Application Note: 40781

The interference-free determination of arsenic in a variety of matrices

XSeries^{II} ICP-MS with 3rd Generation CCT^{ED}

Key Words

- Arsenic
- Collision Cell Technology (CCT)
- Environmental analysis
- Geochemistry
- Kinetic Energy Discrimination (KED)

Introduction

Arsenic is a toxic element that is regularly required as a determinant in a suite of elements for many laboratories. Traditionally the determination of arsenic for environmental monitoring would be performed by Vapour Generation Atomic Absorption or ICP-OES. Since the latter technique produces low sensitivity for arsenic, US methods using ICP-OES will be withdrawn from federal approval for the measurement of arsenic in drinking waters as of January, 2006. However, ICP-OES determination of arsenic will still be allowed for waste waters and digested materials. ICP-MS has been used for the determination of arsenic in multi-element analysis and this has proven to be very successful in matrices such as waters and even digested materials.

As more complex matrices have been considered and the limits of detection pushed lower for arsenic, problems of data accuracy have been seen due to interference. The main consideration for arsenic is that it is mono-isotopic at mass 75 so when interferences occur it is not possible to switch to an alternative isotope as can be done with many other elements. This also means that consideration must be given to the possibility of false positives for arsenic as there is no other isotope that can be used for confirmation of presence.

The main interference for arsenic is the combination of argon from the plasma gas and chlorine. Wherever possible, chloride based acids should not be used for material digestion in ICP-MS. However, this is not always practical and sometimes chloride is an essential part of the matrix to be measured, examples being hydrochloric acid for the semi-conductor industry, seawater analysis and many body fluids. To remove the ArCl interference has proven to be a key advantage of the recently developed Collision Cell based ICP-MS systems.

The ArCl interference can be removed quite readily using either reactive chemistry or kinetic energy discrimination using the XSeries^{II} ICP-MS from Thermo Electron Corporation. The decision on which technique to use is determined by which other analytes are to be measured, the general matrix and the question of productivity over ultimate performance.

Methods

Pure hydrogen as a reaction gas can effectively remove the ArCl interference, even in high strength HCl solvents for semi-conductor applications. Used in this manner As can be added to a general suite of analytes including K, Ca, and Fe which can be effectively measured under one set of conditions and lead to single figure ppt detection limits for arsenic. Figure 1 shows the calibration of arsenic in a matrix of 5% HCl using conditions appropriate for semi-conductor grade acids.

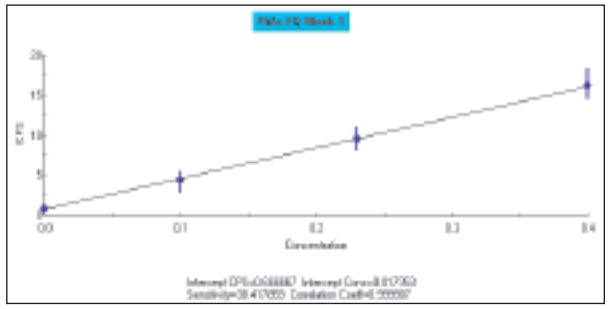

Figure 1: Calibration of arsenic in a matrix of 5% HCl

When the matrix to be measured is more complex the use of reactive gases such as hydrogen can lead to several different tune conditions being required to optimise the data for different analytes. In these circumstances, the reactive nature of the collision cell can produce new unforeseen interferences which can compromise the detection of many analytes. These problems can be overcome by the use of kinetic energy discrimination and an inert gas such as helium. Figure 2 shows a standard additions calibration of NASS-5 seawater diluted 1:10.

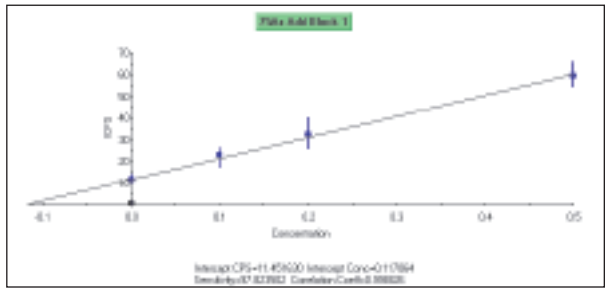

Figure 2: Standard additions calibration of NASS-5 seawater (diluted 1:10)

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The use of kinetic energy discrimination makes the analysis very robust with respect to the matrix such that a variety of sample types can all be analyzed under the same set of conditions. Table 1 shows the recoveries of various reference materials under helium based kinetic energy discrimination conditions:

MATERIAL	REFERENCE VALUE (PPB)	VALUES FOUND (N=4)
NASS-5	1.27 +/- 0.12	1.16 +/- 0.05
CASS-4	1.11 +/- 0.16	1.15 +/- 0.04
SLEW-3	1.36 +/- 0.09	1.44 +/- 0.03

Table 1: Recoveries of reference materials under He-based KED conditions

For some analytes such as selenium, kinetic energy discrimination is not effective due to poor transmission so it can sometimes be more effective to use a hybrid of hydrogen reactive chemistry and energy discrimination under one set of conditions.

The reactivity of gas contaminants in the cell can cause problems when higher levels of calcium are present. This is due to the species $CaOH(H_20)^+$ forming. This needs to be excluded using energy discrimination and the measurement of arsenic in the presence of 100 ppm calcium is one of the standard specification tests for the XSeries^{II}. This is most effective with a mixture of 7% hydrogen in helium using 4-5 ml/min of gas in the cell and approximately +3V difference between the cell pole bias and the quadrupole. Figure 3 shows a typical calibration of arsenic in 100 ppm Ca from XSeries^{II} final test. Figure 4 shows overlaid scans of nitric acid, 100 ppm calcium and 1 ppb arsenic in 100 ppm calcium. Table 2 shows the mean and range data from a batch of 25 XSeries^{II} from final test.

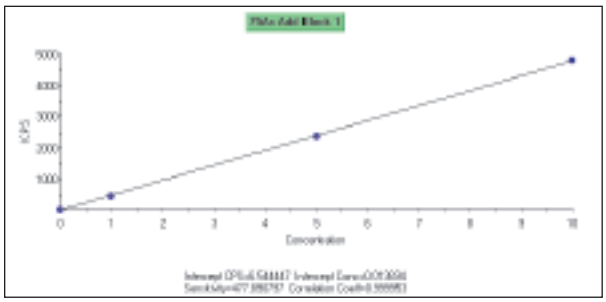


Figure 3: Typical calibration of arsenic in 100ppm Ca

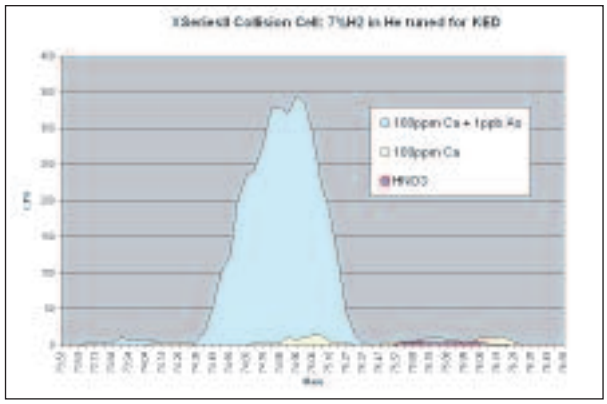


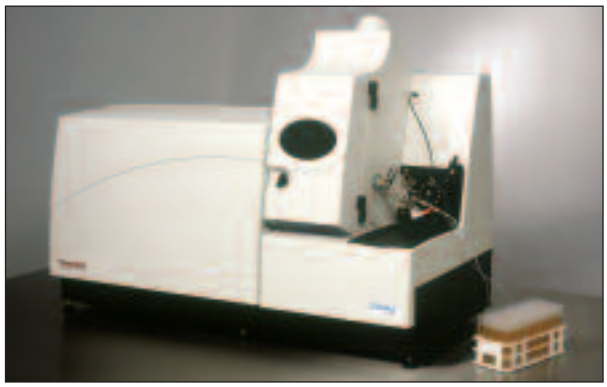
Figure 4: Scans of nitric acid, 100ppm calcium and 1ppb arsenic in 100ppm calcium

RANGE	AS BEC (PPT) IN 100 PPM CA
Mean	12.18
Upper Quartile	14.57
Lower Quartile	10.21

Table 2: Mean and Range data from 25 XSeries^{II} instruments

Conclusion

The XSeries^{II} ICP-MS has the flexibility and detection power to accurately determine the traditionally difficult element arsenic to very low levels in a variety of matrices. Third generation collision cell technology utilizes reactive or inert modes of operation and a unique hybrid mode for the best productivity and flexibility available in a quadrupole ICP-MS.



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