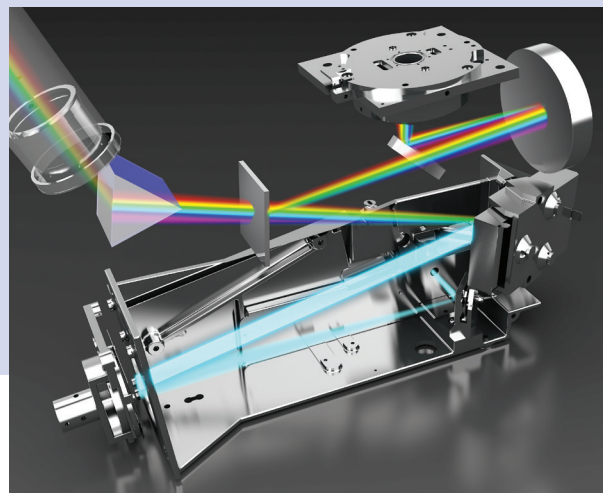


ICP-Optical Emission Spectroscopy

HP-ICP-OES: Using the Avio 550/560 Max to Achieve the Highest Possible Precisions



Introduction

When performing measurements with ICP-OES, precisions (i.e. relative standard deviations, RSDs) of 1-2% are typical. However, applications exist where greater precision is required, such as in the analysis of precious metals, major components in battery materials, and verification of the matrix composition of samples. A simple way of achieving high precision measurements is through high precision ICP-OES (HP-ICP-OES) on the Avio® 550/560 Max systems.

Variables Which Affect Precision

Several factors influence the precision of a measurement: flicker noise, shot noise (i.e. counting statistics), and selection of internal standards.

Flicker noise describes the noise in the analytical signal which originates from the plasma, largely resulting from sample introduction processes. These noise sources include pulsations from the peristaltic pump, the production and transport of the aerosol through the spray chamber and into the plasma, and processes occurring within the plasma itself. Normally, the peristaltic pump is the largest contributor to flicker noise.

When considering shot noise/counting statistics, logic suggests that higher intensities will result in better precisions. While true in principle, higher intensities may also result in slightly worse precisions, if they are not achieved in the optimal manner. For example, one way to increase the intensity of a measurement is to use a longer read time. However, in order to maintain a constant measurement time with longer read times, the number of replicate measurements must decrease. As a result, precisions will be lower. The right balance between measurement time, number of replicates, analyte intensity, and read time must be used.

While performing an analysis, the use of internal standards is very important since they account for both sample matrix and sample introduction effects. Internal standards are added to all blanks, samples, and standards at a constant concentration that gives a reasonably large intensity so that any variations caused by the sample matrix and/or sample introduction will be seen. If the intensity of the internal standard is affected, it is assumed that the intensity of the analyte will be affected the same way. As a result, the analyte intensity will be adjusted based on the signal effect of the internal standard. Usually, 1 or 2 internal standards are used for all elements in an analysis. However, the internal standards may not be measured at exactly the same time as the analytes – even on a simultaneous ICP-OES instrument. The key to achieving the best precisions is to measure the internal standard at exactly the same time as the analyte.

HP-ICP-OES: Principle

High precision ICP-OES (HP-ICP-OES) compensates for all sources of noise by measuring the internal standard at exactly the same time as the analyte: the user sets the read and integration times of the analyte(s) and internal standard(s) to be exactly the same for true simultaneous measurements. Therefore, any variables which affect the analyte signal also affect the internal standard signal at the exact same time, so that the internal standard perfectly compensates for these variations. The result: precisions with RSDs of 0.1% or less.

HP-ICP-OES Example: Battery Materials

HP-ICP-OES has a variety of uses and implementations, as mentioned above. As an example, consider the analysis of major components in battery materials. While a wide variety of battery types exist, lithium (Li), manganese (Mn), and zinc (Zn) are three commonly-used elements in batteries that must be measured with high precision.

In order to achieve high precisions, the analyte must be measured at the exact time as the internal standard. Table 1 shows the method parameters used on an Avio 550/560 Max ICP-OES.

The analyte concentrations represent typical concentrations as would be seen in battery materials diluted 1000 times for analysis. Because of these high concentrations, less sensitive wavelengths were selected for analysis. The concentrations of the internal standards were adjusted so that they had the same integration time as their respective analytes – the key to achieving high precisions with RSDs < 0.1%.

Table 2 shows the results from the analysis of a solution containing Li, Mn, and Zn at the concentrations listed in Table 1. All analytes have RSDs less than 0.1% and recover within 0.3% of their true values, demonstrating the power of HP-ICP-OES.

Conclusion

HP-ICP-OES is only available on the Avio 550/560 Max ICP-OES since true simultaneous analysis is possible when the integration and read times of the internal standard and analyte are the same. The true simultaneous analysis compensates for flicker and sample introduction noise, meaning that RSDs of < 0.1 % are achievable, along with unprecedented accuracy. For users requiring the highest accuracy and precision, HP-ICP-OES is an invaluable tool on the Avio 550/560 Max ICP-OES.

Table 1. Li, Mn, and Zn Method Parameters.

Element	Wavelength (nm)	Function	Concentration (mg/L)	Plasma View	Integration Time (sec)	Read Time (sec)
Li	460.303	Analyte	1000	Axial	0.002	10
Yb	328.937	Internal Standard		Axial	0.002	10
Mn	403.066	Analyte	2000	Radial	0.010	10
Yb	328.937	Internal Standard		Radial	0.010	10
Zn	334.496	Analyte	200	Axial	0.005	10
Sc	361.383	Internal Standard		Axial	0.005	10

Table 2. Analytical Results for Li, Mn, and Zn.

Element	Wavelength (nm)	% RSD	Concentration (mg/L)	% Recovery
Li	460.303	0.07	998	99.8%
Mn	403.066	0.04	2005	100.0%
Zn	334.496	0.09	200.5	100.3%