

Optimization of method conditions for high matrix samples using the Agilent 4210 MP-AES

Technical Overview

4210 MP-AES



Introduction

Analyzing high matrix samples on atomic spectroscopy products presents several spectroscopic challenges for the analyst. The presence of high levels of certain elements in the sample matrix can result in spectral and physical interferences, the easily ionizable element effect, background spectrum changes, and other effects on the plasma. Without careful consideration to method development, these effects will ultimately lead to incorrect results.

The 4210 MP-AES continues Agilent's technology leadership in atomic spectroscopy and has expanded the application reach of the microwave plasma, in particular high matrix samples. The unique waveguide design enables superior analytical performance and torch lifetime. The humidification of the mass flow controlled nebulizer gas also provides greater stability when running tough samples.

Additionally, the MP Expert software has easy to use features such as auto-optimization and a comprehensive wavelength database which enable the analyst to rapidly develop a method that will easily handle high matrix samples.

Developing a method for high matrix samples

The two main factors to consider when running high matrix samples on the 4210 MP-AES are wavelength selection and the nebulizer flow rate. In the examples below, 10 ppm of target elements were analyzed in the presence of increasing levels of Na, K, Mg and Ca at equal concentrations to demonstrate the benefits of optimizing the nebulizer flow rate and wavelength selection.

Wavelength selection

The 4210 MP-AES has complete wavelength coverage from 178 nm–780 nm. This allows the analyst to select from a range of wavelengths to avoid spectral interferences, extend the working range, and to select wavelengths that perform better in the presence of a challenging matrix. In the example below, two Mn wavelengths were selected; Mn 257.610 nm which is an ionic emission line, and Mn 403.076 nm which is an atomic emission line. The figure below compares the recovery of 10 ppm Mn for the two wavelengths in the presence of up to 5000 ppm of the Na, Ca, K and Mg matrix, with the atomic Mn 403.076 nm line giving better performance.

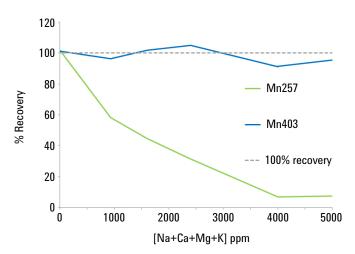


Figure 1. 4210 MP-AES analysis of 10 ppm of Mn 403.076 nm and Mn 257.610 nm in the presence of an increasing concentration of Na, K, Ca and Mg. 5000 ppm of the metal is equivalent to approximately 2% total dissolved solids (as the nitrate salt).

Nebulizer flow rate

The optimization tools in MP Expert allow quick optimization of the nebulizer flow rate for each element and wavelength in a method. When running high matrix samples it is important to optimize the nebulizer flow rate by using a sample that closely represents the matrix to be analyzed, rather than a clean solution with low acid content. Figure 2 shows the recovery of a spike of 10 ppm Cu with the nebulizer optimized in a 1% nitric acid solution, and a solution containing a total of 5000 ppm of Na, K, Ca, and Mg.

As can be seen, the recovery of Cu clusters tightly around 100% when the nebulizer pressure is optimized in the presence of the matrix.

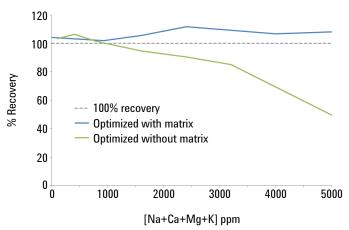


Figure 2. Recoveries of Cu 327.395 nm in increasing strength matrix when using two different nebulizer optimization techniques. When optimized on a complex matrix sample, the optimum nebulizer flow was 0.5 L/min. When optimized on a calibration standard the optimum nebulizer flow was 0.75 L/min.

Other considerations

When running samples with high dissolved solids it is important to use an adequate rinse time between solutions in order to prevent salt buildup on the sample introduction and torch, as well as minimizing sample to sample contamination. The MP Expert software allows for easy setup of rinse times and variable speed fast pump during the rinse time for particularly challenging samples.

As the level of total dissolved solids increases the Advanced Valve System (AVS 4) four port switching valve (Figure 3) accessory can be used to further reduce the matrix loading on the plasma. By only using the minimum sample required for the analysis, the AVS 4 also decreases the need for cleaning the nebulizer, spray chamber and torch.

Conclusion

The 4210 MP-AES is capable of running a wide range of challenging applications. Performance with high matrix samples is inherent to the 4210 MP-AES but can be improved further by optimizing the nebulizer flow rate in the presence of the sample matrix, by careful selection of the analytical wavelength, and by utilizing the integrated AVS 4 switching valve.



Figure 3. The AVS 4 switching valve integrates with the 4210 instrument.

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