

## **Low Level Detection of Priority Inorganic Pollutants in Groundwater**

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### **Abstract**

**Introduction:** Arsenic, selenium, vanadium and chromium are identified in the Priority List of Hazardous substances by the Environmental Protection Agency. These elements have been found in groundwater and surface water due in part to the dissolution of naturally occurring minerals and ores as well as effluents from industrial processes. Current drinking water guidelines for arsenic vary between seven and 25ug/L but some have suggested minimum risk levels should be set below 0.2ug/L. Typical detection limits available for current conventional methods of analysis range between 0.2 and 1ug/L. However, natural occurring salts in groundwater and seawater tend to interfere with the analysis causing reliable detection limits to increase above 1-2ug/L.

Conventional inductively coupled plasma – mass spectrometry (ICP-MS) methods is limited because the presence of chloride, sulphur, potassium and organic carbon in the sample interferes with the detection of some of these pollutants. This may lead to inaccurate detection of these pollutants which could result in unnecessary treatment costs.

**Objectives:** Compare the detection limits for arsenic, selenium and vanadium between the conventional ICP-MS methods and an ICP-MS equipped with dynamic reaction cell technology.

**Methods:** Analysis of arsenic, selenium, vanadium and chromium was performed using the Perkin Elmer Elan 9000 and an Elan 9000 DRCII, equipped with dynamic reaction cell technology.

Samples of typical surface water and groundwater were spiked with known concentrations of arsenic. Also, blank samples were spiked with known concentrations of interfering compounds to determine the detection limit capabilities of this technology for each element. Ammonia was used as the reaction gas in the DRCII to reduce these polyatomic interferences. Interferences by chloride and potassium in the case of arsenic, chromium and vanadium were examined.

**Results:** Preliminary results show that detection limits of arsenic using reaction cell technology can be reduced by 10 fold in the presence of interfering substrates. Similarly, pilot studies examining vanadium, selenium and chromium in waters with interfering substances show dramatically improved detection limits and reduced positive interferences.

The research presented here demonstrates the effectiveness of reaction cell technology for the analysis of these priority substances down to lower levels than were previously achievable by conventional ICP-MS analysis. Comparison of these detection limits to the detection limits suggested by certain agencies appear to be more favorable than what can be achieved using conventional ICP-MS.

### **Introduction**

Heavy metals are found with varying abundance throughout the earth's crust. Many of these metals make their way into the human food chain through plant and animal uptake and through dissolution of metal rich deposits in both ground water and surface water.

Anthropogenic sources of contamination of soil, water and air also contribute to elevated levels that may affect the health children and adults.

A list of priority pollutants is maintained in the United States by the Comprehensive Environmental Response, Compensation, and Liability Act for 2003 (ASTDR, 2003). This list considers both prevalence of the pollutant in the environment and its potential toxicity to human health. The following metals and transitional elements appear on this list: Arsenic, lead, cadmium, beryllium, chromium, cobalt, nickel, barium, zinc, copper, selenium, tin, vanadium, silver, and antimony. The elements of concern in Canada mirror this list with the further inclusion of molybdenum (CCME, 1999).

Arsenic is the top priority pollutant to appear on the CERCLA list (ASTDR, 2003). Elevated arsenic levels are found at 1014 of 1598 sites on the national priority list maintained by the US Environmental Protection agency and it is highly toxic. Arsenic is the 20<sup>th</sup> most abundant element in the earth's crust (Merck, 1989).

Arsenic is potentially highly toxic and is also a known carcinogen. Arsenic is most toxic in its inorganic forms with the trivalent species (arsenite) being more toxic than the pentavalent (arsenate) oxyanions (WHO, 2001a). Arsenic is less toxic in organic forms and the arsenobetaine form found in shell fish is not considered toxic (US Public Health Service, 2000). Unlike other metal cations at neutral pH, arsenate and arsenite are relatively mobile under both oxidizing and reducing conditions – making these species the most commonly found forms of arsenic in surface and groundwater. Similarly, oxyanions of selenium and chromium are relatively mobile at neutral pH, but under oxidizing conditions only (WHO, 2001b).

Because of the prevalence, potential mobility in water and toxicity of these inorganic pollutants, governments in North America have established regulatory guidelines which must be met before a contaminated site can be considered reclaimed. As more is understood about the toxicity of certain pollutants and as exposure pathways are determined, guidelines are modified to reflect the potential impact on human health. In the case of arsenic, current maximum allowable concentration (MAC) vary between 7ug/L in Australia and 50ug/L in the U.S. Canada has a current regulatory guideline of 25ug/L. Due to a more comprehensive understanding of arsenic and its carcinogenicity, several countries are changing the regulatory limits (for example see USEPA, 2001). Recently, Alberta has suggested that the MAC for arsenic be reduced to 5ug/L for municipal water treatment plants. Further, the Office of Environmental Health Hazard Assessment (OEHHA) in California devised a public health goal of 0.004ug/L for arsenic which is estimated to reduce the risk of arsenic induced cancer to 1 in one million.

Typical detection limits of commercial labs for arsenic in water are between 0.2 and 1ug/L, limited by currently available technology and methodology. The OEHHA public health goal (0.004ug/L) is not a regulation, and while the document recognizes the limitations of instrumentation to accurately detect levels this low, there is pressure on laboratories to produce the lowest detection limit possible.

Analysis of arsenic and other heavy metals is primarily performed by spectroscopic methods including atomic absorption spectroscopy (AAS), inductively coupled plasma

optical emission spectroscopy (ICP-OES) and inductively coupled plasma mass spectrometry (ICP-MS). Atomic absorption spectroscopy is limited in its use because it cannot do simultaneous multi-element detection resulting in longer analytical times and a higher costs of analysis. ICP-OES is limited in its abilities to meet the low regulatory limits of several of the priority pollutants in water (Rosen and Hieftje, 2004). As a result, many commercial labs perform metal analysis in water using ICP-MS. ICP-MS is capable of analyzing several analytes in a very short time span at a low detection limit. It also has a large dynamic range so it is capable of analyzing major constituents and trace level contaminants without the need for multiple dilutions.

Basically, the sample is introduced into a plasma which generates the ions of the elements in solution. The species created in the ion beam are then separated on their charge:mass ratio ( $m/z$ ) using a quadropole and quantified by a detector (figure 2). However, the ICP-MS does have limitations. Because the instrument separates elements on the basis of the  $m/z$  ratio, polyatomic species and oxides formed in the plasma by naturally abundant molecules in water such as chloride, organic carbon, calcium, potassium and sulphur can cause interferences (Cave et al. 2000). Chloride, with a mass of 35 will form a polyatomic species with argon, the carrier gas for ICP-MS, to form  $\text{ArCl}^+$  which has the same mass and charge as arsenic. Because of the high background noise created by these interferences, lower detection limits in complex matrices is difficult (Chang et al., 2001). A list of other polyatomic interferences can be found in table 1. While ICP-MS is capable of meeting most of the current regulatory requirements in water, elements such as arsenic, selenium, vanadium and chromium are subject to these major interferences (see Table 1).

*Table 1. List of Polyatomic Interferences in ICP-MS*

As such, standard mathematical corrections are built into the analysis that try to remove these interferences. However, each environmental sample that is submitted to the lab

ISOTOPE	ABUNDANCE	INTERFERENCE
<sup>75</sup> As	100	<sup>40</sup> Ar <sup>35</sup> Cl <sup>+</sup> , <sup>59</sup> Co <sup>16</sup> O <sup>+</sup> , <sup>36</sup> Ar <sup>39</sup> K <sup>+</sup>
<sup>52</sup> Cr	83.76	<sup>40</sup> Ar <sup>12</sup> C <sup>+</sup> , <sup>36</sup> Ar <sup>16</sup> O <sup>+</sup>
<sup>56</sup> Fe	91.66	<sup>40</sup> Ar <sup>16</sup> O <sup>+</sup> , <sup>40</sup> Ca <sup>16</sup> O <sup>+</sup>
<sup>76</sup> Se	9.02	<sup>40</sup> Ar <sup>36</sup> Ar <sup>+</sup> , <sup>38</sup> Ar <sup>38</sup> Ar <sup>+</sup>
<sup>77</sup> Se	7.58	<sup>40</sup> Ar <sup>37</sup> Cl <sup>+</sup>
<sup>78</sup> Se	23.52	<sup>40</sup> Ar <sup>38</sup> Ar <sup>+</sup>
<sup>80</sup> Se	49.82	<sup>40</sup> Ar <sub>2</sub> <sup>+</sup>
<sup>82</sup> Se	9.19	<sup>40</sup> Ar <sub>2</sub> <sup>1</sup> H <sub>2</sub> <sup>+</sup>

has unique levels of analytes that can create these interfering substrates and the corrections tend to be quite general. Therefore, given certain matrices, the corrections that are built into the instrument may decrease the accuracy and precision of the data generated.

Recently, a system capable of selectively removing these interfering ions through chemical conversions has been described (Tanner et al., 2002). In this system, a

dynamic reaction cell (DRC) is pressurized with a reaction gas and the ion beam from the plasma is passed through this chamber (Neubauer and Vollkopf, 1999). The gas reacts with species in the ion beam transforming interfering species into innocuous by products that no longer interfere with the species of interest. Two types of reactions typically occur:

- 1) the reaction gas donates an electron to the interfering substrate thereby removing the charge of the interference resulting in ejection from the quadropole or

- 2) the reaction gas donates a hydrogen ion of 1 atomic mass unit, shifting the charge to mass ratio of the interference from the analyte of interest.

Further, dynamic bandpass tuning changes the stability zone of the reaction cell quadrupole which causes the rejection of intermediate species thereby preventing the formation of other interfering ions in the process (Tanner et al., 1999; Tanner et al., 2000). This tuning is specifically adjusted with each element so that interferences can be selectively removed without significantly increasing the overall run time (Hattendorf and Gunther, 2002). The same instrument can be operated in both the dynamic reaction cell mode (ICP-DRC-MS) or the standard mode (ICP-MS) without chemical alteration of the ion beam.

For example, even though selenium 80 is the most abundant isotope of selenium naturally present in the environment, it cannot be used as a quantifiable isotope in standard ICP-MS mode due to the interference of the Argon dimer that is formed in the plasma. The argon dimer will increase the background noise for selenium 80 resulting in a detection limit far above typical concentrations found in the environment. Because argon is the carrier gas for ICP-MS, the dimer formation is significant and the background noise due to this polyatomic interference exceeds 1000000 counts per second (Figure 1 – 0mL/min gas flow rate). However, in the ICP-DRC-MS, and using methane as the reaction gas, selenium can be differentiated from the polyatomic interferences. By gradually increasing the gas flow in the reaction cell, methane can selectively react with the argon dimer, eject it from the ion path, and differentiate selenium from the background noise.

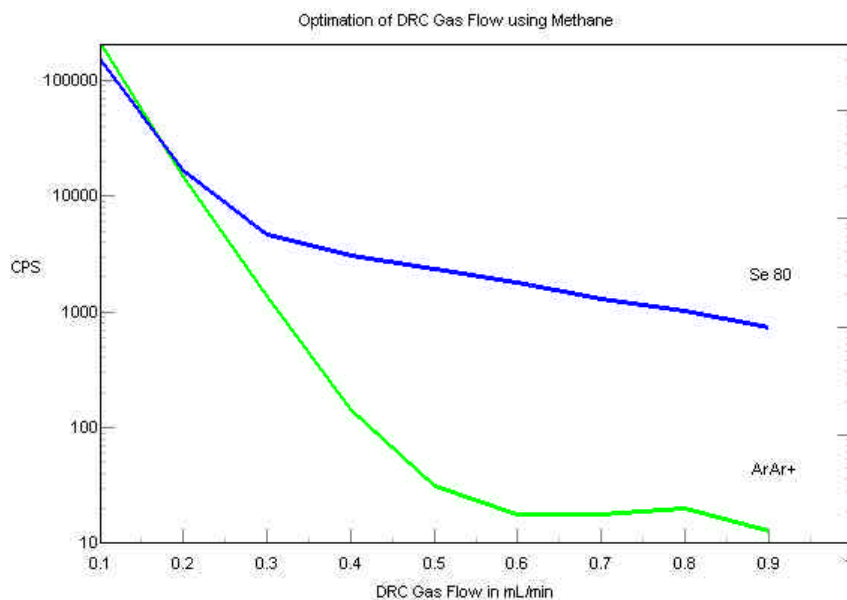


Figure 1. Detection of selenium 80 in the Elan 9000 DRC mode using methane as the reaction gas.

The purpose of this study is to compare the determination of several priority pollutants in the presence of potential interferences using ICP-MS and a ICP-DRC-MS modes. The instrument detection limits in the presence and absence of interfering substrates (chloride

or potassium) were determined for arsenic, selenium, vanadium and chromium. Further, the accuracy of the ICP-MS and ICP-DRC-MS modes were compared by spiking water with low levels of analytes (0.2ug/L and 2 ug/L) in the absence of polyatomic interferences. Finally the accuracy of arsenic quantification in the presences of 2 interfering substrates (chloride and potassium) was examined. The results obtained with the ICP-DRC-MS were compared to an ICP-MS operating in standard mode using built in mathematical corrections for these interferences.

## **Experimental**

### *Instrumentation*

All results were obtained using a PerkinElmer SCIEX Elan DRC II ICP-MS (Concord, Ontario). Analysis was performed in standard mode using polyatomic corrections as outlined in EPA method 200.8 *Determination of Trace Elements in Waters and Wastes by ICP-MS*. Analysis in dynamic reaction cell (DRC) mode had no mathematical correction factors applied, but relied on the DRC to chemically correct for any inferences. The instrument operation parameters are listed in Table 2.

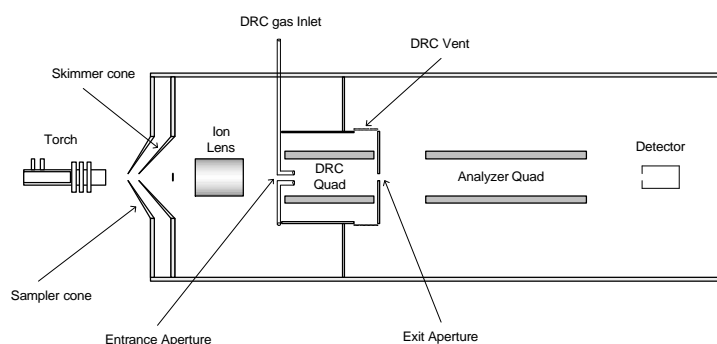
*Table 2. Elan DRC II operation parameters*

Standard mode (ICP-MS) operation is defined as opening the DRC vent to the analyzer chamber, which prevents any significant pressure to build within the dynamic reaction cell. The DRC quadrapole in this mode is simply used to help focus the ions generated by the plasma to the analyzer quadrapole.

RF Power	1200 W
Auxiliary Gas	1.2 L/min.
Plasma Gas	15 L/min.
Nebulizer	0.95 L/min.
Flow	
Reaction Gases	NH <sub>3</sub> , CH <sub>4</sub>
Calibration	external

ICP-DRC-MS mode operation is defined as closing the DRC vent and metering in a reaction gas at such a rate as to consistently build a pressure inside the reaction cell that favors the decomposition of polyatomic interferences. The DRC quadrapole in this mode is used to aid in the focusing and transmission of the ion of interest and the rejection

of unwanted reaction byproducts.



*Figure 2. Elan DRC II ICP-MS.*

### *Reagents*

Gases used in the dynamic reaction cell were Ammonia, Research Grade, 99.999% purity, and Methane, Research Grade, 99.999% purity. (Praxair Distribution Inc., Canada).

External calibration solutions were prepared from mixed stock standards from High-Purity Standards (Charleston, SC, USA.). The 1000 mg/L chloride solution was prepared from 1.648 g/L NaCl, and the seawater matrix solution 31.3 g/L NaCl (Fisher Certified A.C.S.). The 1000 mg/L potassium solution was prepared from 1.907 g/L KCL (EMD Chemicals A.C.S.).

Certified reference materials used included SLRS-2 and SLRS-4 (National Research Council Canada). Standard reference materials used were from SCP Science (St. Laurent, Quebec) included: EnviroMAT Waste Water Low EU-L-1, EnviroMAT Ground Water Low ES-L-1, EnviroMAT Drinking Water Low EP-L-1.

## **Results**

### *Determination of Instrument Detection Limits in ICP-MS and ICP-DRC-MS*

As shown in table 1, arsenic has a major interference with typical salt components found in most environmental water samples. The mathematical correction in standard mode accounts for chloride interference and therefore, should have reasonable accuracy in standard ICP-MS. Three concentrations of chloride were chosen for this experiment: 0mg/L, 1000 mg/L, 19000mg/L. The first two concentrations are a typical range of chloride found in uncontaminated groundwater and surface waters. The last concentration chosen is equivalent to the salt load found in seawater.

An instrument detection limit study was performed in the presence of each of these chloride solutions and the results are presented in table 3. The instrument detection limit data was determined by analyzing ten replicates of a solution that contained the analytes

*Table 3. The chloride matrix effects on the instrument detection limits of ICP-MS.*

<b>Matrix</b>	<b>DRC Mode IDL</b>	<b>Standard Mode IDL</b>
Water	<b>0.028</b>	<b>0.077</b>
1000mg/L Chloride	<b>0.035</b>	<b>0.349</b>
Seawater	<b>0.533</b>	<b>7.898</b>

of interest at or near background levels. The standard deviation ( $s_0$ ) of data was then multiplied by the one sided Student t Variate at a 99% confidence factor for nine degrees of freedom (t) - where the degrees freedom is equal to the number of replicates minus one. Therefore the IDL is a measurement of the precision of the instrument.

In the absence of chloride, the detection limit of arsenic in standard mode is approximately two times higher than with the reaction gas. This may be due to phenomenon known as collisional focusing. However, in the presence of 1000mg/L of chloride, the detection limit for the standard mode operation has increased by five fold, whereas the detection limit with the ICP-DRC-MS has not increased significantly over blank conditions. This correlates to a 10 fold increase in precision using the DRC. Finally, in seawater, the detection limit for arsenic in standard mode is shown to increase by 22.6 times, whereas the DRC shows an increase of approximately 15 fold. The main

cause of the increase is due to a 20 fold dilution to prevent precipitation of the salt in the sample introduction system. So overall, the precision in the detection of arsenic is increased by 10-15 fold using ammonia as the reaction gas for the ICP-DRC-MS.

The instrument detection limits (IDL) for 3 other elements - vanadium, chromium and selenium - were determined in two different matrices: blank water and 1900 mg/L potassium chloride. Results are compared in both the ICP-DRC-MS and standard ICP-MS mode of operation and are presented in figure 3. For the ICP-DRC-MS mode of operation, the reaction gas for chromium and vanadium determinations was ammonia while methane was used for selenium determinations.

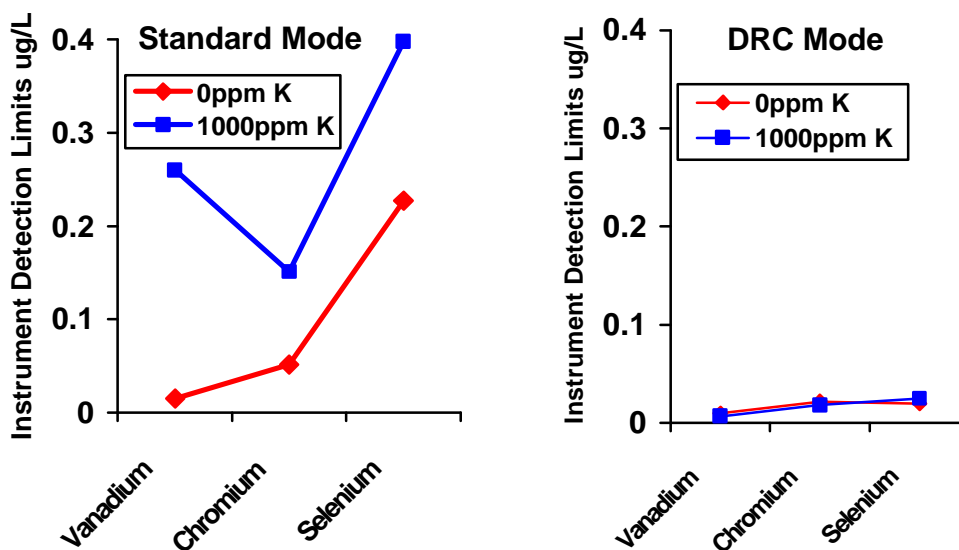


Figure 3. IDLs for Vanadium, chromium and selenium in reagent water and salt solution.

For chromium and vanadium, in the absence of potassium chloride, both the ICP-MS and ICP-DRC-MS have comparable IDLs. However, in the presence of salt, the precision of the data drops dramatically in the ICP-MS while the IDLs in the ICP-DRC-MS are similar to those obtained in the absence of salt.

For the analysis of selenium, the precision of the standard mode is 10-fold higher than that found in the ICP-DRC-MS because the selenium 80 isotope was analyzed in the ICP-DRC-MS mode while selenium 82 was analyzed in the ICP-MS mode. Part of this improvement in DRC is due to the relative natural abundances of each isotope – the natural abundance of selenium 80 is 6 times that of the selenium 82 isotope. It is not possible to analyze the selenium 80 isotope under standard ICP-MS conditions since the large interference due to the formation of the argon dimer masks the detection of selenium 80. In the presence of potassium chloride, the IDLs in standard mode are increased by 2 fold while DRC mode is not affected. The IDLs for the selenium 77 isotope under standard mode are similar to selenium 82. However, in the presence of potassium chloride, the polyatomic interference,  $^{40}\text{Ar}^{37}\text{Cl}^+$ , is formed and the IDLs increase to 2ug/L (data not shown).

*Accuracy and precision of data generated by the ICP-DRC-MS.*

The IDLs are used to determine the precision of data but do not determine if the data is accurate. Accuracy is determined by preparing known concentrations of the element in question and determining if the instrument records the expected result.

Two solutions, at 0.2ug/L and 2ug/L were prepared for each element (vanadium, arsenic, chromium, selenium – figure 4). All solutions were prepared in reagent water without any salt additions. Each determination was performed 10 times and the relative standard deviation (RSD) was determined. The RSD is a comparison of the standard deviation of the 10 results to the mean of the 10 results. The higher the RSD, the lower the precision of the result. The average of the ten results is recorded at the top of each bar in figure 4.

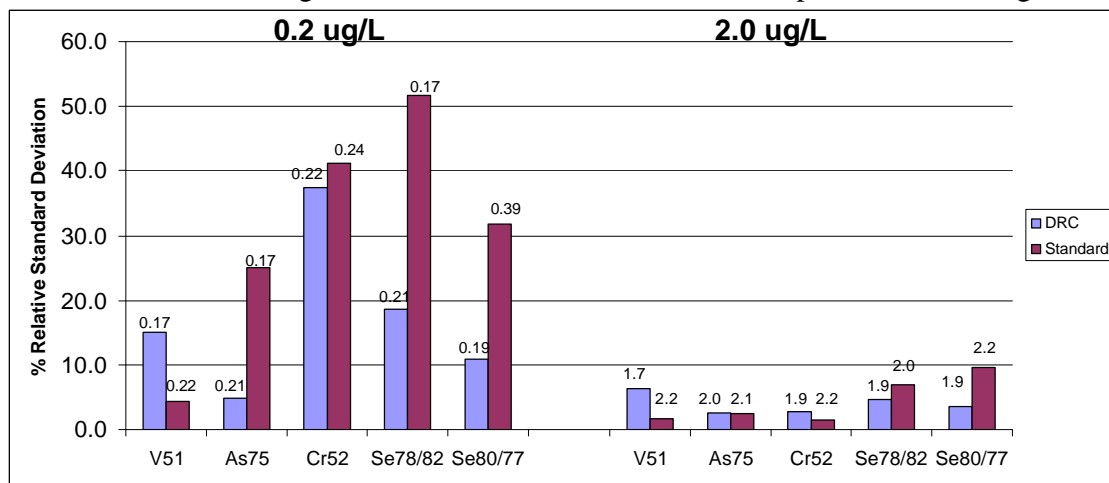


Figure 4. Relative Standard Deviation of each element at 0.2 and 2.0ug/L.

Vanadium was less precise and tended to have a higher RSD in the ICP-DRC-MS. It may be that the reaction gas does not act specifically against the interference but also against the vanadium itself. As a result, the vanadium is being pulled out of the system by the reaction gas and causing a low bias in recovery.

Arsenic has a much lower RSD and the results are more accurate at 0.2ug/L in the ICP-DRC-MS when compared to the standard mode. Both the RSD and accuracy are not significantly different at 2ug/L. The RSD and accuracy for chromium are similar at both concentrations in both modes of operation.

Both selenium 80 and selenium 78 have lower RSD and more accurate results when determined in the ICP-DRC-MS when compared to standard ICP-MS at both 0.2ug/L and 2.0ug/L. Because the ICP-DRC-MS can detect the more abundant species of selenium than the standard mode of operation can, the accuracy and precision are significantly improved at low concentrations.

*The effect of the presence of Interferences on the detection of Arsenic*

Four levels of arsenic were spiked into 0mg/L, 1000mg/L and 19000mg/L chloride (seawater) solutions and samples were run in triplicate on both the standard ICP-MS and the ICP-DRC-MS using ammonia as the reaction gas. In order to prevent the precipitation of salt in the introduction system of the instrument, the seawater sample had to be diluted 20 fold. Figure 5 shows that, overall, there is very little difference between standard mode ICP-MS and ICP-DRC-MS at all concentrations of arsenic in the 0mg/L and 1000mg/L chloride solutions. However low concentrations of arsenic in seawater seem to be “overcorrected” using the standard mathematical corrections while the ICP-DRC-MS is capable of resolving these concentrations very effectively. Therefore, the ICP-DRC-MS is capable of selectively removing interferences and accurately detecting arsenic in all types of matrices tested.

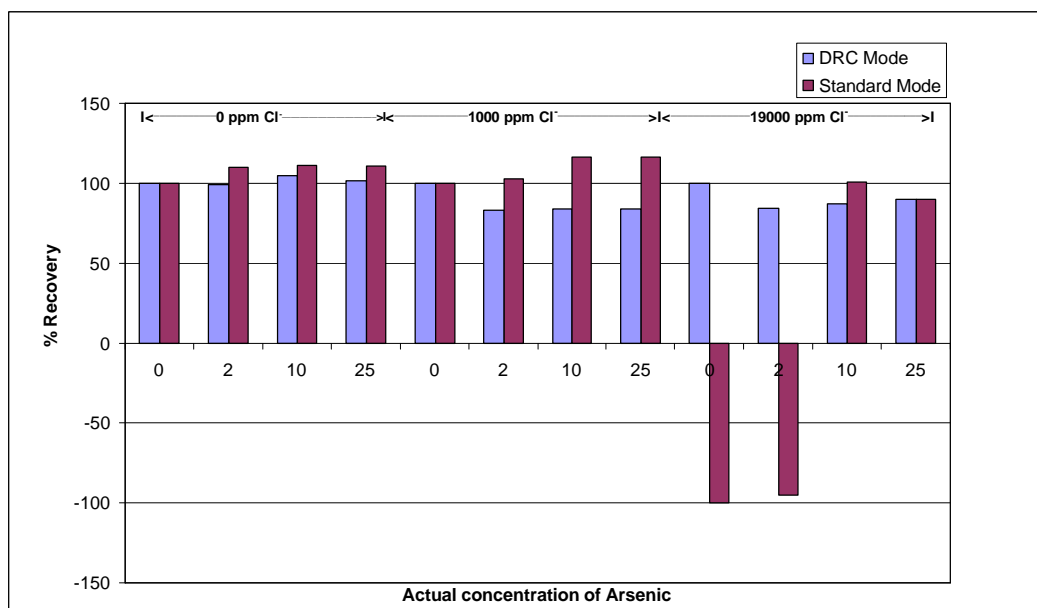


Figure 5. The effect of chloride interference on the detection of arsenic by ICP-MS

Potassium can act as an interference in the detection of arsenic by ICP-MS (table 1). Because potassium binds strongly with soil, levels in groundwater tend to be much lower than chloride. However, it can be present in higher concentrations in a variety of environmental samples due to various industrial processes. In this experiment, a potassium chloride solution containing 1000mg/L of potassium (1900 mg/L KCl) was spiked with various concentrations of arsenic and the recovery of each are presented in figure 6. The results are an average of 3 replicates at each concentration.

According to the Environmental Protection Agency, Method 200.8, laboratory fortified blanks should show a recovery of between 85-115%. All tests achieved this criteria except for the 1000 mg/L solution of potassium operated in standard mode. The average levels of recovery for both the 10ug/L and 25 ug/L spike of arsenic were 120% and 118% respectively. Further, each individual result at these levels exceeded the recovery criteria as per the EPA. The higher recoveries are most likely because the standard calculation for potassium polyatomic interferences are not built into the ICP-MS.

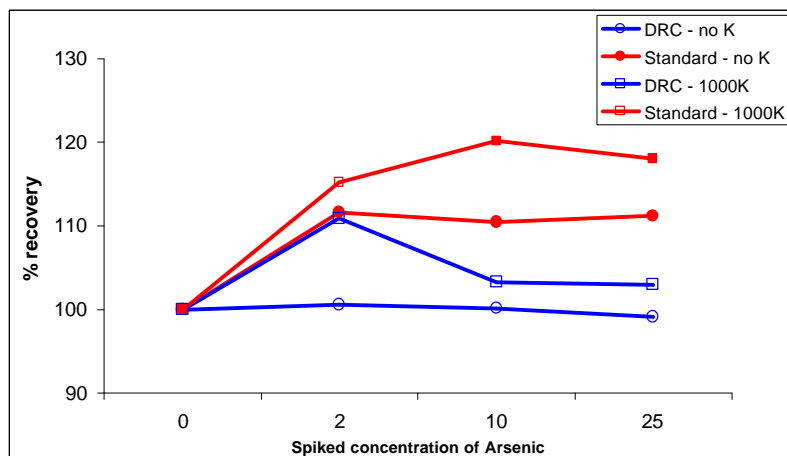


Figure 6. The effect of potassium induced polyatomic interferences on the recovery of arsenic spiked solutions.

## Conclusions

The only way to increase the sensitivity of an ICP-MS is to increase the signal of the element in question compared to the background noise due to interferences. Many techniques are available that perform a pre-concentration step prior to introduction of the sample into the ICP-MS (Cave et al., 1999). These steps focus on the selectivity of certain chemical reactions with specific elements to concentrate these elements and remove interferences. However, each procedure can be time consuming and tends to be specific for only a few elements. Due to the manual nature of these pre-concentration techniques the cost of analysis can be prohibitive.

The dynamic reaction cell uses a technique called chemical resolution to remove all types of interferences that are created in the sample introduction system of ICP-MS without affecting the element of interest (Spectroscopy 1999). Unlike pre-concentration procedures that are specific for only certain elements, the ICP-DRC-MS is capable of using chemical resolution for all elements that are subject to interferences by ICP-MS. Traditional ICP-MS uses mathematical equations of known interferences to correct for each element on each analytical run resulting in a generalization for each sample analyzed. True instrument detection limits are sample and matrix-specific. In simple matrices or at higher concentrations of the element being analyzed, mathematical corrections are sufficient to correct for the interferences to provide an accurate result. However, the application of these corrections to each sample changes the IDL for that particular sample, in that particular matrix. The problem arises when there are enough interferences that the IDL exceeds established regulatory or client requested guidelines. ICP-DRC-MS removes the effect of the interferences making this system capable of maintaining lower IDLs in virtually all matrices.

We have shown that the ICP-DRC-MS technique can improve the precision of low level analysis without compromising the accuracy of the results. The use of specific reaction gases selectively reduces the polyatomic interferences created in the sample introduction system with a small increase to the overall run time resulting in a cost savings compared to pre-concentration techniques.

We have demonstrated that the DRC can be used to reduce the detection limits of elements such as arsenic and selenium by 10-20 fold using ammonia and methane as the reaction gas respectively. Future testing will include the use of other reaction gases to reduce the detection limits for arsenic species below 4 ng/L as outlined in the public health goal of the OEHHA. Also, testing will be evaluated to determine the use of a minimum number of reaction gases to reduce costs of analysis.

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