



Advanced Modes of Inductively Coupled Plasma - Mass Spectrometry (ICP-MS) for Reducing Spectral Interferences on Matrix Based in Food Matrices

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ABSTRACT

Inductively coupled plasma mass spectrometry (ICP-MS) is a highly sensitive analytical technique for determining trace and ultra-trace levels of elements in complex matrices such as food and beverages. Its accuracy can be compromised by spectral and non-spectral interferences arising from the sample matrix. This study explores the application of advanced ICP-MS operational modes, specifically, collision/reaction cell (CRC) technology using helium (He) and hydrogen (H₂) gases to reduce these interferences during the quantification of iron (Fe), arsenic (As), and selenium (Se) in packaged drinking water, representing a food-based matrix. During the analysis, potential spectral interferences such as ArCl⁺ on As-75, ArO⁺ on Fe-57, and Ar₂⁺ on Se-77 were effectively reduced using advanced ICP-MS modes, specifically CRC technology. By introducing helium (He) or hydrogen (H₂) gases into the cell, these interfering polyatomic ions are dissociated or neutralized before detection, allowing accurate and interference-free measurement. The study demonstrates that advanced interference-removal modes in ICP-MS play a crucial role in achieving precise and reliable quantification of trace elements in complex matrices such as food and beverages. The techniques provide high sensitivity, selectivity, and accuracy, making ICP-MS an indispensable tool for ensuring food and water safety in compliance with global quality standards.

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INTRODUCTION

Inductively coupled plasma mass spectrometry (ICP-MS) stands to achieve very low detection limits, which is crucial for identifying toxic elements such as iron, arsenic, and selenium, even at minimal concentrations. Additionally, ICP-MS offers rapid, simultaneous multi-element analysis with excellent reproducibility, making it ideal for evaluating complex food matrices [1]. Prior to instrumental analysis, sample preparation typically involves acid digestion using concentrated nitric acid or a combination of nitric and perchloric acids, often with microwave-assisted digestion to ensure complete mineralization of the organic matter. The choice of analytical method depends on several factors, such as the concentration level of the target metal, matrix complexity, and the required sensitivity [2]. It is well known for its high sensitivity and accuracy, allowing it to detect very low concentrations of harmful metals. ICP-MS measures multiple elements at the same time in a single run, making it fast and efficient. The combination of speed, precision, and multi-element capability makes ICP-MS an essential instrument for modern food safety research and quality control laboratories, where accurate monitoring of contaminants is crucial to protecting public health and meeting

regulatory standards [3,4]. The development and validation of an ICP-MS system equipped with a third-generation collision/reaction cell for the simultaneous quantification of seven elements (V, Cr, Fe, Co, Ni). Food matrices contain large amounts of arsenic (As) and selenium (Se), both of which are susceptible to spectral interferences. To minimize these interferences, the collision cell parameters were statistically optimized using experimental design techniques aimed at streamlining the analysis. Because the collision/reaction technique primarily resolves spectral interferences, additional tests were performed to evaluate the effects of different nebulizer gas flow rates and different sample dilutions on no spectral interferences. The final method was validated and complies with the relevant French and EU regulatory standards [5]. In ICP-MS, matrix effects can change the accuracy of results by affecting the analyte signal. These effects mainly happen in two ways: spectral overlaps and changes in signal intensity. This explanation focuses on signal intensity changes, which usually rely on signal suppression caused by high concentrations of other elements (matrix elements). At higher nebulizer flow rates, suppression is less effective, but signal enhancement may sometimes

occur under certain conditions. Plasma power and sampling depth don't greatly affect these matrix effects. Heavier matrix elements and analytes with lower mass are more affected, especially if the matrix has a low ionization potential. The effects are more related to the absolute amount of matrix elements, not their ratio to the analyte, so diluting the sample helps reduce the issue [6].

INSTRUMENTATION

Element Entrance Systems

ICP-MS is a very versatile technique that can handle different types of samples using different sample introduction systems. For liquid samples, the process starts with a nebulizer, which turns the liquid into a fine mist (aerosol) before it enters the plasma. Most systems use a peristaltic pump and auto sampler to deliver the sample, although self-aspirating nebulizers can also be used without a pump [7].

Nebulizers

The most commonly used pneumatic nebulizers in ICP-MS systems are the concentric and crossflow designs. The concentric design works best with clean samples, whereas the crossflow design is generally more tolerant of samples that contain higher levels of solids or particles. The sample introduction system helps turn a liquid sample into a fine mist (aerosol) so it can be analyzed. A small pump (peristaltic pump) pushes the liquid into the nebulizer at a steady rate. The nebulizer uses fast-moving gas to break the liquid into tiny droplets, like a spray can. Some nebulizers don't need a pump; they pull the liquid in using gas pressure, like a vacuum effect [8,9].

Spray Chambers

The spray chamber in an ICP-MS system plays an important role in preparing the sample before it enters the plasma. Its main function is to filter large droplets from the aerosol produced by the nebulizer and to smooth the flow of the sample, which may pulsate unevenly due to the action of the peristaltic pump. This helps ensure a steady and consistent sample introduction into the plasma. The droplets move through this tube, and the bigger ones, which are larger than about 10 micrometers in size, fall out due to gravity and exit through the drain tube at the end of the spray chamber. The smaller droplets, which are between 5 and 10 micrometers in size, pass between the outer wall and the central tube, and then they come out of the spray chamber and go into the sample injector of the plasma torch [10].

The Plasma Torch

The plasma torch in an ICP-MS system is a key component where the plasma is generated to ionize the sample. It is typically made of three concentric quartz tubes: an outer tube, a middle tube, and a sample syringe (the innermost tube). These tubes can be fused together or designed to be detachable for easy cleaning or replacement. To create the plasma, argon gas is used; it flows between the outer and middle tubes at a high rate (about 12–17 liters per minute) to maintain the plasma itself. A second flow of argon, called the auxiliary gas, passes between the middle tube and the sample injector at a much lower rate (about 1 liter per minute) to help properly position the base of the plasma flame. The third gas flow, known as the nebulizer gas, also flows about 1 liter per minute, and its main role is to carry the

sample in the form of a fine mist from the nebulizer into the center of the torch. This mist travels through the sample injector and into the hottest part of the plasma, where the sample is dried, vaporized, atomized, and ionized, so it can be analyzed by the mass spectrometer. The carefully control of these gas flows and the structure of the torch ensures that the sample is efficiently turned into ions while maintaining a stable plasma for accurate analysis [11].

Ion Source

ICP plasma as a sources convert the atoms into ions, then separates and is detected by the mass spectrometer. Plasma is ionized inductively heating the gas with an electromagnetic coil. Argon (Ar) gas is electrically heated to a high temperature (> 6000K) plasma. Plasma is one of the four states of matter. It consists of free electrons, positive ions, and neutral atoms or molecules. The ions enter into an electric field and are separated according to their mass/charge (m/z) ratio [12].

Ion Optics

The ion optics in an ICP-MS system are a set of metal components (such as plates or cylinders) placed between the skimmer cone and the mass analyzer. To guide the ions from the high-pressure plasma into the vacuum area of the mass spectrometer without losing or damaging them. These ion optics use electric voltages to focus and steer the ions into the analyzer while blocking unwanted stuff like dust particles, neutral atoms, and light (photons) that can interfere with the measurements. Some designs use metal disks to physically block these unwanted particles, while others slightly offset the ion path so that only charged ions can pass through. This helps keep the signal clear and accurate [13].

Mass Analyzer

After passing through the ion optics system, the ions are directed into the mass analyzer, where they are separated based on their mass-to-charge ratios. A variety of mass analyzers can be employed in inductively coupled plasma mass spectrometry (ICP-MS), including quadrupole, magnetic sector, and, less commonly, time-of-flight (TOF) systems. Among these, the quadrupole mass analyzer is the predominant choice for routine applications in clinical biochemistry due to its robustness, relatively low cost, rapid scanning capability, and ease of operation [14].

Quadrupole

A quadrupole mass filter is a device used to detect and measure different ions based on their mass-to-charge ratio. It has four metal rods, where the opposite pairs are given a combination of direct current (DC) and rapidly changing radio frequency (RF) voltages. This electric field allows only ions with a specific mass-to-charge ratio to pass through the center of the rods to reach a detector, while all other ions are pushed out of the path. For example, if we want to detect an iron ion (^{57}Fe), the voltages are set so only Fe ions can pass through and hit the detector, creating electrical pulses that are counted. The instrument scans quickly by changing the voltages to allow other ions through one at a time. This scanning process repeats for all the ions, allowing the machine to detect many elements in just a couple of minutes [15].

MODES AND INTERFERENCES IN ICP-MS

Standard (No Gas) Mode

Standard mode in an ICP-MS is the basic operation mode where no collision or reaction gases are introduced into the system. The ion beam passes directly to the mass analyzer without any reaction gas. In this mode, ions generated in the plasma are directed into the mass analyzer without any additional interference removal processes beyond the quadrupoles' mass filtering capability.

Polyatomic interferences

Formed when elements from the plasma, matrix, or solvents combine. Example ArO^+ interfering with 56Fe^+ , ClO^+ interfering with 75As^+ .

Isobaric interferences

when two isotopes from different elements have the same mass-to-charge ratio (m/z). Example 40Ca^+ overlapping with 40Ar^+ .

Doubly charged ion interferences

Ions that lose two electrons during ionization and appear at half their mass. Example Ba^{2+} interfering with certain lighter isotopes like Ce^+ .

Matrix effect

High concentration of certain elements in the sample can suppress or enhance the signal of analyte ions. When to use standard mode.

Simple matrices

Environmental samples with low concentration. Deionized water or a simple aqueous solution.

Elements unaffected by interferences

Example Na, K, Rb and Cs, which have few polyatomic or isobaric interferences [16,17].

Collision/Reaction Cell (CRC) Mode

In collision mode, an inert gas, typically helium, is introduced into the collision cell. An inert or reactive gas (like He, H_2 , or O_2) is introduced into a collision/reaction cell to remove or reduce interfering species before ions reach the detector. The main mechanism for removing interferences in this mode is kinetic energy discrimination (KED). Polyatomic interferences have larger sizes and higher energy than target analyte ions. These interfering loss kinetic energy as they collide with the helium atoms in the cell. A process called kinetic energy discrimination (KED) is applied.

Polyatomic interferences

Generated from plasma gases, sample matrix, or the solvent. Example ArO^+ interfering with 56Fe^+ . ClO^+ interfering with 75As^+ .

Effective for low-mass SO+(SULFUR-OXYGEN) interferes with 48Ti^+ : Works best for elements with mass < 100 amu.

Interference is unwanted signals that affect the accuracy of the measured ion counts. There are two main types: a. Spectroscopic (polyatomic or isobaric) interference occurs when ions of similar or the same mass/charge (m/z) overlap with the target elements' signal. b. Non-spectral interferences are caused by matrix effects such as

high total dissolved solids, ionization suppression, or transmission effects in the plasma [18].

MATERIALS AND METHODS

Chemicals and Reagents

The following chemicals were used to prepare the water samples: deionized water ($18.2 \text{M}\Omega \text{cm}$) obtained using a Milli-Q system (Direct-Q-3®), nitric acid (HNO_3 , 67-70% Suprapur® (for trace analytical grade), Supelco®) and hydrogen peroxide (H_2O_2 , 30.0-32.0% Suprapur® is trace analytical grade, Supelco®). Certified reference materials (Heavy metals) Inorganic Ventures Iron (Fe), Concentration Ion (1000 mg/L), Traceability (CGFE1), Purity $1000 \pm 4 \text{mg/L}$. Arsenic(As), Concentration Ion(1000 mg/L), Traceability(AAAS1), Purity $1000 \pm 10 \text{mg/L}$. Selenium (Se), Concentration Ion(1000 mg/L), Traceability (CGSE (4)1), Purity $1002 \pm 4 \mu\text{g/mL}$.

ICP-MS Instrument -Thermofisher Scientific ICAP RQ (13031).

Sample preparation

Packaged drinking water (Bisleri) sample collected from the market. Add 1-mL of concentrated nitric acid (HNO_3) to a defined amount of sample to acidify and preserve the sample, ensuring that the metal ions remain in solution. The mixture was diluted to a final volume of 50 mL with ultrapure water, ensuring that the sample conditions were consistent with the calibration standards. The prepared sample solution was analyzed under the same instrumental conditions as the standards to determine the concentrations of iron, arsenic, and selenium in packaged drinking water [19,20].

Standards preparation

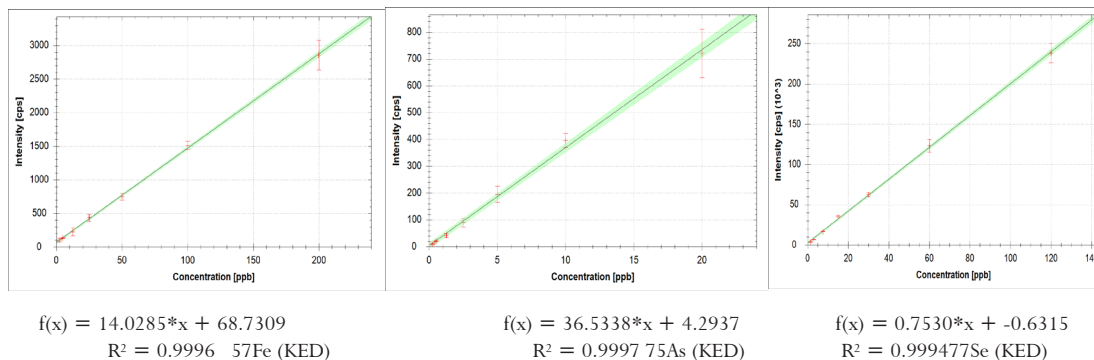
For the preparation of the Standards, mixtures of Fe, As, and Se, concentrated stock solutions were diluted to obtain working standards at a concentration level, which served as the source for preparing the final calibration standards. Each intermediate mixture was prepared in a 10 mL volumetric flask using appropriate aliquots from the respective stock solutions. For iron (Fe), dilute 0.025 mL (25 μL) of the 1000 mg/kg stock solution to 10 mL with deionized water to obtain an intermediate concentration of 2.5 mg/kg (2500 $\mu\text{g/kg}$). Similarly, for arsenic (As) and selenium (Se), 0.25 mL (250 μL) of the 10 mg/kg stock solution was diluted to 10 mL to obtain an intermediate mixture with a concentration of 0.25 mg/kg (250 $\mu\text{g/kg}$). The use of these Standards ensures consistency and precision in the preparation of low-level working standards required for the linearity and quantification of Iron, Arsenic and Selenium in packaged drinking water. Internal Standard Preparation. Additionally, 100 μL of an internal standard solution (prepared from a 10 mg/kg intermediate standard) is added to correct for any instrumental or matrix-related variations during analysis [21].

Calibration Standard Solutions

Calibration standards were prepared at seven different concentrations of iron (Fe): 2.5, 5, 12.5, 25, 50, 100, and 200 $\mu\text{g/kg}$. For arsenic (As) and selenium (Se), the standard concentrations were 0.25, 0.5, 1.25, 2.5, 5, 10, and 20 $\mu\text{g/kg}$, respectively. These prepared standards were analyzed under the same instrument conditions to generate calibration curves for each element. The linearity of these

Table 1: Elements iron (Fe), arsenic (As), and selenium (Se) result and selected mass

Element	Mass (m)	Correlation coefficient (R ²)	Packaged drinking water (Bisleri) MRL(mg/kg)	Element result (mg/kg)
Iron (Fe)	57	0.9996	0.1	BLQ
Arsenic (As)	75	0.9997	0.01	BLQ
Selenium (Se)	77	0.9994	0.01	0.001

**Fig.-1:** Linear Graph of Elements Iron (Fe), Arsenic (As), and Selenium (Se)

curves confirms that the analytical method can accurately quantify trace amounts of Fe, As and Se in packaged drinking water samples within the specified concentration range [22].

RESULTS AND DISCUSSIONS

Packaged drinking water (Bisleri) samples were analyzed for iron (Fe), arsenic (As), and selenium (Se) using ICP-MS, with the isotopic masses being Fe (m/z 57), As (m/z 75), and Se (m/z 77), respectively. The method showed good linearity with correlation coefficients (R²) of 0.9996 for Fe, 0.9997 for As, and 0.9994 for Se, confirming the accuracy and reliability of the calibration curve shown in Table-1. The sample results showed that Fe and As are below the limit of quantification (BLQ), while Se is detected at a very low concentration of 0.001 mg/kg, well below the maximum residue limit. This response shows that the packaged drinking water meets safety standards for these trace elements. However, during ICP-MS analysis, spectral interferences can affect accuracy. For example, Fe-57 suffers interference from ArO⁺ or CaO⁺ ions, As-75 is affected by ArCl⁺ (m/z 75), and Se-77 suffers interference from Ar₂⁺ species. These interferences cause false signals or elevated readings. To eliminate this interference, the collision/reaction cell (CRC) mode of ICP-MS was used. In this mode, helium (He) or hydrogen (H₂) gas is introduced into the cell to break down or neutralize interfering polyatomic ions before they reach the detector. This response ensures that only the target element ions are measured, improving signal accuracy and detection limits. In the ICP-MS instrument, each analyte is detected based on its unique mass-to-charge ratio (m/z). In this analysis, the measured masses of iron (Fe) 57, arsenic (As) 75, and selenium (Se) 77 are the most stable and non-interfering isotopes. The instrument ionizes the sample and separates these ions according to their masses for precise quantification. This demonstrates that ICP-MS provides an accurate and consistent response across the entire concentration range, ensuring reliable detection of trace metals in packaged drinking water samples linearity graph is provided in Fig no.1 [23,24].

Explanation Of Interference Control In Icp-Ms

Iron (Fe, m/z = 57)

Possible interferences: ArO⁺ (m/z = 56) and CaO⁺ (m/z ≈ 56–57). Since Fe(57) is a minor isotope and there is less overlap, interference is already minimal. In He mode, any remaining molecular ions are further reduced by collisions.

Signal below quantification (BLQ) + Fe content is negligible in the sample.

Arsenic (As, m/z = 75)

Possible interference: Ar⁺ + Cl⁺ ArCl⁺ (m/z=75). This ArCl⁺ The ion has the same mass number as As⁺ (75), so both appear at m/z = 75. Reaction solution: The interfering ions are either shifted to a new mass or neutralized, leaving only As⁺ detected at m/z = 75. When helium is used (collision), no chemical reaction occurs; instead, helium atoms collide more efficiently with ArCl⁺ than with As⁺. Because ArCl⁺ is larger, it causes kinetic energy discrimination when As⁺ passes through, the interference signal have be weakened. Result data: As = BLQ → after the reaction process, no significant As⁺ signal remains, confirming extremely low As⁺ content. When using a reaction gas (H⁺): ArCl⁺ + H₂⁺ ArClH⁺ (m/z = 76) or neutral species.

Selenium (Se, m/z = 77)

Main interferences: Ar₂⁺ (m/z = 80), Ar₂H⁺ (m/z ≈ 77), ArCl₂⁺ (m/z ≈ 77). Reaction solution: Using H⁺: Ar₂H⁺ + H₂⁺ neutral or shifted, ions Larger polyatomic ions were removed by kinetic energy filtering using He: He collisions. Only true Se⁺ passes to the detector → stable, accurate reading (0.001 mg/kg) [25].

CONCLUSION

The analysis of trace elements iron (Fe), arsenic (As), and selenium (Se) in packaged drinking water (Bisleri) is successfully performed using Inductively Coupled Plasma Mass Spectrometry (ICP-MS). The selected isotopic mass numbers of Fe (57), As (75), and Se (77) showed good linearity with correlation coefficients (R²), confirming

the reliability and accuracy of the analytical method. During the analysis, potential spectral interferences such as ArCl^+ on As-75, ArO^+ on, Fe-57, and Ar_2^+ on Se-77 were effectively reduced using advanced ICP-MS modes, specifically collision/reaction cell (CRC) technology. By introducing helium (He) or hydrogen (H_2) gases into the cell, these interfering polyatomic ions are dissociated or neutralized before detection, allowing accurate and interference-free measurement. Overall, the study demonstrates that advanced interference-removal modes in ICP-MS play a crucial role in achieving precise and reliable quantification of trace elements in complex matrices such as food and beverages. The method provides high sensitivity, selectivity, and accuracy, making ICP-MS an indispensable tool for ensuring food and water safety in compliance with global quality standards.

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AUTHOR STATEMENT

On behalf of all authors of the manuscript entitled Advanced Modes of Inductively Coupled Plasma - Mass Spectrometry (ICP-MS) for Reducing Spectral Interferences on Matrix Based in Food Matrices, written by Ravi. Sethi, Priyanka. Gour and Purnima. Dashora, I state that we have seen and approved the final version of the manuscript being submitted. We warrant that the article is our original work, has not received prior publication and is not under consideration for publication elsewhere.

CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest with respect to this work.

AUTHOR CONTRIBUTIONS

All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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