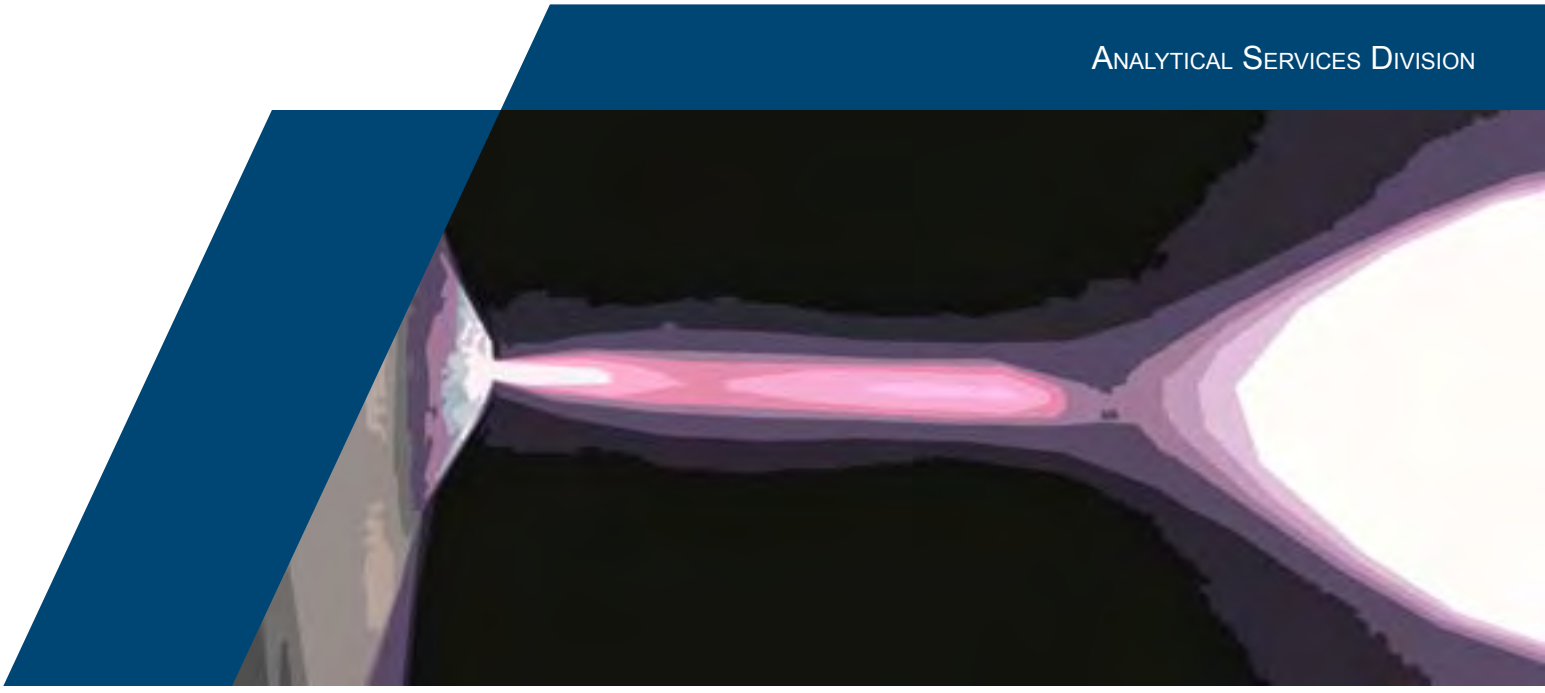
Advanced Analytical Services for the Determination of

Trace Metals by ICP-MS and Dynamic Reaction Cell Technology

Analytical Services Division



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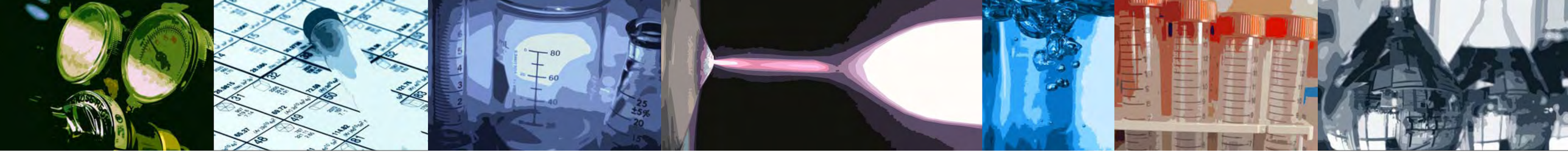
MEANINGFUL METALS DATA



Inductively coupled plasma – mass spectrometry (ICP-MS) is widely recognized as one of the most accurate and precise analytical techniques for the determination of many trace metals in a wide variety of sample types. However, analyses for a small number of these elements using a conventional instrument configuration have also been persistently challenging due to spectral interferences that can severely compromise the accuracy of reported results.

These spectral interferences occur due to the formation of polyatomic ions that have the same nominal mass as the analytes of interest and that cause erroneously large signals for the intended analyte at the detector, resulting in unnecessarily elevated detection limits and mistakenly elevated final concentrations.

Polyatomic ions form when the various constituents of the plasma gas, preparation reagents, and the sample matrix combine in the ion beam and prior to reaching the detector. Procedurally introduced elements, such as the argon that fuels the plasma, as well as the chlorine, hydrogen, nitrogen, and oxygen found in the diluents and acids used in sample preparation, unavoidably contribute to the formation of many of these polyatomic ions, even in relatively low level samples.

Isotope	Abundance	Polyatomic Ions
^{75}As	100%	$^{40}Ar^{35}C^+$, $^{38}Ar^{37}Cl^+$, $^{43}Ca^{16}O_2$
^{52}Cr	84%	$^{40}Ar^{12}C^+$, $^{36}Ar^{16}O^+$, $^{35}Cl^{16}O^1H^+$
^{53}Cr	10%	$^{37}Cl^{16}O^+$, $^{38}Ar^{15}N^+$, $^{38}Ar^{14}N^1H^+$
^{56}Fe	92%	$^{40}Ar^{16}O^+$, $^{40}Ca^{16}O^+$, $^{40}Ar^{15}N^1H^+$
^{58}Ni	68%	$^{23}Na^{35}Cl^+$, $^{40}Ar^{18}O^+$, $^{40}Ca^{18}O^+$
^{60}Ni	26%	$^{44}Ca^{16}O^+$, $^{23}Na^{37}Cl^+$, $^{43}Ca^{16}O^1H^+$
^{76}Se	9%	$^{40}Ar^{36}Ar^+$, $^{38}Ar^{38}Ar^+$
^{78}Se	24%	$^{40}Ar^{38}Ar^+$, $^{38}Ar^{40}Ca^+$
^{80}Se	50%	$^{40}Ar_2^+$, $^{32}S^{16}O_3^+$
^{82}Se	9%	$^{12}C^{35}Cl_2^+$, $^{34}S^{16}O_3^+$, $^{40}Ar_2^1H_2^+$
^{51}V	100%	$^{34}S^{16}O^1H^+$, $^{35}Cl^{16}O^+$, $^{38}Ar^{13}C^+$

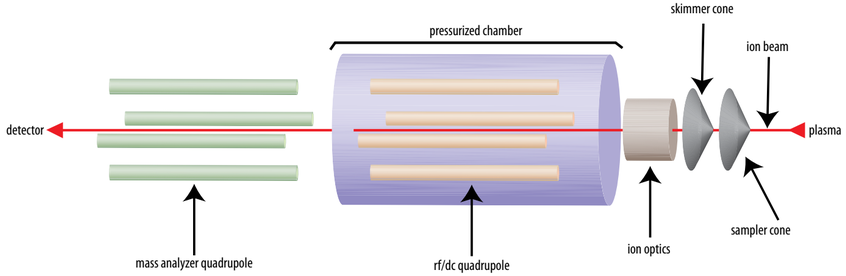
The analyses of samples that contain abundant amounts of particular elements, such as calcium, carbon, chlorine, magnesium, potassium, sodium, or sulfur, can dramatically further increase the formation of polyatomic ions to levels that can easily exceed even those of the analytes of interest.

In some instances, less abundant isotopes that may not be affected as severely by these interferences can be targeted for detection, but not without the potential for a significant loss in analytical sensitivity. Moreover, the effects of these interferences are not readily apparent by reviewing the results of typical quality control analyses, such as the recoveries of matrix spikes, since the interferences can contribute to each of the analyses equally.

Numerous elements, especially arsenic, chromium, iron, nickel, selenium, and vanadium, are known to be frequently affected by these spectral interferences, with just a few of the common polyatomic ions listed in the adjacent table.

Without an analytical technique to eliminate these interferences, costly decisions might be made based on inaccurate data.

A relatively recent advance for overcoming the challenges associated with these interferences, PerkinElmer's Dynamic Reaction Cell™ (DRC) improves significantly upon conventional analysis by ICP-MS. Located between the ion optics and the mass analyzer vacuum chambers of an ICP-MS instrument, the DRC is an enclosed and positively pressurized chamber with an independent quadrupole that can be configured to nearly eliminate polyatomic spectral interferences.



In a process described as chemical resolution, a highly reactive gas is injected into the DRC chamber, resulting in gas-phase chemical reactions that can either breakdown polyatomic ions into neutral species or convert the analyte of interest to a different ionic species with a different nominal mass. The reaction gas and gas flow rate ultimately selected are based upon the predictability of controlled and specific reactions.

The ion beam maintained by the frequency of the quadrupole guides ions through to the detector; non-ionized atoms and molecules will not persist in the ion beam or proceed to the detector. Using the appropriate reaction gas, polyatomic ions entering the DRC chamber will effectively transfer their charge to the molecules of the gas and then fall out of the ion beam.

According to the specifics of thermodynamic reaction kinetics, charge transfer reactions between ions and reaction gas molecules will occur if the ionization potential of the reaction gas molecule is significantly less than that of the ion. Conversely, if the ionization potential of the ion is less than that of the reaction gas molecules, a charge transfer will not occur. Ammonia is used as one reaction gas since it has an ionization potential that is intermediate between those of many analyte elements and most polyatomic ions, making it highly effective at chemical resolution. Examples of this process can also be expressed thusly:

$$Ar^+ + NH_3 \rightarrow Ar + NH_3^+$$

$$ArO^+ + NH_3 \rightarrow Ar + O + NH_3^+$$

$$Fe^+ + NH_3 \not\rightarrow Fe + NH_3^+$$

For elements that have ionization potentials similar to those of the interfering polyatomic ions, an alternate technique can be employed to deliberately form an ionic oxide species with the analyte by using oxygen as the reaction gas, resulting in a species with a mass unaffected by the polyatomic ions. An example this process can be expressed thusly:

$$As^+ + O \rightarrow AsO^+$$

The byproducts of the chemical resolution process may however maintain the potential for secondary reactions and form further polyatomic interferences if they are not effectively removed from the chamber. In addition to focusing the analyte ions through the chamber, the electrical fields of the DRC quadrupole can be optimized to behave as a selective bandpass mass filter.

In a process described as dynamic bandpass tuning, the amplitude and frequency of the DRC quadrupole are adjusted to establish precisely defined stability regions that act as a low- or high-mass cut off. Ions that do not fall within this window will be ejected from the chamber, preventing the secondary reactions that can lead to new interferences.

By expert configuration of the DRC with the appropriate reaction gas for chemical resolution and precise dynamic bandpass tuning, polyatomic spectral interferences are largely eliminated and optimal transmission of the analytes of interest into the mass analyzer quadrupole for conventional mass separation and detection can occur.

Brooks Rand Labs has developed numerous matrix and analyte specific methods employing the DRC to produce some of the lowest detection limits and most accurate data available.

To learn more about our innovative analytical methods and how they can benefit your projects, contact us today.

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Hydrofluoric Acid Digestions

Most digestion procedures for metals in sediment samples prescribed by the EPA and followed by routine environmental laboratories use mineral acids, such as nitric and hydrochloric, to extract the metals that tend to be relatively soluble, and therefore potentially bioavailable in the environment. Such digestions do not necessarily achieve complete dissolution of all of the metals, since some mineral- or chemically-bound metals are resistant to dissolution by these acids. These soluble fractions are typically described as the “total recoverable metals” concentrations.

Occasionally, a more comprehensive evaluation is required and the true “total metals” concentrations of solid samples must be determined. The soils and sediments surrounding decommissioned military bases, legacy mining sites, and former chemical manufacturing plants may have extensive metals contamination that are bound up in forms of complex chemical compounds. Industrial feedstock and byproducts are subjected to conditions far more extreme than environmental samples, and often must be tested to evaluate their contribution to the metals concentrations in related waste streams.

Brooks Rand Labs has extensive experience offering a more strenuous digestion process for the determination of true “total metals” in solid samples using a high-temperature, high-pressure extraction with hydrofluoric acid, which can dissolve even the most tightly bound metals.

Microwave Digestions

The reliability of typical digestion procedures for the determination of trace metals in a variety of sample types is usually demonstrated by the analytical recoveries of expected values for individual analytes from various certified reference materials (CRM). However, the availability of CRM types and the range of expected values for specific analytes is limited, and they do not always match as closely as might be desired for unique sample types or projects.

Therefore, projects that demand exceptional quality assurance often design digestion procedures particular to specific sample types and program objectives. Employing the latest in microwave digestion technology, Brooks Rand Labs offers the capability to design and perform highly customized digestion procedures using the optimal reagents, temperature, and pressure, over nearly any time scheme.

Moreover, our ultra-clean system allows for the constant optical monitoring and reporting of temperature and pressure levels for each individual sample throughout the digestion process, alleviating the need to rely solely upon CRM recoveries. Highly reproducible and dependable reactions permit for a level of certainty unavailable by other techniques. We have developed numerous matrix and project specific microwave digestion procedures to accommodate even the most demanding quality assurance objectives.

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About Brooks Rand Labs

Specializing in superior quality trace metals analytical services, Brooks Rand Labs focuses on metals speciation, trace level detection limits, and the analysis of complex matrices.

Experience & Expertise

Since 1982, Brooks Rand Labs has specialized in providing the highest quality trace metals analytical services and instrumentation. Low detection limits, outstanding data quality, and unparalleled customer service have established Brooks Rand Labs as the premier specialty metals analytical laboratory services provider.

As experts in the determination and characterization of trace metals in complex matrices, we have the capabilities to meet even the most challenging project requirements. Ultra-clean, pre-tested sampling equipment delivered directly, fast turn-around-time options, and custom reporting packages at competitive prices ensure that our clients receive the quality data they require to make critical decisions.

Ultra-low Detection and Reporting Limits

Brooks Rand Labs consistently strives to achieve detection and reporting limits that are among the lowest commercially available. Performance based evaluations of laboratory techniques ensure the most accurate and precise measurements at even the lowest concentrations. Ultra-clean facilities, laboratory equipment, and reagents, custom designed state-of-the-art instrumentation, and constantly improving methods allow Brooks Rand Labs to provide meaningful metals concentrations data in even the most challenging matrices.

Outstanding Data Quality

As an ultra-low level trace metals analytical laboratory, Brooks Rand Labs appreciates how important high quality data is for our clients. All our data is subjected to a rigorous multi-level review process to ensure only the most credible and scientifically defensible data is provided to our clients

The accuracy and precision of our data are constantly proven by internal and client-requested laboratory audits; consequently, Brooks Rand Labs is proudly NELAP accredited through the State of Florida Dept. of Health and certified in many additional states.

Unparalleled Customer Service

The project management team at Brooks Rand Labs provides clients with an unparalleled level of support from initial project planning, to data validation and interpretation, and project follow-up. They review quality assurance and sampling plans, advise on sampling procedures, and produce custom reporting packages that are superior to other analytical laboratories.