.79	0.16
CaO	7.964	0.13
MgO	3.724	0.09
Al_2O_3	1.147	0.05
K_2O	0.347	0.016
SO_3	0.226	0.011
Fe_2O_3	0.210	0.010
TiO_2	0.074	0.0035
BaO	0.025	0.0052
Cl	0.024	0.0011
CuO	0.018	0.0009
MnO	0.012	0.0007
ZrO_2	0.010	0.0005
P	0.008	0.0006
Cr_2O_3	0.007	0.0006
SrO	0.007	0.0003
SnO_2	0.002	0.0009
ZnO	0.002	0.0004
StdErr = Estimated uncertainty		

Table 1. UniQuant elemental determination of the glass sample.

Element	Shiny particle	Glass surface
Al	0.74	0.50
Cu	24.34	0.02
Fe	1.57	0.16
Sn	1.19	0.50
Si	34.84	70.46

Table 2: Spot analyses (all values in %) using the 0.5mm collimator.

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