Poster Note

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Analysis of gold alloys by macro-LA-ICP-OES NWR266macro

Introduction

Elemental analysis of gold samples has traditionally been most commonly accomplished by acid digestion followed by ICP-OES, or spark source ICP-OES. Acid digestion consumes more sample, produces more hazardous waste than solid sampling and takes hours to digest (some precious metal alloys take even longer). Spark source is increasingly unavailable and uses a large sampling area, which creates excessive debris and cannot be used for spatial analysis or the interrogation of inclusions. Macro laser ablation ICP-OES provides results with comparable figures of merit to spark source OES, but with more efficient sampling which allows for smaller testing regions. Both the laser and ICP are bench-top models, which minimizes consumed lab space. Results with macro-LA-ICP-OES are fast, accurate, and robust enough for routine analysis in high throughput, industrial environments.

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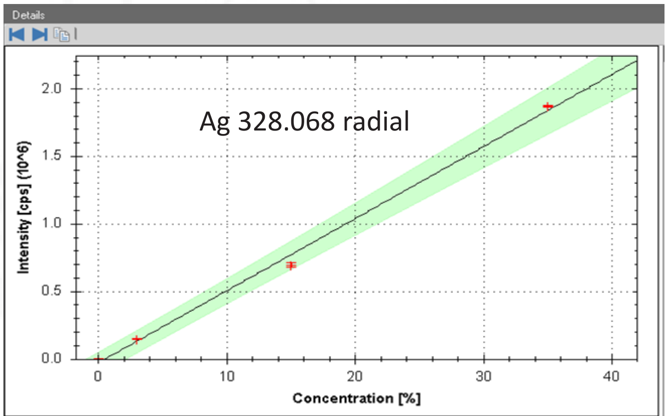
Method

Four in-house standards of gold alloy were analyzed using an Elemental Scientific NWR266macro laser ablation system and an iCAP 7600 dual view ICP (Thermo Scientific). The use of the macro laser ablation system is important because the high energy allows an extremely large beam to be employed, averaging out heterogeneity in the sample. The samples were analyzed for Au, Ag, Cu, Zn, Co, Fe, Ni, and Pb. Multiple wavelengths were chosen for each element. Ni, Pb, and Co were analyzed axially, while Au, Ag, Cu, Zn, and Fe were analyzed radially, based on the anticipated signal intensity for each element.

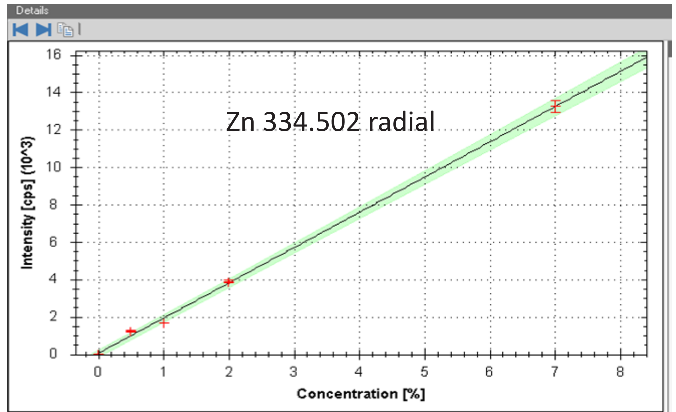
Table 1. Parameters of the analysis. The 780 μm spot was utilized for maximum sensitivity, presenting large amounts of sample to the ICP.

Parameters Employed

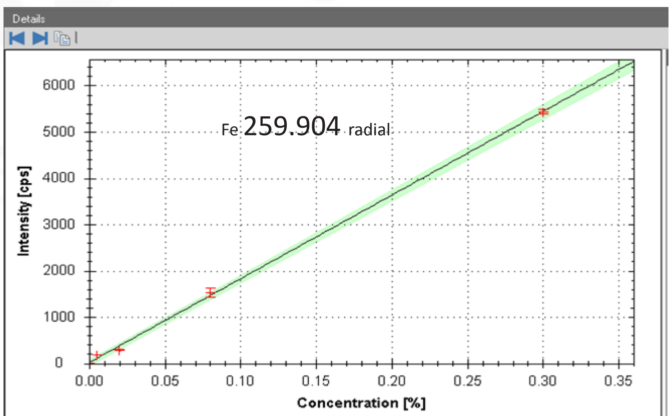
Parameter	Ablation	Pre-ablation
Spot diameter	780 μm	780 μm
Fluence	4.3 J/cm^2	2 J/cm^2
Repetition rate	10 Hz	10 Hz
Scan speed	40 μm/s	75 μm/s
Nebulizer flow	0.45 L/min	
Aux flow	0.8 L/min	
RF power	1350 W	
Integration time	20 s UV axial 5 s Vis Radial	



f(x) = b*x + a				
Parameter	Value	Confidence Delta at 90%	StdErr	Rel. StdErr [%]
b	53,321.989	+/-4,327.311	2,294.925	4.304
a	-29,706.845	+/-82,644.837	43,829.464	147.540
R^2	0.996309			
BEC	-0.557 %			
IDL(LOD)	0.009672 %			



f(x) = b*x + a				
Parameter	Value	Confidence Delta at 90%	StdErr	Rel. StdErr [%]
b	1,884.076	+/-60.936	37.208	1.975
a	50.261	+/-200.720	122.562	243.852
R^2	0.998831			
BEC	0.027 %			
IDL(LOD)	0.002918 %			



f(x) = b*x + a				
Parameter	Value	Confidence Delta at 90%	StdErr	Rel. StdErr [%]
b	18,075.771	+/-496.310	303.053	1.677
a	18.861	+/-69.066	42.172	223.596
R^2	0.999157			
BEC	0.001 %			
IDL(LOD)	0.000218 %			

Figure 1. Selected calibration curves showing excellent linearity and low detection limits.

Results

Calibration curves for all analyzed elements had correlation coefficients (R^2 values) of 0.995 or higher. Example calibration curves are shown in Figure 1, and demonstrate linearity and sensitivity. Absent a sample blank, the detection limits were calculated from the variance of the calibration curve, shown in Table 2.

With the decreasing availability of spark source OES, new solid sampling solutions are required. The absence of sample preparation, low detection limits, and turnkey operation make macro-LA-ICP-OES with Elemental Scientific's NWR266macro ideal for analysis of gold and other metallic samples or alloys in industrial environments.

Table 2. Detection limits (LOD) for each wavelength, calculated from the variance of the calibration curve.

Ag 328.0 ppm	Ag 338.2 ppm	Cu 219.9 ppm	Cu 211.2 ppm	Zn 334.5 ppm	Zn 330.2 ppm	Fe 259.9 ppm	Fe 261.1 ppm
96.72	85.12	13.43	109.4	29.18	69.12	2.18	1.281

Ni 221.6 ppm	Ni 231.6 ppm	Pb 220.3 ppm	Pb 182.2 ppm	Co 228.6 ppm	Co 230.7 ppm
0.1416	0.3011	1.098	0.2194	0.2555	0.2096

Elemental Scientific LASERS

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