

ICP - Mass Spectrometry

Interferences in ICP-MS: Do we still have to worry about them?

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When the first commercial ICP mass spectrometer (ICP-MS) was introduced in 1983, it was of general opinion that this new technology freed analysts from worries about interferences. Now, many years later, we know that interferences do exist in ICP-MS, but we have a good understanding of them.¹ Over the years, new techniques and technologies have been developed to reduce or eliminate them.

In routine ICP-MS analyses, two types of interferences exist: non-spectral (physical and matrix effects) and spectral (isobaric and polyatomic). Let's look in more detail at both non-spectral and spectral interferences.

Non-Spectral Interferences

Physical interferences are generally related to the sample's viscosity and surface tension, affecting their transport through the autosampler probe, nebulizer and spray chamber. Due to the presence of sample matrix, an analyte signal can be suppressed or enhanced in comparison to standards without matrix, resulting in incorrect analytical answers. Matrix-matching of calibration standards and the use of internal standards are common techniques to compensate for these types of interferences.

One form of matrix interference is related to changes in the ionization efficiency of the analyte in the plasma. Samples with high total dissolved solids (TDS) or organic matrices can lower the energy of the plasma and decrease the ionization efficiency, affecting sensitivity in comparison to non-matrix standards. Plasma loading effects are compensated for by internal standards, hot plasma conditions and lower nebulizer flow.

Ion sampling or space-charge effect is another type of matrix interference. Behind the cones, the plasma changes into an ion stream due to the loss of electrons. Since ions have a positive charge, heavy ions repel lighter ions, causing mass-dependent matrix effects where lower mass ions are affected more. The Triple Cone Interface (Figure 1), available only in the NexION® family of spectrometers, provides a three-step reduction in pressure between the plasma (atmospheric pressure) and the Quadrupole Ion Deflector (vacuum), resulting in a substantial reduction in the divergence of the ion beam and consequently in reduction of space-charge effects. In the NexION 5000 multi-quadrupole ICP-MS, a patented hyper-skimmer cone with OmniRing™ assembly in the Triple Cone Interface reduces matrix effects even further.²

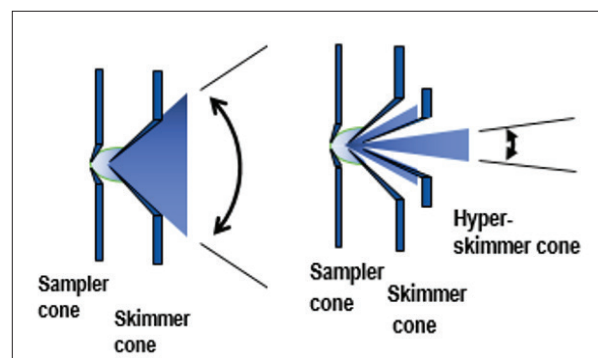


Figure 1. The two-cone design on the left shows a wide ion beam. The NexION series' three-cone design on the right shows a well-confined ion beam with minimal divergence, resulting in less matrix effects.

After the Triple Cone Interface is the Quadrupole Ion Deflector (QID) – also unique to the NexION family – which directs ions at a right angle, separating the positive ion beam from non-ionized particles, neutrals and photons (Figure 2). The QID is an electrostatic analyzer which transfers a selected mass range into the spectrometer, enhancing sensitivity and preventing unwanted species (e.g., neutrals and photons) from reaching the downstream ion optics. This action also lowers matrix effects and is the first step of mass resolution.

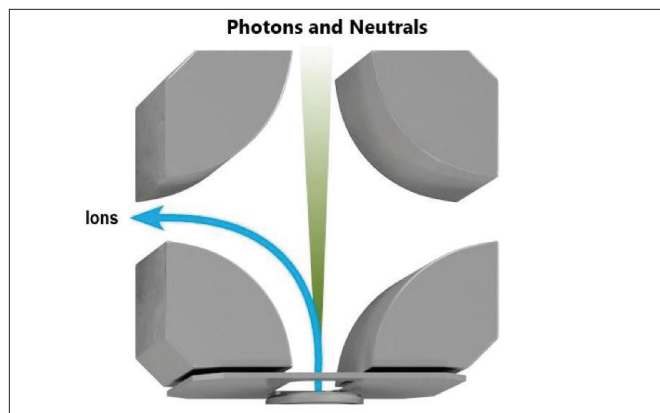


Figure 2. Diagram of the NexION series' Quadrupole Ion Deflector.

As mentioned previously, one of the analytical ways to compensate for analyte signal suppression or enhancement due to matrix effects is the use of internal standards. This technique can be extended to compensate for matrix effects by matching the internal standard ionization potential and mass to the analyte. It is desirable that internal standards are not present in samples or are at least present at very low concentrations.

Matrix effects due to plasma overloading can be reduced by gas dilution (using AMS – All Matrix Solution) where a flow of argon is introduced into the neck of the spray chamber via the AMS port to dilute and dry the tertiary aerosol stream (Figure 3). This reduces the amount of matrix reaching the plasma, lowers the level of oxides, and allows for more efficient ionization by increasing the plasma robustness.



Figure 3. Spray chamber with the AMS port for the NexION ICP-MS series.

Spectral Interferences

Spectral interferences, both isobaric and polyatomic, are caused by other ionic species having the same or very similar mass to the analyte. In quadrupole-based instruments operating with a resolution of ~ 1 amu, it means a direct overlap of an interference on an element of interest.

Isobaric interferences occur when there are naturally occurring isotopes for more than one element at the same mass (i.e. Ba⁺, Ce⁺, and La⁺ at mass 138), or when a doubly-charged ion occurs at the same mass as an analyte (i.e. Pb²⁺ on Rh⁺ at mass 103).

Polyatomic interferences occur when elements present in a matrix combine with each other or with plasma gases forming molecular ions at the same mass as an analyte. The most common polyatomic ions are argides (i.e. ArCl⁺), oxides (i.e. ClO⁺), hydrides (i.e. BrH⁺), hydroxides (i.e. CaOH⁺) and dimers (i.e. Ar₂⁺).

Historically, when ICP-MS instruments did not have a collision/reaction cell, elemental equations were used to correct for spectral interferences when no interference-free isotopes of the analyte could be found. That approach had many limitations and could lead to incorrect results.

A big breakthrough in dealing with spectral interferences came with the incorporation of Universal Cell Technology into PerkinElmer ICP mass spectrometers (Figure 4).

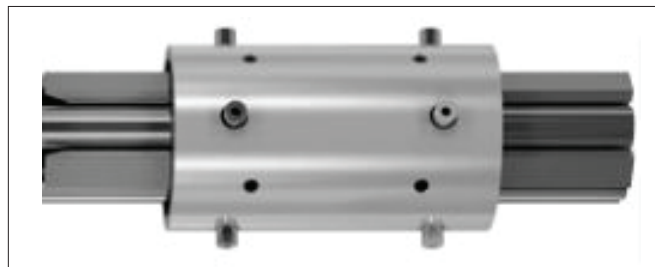


Figure 4. NexION series' Universal Cell.

The Universal Cell, a quadrupole-based mass filter, can perform two different tasks depending upon the nature of the introduced gases and electrical parameters. The first one is the capability to act as a Dynamic Reaction Cell (DRC) with reactive gases, such as NH₃, O₂, CH₄ or H₂. The second is the ability to use the cell as a collisional device, employing kinetic energy discrimination (KED) with a non-reactive gas, such as He and Ar.

Universal Cell – DRC Mode in a Single-Quad ICP-MS

In DRC mode³, the Universal Cell is pressurized with a reactive gas. For the reaction gas to be considered suitable for interference correction, three criteria need to be met. First, the reaction of the gas should be preferentially allowed to proceed thermodynamically with either the interferent species or the analyte of interest through an exothermic reaction. Second, the reaction kinetics in the gas phase suggest that the reaction between the reaction gas and the target polyatomic or analyte ion should be relatively fast. Finally, the use of a selected reaction gas should not lead to reaction by-product species that would be considered as new interferences on the target mass or

other analytes. However, the latter has been a major limitation of reaction cells that do not employ dynamic bandpass tuning, such as those utilizing static filtering or no filtering. This is especially important when analyzing samples with complex matrices where unwanted side-reactions between the reaction gas and other sample matrix constituents can proceed unhindered and cause new interferants on other masses of interest. In such cases, the user is often forced to utilize an alternate and less efficient reaction gas or resort to diluting the reaction gas with an inert gas (e.g., He). In both cases, the efficiency of interference removal in the cell is compromised due to lower reaction kinetics.

A classic example of converting an interference into a different form is the determination of calcium (Ca) at mass 40 where the major isotope of argon (Ar) co-exists. Argon ions react with ammonia according to the following fast exothermic reaction: $Ar^+ + NH_3 \rightarrow NH_3^+ + Ar$ with a reaction rate constant of $1.6 \times 10^{-9} \text{ cm}^3 \text{ molecule}^{-1} \text{ s}^{-1}$. Argon ions are changed into Ar atoms which do not interfere with Ca 40^+ . Calcium ions practically do not react with ammonia: $Ca^+ + NH_3 \rightarrow \text{slow reaction}$ (reaction rate 1.2×10^{-12}), so Ca⁺ can be determined at mass 40 without interferences.

As mentioned above, a major advantage of utilizing a quadrupole as the Universal Cell is the presence of a well-defined stability diagram for each analyte. This allows parameter RPq (related to RF voltage) and RPa (related to DC voltage) to create a dynamic bandpass (DBT) with both a low and a high mass cut off (Figure 5). The DBT filters out undesirable masses, making gas-phase reactions more specific and permitting only desirable reaction products to enter the analyzing quadrupole.³

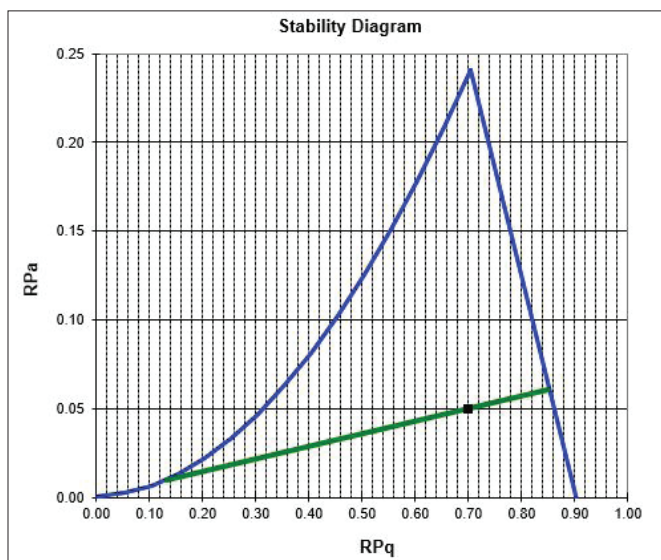


Figure 5. Quadrupole stability diagram for V 51 with RPq=0.7 and RPa=0.05.

A good example of how RPq is used as a low mass filter is the removal of ClO⁺ interference on V⁺ at mass 51. Ammonia reacts rapidly with ClO⁺, creating an intermediate product of Cl⁺, which can then react with NH₃ to form another interference: ClNH₂⁺ at mass 51. However, because the RPq parameter serves as a mass filter, it can be set to a value that makes ³⁵Cl⁺ unstable in the cell, preventing the formation of ClNH₂⁺ and allowing V⁺ to be accurately measured, free of interferences. As is shown in Figure 6, the background signal for 10% HCl measured at mass 51 decreases sharply with only a small flow of NH₃ when RPq is set to 0.7 (blue line). Nevertheless, a lower value of RPq requires a much higher flow of NH₃ to remove all chlorine-related spectral interferences (green line).

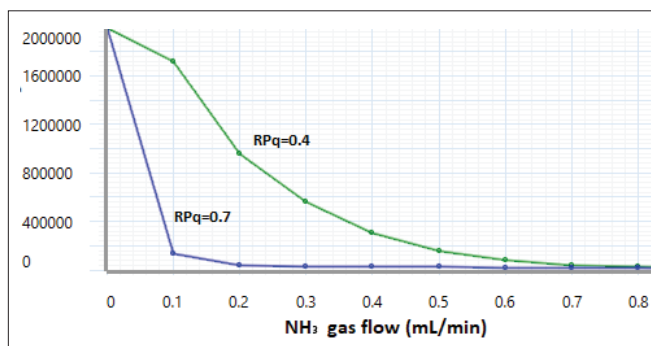


Figure 6. Signal intensities at mass 51 as a function of NH₃ flow in 10% HCl with two RPq values (blue line for RPq=0.7 and green line for RPq=0.4).

Universal Cell – KED Mode

The NexION Universal Cell can also work as a collision cell with kinetic energy discrimination (KED) when a non-reactive gas, such as He, is introduced. The gas collides with all ions in the cell, but more collisions occur with mono-atomic and polyatomic ions that have larger cross-sectional diameters than analyte ions. Consequently, the kinetic energy of the larger ions is reduced, and they are prevented from being transmitted into the analyzer quadrupole by kinetic energy discrimination, an energy barrier established between the cell and the analyzing quadrupole. Kinetic energy discrimination interference removal is independent of the matrix and is applicable mostly in single-quadrupole ICP-MS spectrometers, when appropriate reaction gases cannot be found. For example, KED works relatively well for the reduction of PO²⁺ and SO²⁺ interferences on Cu and Zn.

KED mode is not as effective in the removal of interferences as DRC technology for isobaric, doubly-charged and many polyatomic interfering ions due to small collisional cross-section differences between these interferences and analytes. Another drawback of KED mode is a significant loss of analyte sensitivity, especially at lower masses. Therefore, KED mode is best suited for semi-quantitative analysis, environmental monitoring and other analyses without a high demand for low BECs and DLs.

Cold Plasma Mode

By reducing the plasma power and/or increasing nebulizer flow, a less energetic plasma (known as a cold or cool plasma) is created. Under these conditions, the ionization of Ar is much less efficient, and thus Ar ionic interfering species like Ar⁺, ArO⁺, and ArH⁺ are not readily formed. Several low mass and easily ionized elements show significant signal enhancement due to lower Ar ion concentrations in the ion stream, creating less space-charge effects. On the other hand, the sensitivity of elements that have relatively high ionization potentials or a strong energy bond with oxygen drops drastically. Moreover, matrix decomposition is quite poor, so the use of cold/cool plasma conditions is not suitable for samples with high TDS.

Cold plasma conditions can be used for analysis not only in Standard mode but also in DRC mode with NH₃, allowing lower BECs to be achieved for some elements, such as, K, Ca, Cr, Fe and few others, by reducing the formation of polyatomic and isobaric interferences, enhancing sensitivity and minimizing the background from the instrument, including the cones. The plasma generator designed for the NexION family of instruments (i.e. NexION 1000, NexION 2000, NexION 5000) enables fast power switching between cold and hot plasma, thus increasing throughput when both conditions are used in a single method.⁴ In Figure 7, calibration curves for Mg and K in Cold Plasma mode and DRC mode with NH₃ in 20% HCl demonstrates excellent linearity and sub-ppt BECs and DLs.

Universal Cell with Multi-Quadrupole Technology

A significant improvement in spectral interference removal happens when Universal Cell Technology is combined with multi-quadrupole technology, as in the NexION 5000 ICP-MS. A schematic of the system is shown in Figure 8 and is described in the NexION 5000 Multi-Quadrupole ICP-MS product note.⁵

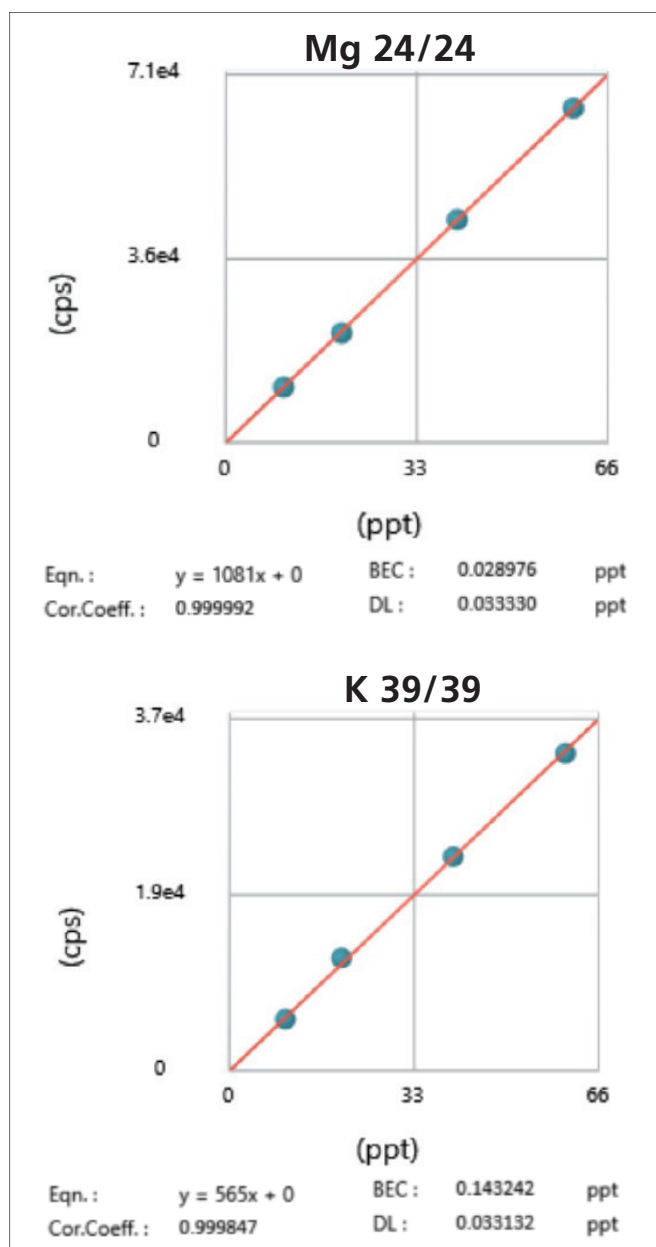


Figure 7. Mg and K calibration curves in Cold Plasma mode in 20% HCl.

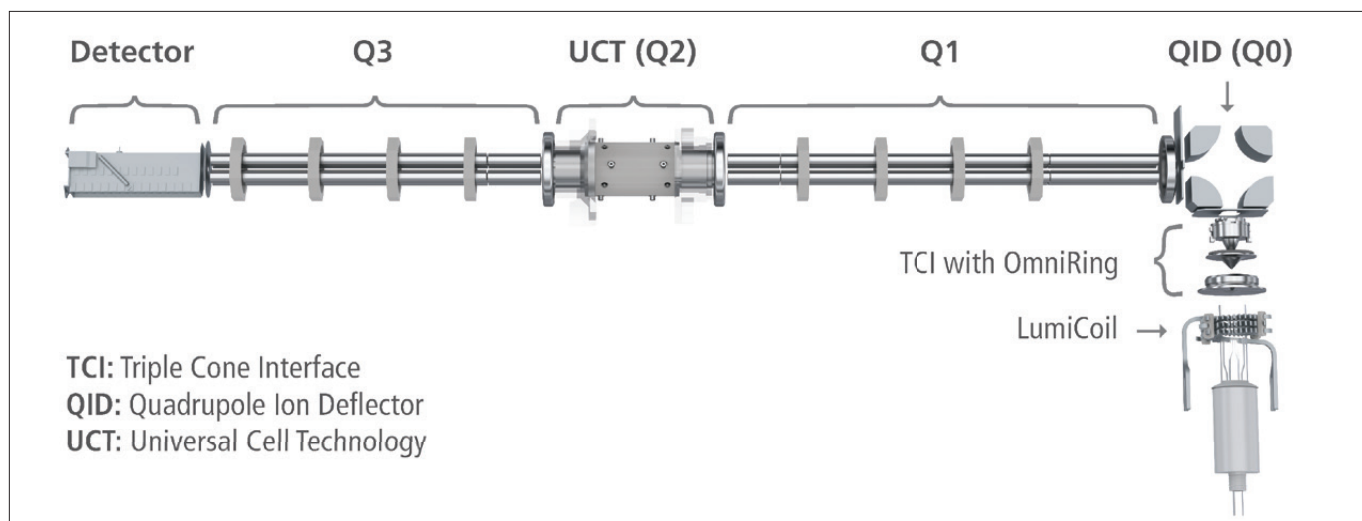


Figure 8. Schematic of the ion path of the NexION 5000 ICP-MS.

The Quadrupole Ion Deflector (QID – Q0) has the same role as described previously in this document, rejecting photons and neutral species. At the same time, the QID transfers a selected mass range into the spectrometer, enhancing sensitivity and in some cases even rejecting some of the sample matrix. The second quadrupole (Q1), placed before the Universal Cell (Q2), can work as an ion guide in Single Quad mode, transmitting all ions into the Universal Cell, or as a mass-filtering device with a nominal resolution of 0.7 amu. The mass-filtering ability of Q1 allows only ions of a specific mass to be passed into the Universal Cell, while all other ions not at this mass from the matrix, solvents and plasma are rejected. This capability has a significant impact on the reduction of spectral interferences which may arise from reactions of ions with a lower mass than the analyte and result in polyatomic interferences on the mass of interest. Q1 works together with the Universal Cell where up to four reactive or non-reactive gases can be used to create desirable reactions or collisions. Q3, the fourth quadrupole in the NexION 5000 ICP-MS, works predominantly as a resolving transmission analyzer quadrupole, but can also serve as an ion guide if Q1 is set as the resolving quadrupole.

As shown in Table 1, the NexION 5000 ICP-MS allows an analyte to be measured in four different analytical modes using either Standard, DRC or KED cell conditions. In analytical methods, two Single Quad modes can be typically used for analytes without spectral interferences, whereas the use of MS/MS or Mass Shift modes in concert with the DRC mode ensures the most effective interference removal. In MS/MS mode, Q1 and Q3 are set to the same mass, while in Mass Shift mode, Q1 is set to the mass of interest and Q3 is set to a higher mass where the resulting cluster ion resides.

Four scanning modes are a valuable tool during method development where the Product Ion Scan mode looks at resulting masses after a reaction, and Precursor Ion Scan mode looks at masses that can create a specific product ion. Q1 and Q3 scans can operate in all three cell modes to demonstrate spectra in selected mass ranges and conditions.

Interference Elimination with UCT and Multi-Quadrupole Technology

Examples of specific interference elimination mechanisms are described in detail in several semiconductor application notes, where it is shown how a combination of pure reaction gases (NH₃, H₂ and O₂) together with multi-quadrupole technology can improve BECs and DLs for Si, S, P, Ti, V, As, Cr and many other elements.^{6,7,8,9} Below, additional interesting cases of spectral interference removal are discussed.

Resolving Interferences on Pb isotopes

Pb isotopes 204, 206, 207 and 208 are considered stable, but their natural abundances can differ depending on place of origin, so there is interest in measuring the isotope ratio accurately. One of the Hg isotopes at mass 204 directly overlaps Pb 204. In Figure 9, a scan from mass 195 to 209 in Standard mode shows all Hg and Pb isotopes with the overlap Hg 204 on Pb 204.

Taking advantage of the fact that Hg reacts with NH₃ but Pb does not, the Hg interference can be removed. During a charge transfer reaction of Hg with NH₃ in the Universal Cell, Hg⁺ changes into Hg⁰, and as a neutral particle, it is removed from the cell. Optimization of the NH₃ flow, shown in Figure 10, indicates that an NH₃ flow of 0.3 mL/min is sufficient to remove Hg, but maximum sensitivity for Pb is achieved at a higher flow of 0.8 mL/min due to collision focusing.

Table 1. Modes of analysis and scanning in the NexION 5000 ICP-MS.

Mode	Q1	Cell Modes	Q3
Single Quad (Q1)	Resolving	STD/DRC/KED	RF-only guide
Single Quad (Q3)	RF-only guide	STD/DRC/KED	Resolving
MS/MS	Resolving	STD/DRC/KED	Resolving
Mass Shift	Resolving	DRC	Resolving
Precursor Ion Scan	Set to specific mass range	DRC	Set to specific mass
Product Ion Scan	Set to specific mass	DRC	Set to specific mass range
Q3 Scan	RF-only guide	STD/DRC/KED	Set to specific mass range
Q1 Scan	Set to specific mass range	STD/DRC/KED	RF-only guide

The mass scan in Figure 11 verifies that through the reaction with NH₃, Hg is removed and the Pb isotopes show natural abundances for all isotopes.

To further prove that Hg interferences on Pb are removed, isotope ratios of Pb 204 to Pb 206, 207 and 208 were measured with different concentrations of Hg. The results in Table 2 indicate that the ratio of Pb 204 to other Pb isotopes remains the same with or without Hg presence, confirming the elimination of Hg 204.

Other elements, such as Os, Ir, Pt and some rare earth elements (REE), if present in samples, could react with NH₃ creating interferences on Pb isotopes. However, the NexION 5000 ICP-MS operating in MS/MS mode does not allow these elements to reach the Universal Cell and create interfering species on Pb isotopes. The MS/MS mode is a powerful tool in preventing the creation of spectral interferences within the Universal Cell since Q1 operates at nominal 0.7 amu resolution, allowing only elements of interest to be stable and pass through.

The last row of Table 2 shows that even in the presence of 50 ppb REE, the Pb isotope ratios can be measured accurately.

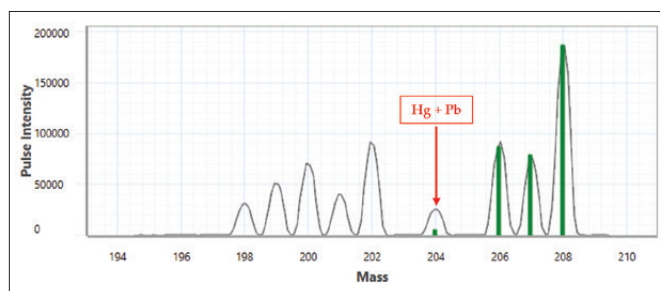


Figure 9. Standard mode scan of 1 ppb Hg and 1 ppb Pb. Pb isotope abundances shown as green bars.

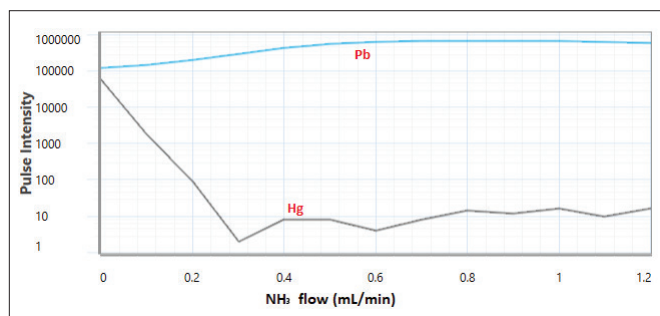


Figure 10. Hg signal attenuation with flow NH₃ (mL/min) in the presence of Pb at m/z 204.

Table 2. Pb 204 ratio to Pb 206, 207 and 208 in presence of Hg and REE.

Sample	IR Pb 204/206	IR Pb 204/207	IR Pb 204/208
1 ppb Pb	0.0546	0.0653	0.0265
1 ppb Pb + 1 ppb Hg	0.0548	0.0653	0.0264
1 ppb Pb + 1 ppb Hg	0.0542	0.0654	0.0265
1 ppb Pb + 1 ppb Hg	0.0533	0.0652	0.0262
1 ppb Pb + 50 ppb REE	0.0538	0.0651	0.0263

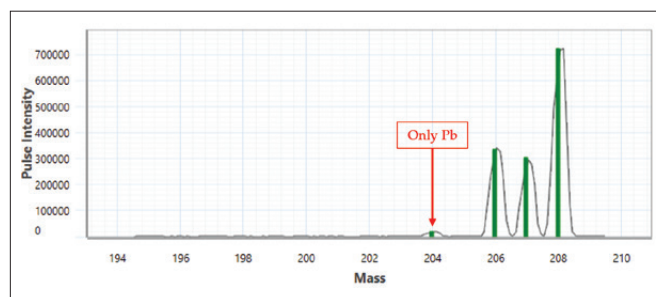


Figure 11. DRC-NH₃ scan of 1 ppb Hg and 1 ppb Pb.

Resolving Rb 87/Sr 87 Overlap

Accurate measurement of Rb 87/Sr 87 and Sr 87/Sr 86 ratios allows geochronology to determine the age of rocks and minerals containing these two elements.^{10, 11} Rb 87 has a long half-life time, but eventually it changes into a stable isotope of Sr 87, so the ratio Sr 87/Sr 86 could indicate the age of the rock formation.

High resolution mass spectrometers do not have enough resolving power to separate Rb 87/Sr 87; therefore, generally, a tedious chemical separation before analysis is required. However, today the power of chemical reactions combined with the multi-quadrupole technology in the NexION 5000 ICP-MS allows Sr isotopes to be resolved from Rb. Sr is oxidized when N₂O, a strong oxidizer, is used in the Universal Cell, while Rb is practically non-reactive. The Mass Shift mode of the NexION 5000 ICP-MS allows the measurement of Sr as SrO⁺ in a spectral region where the background is free from any other species. If such measurement is conducted in a single quadrupole, it is a possibility that non-reactive Rh and Pd, if present in the sample, would interfere with the accurate determination of Sr isotopes.

Figure 12 shows a scan of 1 ppb Rb and 1 ppb Sr, where Sr isotopes are completely resolved from Rb isotopes by converting Sr⁺ into SrO⁺ through the reaction with N₂O. Now the Sr 87/Sr 86 ratio can be accurately measured as SrO 103/SrO 102.

To find out how much N₂O is required to completely convert Sr⁺ into SrO⁺, an optimization of the N₂O flow was performed (Figure 13). Rb 85 and Sr 88 were used for this test. The result indicates that 1.2 mL/min of N₂O is sufficient to change Sr⁺ into SrO⁺, while the Rb 85 signal is stable and even slightly enhanced by the collisional focusing.

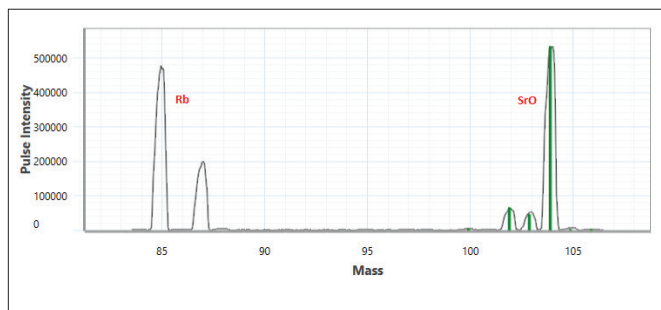


Figure 12. Mass spectrum of 1 ppb Rb and 1 ppb Sr using N₂O reaction gas to convert Sr⁺ into SrO⁺.

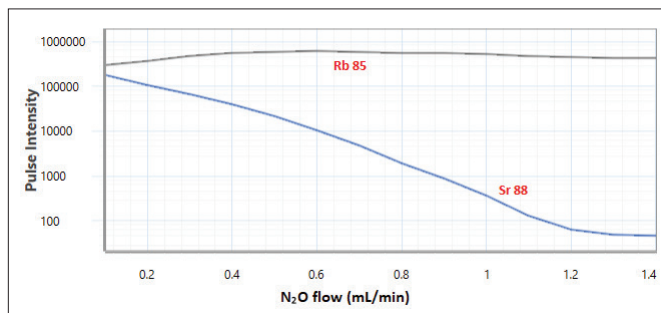


Figure 13. Rb 85 and Sr 88 signals as a function of N₂O flow.

To ensure that the isotope ratios of Sr isotopes are not changed during the conversion to SrO, or in the presence of Rb, the measurements of Sr 87/Sr 86 isotopes were performed in Standard mode at original masses and in DRC mode with N₂O at SrO 103/SrO 102 masses (Table 3).

Table 3. Sr 87/86 Isotope ratio using Standard MS/MS mode and DRC-N₂O Mass Shift mode.

Sample	Mode	Sr 87/86 Ratio (std. dev.)
1 ppb Sr	STD MS/MS	0.7334 (0.0008)
1 ppb Sr	DRC-N ₂ O Mass Shift	0.7368 (0.0015)
1 ppb Sr + 1 ppb Rb	DRC-N ₂ O Mass Shift	0.7347 (0.0022)

No mass discrimination correction was applied, but from the results summarized in Table 3, all three ratios are very similar, verifying that the approach of using N₂O as a reaction gas to convert Sr⁺ to SrO⁺ solves the problem of the Rb 87 and Sr 87 overlap. Each ratio is an average of five measurements with five replicates and 4000 ms integration time per isotope. The standard

deviation is a function of integration time per replicate. In this test, the integration time was relatively short in comparison with what is typically used in isotope ratio analysis; nevertheless, the precision of the measurements was very good.

During the Sr-Rb experiments, a peak at mass 88 was observed while 1% HNO₃ was scanned with N₂O. It was concluded that this is a cluster of (N₂O)₂ at mass 88 formed in the cell (Figure 14). Even though it would not have had any effect on SrO measurements, the Universal Cell of the NexION 5000 has an easy way of removing such by-products of reactions.

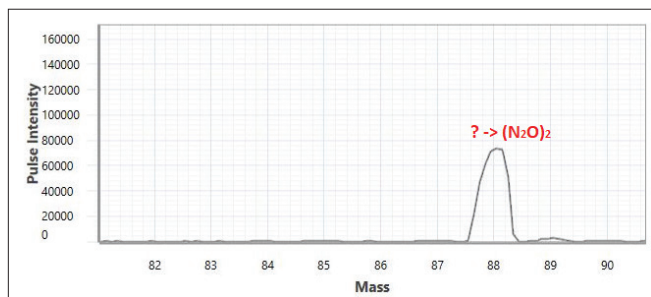


Figure 14. (N₂O)₂ peak observed at mass 88 with low RPq value of 0.35.

A dynamic bandpass, a main attribute of the quadrupole Universal Cell, can be tuned and this unwanted reaction producing the (N₂O)₂ cluster stopped by increasing the RPq value (Figure 15).

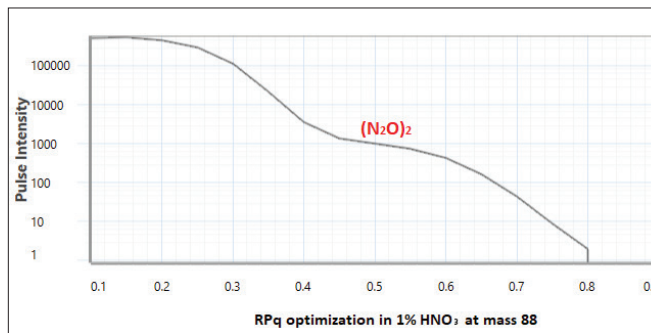


Figure 15. RPq optimization in 1% HNO₃ at mass 88.

Resolving Ba interference on Cs 137

Caesium 137 is a radioactive isotope with relatively short half-life (30 years) obtained from the nuclear fission of U 235, so its toxicity is high, even in small amounts.¹² Cs 137 is used in medicine for radiation therapy and is also an important indicator of environmental and living organism contamination after nuclear tests and nuclear accidents, such as Chernobyl or Fukushima.

One of the Ba isotopes exists at mass 137, which interferes with Cs 137 determination. A similar technique as was used in resolving Sr 87 from Rb 87 can also be used in this case: Ba reacts very efficiently with N₂O creating BaO⁺ at higher masses, while Cs does not react and can be measured at the original mass.

In the scan shown in Figure 16, all Ba isotopes were moved 16 masses higher, allowing Cs to be measured at the original mass free from isobaric interference. A stable isotope of Cs 133 served as a surrogate for radioactive Cs 137 in this example. Although such analysis can be conducted using a single-quadrupole ICP-MS, the excellent resolution and sensitivity of the NexION 5000 guarantees a lower detection limit, which is necessary for Cs 137 measurements.

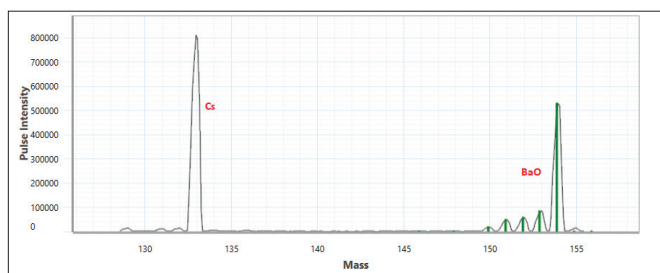


Figure 16. Mass spectrum of 1 ppb Cs and 1 ppb Ba using N_2O reaction gas to convert Ba^+ into BaO^+ .

In order to determine the amount of N_2O needed to completely convert Ba^+ into BaO^+ , an optimization of the N_2O flow was performed using 1 ppb Cs 133 and 1 ppb Ba 138 (Figure 17).

The result indicates that 1.1 mL/min of N_2O is sufficient to change Ba^+ into BaO^+ , while the Cs 133 signal is stable and even slightly enhanced due to collisional focusing.

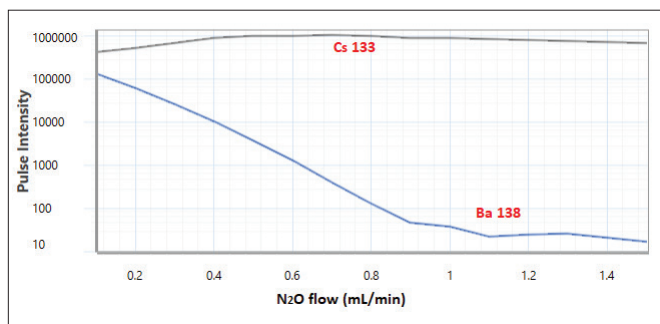


Figure 17. Cs 133 and Ba 138 signals as a function of N_2O flow.

Conclusion

To answer the original question, interferences will always occur in ICP-MS and need to be dealt with. However, the NexION 5000 multi-quadrupole ICP-MS with triple-quadrupole technology, in combination with the Universal Cell, is able to effectively and reproducibly remove spectral interferences leading to improved accuracy, repeatability and reproducibility, while solving problems difficult for single-quadrupole or even high-resolution ICP-MS

instruments. Furthermore, due to the unique designs of the introduction system, RF generator and interface region, the NexION 5000 ICP-MS can also easily manage physical and matrix interferences.

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