APPLICATION NOTE

ICP-Optical Emission Spectroscopy

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Analysis of Impurities in Gold With the Avio 550 Max ICP-OES Following ASTM B562-95

Introduction

Gold (Au) is one of the most precious and useful metals on earth, due to a unique combination of characteristics: resistance to tarnish/corrosion, ability to conduct electricity, and ability to be easily formed into a variety of shapes (i.e. thin wires and sheets, intricate shapes). Because of this versatility, gold is widely used in jewelry, artwork, currency, electronics, medicine/dentistry, aerospace, and ornamental decorations.[1]

The price of Au is highly dependent on the impurity levels present within – not all uses require the same purities. Towards this end, ASTM has developed specifications for various purities of refined gold in method B562-95: Standard Specification for Refined Gold.[2]

This work focuses on the analysis of impurities in gold with ICP-OES, using the levels specified in ASTM B562-95 as a guideline. The Avio® 550 Max ICP-OES was chosen for this work due to its low cost of operation, minimal maintenance requirements and its speed of analysis. The Avio 550 Max's Flat Plate™ plasma technology consumes significantly less argon, leading to a much lower cost per analysis. The Avio 550 Max's ability to handle high levels of dissolved solids running over long periods of time is due to PlasmaShear™ technology, generating a thin stream of air which cuts off the top of the plasma, eliminating deposition on the interface window, resulting in exceptional stability in difficult matrices with no maintenance required. Finally, the advanced optical system of the Avio 550 Max provides fully simultaneous analysis with excellent stability and accuracy for faster sample-to-sample time.

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Experimental

Samples

All analyses were performed in a 1% gold solution in 10% HCl to simulate a digestion diluted 100 times. Accuracy was determined by spiking the 1% gold solution with the elements at their specified levels for 99.99% and 99.995% gold, as shown in Table 1. (Neither arsenic (As) nor nickel (Ni) are specified in 99.995% gold in ASTM method B562-95.) The associated concentrations in solution are also shown in Table 1. All measurements were made against external calibration curves prepared in 8% aqua regia at 0.5, 1, and 1.5 times the specified concentrations. Yttrium (Y) was used as an internal standard and added to all calibration standards and samples.

Table 1. ASTM Method B562-95 Specifications for Impurities in Gold and Associated Concentrations in Solution with 100 x Dilution.

	99.99% Gold		99.995% Gold	
Element	Specification (wt%)	Concentration in 1% Solution (mg/L)	Specification (wt%)	Concentration in 1% Solution (mg/L)
Ag	0.009	0.9	0.001	0.1
As	0.003	0.3	---	---
Bi	0.002	0.2	0.001	0.1
Cr	0.0003	0.03	0.0003	0.03
Cu	0.005	0.5	0.001	0.1
Fe	0.002	0.2	0.001	0.1
Mg	0.003	0.3	0.001	0.1
Mn	0.0003	0.03	0.0003	0.03
Ni	0.0003	0.03	---	---
Pb	0.002	0.2	0.001	0.1
Pd	0.005	0.5	0.001	0.1
Si	0.005	0.5	0.001	0.1
Sn	0.001	0.1	0.001	0.1

Instrumentation

All analyses were performed on the Avio 550 Max fully simultaneous ICP-OES using the parameters in Table 2 and wavelengths in Table 3. The standard sample introduction components were used, along with a torch position of -4. Having the ability to adjust torch position gives the flexibility to optimize the application for sensitivity and robustness. Wavelengths were selected which were interference-free, although Multicomponent Spectral Fitting (MSF)[3] within Syngistix™ software was applied to As 188.979 nm to remove the spectral interference from gold. (This analysis can also be accomplished on the Avio 220 Max hybrid simultaneous ICP-OES.) Combining the rapid, fully simultaneous analysis of the Avio 550 Max with a total argon consumption of only 9 L/min provides significant savings with regard to argon use which translates directly to a lower cost of analysis per sample.

Table 2. Avio 550 Max ICP-OES Instrumental Parameters.

Parameter	Value
Nebulizer	MEINHARD® K-1
Spray Chamber	Baffled Glass Cyclonic
RF Power	1500 W
Injector	2.0 mm Ceramic
Plasma Flow	8 L/min
Auxiliary Flow	0.2 L/min
Nebulizer Flow	0.7 L/min
Torch Position	-4
Sample Uptake Rate	1 mL/min
Replicates	2
Plasma View	Axial
Read Time Range	Min = 1 sec; Max = 5 sec

Table 3. Wavelengths.

Element	Wavelength (nm)
Ag	328.068
As	188.979
Bi	223.061
Cr	205.560
Cu	327.393
Fe	259.939
Mg	285.213
Mn	259.372
Ni	341.476
Pb	220.353
Pd	340.458
Si	251.611
Sn	189.927
Y (int std)	371.029

Results and Discussion

For accuracy determination, 1% gold solutions were spiked at the specification levels for 99.99% and 99.995% gold and measured against external calibration curves. Figure 1 shows the spike recoveries at both specification levels. With all recoveries within 10%, accuracy is shown, demonstrating that with the proper selection of wavelengths and analytical conditions, a 1% gold matrix does not inhibit impurity analysis.

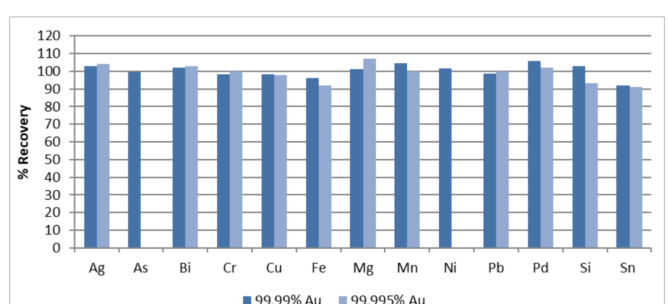

Figure 1. Analyte spike recoveries in 1% Au at the 99.99% (dark blue) and 99.995% (light blue) impurity specification levels.

With the accuracy established, detection limits in 1% Au were determined by measuring an unspiked 1% Au solution seven consecutive times against a calibration curve. The standard deviation of the measured concentrations was then multiplied by 3.14. The resulting detection limits (shown in Figure 2, orange bars) are at least one order of magnitude lower than the specification limits and, in most cases, two orders of magnitude lower, demonstrating the possibility of measuring even lower concentrations than those specified in ASTM B562-95.

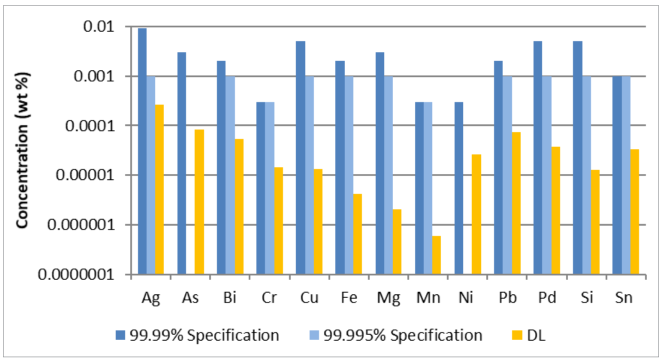

Figure 2. Analyte detection limits (orange bars) in 1% gold plotted alongside the ASTM method B562-95 impurity specifications for 99.99% Au (dark blue) and 99.995% Au (light blue).

Finally, to assess stability, the internal standard signal was monitored over 10 consecutive analyses of a 1% Au solution. The resulting stability (Figure 3) shows signal variations of less than 2%, demonstrating exceptional system stability and no matrix effects using the Avio 550 Max ICP-OES.

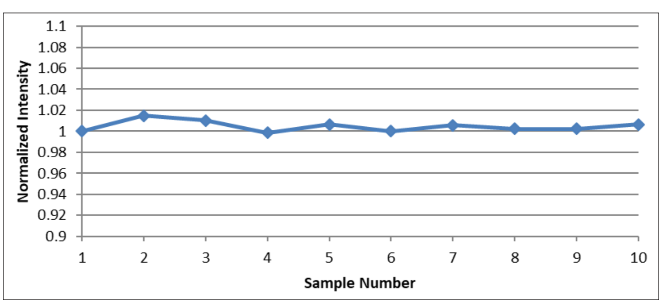

Figure 3. Internal standard signal over 10 analyses of a 1% Au solution. All signals were normalized to the first sample.

Conclusion

This work has demonstrated the ability of the Avio 550 Max fully simultaneous ICP-OES to successfully measure impurities in gold at the 99.99% and 99.995% purity levels specified in ASTM method B562-95. These analyses were accomplished with standard Avio 550 Max instrumental conditions. Matrix-induced spectral interferences from the matrix were overcome with the proper wavelength selection and the use of Multicomponent Spectral Fitting, where required. This combination allowed accurate, low-level analyte analysis with no matrix-induced instrument drift. Detection limits of 1-2 orders of magnitude lower than the specification limits suggest the ability to measure even lower impurity levels.

Reference

1. https://geology.com/minerals/gold/uses-of-gold.shtml.
2. ASTM Method B562-95: "Standard Specification for Refined Gold", 2012.
3. "Multicomponent Spectral Fitting", PerkinElmer Technical Note, 2016.

Consumables Used

Component	Part Number
Sample Uptake Tubing, Black/Black (0.76 mm id), PVC	N0777043 (Flared) 09908587 (Non-Flared)
Drain Tubing, Gray/Gray (1.3 mm id), Santoprene	N0777444
Arsenic Standard, 1000 mg/L	N9300180 (125 mL) N9300102 (500 mL)
Bismuth Standard, 1000 mg/L	N9303761 (125 mL) N9300105 (500 mL)
Chromium Standard, 1000 mg/L	N9300173 (125 mL) N9300112 (500 mL)
Copper Standard, 1000 mg/L	N9300183 (125 mL) N9300114 (500 mL)
Iron Standard, 1000 mg/L	N9303771 (125 mL) N9300126 (500 mL)
Lead Standard, 1000 mg/L	N9300175 (125 mL) N9300128 (500 mL)
Magnesium Standard, 1000 mg/L	N9300179 (125 mL) N9300131 (500 mL)
Manganese Standard, 1000 mg/L	N9303783 (125 mL) N9300132 (500 mL)
Nickel Standard, 1000 mg/L	N9300177 (125 mL) N9300136 (500 mL)
Palladium Standard, 1000 mg/L	N9303789 (125 mL) N9300138 (500 mL)
Silicon Standard, 1000 mg/L	N9303799 (125 mL) N9300150 (500 mL)
Silver Standard, 1000 mg/L	N9300171 (125 mL) N9300151 (500 mL)
Tin Standard, 1000 mg/L	N9303801 (125 mL) N9300161 (500 mL)
Yttrium Standard, 1000 mg/L	N9303810 (125 mL) N9300167 (500 mL)

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