APPLICATION NOTE

No. AN41670

# Small spot and mapping analysis of gold alloys with Thermo Scientific ARL PERFORM'X Series Advanced X-Ray Fluorescence Spectrometers

Christopher R. Shaffer, Thermo Fisher Scientific, Ecublens, Switzerland

**Key words**
ARL PERFORM'X 4200 W, Gold alloys, X-ray fluorescence, XRF, Small spot, Mapping

## Goal

Determination of homogeneity in gold samples for quality control

## Introduction

Gold is one of the most sought-after and therefore valuable precious metals in the world. It has been used in the manufacturing of coins, jewelry, ornaments and decorations for thousands of years. In recent years, the market value of gold has increased dramatically as it is seen as a stable investment. Many new companies and industries for recycling and refinement have sprung up all over the world.

The production of gold alloyed products is based on the amount of gold used in the alloy process. This is because Au is the most valuable element in these products.

The precision and accuracy in the analysis of gold is highly demanding due to its high price. A small error in concentration determination can equate to a large amount of money. It is for this reason that high power wavelength dispersive X-ray fluorescence (WDXRF) is one of the primary methods for quality control in gold analysis.

## 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.

ThermoFisher SCIENTIFIC

## Calibration

A set of certified reference standards were used in the creation of elemental regression plots. These graphs are linear regressions of the known concentrations plotted against the measured intensities. One set of regression calibrations were made using the ARL PERFORM'X's small spot aperture at 0.5 mm.

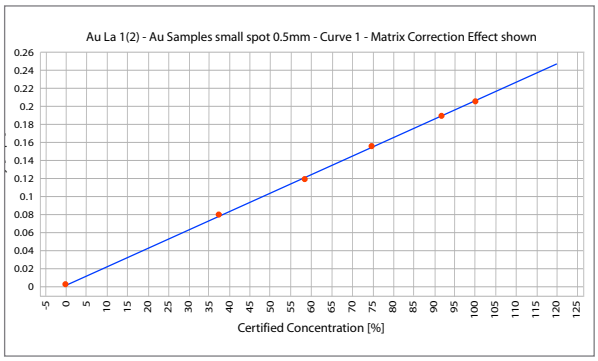


**Figure 1: Au calibration at 0.5 mm aperture**

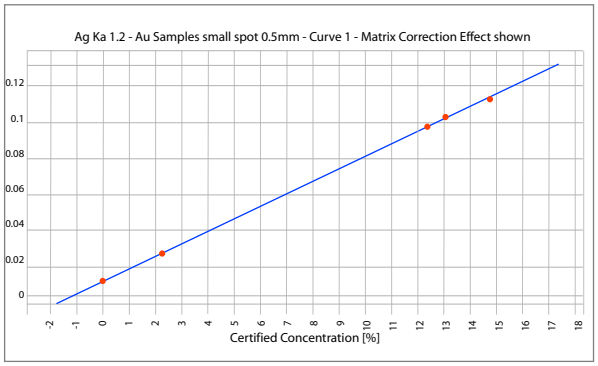


**Figure 2: Ag calibration at 0.5 mm aperture**

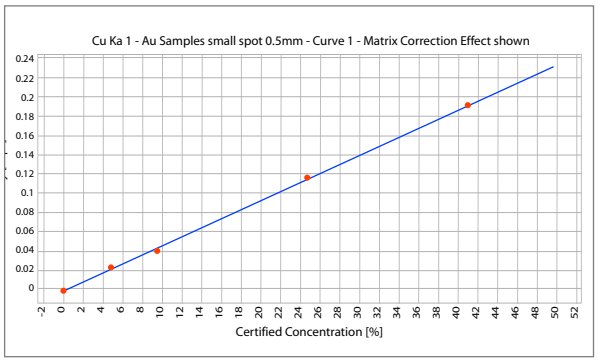


**Figure 3: Cu calibration at 0.5 mm aperture**

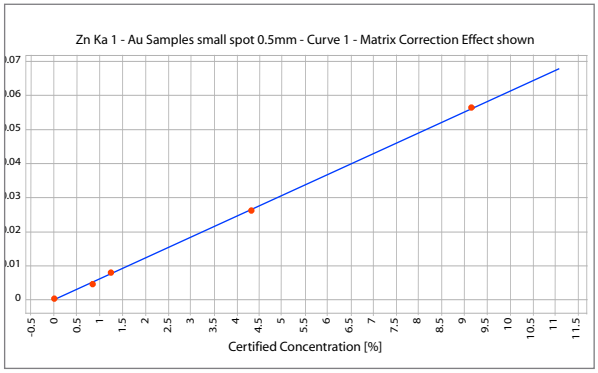


**Figure 4: Zn calibration at 0.5 mm aperture**

## Results

Precision tests on gold alloys were carried out by running the sample for ten repeat analyses at 100 seconds counting time. The results are summarized in Table 1. The standard estimates of error (SEE), the standard deviations (SD) and the relative standard deviations (RSD) are the typical deviations achieved from the above regressions.

Table 2 represents the precision results using the 0.5 mm small spot feature for this sample. This feature allows for analysis of very small and/or oddly shaped samples or small defects in samples.

| Elements | Conc. (%) | SEE (%) | SD (%) | RSD (%) |
|---|---|---|---|---|
| Au | 75.5 | 0.0012 | 0.58 | 0.77 |
| Ag | 14.1 | 0.0046 | 0.14 | 0.99 |
| Cu | 8.8 | 0.0014 | 0.12 | 1.41 |
| Zn | 0.8 | 0.0067 | 0.03 | 3.78 |

Table 1: Typical gold alloy calibration at 0.5 mm aperture

## Small spot analysis

A South African Krugerrand coin was analyzed at two separate locations using the small spot feature of the ARL PERFORM'X. The below analytical results show the precision of the small spot capability achievable by the ARL PERFORM'X.

**Figure 5: Photo of spot #1**

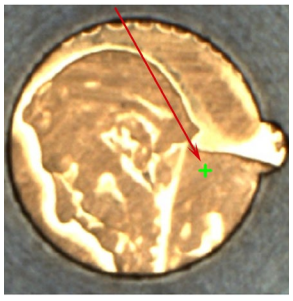

**Figure 6: Photo of spot #2**

| Samples | Au (%) | Ag (%) | Cu (%) | Zn (%) |
|---|---|---|---|---|
| Spot #1 | 97.5616 | 0.1314 | 2.3382 | LoD |
| Spot #2 | 97.4032 | 0.1011 | 2.3148 | LoD |

Table 2: Small spot results on Krugerrand
*Note: LoD = Limit of Dectection*

## Elemental mapping analysis

The ARL PERFORM'X can add a new dimension of sample analysis with its elemental mapping functionality. The mapping capability enables sample inhomogeneity, contamination, gradient segregation and inclusion analysis.

The ARL PERFORM'X mapping can construct detailed composite maps of elemental distributions within a sample. The cartography control and overlay has an ultra fine resolution of 0.1 mm steps providing superior analysis allowing for process improvement and problem solving applications. An example of this feature can be seen below in the analysis of an inhomogeneous gold sample.

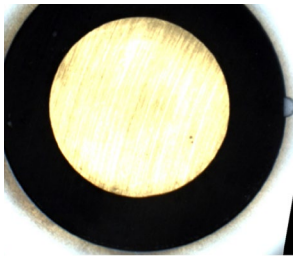

Figure 7: Photo of sample

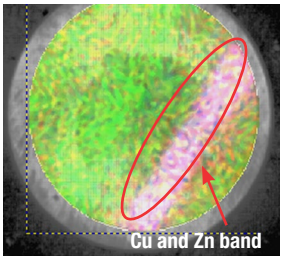


Figure 8: Elemental overlay

The four primary elements (Au, Ag, Cu and Zn) were mapped over the entire sample surface. Each element is represented by a different color (Au–green, Ag–red, Cu–blue, Zn–yellow). The overlay shows a distinct band of high concentrations for Cu and Zn and low concentration of Au. This segregation is not visible by the naked eye.

From the overlay, each element color can be shown separately. Here we can easily see the elemental concentration differences in the samples.

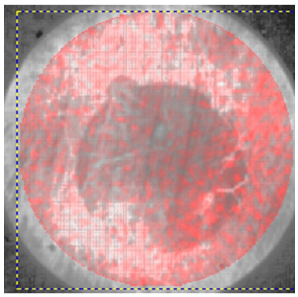

Figure 9: Ag

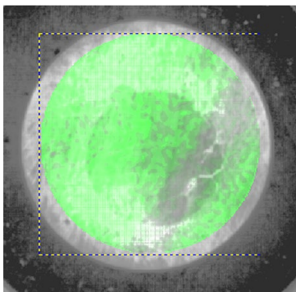

Figure 10: Au

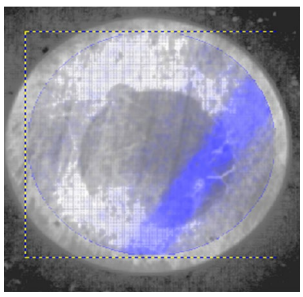

Figure 11: Cu

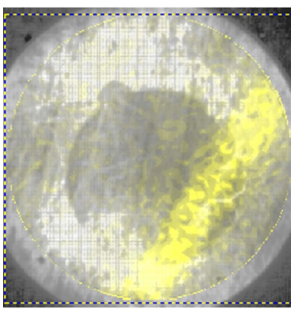

Figure 12: Zn

These differences can also be illustrated in 2D, with the highest concentration being the brightest and the lowest the darkest.

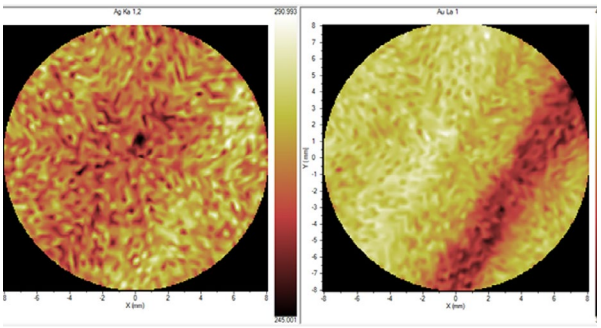

Figure 13: Ag

Figure 14: Au

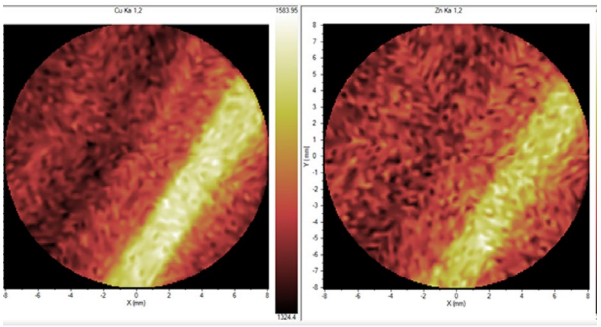

Figure 15: Cu

Figure 16: Zn

Or for a more graphic depiction of the segregation, the ARL PERFORM'X can illustrate the results in a 3D representation, with the highest concentration being the tallest and the lowest concentration the shortest.

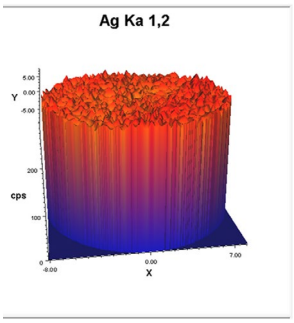


Figure 17: Ag

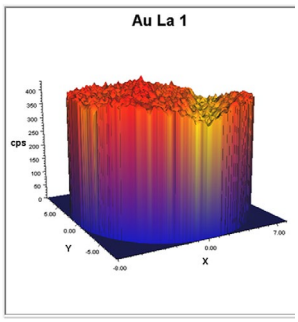


Figure 18: Au

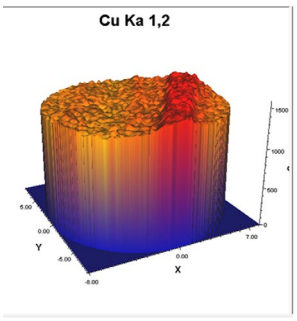


Figure 19: Cu

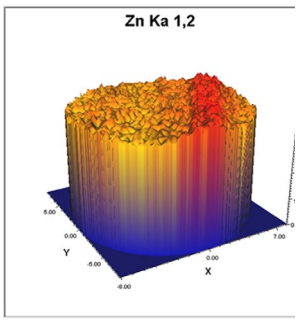


Figure 20: Zn

thermo scientific

## Conclusion

The results show that gold alloy 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 for routine and non-standard analysis.

The ARL PERFORM'X is not only an ideal analytical instrument for quality control, but also for identifying inhomogeneity, contamination, gradient segregation and inclusions in gold samples.

Furthermore, operation is made easy through the newest and most advanced state-of-the-art Thermo Scientific OXSAS WDXRF software which is able to operate with the latest Microsoft Windows® 7 packages.

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

ThermoFisher SCIENTIFIC