

Agilent

Trusted Answers

Single Quadrupole ICP-MS vs Triple Quadrupole ICP-MS

What is the difference and which one would suit your application?

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In the 1980s, we introduced our first single quadrupole inductively coupled plasma mass spectrometer (ICP-MS).

In 2012 we introduced the first triple quadrupole inductively coupled plasma mass spectrometer (ICP-QQQ), followed by a second-generation instrument in 2016.

Today, Agilent ICP-MS instruments are used in practically every industry, including:

- routine environmental monitoring of water, soil, sediments, waste, biota, and airborne pollutants
- detection of novel and emerging contaminants such as nanoparticles and microplastics
- consumer product testing
- food and pharmaceutical safety applications
- life science and clinical research
- mining, materials, and metals analysis
- geochemistry, nuclear, and renewable energy
- petrochemicals and biofuels
- semiconductor process chemicals analysis



Agilent single quadrupole ICP-MS (left) and triple quadrupole ICP-MS (right, with Agilent HPLC)

What is ICP-MS?

ICP-MS—inductively coupled plasma mass spectrometry—is a fast, multielement, atomic spectrometry technique used for inorganic, elemental analysis. ICP-MS can measure almost every element and provides low detection limits, high sample throughput, and measurement over a wide concentration range.

The ICP-MS technique was developed commercially in the 1980s when existing ICP technology (from ICP-OES) was combined with quadrupole mass spectrometer technology already in use in residual gas analyzers and GC/MS.

ICP-MS uses the ICP as a high temperature plasma ion source to convert a sample to individual ions, which are then extracted into the mass spectrometer (MS) to be separated and detected. Most commercial ICP-MS instruments use a quadrupole MS, as it provides a good combination of fast scanning speed, single unit mass resolution, and relatively low cost.

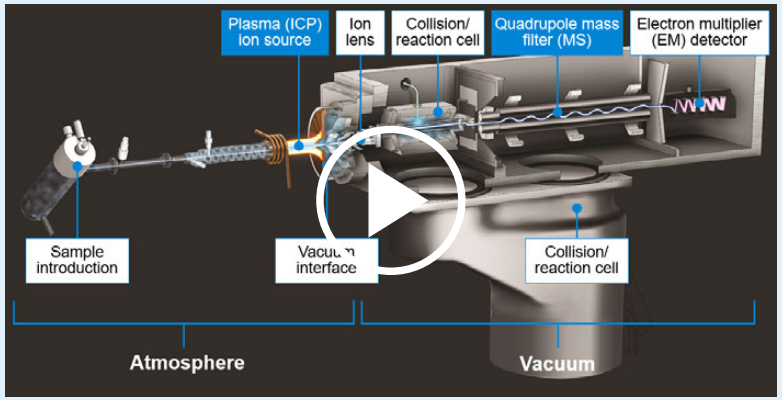
The ICP operates at atmospheric pressure and the mass filter and detector require a high vacuum, so an ICP-MS also needs an interface to transfer the ions from the ICP to the MS, while maintaining the low pressure in the vacuum system. Ion lenses—metal plates with voltages applied to them—are used to steer and focus the ions through the interface and into an enclosed collision/reaction cell. The collision/reaction cell resolves common spectral interferences in a chamber that can be pressurized with an inert (collision mode) or reactive (reaction mode) cell gas. The ions then pass to the quadrupole mass filter, which separates the ions into their different masses and transfers them to the detector, where the individual ions are counted. Elemental concentrations are calculated by comparing the measured count rate at each mass to the signal in a known standard.

Agilent offers two configurations of quadrupole ICP-MS:

1. Conventional or “single quadrupole” ICP-MS, and
2. “Triple quadrupole” ICP-MS, also known as ICP-QQQ, ICP triple quad, or “TQ”.

Teach me in 10

This short video from LabTube is a high-level introduction to the technique of ICP-MS. It explains the technology and key performance capabilities of the technique.



What Are the Differences Between Single Quadrupole ICP-MS and ICP-QQQ?

The most common configuration of ICP-MS is a “single quadrupole” instrument, which has one analyzer mass filter, located between the collision/reaction cell and the detector. A triple quadrupole ICP-MS configuration has two quadrupole mass filters, one before and one after the collision/reaction cell. The triple quadrupole configuration can perform two mass filtering steps, which is why the technique is also known as tandem ICP-MS or ICP-MS/MS.



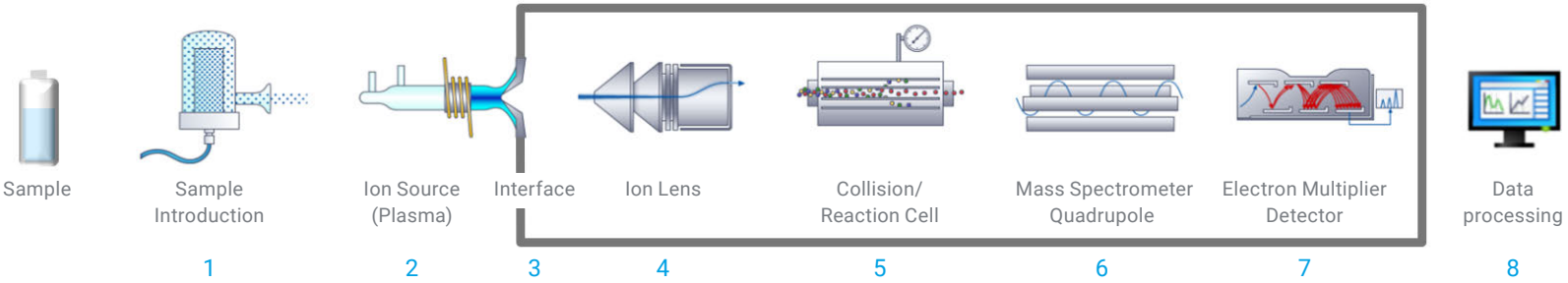
How Does an ICP-MS Work?

An ICP-MS instrument uses a plasma (ICP) to ionize the elements in a sample and then measures the ions using a mass spectrometer (MS).

The main components of a single quadrupole ICP-MS instrument are:

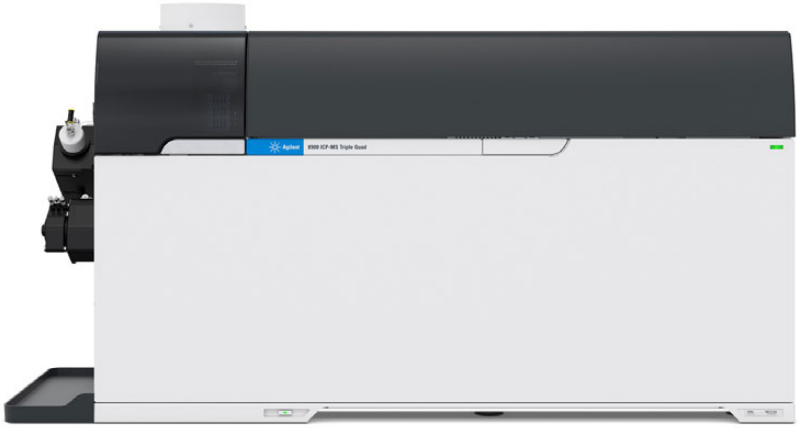
1. Sample introduction system to form a fine aerosol mist from the liquid sample
2. Plasma (ICP) to convert the elements in the sample aerosol to ions
3. Interface to extract the ions into the vacuum system
4. Ion lens to focus the ions and separate them from background signals
5. Collision/reaction cell (CRC) to resolve the analyte ions from interfering ions
6. Mass spectrometer (MS) to filter the analyte ions by mass
7. Electron multiplier (EM) detector to count the number of ions at each mass
8. Data processing software to convert the measured signals to concentrations

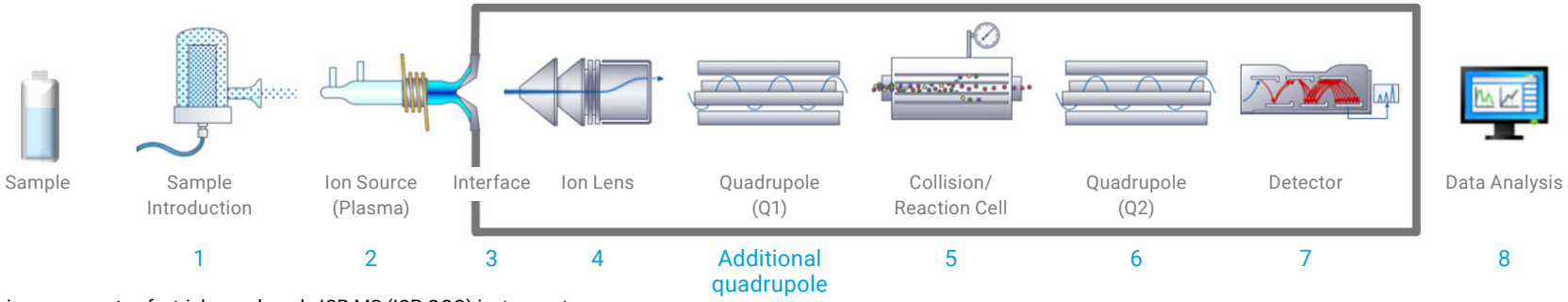




Main components of a single quadrupole ICP-MS instrument.

A triple quadrupole ICP-MS includes all the components of a single quadrupole instrument, but with the addition of another quadrupole mass spectrometer between the ion lens (4) and the CRC (5). The additional mass spectrometer (Q1) of an ICP-QQQ filters the ion beam before the ions reach the CRC, so only a single mass enters the cell at any given time. This double mass filter or "tandem" (MS/MS) configuration means that reactive cell gas methods can be reliably used to resolve interferences, leading to lower detection limits and better accuracy for some difficult analytes.



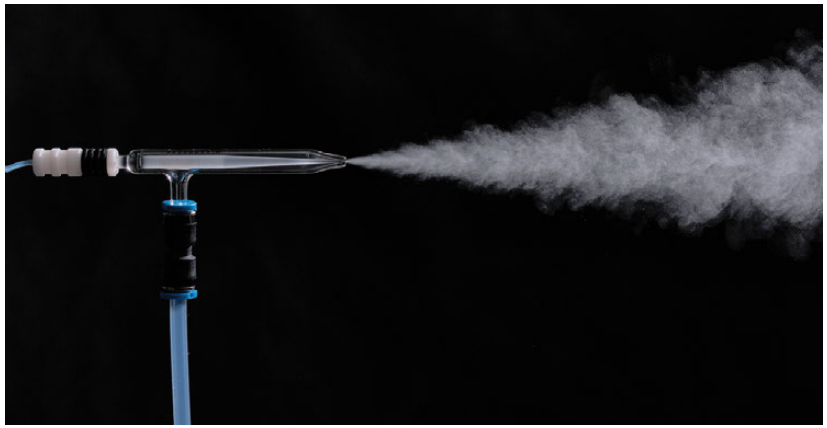


Main components of a triple quadrupole ICP-MS (ICP-QQQ) instrument.

Let's start by looking in a little more detail at the main components of an ICP-MS instrument.

1. The sample introduction

ICP-MS is usually used to measure liquid samples, although solids and gases can be handled with the addition of appropriate accessories. The sample solution is pumped into a nebulizer, where the liquid is converted into a fine spray or aerosol mist using a jet of argon gas. The aerosol mist passes through a spray chamber, where the larger droplets are removed. The fine droplets are carried by the argon gas flow to the ICP plasma torch.



A nebulizer turns the liquid sample into an aerosol.



The aerosol passes through a spray chamber where the larger droplets are removed.

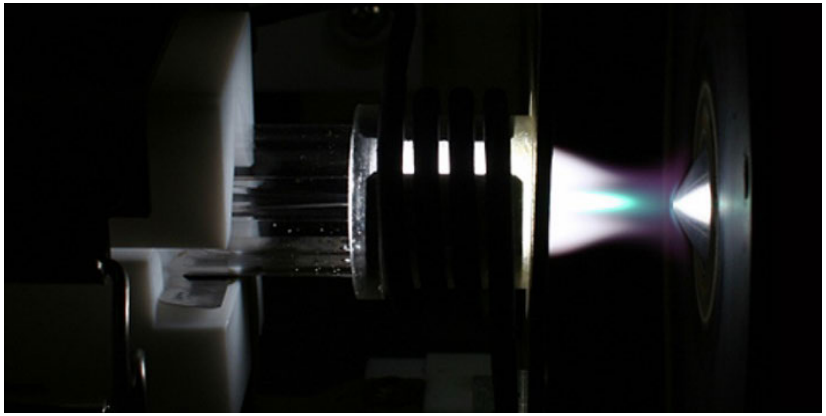
The sample introduction components are selected and optimized to work in combination with the ICP. This means that the nebulizer and spray chamber are designed to produce an aerosol that can be effectively processed by the plasma. The overall solution load, the aerosol density (number of droplets), and the droplet size are key factors that affect the ability of the plasma to dry and decompose the sample material carried in the droplets. The plasma is most effective at producing ions if the aerosol contains small droplets of uniform size, dispersed across the central channel of the plasma.

2. The ICP (plasma) ion source

The term "plasma", which refers to a substantially ionized gas, can apply to things as diverse as a fluorescent light bulb and the sun. In the plasma used in ICP-MS, the ionized gas is argon, which flows through a quartz tube. The energy is provided by a radio frequency (RF) generator operating at about 1.5 KW – around half the power of a typical domestic kettle. The RF energy is transferred to the argon gas flow by inductive coupling from a load coil wrapped around the outside of the quartz tube. The RF field causes free electrons in the argon gas flow to oscillate; causing them to collide with argon atoms with enough energy to remove an electron, ionizing the argon atoms. The new argon ions and electrons also oscillate in the RF field, leading to further collisions and further ionization in a process that distributes energy through the gas stream to form the plasma. The energy density in the ionized argon gas is very high, so the ICP reaches a temperature of 10,000 degrees Celsius – hotter than the surface of the sun!



The plasma is formed from argon gas flowing in a quartz tube.

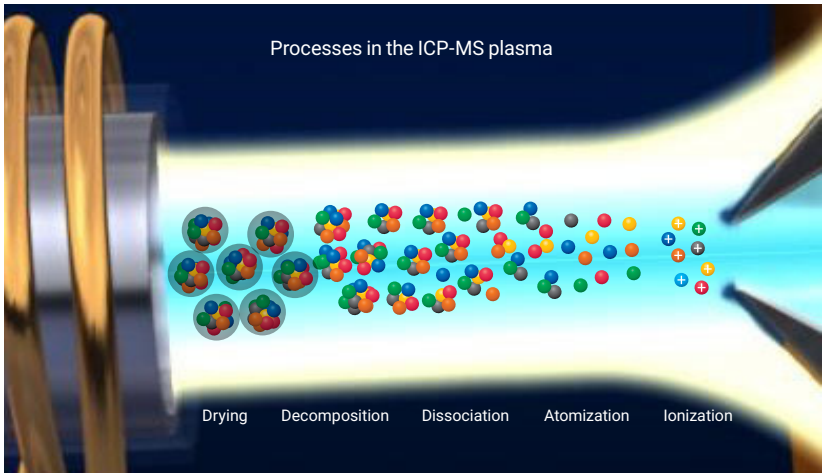


The argon gas is ionized by a radio frequency generator. The sample aerosol is carried through the center of the plasma.

The argon gas passing through the outer quartz tube flows at a rate of around 15 liters per minute. This "plasma gas" forms the plasma and cools the quartz tube to prevent it from melting. Two additional, smaller quartz tubes are positioned concentrically inside the outer tube, together making up the plasma "torch". The middle quartz tube carries an auxiliary gas flow which pushes the base of the plasma away from the inner quartz tubes to prevent them from melting. The smallest tube – known as the "injector" – carries the aerosol droplets from the spray chamber to the plasma at a flow rate of around 1 liter per minute. The injector tube typically has an internal diameter (ID) of around 2 mm or more, so the carrier gas travels at high speed, punching a hole through the plasma to form an elongated "torus" or donut shape. The aerosol droplets are carried through the center of the plasma, where the droplets are dried and the sample material is decomposed and dissociated into individual atoms, which are then ionized.

Almost all ICP-MS instruments use an RF generator operating at a frequency of 27.12 MHz (27.12 million cycles per second). This is lower than the standard 40.68 MHz frequency traditionally used for ICP-OES instruments. The lower frequency plasma is preferred for ICP-MS as it has a thicker "skin depth", which means the plasma energy extends closer to the central channel where the aerosol droplets are processed. This is important for the performance of an ICP used for MS, where the higher central channel temperature provides better ionization and so higher sensitivity.

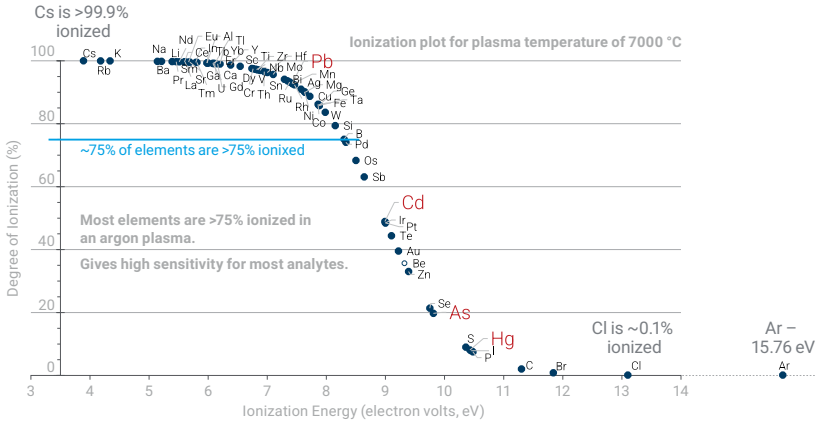
The plasma of an ICP-MS plays a critical role in decomposing the sample matrix, dissociating potential interferences, and ionizing the analyte atoms. Indeed, the overall performance of an ICP-MS is highly dependent on the plasma torch design and operating conditions. For a given carrier gas flow rate (e.g. 1 liter per minute), the linear gas velocity through the torch injector is inversely is proportional to the square of the injector ID. Changing from a torch with an injector ID of 1.5 mm to one with an injector ID of 2.5 mm therefore causes almost a 3-fold decrease in carrier gas velocity, from 9.43 m/s to 3.4 m/s. The plasma only has a few milliseconds to process the aerosol droplets to form ions, so slowing down the carrier gas flow dramatically improves these processes. A wider torch injector causes a lower carrier gas velocity, which means the aerosol droplets spend more time exposed to the hottest part of the plasma. This leads to better matrix decomposition, lower levels of spectral interferences, and higher ionization of analyte ions, all of which are critical parameters in most ICP-MS applications.



Processes to convert aerosol droplets to ions in the plasma.

Some ICP-MS applications use different torch designs. Options include a demountable torch with a platinum or sapphire injector tube for use with samples prepared in highly corrosive acids like HF. Torches with a narrow injector are also available and are typically used for analysis of volatile solvents and other specialized applications.

The degree of ionization of each element in the plasma depends on the element's ionization energy (or ionization potential, IP) and the plasma temperature. The IP is the amount of energy input that causes one electron to be removed from a neutral atom to create an ion. Argon is an ideal support gas for ICP-MS because it has a first IP that is above the first IP of most other elements, but below their second IP (the energy input required to remove two electrons). This property of argon means that most elements that pass through the ICP-MS plasma are converted efficiently to singly charged positive ions (M^+), with only a small percentage of doubly charged ions (M^{2+}) being formed. Under typical plasma conditions, most naturally occurring elements are at least 75% ionized, which is one of the main reasons for the very high sensitivity of ICP-MS. A few important elements are less well ionized, however, notably Zn, Be, As, Se, Cd, Hg, and some of the noble metals. Optimizing for a robust, high temperature plasma is important to achieve high sensitivity and low detection limits for these elements.

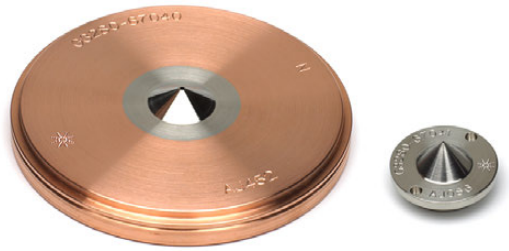


Plot showing degree of ionization (% of atoms converted to ions) plotted against first ionization potential for all elements.

3. The vacuum interface

The plasma ion source and the quadrupole mass spectrometer are separated by a vacuum interface, which transfers ions from the plasma (at atmospheric pressure) to the mass spectrometer (in a vacuum chamber). The interface consists of a series of cooled metal plates or "cones" with small holes in them to allow the ions to pass through. The typical arrangement uses a sampling cone to extract ions from the plasma, followed by a skimmer cone to sample the central portion of the ion beam to pass to the high vacuum region.

Small cone apertures – around 0.5 to 1.0 mm – are preferred as they provide optimum vacuum conditions for operation of the quadrupole mass filter and detector. Lower vacuum pressure (less residual gas) leads to higher ion transmission, less peak broadening due to scattering, and lower background. But small aperture cones are more difficult to manufacture precisely and lead to fewer ions being extracted from the plasma and passed through the interface. Smaller cone apertures would also be more susceptible to clogging if the sample matrix was not completely dissociated in the plasma. The benefits of an optimum interface cone geometry are only achieved in combination with optimized design of the other ICP-MS components, such as the plasma and ion lens.



An ICP-MS interface includes a sampling cone (left) and skimmer cone (right).

4. Ion focusing and separation

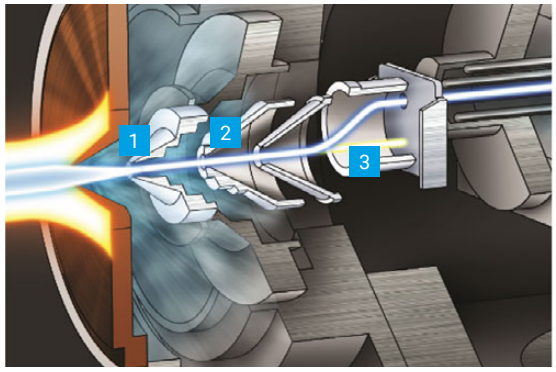
After the ions pass through the interface cones, they are focused into a narrow beam using an ion "lens". The lens is composed of several metal plates with adjustable voltages applied to them. A plate with a positive voltage repels the positively charged ions while a plate with a negative voltage attracts the ions. A combination of lens plates with different voltages is used to steer and focus the ions.

As well as focusing the ions to maximize transmission and therefore sensitivity, the ion lens also separates the ions from the neutral particles and photons extracted from the plasma. These uncharged particles would cause a high background signal, so they must be prevented from passing through the vacuum system and reaching the detector. This is usually achieved by deflecting the ions off axis, while the photons and neutrals, being uncharged, continue in a straight line and so are removed from the ion beam. Several different ion lens designs are used in ICP-MS, but all have the same goal of providing high transmission for ions of all masses, while minimizing background signals due to photons and neutral particles.

It is worth noting that a lower voltage, gentler deflection is preferred, as it will cause less variation in the angle of deflection for different masses. Variable deflection for different masses introduces mass bias (differences in transmission for different masses), which is an undesirable characteristic for a multielement analyzer. An ion lens that causes minimal mass bias will provide uniform ion transmission and therefore consistently high sensitivity and low detection limits for all analytes across the mass range.

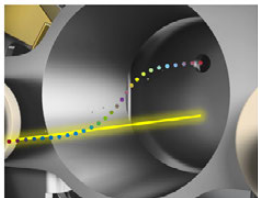
The ion lens

1. Extracts and guides the ions as they pass through the interface
2. Focuses all masses efficiently to provide good sensitivity across the mass range
3. Separates the ions from photons & neutrals



When beam is deflected at low voltage, transmission is high for all mases

Photons & neutrals removed so background is low

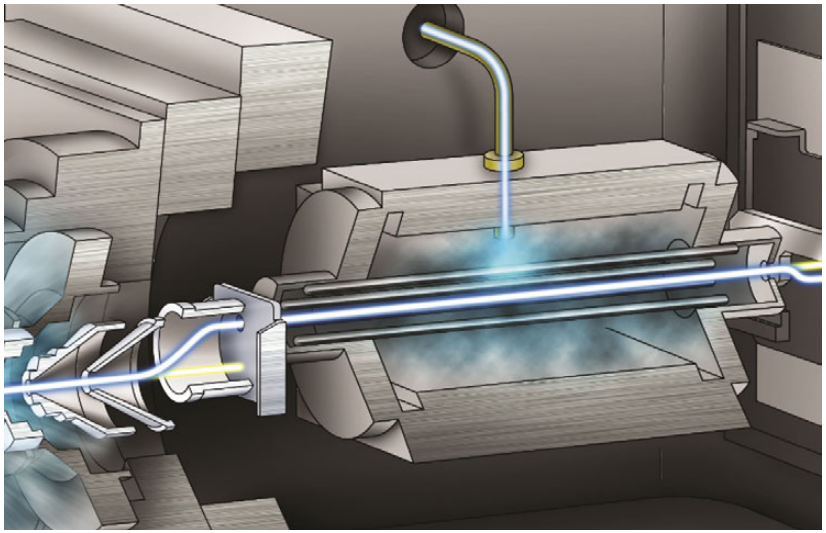


Cutaway showing position and operation of the ion lens.

5. Collision/reaction cell

Since the early 2000s, nearly all new ICP-MS systems have included a collision/reaction cell (CRC) to resolve spectral overlaps caused by unwanted ions that appear at the same mass as the analyte ions being measured. By far the most significant spectral overlaps in ICP-MS are caused by molecular (or "polyatomic") ions. A polyatomic ion overlap occurs when a combination of atoms forms an ion at the same mass as an analyte of interest. For example, ^{40}Ar can combine with ^{16}O to create an ArO^+ polyatomic ion at mass 56, overlapping the major isotope of iron (^{56}Fe). Similarly, ^{40}Ar can combine with ^{35}Cl to form an $ArCl^+$ polyatomic ion at mass 75, overlapping the only isotope of arsenic (^{75}As). Polyatomic ion overlaps have been a longstanding problem in ICP-MS, so the development of CRCs to address these overlaps was a major factor in improving data accuracy and increasing adoption of ICP-MS across many industries performing routine measurements.

A CRC consists of an ion guide – usually an octopole or quadrupole – positioned in an enclosed chamber (or "cell"), that can be pressurized with a gas. Small entrance and exit apertures maintain the cell pressure while allowing the ions to enter and exit the cell. The process used to remove the interferences depend on the gas (or gases) added to the cell and can be broadly divided into collision and reaction modes.



Cutaway showing position of the collision/reaction cell between the ion lens and mass filter of a single quadrupole ICP-MS. The cell can be pressurized with a collision or reaction gas to resolve interfering ions.

Collision mode

In collision mode, the CRC is pressurized with a nonreactive gas – usually helium (He). Ions passing through the CRC collide with the He atoms, losing a small amount of kinetic energy with each collision. Polyatomic ions are always larger than analyte ions of the same mass, so polyatomic ions collide more often than the analyte ions do. The different collision rates mean the polyatomic ions emerge from the cell exit with lower residual energy than the analyte ions. The low energy ions are rejected from the ion beam by a positive bias voltage "step" using a process called kinetic energy discrimination (KED).

Collision mode requires that all the ions enter the cell with a narrow ion energy spread. Also, the ion guide must minimize scattering losses to provide high ion transmission at the high cell gas pressures and high collision rates required for effective KED.

He KED can be considered as a universal CRC mode, as the same cell conditions can be used to resolve multiple polyatomic ion overlaps on multiple analyte masses in multiple different sample types.

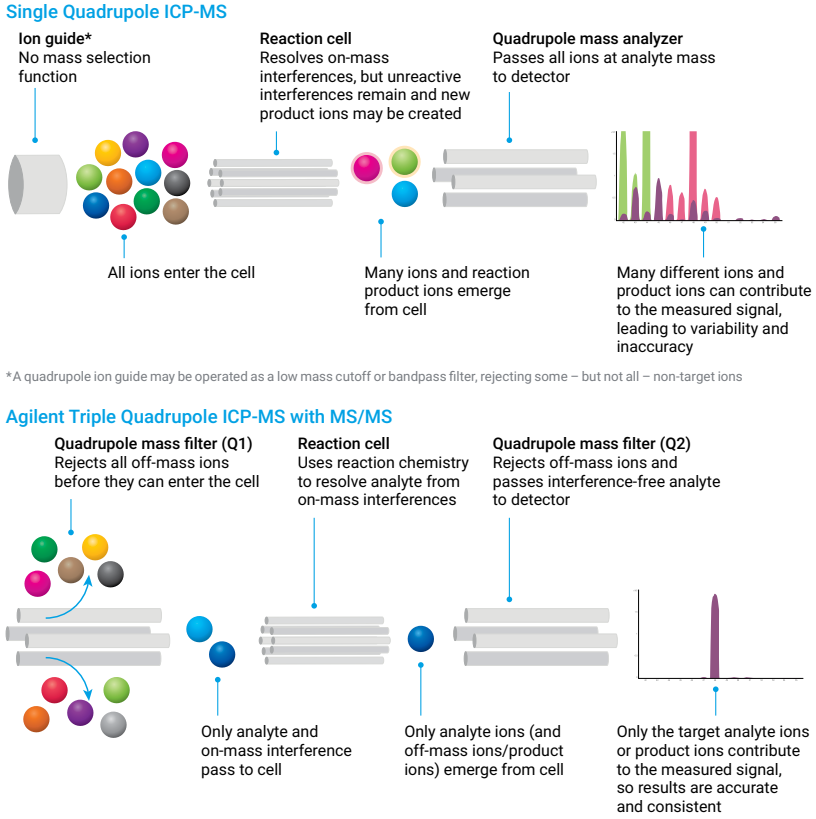
Reaction mode

Reaction mode uses the same CRC hardware as collision mode, but the cell is pressurized with a reaction gas such as H_2, O_2, NH_3, CH_4, N_2O, or CH_3F, rather than He. Reaction mode can be extremely efficient, as a reaction between the ion and the cell gas molecule often occurs within the first few collisions, whereas He KED requires multiple collisions and therefore higher cell gas pressure. Reaction gas methods are also more specific than He KED, because the gas must be selected based on its different reaction chemistry with the interfering ion and the analyte isotope. Analysts must therefore know which interfering ions they are trying to resolve before they select the reaction gas.

Reactive cell gases differ from He KED in that they can be used to resolve interferences other than polyatomic ions. These other spectral overlaps – such as isobaric overlaps, doubly charged ion overlaps, and adjacent mass peak-tail overlaps – are less common than polyatomic interferences but can still be significant.

The other major difference compared to He KED is that reactive cell gases can form new reaction product ions that may overlap a required analyte. The potential for new product ion interferences has meant that single quadrupole ICP-MS users tend to use reaction gas methods only when the sample matrix is consistent and the interferences are predictable. This limitation does not apply to triple quadrupole ICP-MS (ICP-QQQ) in MS/MS mode, however. The additional quadrupole mass filter before the CRC of an ICP-QQQ instrument means the composition of the ion beam entering the CRC is consistent even when the sample matrix varies. As a result, MS/MS controls the reaction chemistry and removes the possibility of cell-formed product ion overlaps being formed.

The way the additional mass filter (Q1) of a triple quadrupole ICP-MS controls the reaction chemistry in the CRC is illustrated in the schematics below.

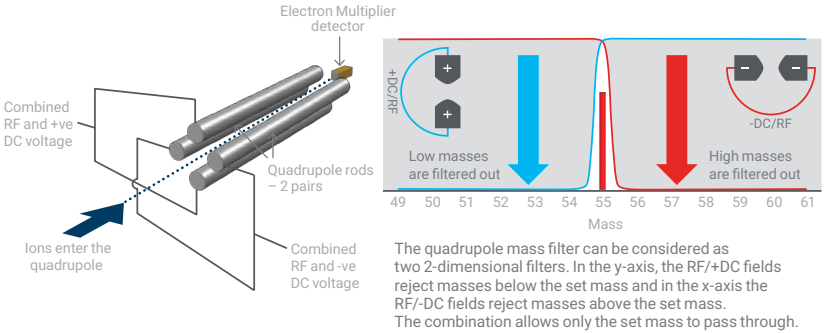


A schematic comparison of reaction mode on a single quadrupole ICP-MS (top) and a triple quadrupole ICP-MS (bottom)

6. The mass spectrometer (MS)

Most ICP-MS instruments use a quadrupole (or "quad") mass spectrometer to filter ions by mass, or, more accurately, by mass to charge ratio (*m/z*). Since most ions produced by the ICP are singly charged, *m/z* = mass, so ICP-MS can usually be considered to produce a "mass spectrum".

A quadrupole mass filter consists of two pairs of rods; opposite pairs of rods are connected to separate electrical supplies. An out of phase RF voltage and a positive or negative DC voltage is applied to the two pairs of rods (RF/+DC on the y-axis rods and RF/-DC on the x-axis rods). The electric field that is set up in the space between the rods determines the "set mass" of ions that can travel stably along the quadrupole axis and pass through the mass filter. The alternating electric fields destabilize the trajectories of all ions above and below the set mass, so ions at any mass other than the set mass are rejected from the ion beam.



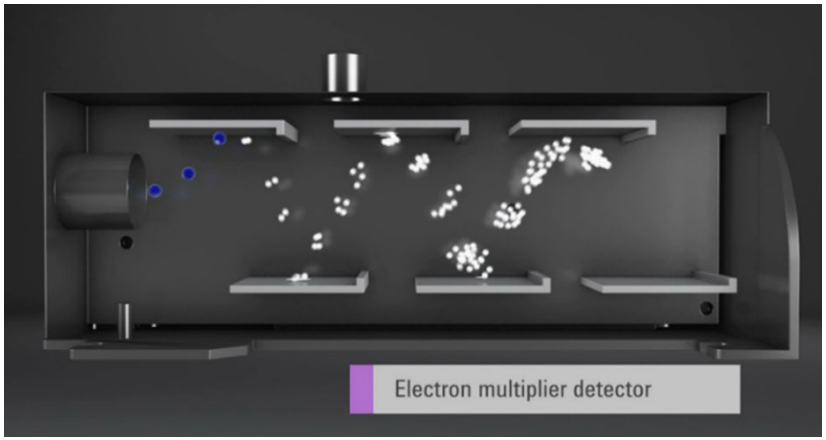
Quadrupole mass filter used in most ICP-MS instruments. RF and DC voltages filter the ions by mass to charge ratio (*m/z*), allowing only 1 *m/z* at a time to pass through to the detector.

The quadrupole set mass is controlled by the applied rod voltages, which can be changed very rapidly. This means the quadrupole can scan very rapidly across the mass range, for example from Li (mass 7) to U (mass 238) more than 10 times per second. With each quadrupole scan, the ions present at each mass are passed to the detector and counted.

In normal quantitative "spectrum" and isotope ratio analysis, the mass spectrum is built up from multiple scans across the selected masses. Other measurement modes include time resolved analysis (TRA), where the ion counts collected in each quadrupole scan are saved on a time-based scale. Single mass monitoring is also available and is typically used for measurements such as single nanoparticle analysis.

7. The electron multiplier detector

The detector used in most ICP-MS systems is a discrete dynode electron multiplier (DDEM). The EM uses a high voltage electrode (or dynode) positioned so that ions that emerge from the quadrupole strike the dynode. Each ion that strikes the first dynode releases one or more electrons from the dynode surface. These electrons strike the second dynode, releasing further electrons to strike the third dynode and so on down the detector. By the final dynode, the cascade of electrons has built up to a high enough level that it can be recorded as a pulse or "count" by the EM electronics. At very high ion count rates – above about 1 million counts per second (cps), the detector switches automatically to a low-gain (or "analog") mode. This allows intense signals from high concentration elements to be measured without overloading the detector.

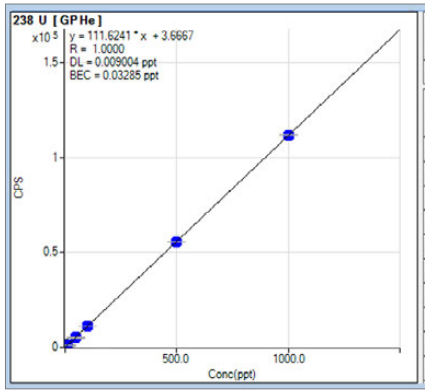


Electron multiplier (EM) detector showing build-up of electron cascade down the detector dynodes to generate an ion pulse or count.

The EM detector can detect individual ions, so ultralow concentrations can be detected. But the EM also has extremely wide dynamic range – around 10 or 11 orders of magnitude. In practical analytical terms, this means ICP-MS can detect a trace element like uranium at a concentration below 0.1 ppt (0.0000001 ppm) while also measuring a major element, such as sodium in seawater, at 1.18% (11,800 ppm). A total concentration range of more than 11 orders of magnitude, measured in the same acquisition.

8. Data processing

For each mass measured, the counts registered by the detector are processed by the data analysis software on the computer controlling the instrument. For conventional quantitative analysis, the software calculates the concentration of each element in the unknown samples by comparing the measured counts in the sample to the counts in a known-concentration reference solution. Usually several reference solutions or “standards” are measured to create a calibration plot of counts vs known concentration for each element.



Analyte Information (238 U [GP He])

Current Sample	Calc Conc.	CPS	Ratio	Det.	Con...
Blank	-0.003	3		P	-43

Calibration

Level	Rjct	Conc.	Calc Conc.	CPS	Ratio	Det.	RSD
1		0.000	0.000	4		P	9.1
2		0.100	0.107	16		P	15.
3		0.500	0.493	59		P	2.6
4		1.000	0.998	115		P	3.3
5		5.000	4.906	551		P	0.8
6		10.000	9.833	1101		P	2.9
7		50.000	45.715	5107		P	1.5
8		100.00	98.213	10967		P	0.3
9		500.00	497.168	55500		P	0.3
10		1000.0	1001.811	111830		P	0.7

A typical calibration plot for ICP-MS quantification of uranium using 10 standards prepared at concentrations between 0.1 and 1000 ng/L (ppt).

Other types of ICP-MS measurements include speciation analysis, which uses chromatographic data analysis tools such as peak integration, and single particle and single cell applications, where specialized data processing tools are used.

Find out more about the Agilent 7850 ICP-MS, 7900 ICP-MS and 8900 ICP-QQQ models, and see how they could transform your laboratory.



What Is ICP-MS Used For?

Labs that perform elemental analysis by ICP-MS are often in industries that analyze a lot of samples and measure a lot of elements in each sample. Examples of these industries include environmental monitoring (of river water, drinking water, estuarine and seawater, waste, soil and sediment digests, biota, etc.), routine measurements for food safety, pharmaceutical materials analysis, and consumer product testing (toys, textiles, etc.). Other industries where ICP-MS is widely used include semiconductor and pure chemicals manufacturing, life science, clinical research, geochemistry, mining, nuclear sciences, petrochemicals and renewable energy, materials research, metrology, and academia.

Liquid sample analysis

Liquid sample introduction is by far the most common route by which samples are introduced into an ICP-MS instrument, partly because liquid calibration standards are widely available and can easily be prepared with all the required analytes at the appropriate concentrations. Because liquid sample analysis is so convenient, solid samples are often converted to liquids using acid digestion to dissolve the sample matrix, or acid extraction to extract the analytes into a solution for analysis. Most routine ICP-MS applications use a conventional pneumatic nebulizer to introduce the liquid samples. Total concentrations of the elements of interest are measured against a calibration generated from the analysis of standards of known concentration.

When very large numbers of samples must be measured, labs will often employ a "discrete sampling" device. These devices work by loading the sample into a loop and then injecting the contents of the loop into a carrier stream flowing to the ICP. Discrete sampling speeds up ICP-MS analysis by enabling the next sample to be loaded into the loop while the previous sample is being measured. This allows faster overall ICP-MS analysis, reducing analysis time from a typical value of around 3 minutes per sample to less than 1 minute per sample for a triplicate multielement analysis.

ICP-MS applications include more unusual measurements where the relative abundances of the different isotopes of an element are of interest, as in isotope ratio or isotope dilution analysis. ICP-MS can also run a quick scan across the whole mass range, giving approximate or "semi-quantitative" results for all elements without needing specific calibration standards. This type of screening analysis is extremely useful for contaminant surveys of environmental or food samples, identifying the cause of heavy metal poisoning, and for fault finding defects in products.

In some applications, a normal "total" elemental concentration measurement does not give sufficient information. In these cases, separate analysis of the different chemical forms or "species" of an element may be required.



Speciation analysis

ICP-MS speciation analysis involves coupling the ICP-MS to some form of chromatography device to separate the different chemical forms of an element. The ICP-MS detects the elemental signal as normal, but the different chemical forms are introduced sequentially to the ICP-MS for separate detection and quantification. High performance liquid chromatography (HPLC, often just called "LC") is the most widely used separation technique, but ion chromatography (IC), gas chromatography (GC), capillary electrophoresis (CE), field flow fractionation (FFF), and hydrodynamic chromatography (HDC) are among the other separation techniques that have been coupled to ICP-MS. Examples of applications where speciation analysis is required include separating the different forms of chromium (Cr), where the trivalent form (Cr(III)) is harmless, but the

hexavalent form (Cr(VI)) is toxic *(1)*. Similarly, inorganic arsenic (As) is much more harmful than the common organic forms (such as arsenobetaine) found at high levels in some seafoods *(2)*. And food safety checks may require specific determination of the level of methyl mercury and organotin compounds in fish and shellfish, rather than just total mercury and total tin.

Speciation analysis with ICP-MS extends far beyond separating toxic and non-toxic forms of an element in environmental and food samples, though. Many industrial processes require assessment of the chemical form of the element, either to ensure the product performs as intended, or to identify contaminants that may affect the process or lead to unwanted emissions. Examples of these types of industrial speciation measurements include measuring contaminant compounds in metallodrugs, and monitoring mercury compounds in petrochemical refinery feedstocks to avoid catalyst poisoning and corrosion in the cracking process equipment. In semiconductor manufacturing, GC-ICP-MS is used to measure trace hydride gas contaminants in the arsine gas used as a precursor in non-silicon semiconductor device fabrication.

Nanoparticles and single cells

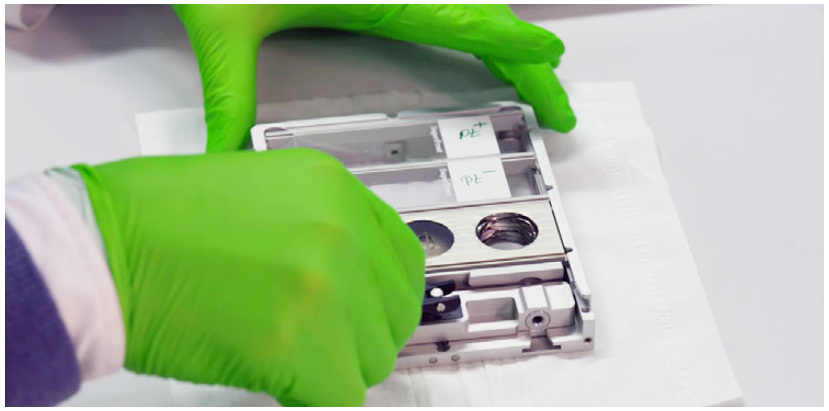
As well as measuring the amount of the elements dissolved in the sample solution, ICP-MS can also be used to measure the composition of tiny particles suspended in the liquid. Large (micron size) particles would be removed by the nebulizer and spray chamber of the ICP-MS, so cannot be measured. But very small (nano-scale) particles remain suspended in the aerosol droplets and are carried to the plasma. When these "nanoparticles" (NPs) are destroyed and atomized in the plasma, they give a "plume" of ions, which can be detected as a signal pulse above the constant background signal from the dissolved element. Nanoparticle analysis using single particle ICP-MS (spICP-MS) is of increasing interest as researchers strive to understand more about the impact of NPs on environmental and biological systems. NPs are also becoming more widely used in industrial processes, consumer products, paints, and coatings, and for applications such as drug delivery and agrochemicals. A similar analysis uses a special, low flow sample introduction system to deliver intact single cells to the plasma – single cell ICP-MS (scICP-MS). This approach allows the metal content of individual cells to be measured to aid understanding of biological and biopharmaceutical processes.

1. L. M. Calder, in: J. O. Nriagu and E. Nieboer, Eds., Chromium in the Natural and Human Environments, Wiley and Sons, New York, 1988, 215–229
2. European Food Safety Authority, Scientific opinion on arsenic in food, EFSA Journal, 2009, 7, 1351

Solid sample analysis

While liquid sampling dominates, solids can also be analyzed by ICP-MS using an appropriate accessory. A laser ablation (LA) device can be connected to an ICP-MS to perform direct solids analysis by LA-ICP-MS. LA-ICP-MS involves placing the sample into a chamber on a special mount, then focusing a high energy beam from a pulsed laser onto the sample surface. Solid particles ablated and vaporized from the sample surface are swept up with a gas stream (usually helium) and carried to the ICP torch, where they are decomposed, dissociated, atomized, and ionized in the same way as for normal aerosol droplets.

LA-ICP-MS is sometimes used for bulk (whole sample) analysis in applications such as quality control of metals, alloys, glasses, and ceramics where digesting the material might be difficult. But the real benefit of LA-ICP-MS is that the laser can be focused to a beam size of only a few microns, so analysis of small samples or a very tiny part of a larger sample is possible. This capability enables nearly non-destructive analysis of precious samples such as gemstones, archaeological artifacts, pottery, coins, paint fragments, and so on. A similar approach can be used for LA-ICP-MS analysis of airborne particulate analysis, where particles are collected on a sampling filter, providing composition information of individual particles for environmental and workplace dust analysis. LA-ICP-MS is also applied in criminalistics, the analysis of crime scene debris such as glass fragments from a road traffic accident, pen ink from a ransom note, fibers from a garment, soil from the sole of a shoe, gunshot residue, and many other such materials.



Loading solid samples into a sample holder for laser ablation, in this case, biological samples on microscope slides.

When combined with precise automated sample position control, LA-ICP-MS can be used for 2D or 3D mapping of the distribution of elements within a sample – often termed “imaging”. By continually scanning the ICP-MS while slowly moving the laser ablation “spot” across the sample surface, a chemical profile or map of the sample composition can be produced. LA-ICP-MS imaging is used in geological studies (geoimaging) and the same technique can be applied to biological materials, such as teeth, bones, and tissue sections (bioimaging).

Gas analysis

For direct analysis of elements in gases, a special gas handling device is used to introduce gases into the ICP. ICP-MS gas analysis applications include measuring volatile organometallic compounds in natural gas, headspace sampling for flavor and fragrance testing of ingredients and foodstuffs, and measuring trace contaminants in raw materials in chemical and petrochemical manufacturing. Many of these types of applications can be performed using GC-ICP-MS, where a gas chromatograph (GC) is connected to an ICP-MS instrument using a heated transfer line. The GC processes the sample using standard gas sampling accessories, and the ICP-MS detects and quantifies the compounds as they emerge from the GC column. GC-ICP-MS applications often have parallels in organic GC mass spectrometry (GC/MS) but, for compounds that contain a heteroelement that can be measured by ICP-MS, the ICP-MS detector enables lower detection limits, better specificity, and the potential for compound-independent calibration.

Direct analysis of gaseous samples is also becoming increasingly important in applications such as process control and workplace monitoring in semiconductor fabrication, where organometallic gases may be used in manufacturing processes. Methods have been developed that employ an ICP-MS instrument fitted with a gas-exchange device (GED) where compounds in the process gas are transferred by gas exchange into an argon stream, which is then passed to the ICP.



What Type of ICP-MS Will Suit Your Application? ICP-MS or ICP-QQQ?

Single quadrupole ICP-MS has a lower purchase price and slightly lower running costs than ICP-QQQ. ICP-QQQ requires a somewhat higher level of skill to setup and operate, although this depends on the methods being run. Many methods developed for a single quadrupole ICP-MS can be run without modification on ICP-QQQ. And methods that use reactive cell gases are much more consistent and reliable when run on ICP-QQQ.

Why ICP-QQQ?

ICP-QQQ has significantly higher sensitivity and much lower backgrounds than single quadrupole ICP-MS. This means ICP-QQQ provides generally around ten times lower detection limits for most elements.

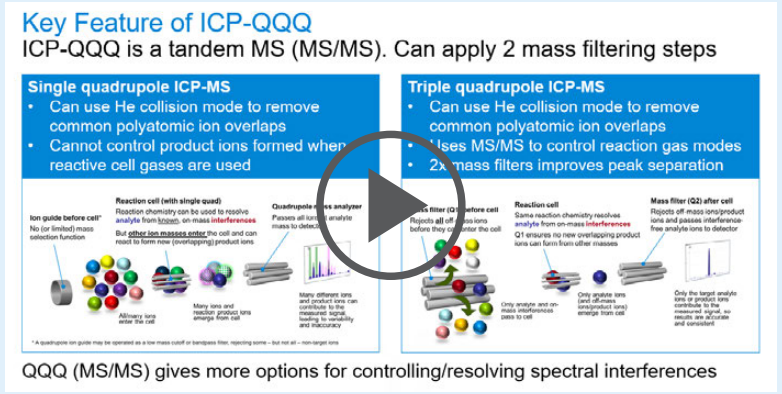
The double mass selection capability of ICP-MS/MS also offers superior separation of adjacent mass overlaps, enabling the technique to separate trace peaks from major peaks (trace Mn in an Fe matrix, B in a C matrix, and Np in a U matrix, for example).

However, the main advantage of ICP-QQQ is that the configuration supports MS/MS operation mode, where both quadrupole 1 and quadrupole 2 are operated as unit mass filters. MS/MS allows ICP-QQQ to use reactive cell gases to resolve spectral interferences that single quadrupole ICP-MS cannot address. Q1 selects the analyte ion mass that enters the cell, rejecting all other masses. This ensures that the ions in the cell are consistent and largely independent of the sample composition, so reactive cell gas methods can be used reliably to deliver consistent results for variable sample types.

Control of the reaction chemistry allows ICP-QQQ to resolve problematic interferences that cannot be addressed using conventional single quadrupole ICP-MS. These include resolving direct isobaric overlaps (such as ^{204}Hg on ^{204}Pb and $^{176}Yb/Lu$ on ^{176}Hf) and separating doubly charged ion overlaps (for example REE^{2+} overlaps on As and Se). Reactive cell gases can also be used to remove very intense background interferences. For example, the elements Si, P, and S suffer intense spectral interferences (N_2 on ^{28}Si, NOH on ^{31}P, and O_2 on ^{32}S) when measured with single quadrupole ICP-MS, so detection limits are relatively poor. An ICP-MS/MS method with a reactive cell gas can resolve these interferences, allowing these difficult elements to be measured at trace levels.

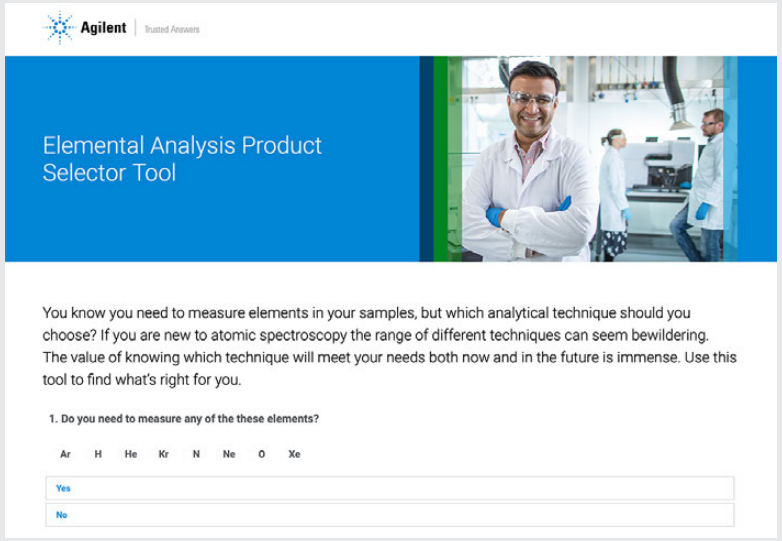
Teach me in 10

This short video from LabTube is a high level introduction to triple quadrupole ICP-MS (ICP-QQQ). It compares ICP-QQQ to single quadrupole ICP-MS and shows practical application benefits of ICP-QQQ.



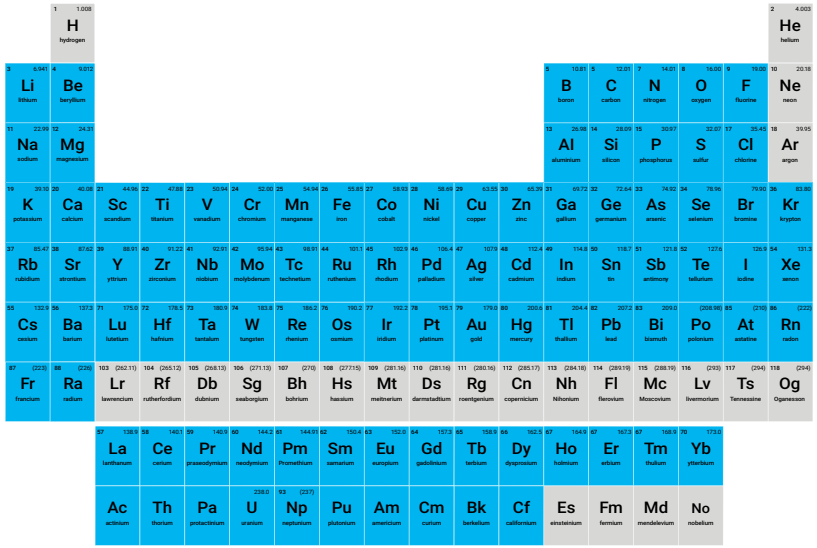
Not sure which elemental analysis technique will suit your needs?

Use our product selector tool to find out:
https://explore.agilent.com/atomic-portfolio-selector



Which elements can be measured?

ICP-MS and ICP-QQQ can measure virtually every naturally occurring element plus many non-natural "radiogenic" isotopes such as technetium, neptunium, plutonium, and americium. The only elements that can't be measured are H and He (which are below the mass range of the mass spectrometer), Ar, N, and O (which are present at high level from the plasma and air), and fluorine and neon (which can't be ionized in an argon plasma). The other noble gases, krypton and xenon, are technically measurable using a gas sampling device coupled to an ICP-MS or ICP-QQQ, but this is not a standard, routine application. Of the "impossible" elements, even fluorine can be analyzed indirectly using an ICP-QQQ – see sidebar.



An ICP-QQQ can measure all the elements colored in blue.

Measuring fluorine (F) with ICP-QQQ

F has a very high first ionization potential, much higher than that of argon, so F forms practically no positively charged F^+ ions in an argon plasma. Also, the background at mass 19 is very high, due to $^{18}O^1H^+$. It has been suggested that measuring F as its negative ion (F^-) might be a better solution, but the background from OH^- is also extremely high. However, F can be measured as a positive ion (like normal ICP-MS analytes) by using online addition of a barium solution to form BaF^+ ions in the plasma. If we add an excess of Ba (around 100 ppm), the BaF^+ formed will be proportional to the F concentration in the solution. So we can measure BaF^+ ions and use the signal to calibrate and quantify the F concentration in a sample.

The reason ICP -QQQ is needed for this analysis is that there are several other ions that overlap the BaF^+ ions at the mass used for the analysis ($^{138}Ba^{19}F^+$ is measured at mass 157). The main interference is from $BaOH^+$, but we can use a reaction gas method (with either O_2 cell gas or NH_3 cell gas) to resolve the BaF^+ ions from the $BaOH^+$ overlap. Using MS/MS (where both Q1 and Q2 of the ICP-QQQ are operated as mass filters) means that any other ions that might form new overlaps on the BaF^+ product ions are eliminated.

Which Applications Are ICP-MS and ICP-QQQ Ideal For?

Compared to the other atomic spectroscopy techniques (AAS and ICP-OES), ICP-MS and ICP-QQQ offer a unique combination of fast, multielement analysis (like ICP-OES) and low detection limits (like GFAAS). If your lab needs to measure ten or more elements at concentrations lower than 100 ppb and you have more than 50 samples per day to measure, then ICP-MS is a better option than the other atomic spectroscopy techniques. ICP-MS can measure high concentrations as well as trace levels, but, if only high concentrations are required and the number of elements and the number of samples is low, a single-element technique such as Flame AAS or MP-AES may be more cost effective.

Advantages of ICP-MS for metals analysis

ICP-MS offers:

- Fast measurement of almost every element in a 3-minute sample reading (1 minute per sample if you use a discrete sampling switching valve)
- High sensitivity and low detection limits (down to ng/L (ppt) or sub-ppt levels) for most elements. Elemental isotope ratios can also be measured
- The ability to measure trace and major elements (from concentrations of less than 0.1 ppt to more than 1000 ppm) in the same measurement
- The capability to avoid or overcome almost all spectral interferences
- The ability to measure a wide range of sample types, including organic solvents and solutions with dissolved solids levels up to 25%
- Connection to other techniques such as liquid or gas chromatography for chromatographic separations, or laser ablation (LA) for direct solid sampling
- Unattended operation, with the use of an autosampler

Single quadrupole ICP-MS applications

ICP-MS is ideal for those labs that:

- Run typical, routine or regulated ICP-MS methods, often analyzing common sample types such as environmental, food, or pharmaceuticals
- Run advanced applications (speciation, nanoparticles, laser ablation, etc) where the required detection limits and potential interferences are not unusually demanding
- Measure a typical range of trace elements, not including low level analysis of “difficult” elements such as Si, P, S, Cl, or F
- Do not need to measure elements at the lowest, ultra-trace (less than ppt) levels
- Do not need to resolve problematic spectral interferences such as isobaric overlaps, peak tail overlaps, and interferences that cannot be addressed using helium collision mode

Triple quadrupole ICP-MS applications

ICP-QQQ is ideal for those labs that:

- Run demanding applications–such as complex, high matrix materials–where intense or difficult to overcome interferences need to be resolved
- Measure ultra-trace levels (single- or sub-ppt) of elements in high purity materials or semiconductor process chemicals
- Need to analyze trace levels of difficult elements such as Si, P, S, Cl, or F
- Need to resolve direct isobaric (elemental) overlaps, such as $^{204}Pb/^{204}Hg$, $^{87}Sr/^{87}Rb$, or $^{176}Hf/^{176}Lu$, or overlaps on radiogenic isotopes, as needed in some geochemical or nuclear applications
- Need to measure low concentrations of elements at the mass next to a major element, for example B in a carbon matrix, Mn in steel, Np in uranium
- Perform research or metrology applications that require analysis of the broadest range of sample types and elements, now or in the future

Side by side comparison: ICP-MS vs ICP-QQQ

The following table shows the Agilent ICP-MS and ICP-QQQ instruments compare, relative to each other.

	ICP-MS	ICP-QQQ
Relative Price	$ $ $ $	$ $ $ $ $
Relative Cost per Sample	$ $ $	$ $ $
Relative Sensitivity		
Reviews	SelectScience	SelectScience
Maximum samples per Day	400 (1200)[1] (50 elements)	400 (1200)[1] (50 elements)
Dynamic Range of Measurement	<1 ppt to 1000 ppm	<1 ppt to 1000 ppm
Relative Sample Volume Required		
Relative Tolerance of Solids in Sample		
Element Measurement	Simultaneous	Simultaneous
How Many Elements Can Be Measured?	86	87 (including F)
Relative Routine Maintenance Requirements		
Relative Operator Skill Required	[2]	[2]
Can Be Left Unattended	Yes	Yes
Part 11/Annex 11 GMP Compliance	Yes (with optional software)	Yes (with optional software)

	ICP-MS	ICP-QQQ
Relative Operational Power Use		
Dimensions (mm – width x depth x height)	730 x 600 x 595	1060 x 600 x 595
Weight	100 kg	139 kg
Gas Requirements	99.99% Argon	99.99% Argon[4]
Warranty[3]	12 months	12 months
Accessories		
Autosampler	Optional	Optional
Water Cooling System	Required, not included	Required, not included

1. The higher sample number indicates throughput for an instrument fitted with a switching valve
2. A simplified interface (ICP Go) and pre-set methods are available for routine analysis, which would significantly reduce the operator skill level required
3. Agilent has various extended warranty and support options
4. For applications where ultratrace contamination must be controlled, for example measuring impurities in semiconductor chemicals, argon purity of at least 99.999% may be required

Application Examples

Multielement analysis of drinking water samples

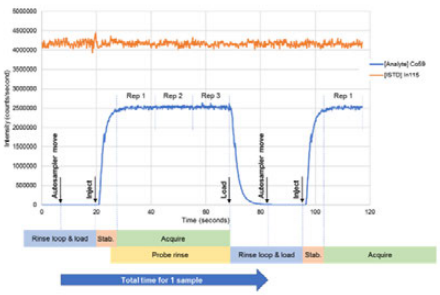
Routine drinking water analysis can be performed on several different atomic spectroscopy techniques, but ICP-MS is the only technique that allows all the major and trace elements (including As, Cd, Hg, and Pb) to be analyzed in combination with high sample throughput.

This application scenario typically involves:

- Routine monitoring of around 100 samples per day, but can be much higher in a busy water lab
- Analyze up to 35 elements in each sample
- Concentration levels vary from minerals (ppm) to traces (0.1 ppb)
- Water labs usually have very high productivity requirements

Why use ICP-MS?

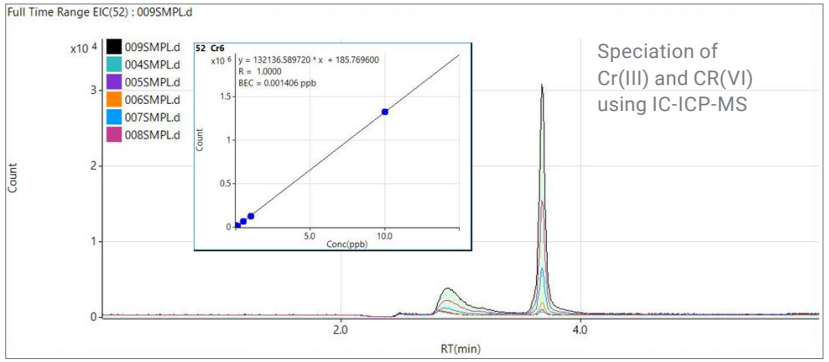
ICP-MS can routinely analyze large numbers of drinking water samples, handling up to 1200 samples per day if fitted with a switching valve.





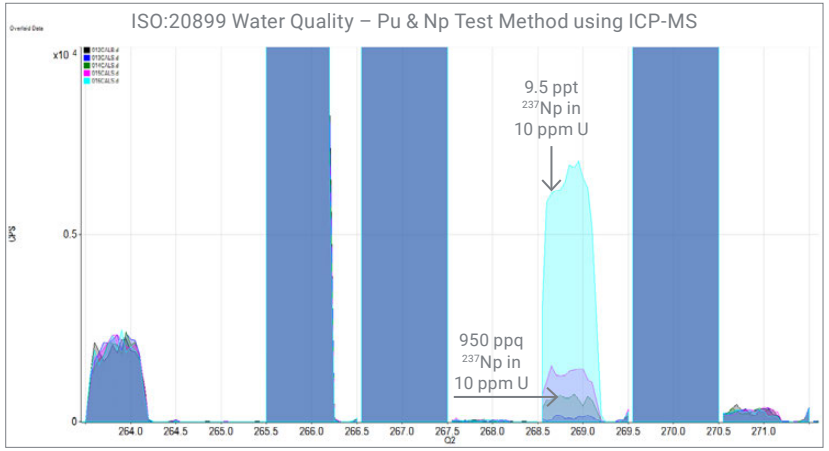
A switching valve gives multi-element analysis in <1 minute

ICP-MS can measure all the regulated major and trace elements that would otherwise need multiple techniques (ICP-OES for majors, and GFAAS, hydride generation, and atomic fluorescence for the traces). ICP-MS is also future-proof as it can measure low levels of emerging contaminants that may be added to future regulations. And ICP-MS can perform speciation analysis – for example to quantify inorganic arsenic, hexavalent chromium, and methyl mercury – and detect nanoparticles, which are an increasing focus of regulatory attention.



Advantages of ICP-QQQ?

- Most routine water quality monitoring labs cannot justify the extra cost of ICP-QQQ. But labs that have to measure difficult water samples – such as coastal and open ocean seawater, or highly saline groundwater – may benefit from the lower detection limits and better control of interferences that ICP-QQQ provides
- ICP-QQQ has the capability to measure emerging contaminants such as Pu and Np, as specified in ISO 20899:2018, for example. These trace contaminants are levels too low to be measured accurately using single quadrupole ICP-MS
- ICP-QQQ provides higher sensitivity and lower background, which are critical factors in measuring the smallest nanoparticles



Multielement analysis of food samples

Food analysis takes many forms, from routine certification of nutrient content to ensuring food safety by quantifying toxic trace elements and pesticide residues. Food producers and regulators also use trace element fingerprinting for food provenance.

The routine food analysis application scenario typically involves:

- The lab analyzes between 10 and 100 samples each day
- They analyze four nutrient elements, plus a suite of trace elements that varies depending on food type
- Detection and quantification limits are from single ppb to 1000s ppm level

Why use ICP-MS?

ICP-MS has the unique capability to measure all required elements across a very wide concentration range, so it can measure major and trace nutrients and toxic heavy metals in one fast analysis.

Element	Instant Non-Fat Dry Powdered Milk	Buttermilk Powder Milk	Whole Powdered Goat Milk	Sweet Cream Buttermilk Powder	Non-Fat Dry Milk Powder	Whole Milk	Fat Free Milk
11 B	3053	2931	2337	2490	2182	443.5	406.4
23 Na	3919	3870	2804	4335	3871	371	378
24 Mg	1152	1017	1094	931	1091	106	107
31 P	10215	90965	8221	8161	9845	965	985
39 K	15827	30554	15912	14590	17195	1519	1560
44 Ca	11762	9963	8486	7950	11077	1104	1123
47 Ti	442.5	221.2	201.3	256.3	253.2	<DL	<DL
51 V	24.40	25.55	27.95	28.97	15.29	9.51	9.70
52 Cr	<DL	<DL	<DL	712.26	<DL	<DL	<DL
55 Mn	170.1	220.3	292.9	193.7	219.2	20.20	19.86
56 Fe	1836	6468	2401	9760	1935	285.0	306.7
59 Co	5.90	5.75	<DL	8.45	<DL	<DL	<DL
63 Cu	308.1	609.5	899.6	485.8	391.7	35.94	66.12
66 Zn	41	30	27	28	41	4.0	4.0
75 As	<DL	<DL	<DL	<DL	<DL	<DL	<DL
78 Se	365.8	349.8	188.0	418.3	526.4	31.15	28.91
95 Mo	319.67	1946	104.73	2440	299.1	39.92	34.33
111 Cd	<DL	<DL	<DL	<DL	<DL	<DL	<DL
118 Sn	3.98	18.23	12.56	5.59	12.36	<DL	2.89
121 Sb	<DL	<DL	<DL	<DL	<DL	<DL	<DL
137 Ba	862.0	970.9	523.7	498.7	694.9	77.87	85.69
202 Hg	<DL	<DL	<DL	<DL	<DL	<DL	<DL
205 Tl	3.49	2.34	4.87	2.46	2.57	0.58	0.49
208 Pb	2.39	3.39	3.85	3.03	1.96	0.61	1.27

Concentrations in ppb except Na, Mg, P, K, Ca, and Zn (ppm)

ICP-MS can measure the 4 major nutrient elements: **Na, Mg, K, and Ca**

ICP-MS can also measure other (trace) elements: **P, Fe, Cu, Zn, Se, etc**

And ICP-MS can monitor toxic contaminants: **Cr, As, Cd, Hg, and Pb**

Plus, ICP-MS method can be extended to almost every other element.

Advantages of ICP-QQQ?

- As with routine water analysis, the benefits of ICP-QQQ center on the lower detection limits and the ability to provide more reliable trace element analysis in complex and varied samples
- Food samples are also subject to regulations that are changing to reflect better understanding of the harmful effects of some contaminants. ICP-QQQ provides lower detection limits for novel contaminants such as americium, which has an FDA intervention level of 15.8 ppq. ICP-QQQ also supports low level analysis of potentially harmful organometallic species, such as methyl mercury in seafood
- ICP-QQQ enables analysts to detect and quantify lower levels and smaller particle sizes for nanoparticles based on elements such as titanium (E 171 TiO_2 food additive)

Multielement analysis of mineral exploration samples

Labs that support mineral exploration need to quickly characterize very large numbers of samples collected from surveys and test drill cores. Wide elemental coverage and high productivity are essential.

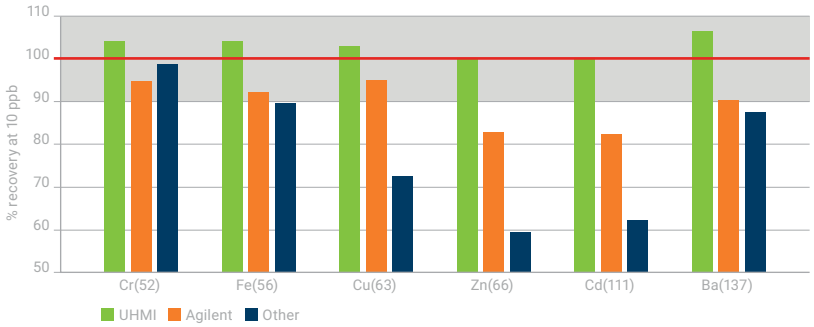
This application scenario typically involves:

- Exploration labs may have thousands of samples to measure each day
- Often would like to measure "all" (50+) elements in each sample
- Concentrations are unknown and very variable, but levels range from majors (%) to ppb

Why use ICP-MS?

An ICP-MS can routinely analyze large numbers of high matrix ore and mineral samples prepared in 30% aqua regia (3:1 HCl:HNO_3). An existing, standard ICP-OES sample preparation can be used. No need to develop new digestion methods or use high dilutions to run samples on ICP-MS

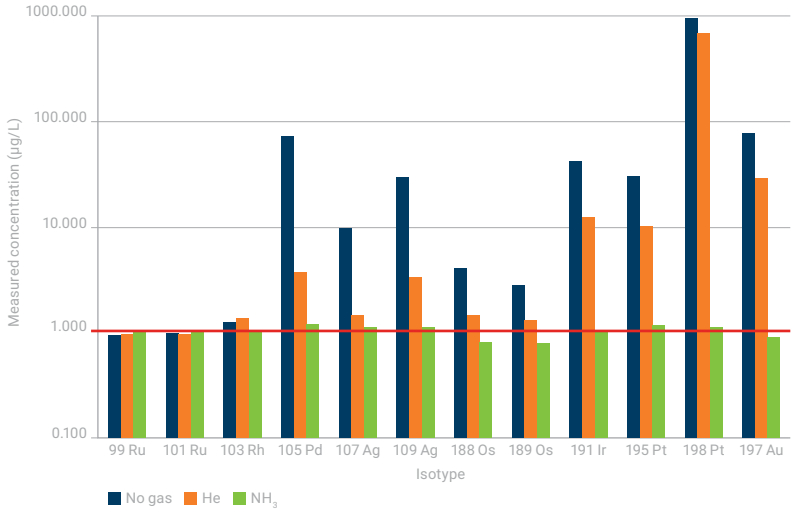
- Labs can use an ultra high matrix introduction (UHMI) aerosol dilution system to directly analyze high matrix samples without needing offline or online liquid dilution
- ICP-MS adds value by quantifying to lower levels and reporting a wider range of analytes
- Very robust UHMI conditions also ensure consistent accurate recoveries in varied high matrix samples, simplifying calibration and QC
- The wide dynamic range of an ICP-MS allows major elements Na, Mg, Al, K, Ca, Fe, Ti, etc to be measured in same run as trace elements, REEs, PGEs, TCEs etc
- Using helium in the collision/reaction cell resolves common matrix interferences, providing accurate results for typical analytes



Spike recovery in high salt matrix (shaded area is ± 10% range) using a single quadrupole ICP-MS. UHMI aerosol dilution gives consistent results in high matrix levels.

Advantages of ICP-QQQ?

- Using an ICP-QQQ allows even lower detection limits and fewer errors
- Matrix overlaps can give errors on single quadrupole ICP-MS, which can cause the inaccurate reporting of low concentration elements such as precious metals
- Using a reaction cell gas method on an ICP-QQQ can improve data quality by resolving spectral overlaps on critical analytes



Accurate recovery of a 1 µg/L spike of platinum group elements using NH_3 in the reaction cell of an ICP-QQQ (green). ICP-MS/MS method removes matrix interferences that give errors in single quadrupole ICP-MS methods using either no gas (blue) or helium collision gas (orange). The sample matrix included Sc, Ni, Cu, Zn, Rb, Sr, Y, Mo, Zr, Nb, Ta, Hf, W, and the rare earth elements at 10 ppm each, and 1 ppm of Hg. (*3*)

3. Demar D., Jean-Soro L., Desprez, A., Analysis of Platinum Group Elements (PGEs), Silver, and Gold in Roadside Dust using Triple Quadrupole ICP-MS, Agilent publication, 2022, 5991-6768EN.

Analysis of trace and ultratrace contaminants in high purity chemicals

High purity chemicals, such as those used in semiconductor wafer fabrication or other electronic manufacturing processes must have very low levels of metal impurities.

This application scenario typically involves:

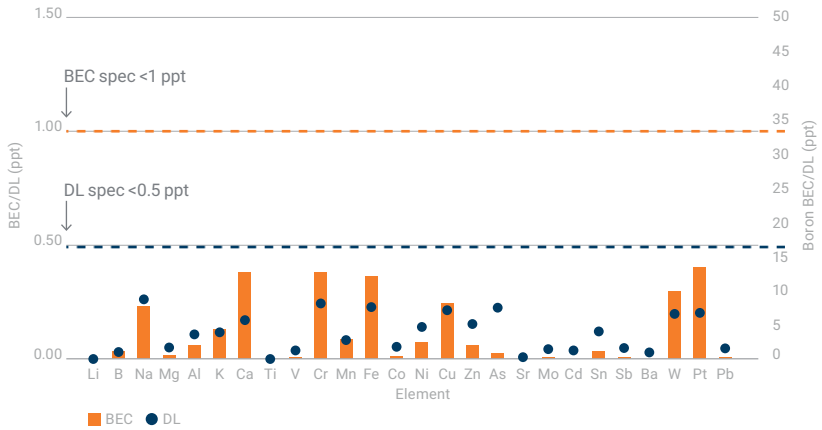
- Variable number of samples each day, but can be up to 50
- Around 20 elements are typically measured, with a range of high purity sample types
- Concentrations are typically single ppb but can be down to single- or sub-ppt

The low detection limits of ICP-MS are necessary for this industry, however single quadrupole ICP-MS may not be able to meet the sub-ppt levels required by some applications.

Why use an ICP-QQQ?

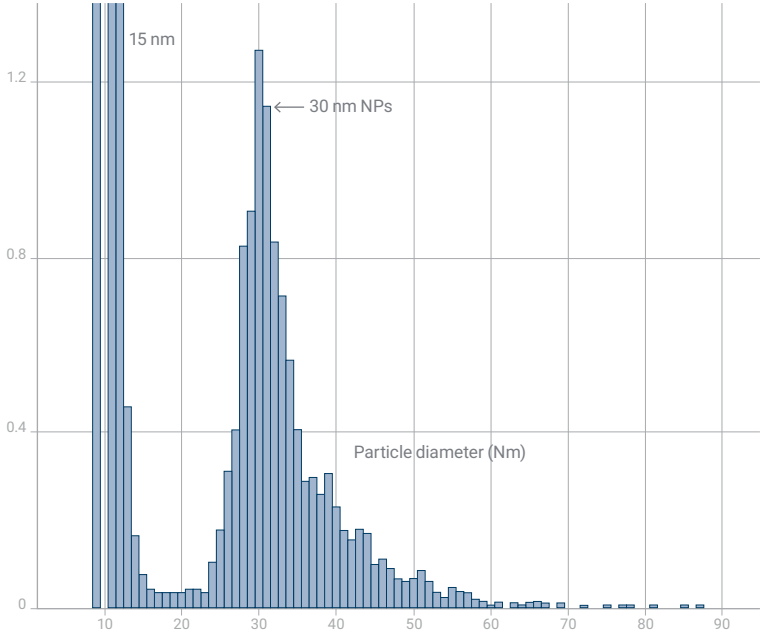
Using MS/MS mode and reactive cell gas methods, ICP-QQQ is able to resolve spectral overlaps and produce even lower detection limits and better accuracy than single quadrupole ICP-MS.

For example, ultrapure water used in electronics manufacturing often has a specification of <1 ppt contaminants (<50 ppt for boron) and requires detection limits of <0.5 ppt (<15 ppt for boron). An ICP-QQQ can easily meet these requirements.

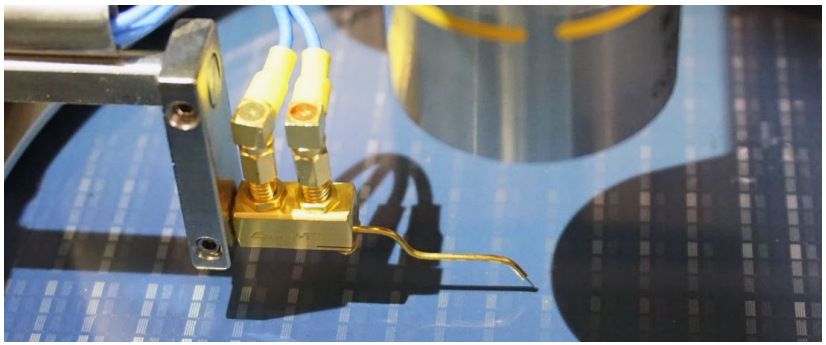


The Background equivalent concentration (BEC) measured using ICP-QQQ is well below the electronics manufacturing ultrapure water specification of <1 ppt. The detection limits of an ICP-QQQ are also well below the specified <0.5 ppt.

- ICP-QQQ improves data quality for interfered elements such as Ti and Zn in H_2SO_4, V in HCl, and Cr in organic solvents
- ICP-QQQ can measure nanoparticles including SiO_2, Fe_3O_4, which are difficult to measure with single quadrupole ICP-MS
- ICP-QQQ has higher sensitivity and lower backgrounds than single quadrupole ICP-MS, which improves analysis of particulate contaminants



The distribution of different sizes of iron nanoparticles in high purity butyl acetate, measured using an ICP-QQQ.



Using the MS/MS mode of an ICP-QQQ measures smaller nanoparticles than single quadrupole ICP-MS in semiconductor manufacturing chemicals, solvents, CMP slurries, etc

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© Agilent Technologies, Inc. 2022
Published in the USA, May 4, 2022
5994-4830EN

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