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APPLICATION NOTE

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Analysis of gold jewellery with ARL QUANT'X EDXRF Spectrometer

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Introduction

Due to the value of precious metals, the accurate analysis of gold and other precious metals has become increasingly important, especially for resellers and processors of these metals. Gold jewellery and gold scrap is typically collected from jewelers, pawnshops, consolidators, dental and medical offices and even electronic manufacturers. The product is either made available for consumer resale or processed to recover the precious metals for use as a raw material in the production of other products.

Energy Dispersive X-ray Fluorescence (EDXRF) challenges the traditional fire assay procedure for analysis of precious metals. It is quickly becoming the method of choice in the precious metal industry as it is a nondestructive technique and therefore used for applications where there is a need to maintain the integrity of the sample.

All material remains intact after analysis allowing for full recovery of the precious metal content. Raw materials in the form of jewellery and scrap in addition to products such as alloys, solders, sterling silver and gold bearing alloys are tested for gold content (karat) and to verify composition. A preliminary scan of various concentrations of elements in a refining lot is easily accomplished through this technique.

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Instrumentation

The Thermo Scientific™ ARL™ QUANT'X Energy Dispersive X-ray Fluorescence Spectrometer is a state-of-the-art elemental analyzer providing the ultimate in accuracy and precision demanded in this industry. The technology is based on the EDXRF principle allowing for the simultaneous measurement of the emission lines for all elements from sodium (Na, Z=11) through uranium (U, Z=92). The Silicon Drift Detector (SDD) with an area of 30 mm² is perfectly suited to detect precious metals.

With the use of a sample-imaging CCD camera and adjustable X-ray beam collimation, the ARL QUANT'X Spectrometer allows positioning of small pieces of jewellery for efficient excitation and analysis. The system combines all the analytical features of selectivity and sensitivity inherent in a bulk-analysis spectrometer with the sampling flexibility typically found in micro analyzers. The operator can visualize and locate the sample before analysis thus aligning small samples or features to improve accuracy of qualitative and quantitative analysis.

Excitation conditions

The ARL QUANT'X Spectrometer achieves superior sensitivity and precision through source-tuned excitation of the sample to fluoresce only the elements of interest. The system offers a virtually unlimited combination of excitation voltages (4-50 kV) and multiple primary beam filters (nine) for optimal background control which improves elemental sensitivity and reduces background in order to provide better performance.

As shown in Table 1, two conditions were used to collect the spectra for gold and associated alloying elements. The tube current is automatically adjusted to optimize the input count rate. The total counting time per analysis is eight minutes while all measurements are performed in an air atmosphere.

Table 1. Analytical settings.

Voltage (kV)	Tube filter	Atmosphere	Live time (s)	Elements
20	Pd Medium	Air	120	Au, Cu
50	Cu Thick	Air	120	Ag

Sample preparation and presentation

All standards and samples were analyzed as received. The standards and samples were placed into a standard XRF sample cell fitted with a 4-micron polypropylene X-ray support film for presentation to the spectrometer.

Calibration

A Fundamental Parameter (FP) approach was used to perform the calibrations. This method is included in the standard quantitative package provided with the ARL QUANT'X Spectrometer and yields excellent results. The FP method establishes a relationship between spectrometer response and element concentration based on a standard set. As the name implies, the software uses a variety of fundamental parameters including the first principles of X-ray absorption and emission to correct for matrix interactions. Any combination of standards ranging from matrix matched to pure element standards can be utilized by the FP software for calibration. The standards employed in this application are shown in Table 2.

Table 2. Elemental concentrations of the calibration standards used for gold analysis.

Standard	Ni (%)	Cu (%)	Zn (%)	Ag (%)	Au (%)
1	11.81	35.32	11.17	0	41.70
2	14.49	30.72	11.58	0	42.06
3	0	40.26	7.97	10.09	41.68
4	0	39.50	5.23	13.30	41.68
5	9.20	23.77	8.55	0	58.48
6	0	29.23	4.71	7.63	58.43
7	0	24.50	4.29	12.82	58.39
8	0	30.33	6.42	4.91	58.34
9	0	28.20	3.65	9.51	58.43
10	5.76	14.08	5.05	0	75.11
11	0	12.48	0	12.48	75.04
12	0	9.86	0	15.08	75.06
13	0	10.66	0	1.73	87.61
14	0	4.91	0.16	3.25	91.38

Calibration results

Figures 1 through 3 show the regression curves for Cu, Ag and Au, the main components of gold jewelry and alloys. The plots demonstrate a good correlation between the given elemental concentrations and the calculated concentrations using the Fundamental Parameter calibration method.

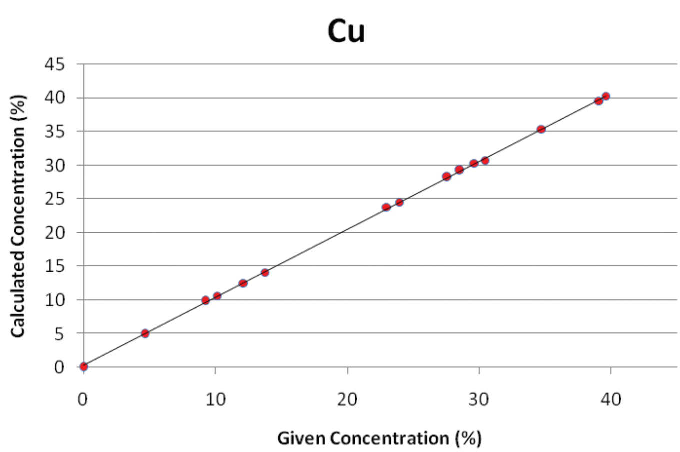

Figure 1. Regression curve for copper.

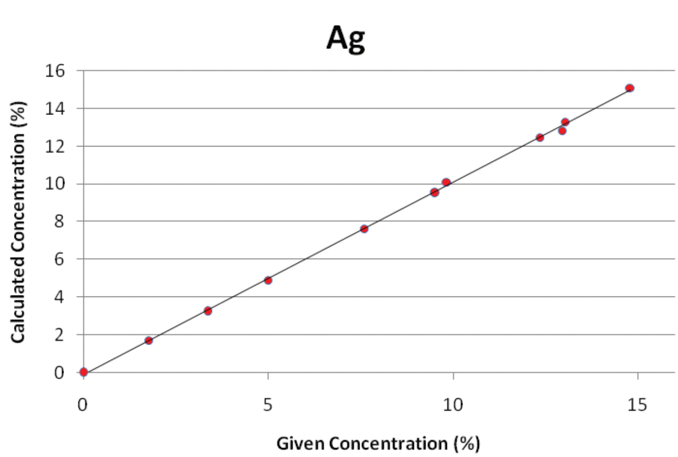

Figure 2. Regression curve for silver.

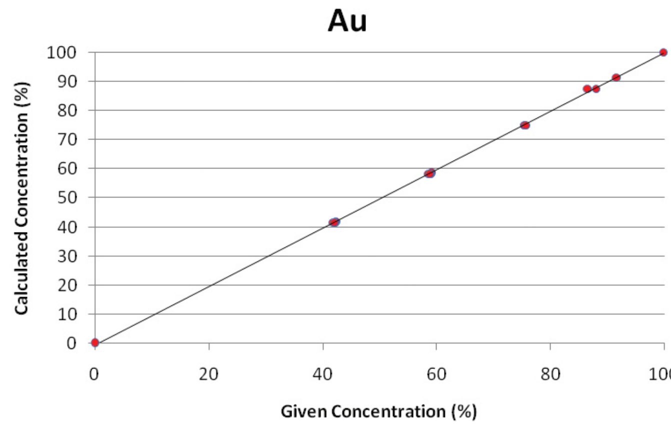

Figure 3. Regression curve for gold.

Repeatability

The primary goal of a sample evaluation is to confirm that the sensitivity and repeatability of the instrument meet the application requirements. Ten replicate measurements of a jewelry sample were made to gauge the reproducibility of the method. The mean concentration and 1-sigma Standard Deviation of these measurements are shown below in Table 3 as well as the certified value of the sample. Measurement times identical to the calibration were applied.

Table 3. Repeatability results of gold jewellery sample.

Run	Au (%)	Ag (%)	Cu (%)
1	51.74	10.55	31.69
2	51.81	10.54	31.65
3	51.77	10.51	31.69
4	51.76	10.51	31.65
5	51.66	10.54	31.66
6	51.68	10.53	31.74
7	51.72	10.55	31.66
8	51.73	10.59	31.60
9	51.72	10.57	31.60
10	51.73	10.59	31.63
Average	51.73	10.55	31.66
Std. Dev.	0.04	0.03	0.04
Certified Value	51.67	10.54	31.40

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Conclusion

The results presented in this study demonstrate the exceptional performance of the ARL QUANT'X EDXRF Spectrometer for the measurement of precious metals in gold jewelry with a high-powered X-ray tube, an efficient SDD, primary beam filters for background control and flexible fundamental parameter software. The ARL QUANT'X Spectrometer delivers the accuracy and precision demanded in the precious metals industry.

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