



**GAS CHROMATOGRAPHY – LIQUID CHROMATOGRAPHY –
INDUCTIVELY COUPLED PLASMA – MASS SPECTROMETRY
PROJECT # 1 : METALS IN SALINE WATER**

Introduction

ALS Environmental (ALSE) has installed an Agilent 7500c Inductively Coupled Plasma Mass Spectrometer (ICP-MS) which may be linked to a High Performance Liquid Chromatograph (HPLC) or a Gas Chromatograph (GC) to offer a range of new services:

1. Determination of Metals in Saline Water to ANZECC 2000 detection levels
2. Determination of Speciated Metals (eg. As^{3+} , As^{5+} , Se^{4+})
3. Determination of Organo Metallics (eg. Butyl Tin, Methyl Mercury)

This Environmental Mail, the first series of three covering this new technology, focuses on the determination of metals in saline water - the concept, and the detection levels available.

Low Level Metal Analysis in Saline Water by Octopole Reaction Cell

ICP-MS is a sensitive analytical technique used for the determination of metals in a diverse range of sample matrices. This robust analytical technique is routinely hampered by high concentrations of Total Dissolved Solids (TDS) that give rise to **Polyatomic Interferences**. These interferences can cause **Ionization Suppression** and **Isobaric Overlap** of target elements. ICP-MS detection is based on the accurate identification and quantification of the target ion mass. A Polyatomic Interference is caused when two or more atoms bond, with a combined mass that is equal to that of a target ion. For example, Arsenic (As) with mass 75 is typically masked by the formation of $^{40}\text{Ar}^{35}\text{Cl}$ (combined mass 75). (Argon (Ar) being the ICP plasma gas and Chloride (Cl) being abundant in saline water)

An alternative to the traditional approach, and a means of overcoming the problems associated with samples of high TDS is the combination of Collision/Reaction Cell technology with ICP-MS. This procedure does not require any specific sample pre-treatment but does require a ten-fold dilution of highly saline water. Inside the Reaction Cell, Polyatomic Ions are reduced by the process of collisions with Helium gas, or reactions with Hydrogen gas.

In **Collision Mode**, Polyatomic Ions with low binding energies are dissociated as they travel across the cell and interact with Helium. Those with high binding energy, are not

dissociated, but rather lose more energy than elemental ions of the similar mass, due mainly to their comparatively larger cross section. Such polyatomics are removed by energy discrimination, which acts as a physical filter for larger ions.

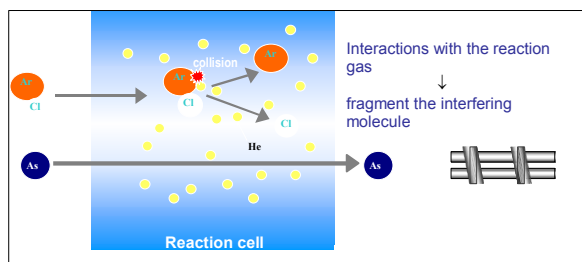


Figure1: Collision with dissociation (Courtesy of Agilent)

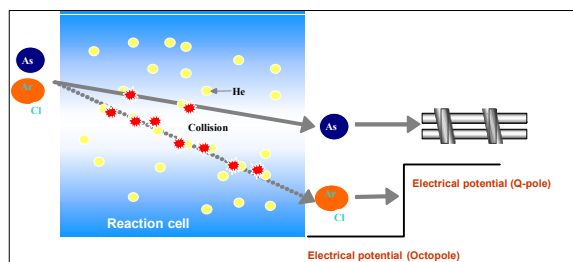


Figure 2: Collision with energy discrimination (Courtesy of Agilent)

Reaction Mode reduces plasma based Polyatomic Interferences through reactions with hydrogen gas. During the atom transfer process, Hydrogen reacts with the Polyatomics, increasing their mass number by one, thereby removing the interference on that mass. The second mechanism is charge transfer, in which a charged Polyatomic will pass the charge onto hydrogen and hence not be detected in the mass spectrometer.

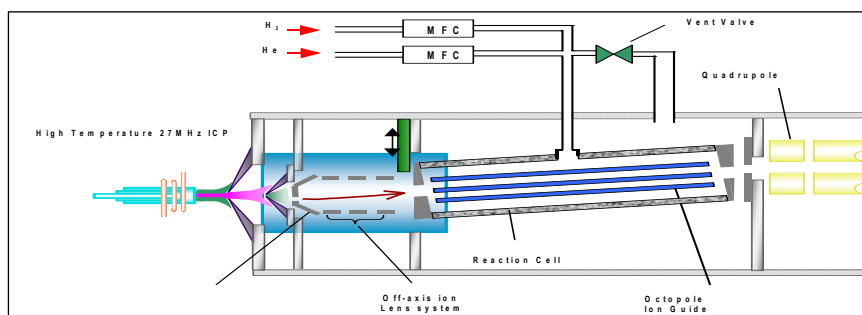


Figure 3: Schematic diagram of ICP-MS with Octopole reaction cell (Courtesy of Agilent)

In addition to these instrumental considerations, **a number of field and laboratory practices also require refinement to realize the sensitivity of the technique.** The instrumentation is housed in a Clean Room laboratory, fitted with HEPA air filtration, Gyprock ceilings, an Air Lock and Restricted access. Sample containers are double washed prior to dispatch in Spec Pure Nitric acid. **Field practices should be modified to consider trace contamination from skin and hair, cross contamination from gloves and atmospheric (dust) contamination.** Statistically proved detection levels and corresponding ANZECC 2000 saline water trigger levels are tabulated below:

Elements	LOR (µg/L)	ANZECC trigger values (µg/L) 95% level of protection	Elements	LOR (µg/L)	ANZECC trigger values (µg/L) 95% level of protection
Ag	0.1	1.4	Mo	0.1	23
Al	10	0.5	Ni	0.5	70
As	0.5	2.3(III) 4.5(V)	Pb	0.2	4.4
B	100	5100	Sb	0.5	270
Ba	1	--	Se	2	3
Be	0.1	--	Sn	5	10
Bi	0.1	--	Sr	10	--
Cd	0.2	5.5	Te	0.5	--
Co	0.2	1	Th	0.1	--
Cr	0.5	27.4(III) 4.4(VI)	Ti	5	--
Cu	1	1.3	Tl	0.1	17
Fe	5	--	U	0.1	--
Li	1.0	--	V	0.5	100
Mn	0.5	80	Zn	5	15

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