

Extending the Analytical Range for Gold Using Agilent UltrAA Lamps

Application Note

Atomic Absorption

Author

John Sanders

Introduction

The accurate determination of gold in ores and minerals provides conflicting demands for the analyst. The majority of samples are near the detection limit yet the occasional sample has a high concentration value. This determination usually requires a multiple pass analysis where all of the samples are determined using the most sensitive set-up. Then those samples with concentration values over the calibration range are either diluted and re-run or re-determined using less sensitive conditions with a wider calibration range. This process is time-consuming and costly for laboratories running contract or exploration analyses.

This study examines the use of the Agilent UltrAA lamp [1] for gold determinations to extend the dynamic range and to achieve better detection levels. Either allows most samples to be determined in a single pass. Careful selection of integration times and system optimization can also provide significant savings in analysis time.



Agilent Technologies

Experimental

Instrumentation

The following were used for this study:

- Agilent SpectrAA 220 spectrometer
- Agilent UltrAA lamp power supply
- Gold UltrAA and conventional hollow cathode lamps
- Agilent SPS-5 autosampler

Reagents and Solutions

All solutions were prepared from BDH Spectrosol 1000 mg/L gold chloride atomic absorption standard (Merck-BDH, Poole, England) in 0.5% hydrochloric acid.

Results and Discussion

Figure 1 shows the calibration graphs obtained using both lamp designs at the 242.8 nm resonance line.

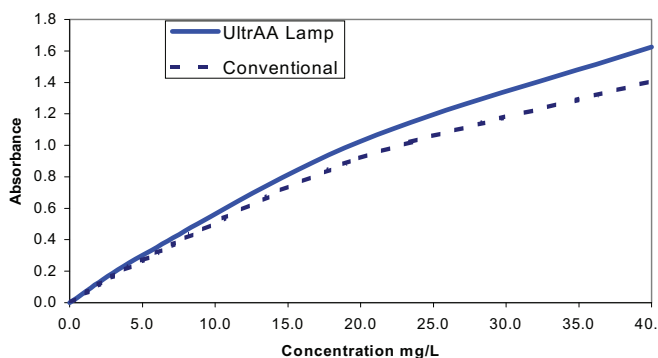


Figure 1: Calibration graph for the 242.8 nm gold line .

Figure 2 shows the calibration graphs obtained using both lamp designs at the less sensitive 267.6 nm resonance line. It is approximately half as sensitive as the 242.8 nm line. The top standard of the calibration using the 267.6 nm line is therefore effectively double the top standard of the calibration using the 242.8 nm line. Using the gold UltrAA lamp, the calibration range can be extended from 40 mg/L to 80 mg/L. This extended calibration range copes with high concentrations and saves time by minimizing the number of samples to be diluted.

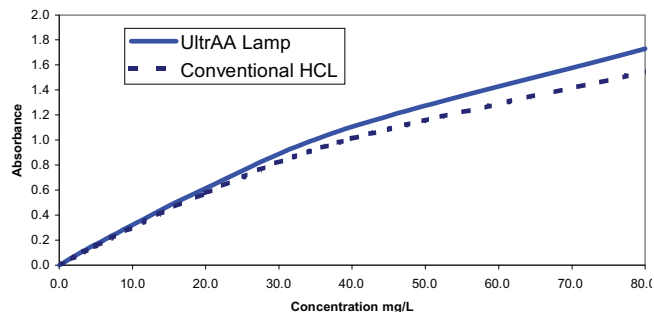


Figure 2: Calibration graph for the 267.6 nm gold line.

The detection limits and characteristic concentrations for the two lamp designs and the two wavelengths are shown in Table 1. The detection limit of the 267.6 nm line using the UltrAA lamp is the same as the 242.8 nm line using the conventional lamp.

Table 1. Results for Various Lamp Designs and Wavelengths

Lamp design	Wavelength (nm)	Characteristic concentration (mg/L)	Detection limit (mg/L)
UltrAA	242.8	0.071	0.0043
Conventional	242.8	0.079	0.0054
UltrAA	267.6	0.13	0.0055
Conventional	267.6	0.14	0.0098

Figure 3 shows the improvement in precision of the gold UltrAA lamp relative to a conventional hollow cathode lamp at two wavelengths. The UltrAA lamp, when used at the alternative wavelength of 267.6 nm has a precision at low concentrations superior to the conventional lamp at 242.8 nm. The brighter emission of the UltrAA lamp gives a better improvement in detection limits than the 10 % improvement in the slope of the calibration graphs and characteristic concentration would suggest.

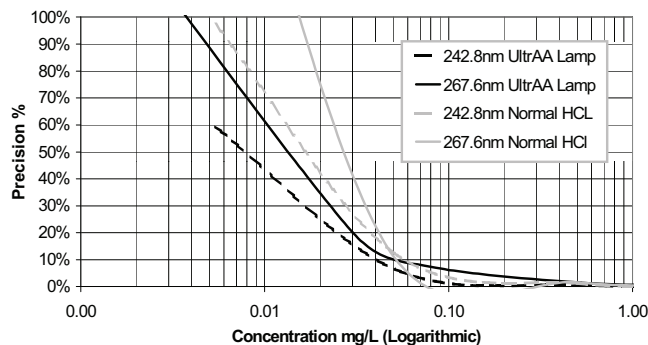


Figure 3: Precision versus concentration.

Analysis Times

The analysis time consists of a number of components: the read delay, the integration period and the autosampler slew time.

The Agilent SPS-5 autosampler was used in this study. The 1 mm i.d. probe was modified to reduce the transfer-line volume and thus provides shorter read delays. The PTFE tubing was cut off 20 mm from the top of the probe and a 550 mm length of 0.64 mm i.d. capillary tube was connected to the nebulizer of the spectrometer.

With these modifications and the nebulizer adjusted for maximum sensitivity, the read delay required was only 5 seconds. Figure 4 shows the effect of integration time on precision. The minimum read time was found to be 3 seconds. An integration time of 3 seconds was used in this study.

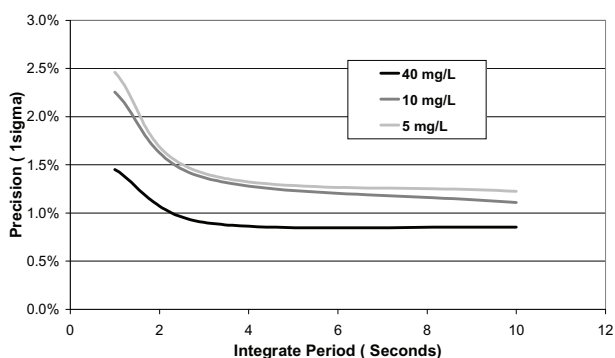


Figure 4: Integration time versus precision.

Table 2 illustrates the analysis times involved in this study. For the calibration standards an extra component is required for a rinse cycle between each solution. There was no rinse between sample solutions.

Table 2. Analysis Cycle Times

Solution type	Analysis time
Calibration Standard	36 seconds
Sample	18 seconds

Conclusion

This study illustrates that the use of the Varian gold UltraAA lamp can effectively double the calibration range without compromising the precision and detection limits based on the 242.8 nm line. This minimizes the number of samples that have to be re-measured. The use of the SPS-5 autosampler provides more savings in time by automating the analysis.

The result is fast, accurate and automated determinations of gold over an extended calibration range.

References

- 1 J. H. Moffett, Varian Instruments At Work, AA-124, September 1996

For More Information

For more information on our products and services, visit our Web site at www.agilent.com/chem

www.agilent.com/chem

Agilent shall not be liable for errors contained herein or for incidental or consequential damages in connection with the furnishing, performance, or use of this material.

Information, descriptions, and specifications in this publication are subject to change without notice.

© Agilent Technologies, Inc., 1999
Printed in the USA
November 1, 2010
A-AA10



Agilent Technologies