

# Small Spot Analysis

## Inclusion in Glass

### Key words

ARL PERFORM'X, small spot analysis, glass, XRF, X-ray fluorescence, inclusion

### Introduction

To widen the usual capabilities of X-ray fluorescence analysis, the Thermo Scientific™ 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.

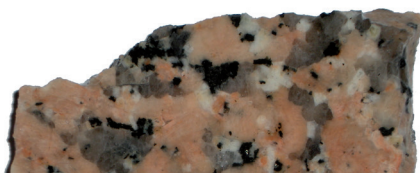


Figure 1: Example of an inhomogeneous sample

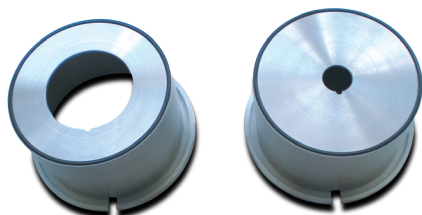


Figure 2: Cups for small spot analysis with 30 mm and 10 mm apertures

### 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.



### 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 inter-elemental effects to provide precise quantitative results.

### 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.



Figure 3: Glass sample with shiny inclusion

As the sample is a glass the results are expressed as oxides (Table 1). Such global analysis does not permit to assess if the shiny inclusion affects the elemental determinations.

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, hence 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 latest Microsoft Windows® 10 system.

Oxide/Element	Conc. %	StdErr %
SiO <sub>2</sub>	69.77	0.23
Na <sub>2</sub> O	12.16	0.16
CaO	7.57	0.13
MgO	3.54	0.09
Al <sub>2</sub> O <sub>3</sub>	1.09	0.05
K <sub>2</sub> O	0.330	0.016
SO <sub>3</sub>	0.215	0.011
Fe <sub>2</sub> O <sub>3</sub>	0.200	0.010
TiO <sub>2</sub>	0.0704	0.0035
BaO	0.0239	0.0052
Cl	0.0228	0.0011
CuO	0.0175	0.0009
MnO	0.0118	0.0007
ZrO <sub>2</sub>	0.0091	0.0005
P	0.0073	0.0006
Cr <sub>2</sub> O <sub>3</sub>	0.0069	0.0006
SrO	0.0066	0.0003
SnO <sub>2</sub>	0.0018	0.0009
ZnO	0.0016	0.0004
StdErr = Estimated uncertainty		

Table 1: UniQuant elemental determination of the glass sample. StdErr% corresponds to the statistical error of analysis.

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 %)