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## Ultrapure Water for Elemental Analysis down to ppt levels

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**Abstract:** Recent enhancements in modern analytical instrumentation have dramatically improved the sensitivity of analysis. Trace elements are now measured at ppt and sub-ppt levels. These levels can be achieved if careful control of the analytical protocol is maintained.

Contamination can result from anything that comes into contact with the sample; the laboratory environment, the air, and anything used during sample preparation.

Limits of detection down to ppt levels can be achieved if special care is taken with the experimental environment as well as with the quality of the reagents. This includes the ultrapure water used to run blanks, dilute samples or prepare standards.

Part of this paper looks at the most sensitive multi-elemental analytical tool, i.e. Inductively Coupled Plasma Mass Spectrometry (ICP-MS). Some of the most common causes of interference will be reviewed, as well as ways of reducing this interference by specific instrument developments and particular care taken during ICP-MS analysis.

Sub-ppt values for limits of quantification are achievable when the ultrapure water used is produced by a system for which all parts have been carefully selected and whose configuration has been specifically developed for ultratrace purposes.

This specific water purification system is described and typical values that can be achieved for blanks with ICP-MS technology are shown.

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### Introduction

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Due to newly developed instrumentation, limits of detection reached by trace analysis are becoming lower and lower. Hyphenated techniques currently used for sample studies and element detection can now reach ng/L levels and even pg/L levels when specific clean conditions and careful experimental handling are ensured. Consequently, instruments and reagents used for blank analysis, standard dilutions and sample preparations need to be of the highest quality.

Depending on the elements studied and the environmental conditions of the analytical laboratory, various instrument configurations are possible.

Flame or Electrothermal Atomic Absorption Spectroscopy (FAAS or ETAAS) and Inductively Coupled Plasma - Optical Emission Spectroscopy or Mass Spectrometry - (ICP-OES or ICP-MS) are the main technologies employed by trace analysis scientists<sup>1,2</sup>.

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### *The Analytical instrument*

*ICP-MS, choice tool for ultratrace analysis.*

Although technical advances are improving the sensitivity and detection limits of most analytical techniques, instrumental limitations, such as difficulties in performing multi-elemental simultaneous studies, still remain in advanced analytical technologies<sup>3</sup>. This is why, in many applications, ICP-MS is often considered as the ideal instrument to perform fast, multi trace-elemental analyses<sup>4</sup>. This technology allows fast, qualitative analysis of unknown samples, and quantitative, multi-elemental analysis down to ppt (ng/L) and even ppq (pg/L) levels. Application fields where this instrument is a key tool are quite varied: they include the medical area<sup>5</sup>, where heavy metals have a significant impact on health; the environmental field, with metal traceability<sup>6</sup>; the nuclear world, with the ability to perform isotope ratio screenings and detect speciation; and the microelectronics industry, with ultratrace analysis in various high purity chemicals, including ultrapure water<sup>7,8,9</sup>. Major software development comes with ICP-MS instruments. However, this method still requires skill and specific experimental conditions in acquiring and interpreting data. The actual detection limits depend on the element, the matrix, the sample preparation and the condition of the instrument. Precise method protocols and experimental conditions are developed to perform some specific element determinations<sup>10</sup>.

### *Interference and contamination*

Most of the experimental optimization includes the special care needed to minimize contamination. It follows that blank optimization, necessary to the newly targeted sub-ppt limits of detection, requires the refinement of sampling, processing and analytical techniques. For example, current analytical capabilities have often exceeded the capacity to collect both uncontaminated and representative environmental samples<sup>11</sup>.

The accurate measurement of trace elements using ICP-MS with sub-ppt detection limits is achievable if one takes into account the possible limitations due to instrumental or experimental contamination and interference.

### *Instrumental interference*

As far as the instrument itself is concerned, spectral interference, occurring when unwanted ions possess the same nominal  $m/z$  value as an analyte ion of interest (see *Table 1*), is one of the greatest obstacles to ICP-MS analysis<sup>12</sup>.

Two categories can be distinguished:

- \* Background polyatomic interference coming from plasma gas, water from the sample aerosol and air in the plasma (For example  $^{40}\text{Ar}^{16}\text{O}$  and  $^{56}\text{Fe}$  or  $^{40}\text{Ar}^{35}\text{Cl}$  and  $^{75}\text{As}$ ).
- \* Isobaric interference, due to elements presenting isotopes of the same  $m/z$  ratio (example of  $^{64}\text{Zn}$  and  $^{64}\text{Ni}$ ).

Although most elemental ions are singly charged within the plasma, doubly charged atomic ions can also cause interference (example of  $^{138}\text{Ba}^{++}$  and  $^{69}\text{Ga}$ ).

For isobaric interference, corrections on peak intensity ( $I$ ) can be applied taking natural isotopic abundance ( $a$ ) into account and using results on other isotopes to establish the correction.

For example, for  $^{64}\text{Zn}$  and  $^{64}\text{Ni}$  interference, the calculation will be:

$$I_{^{64}\text{Zn}}(\text{cor.}) = I_{^{64}\text{Zn}}(\text{mes.}) - (a_{^{64}\text{Ni}}/a_{^{60}\text{Ni}}) * I_{^{60}\text{Ni}}$$

Various experimental method and instrument developments have been dedicated to addressing the problems posed by polyatomic ion interference, such as the ShieldTorch that changes plasma conditions to significantly attenuate ionization thus reducing the formation of polyatomic ions<sup>13</sup>. Analysis by less powered plasma allows the reduction of specific spectroscopic interference. Only species with an ionization potential below 8eV will undergo complete dissociation. This "cold plasma" analysis is often used for elements such as  $^{23}\text{Na}$ ,  $^{39}\text{K}$ ,  $^{40}\text{Ca}$  and  $^{56}\text{Fe}$ <sup>14</sup>.

Other interference is inherent to the instrument used. First of all, matrix effects can lead to a signal drift due to a modification of the surface of the skimmer cone of the ICP-MS, (interface between the torch and the mass spectrograph). This results in a change in the ionization characteristics inside the plasma torch, which modifies the system sensitivity. Finally, some memory effects of specific elements, such as Mercury (Hg), Iodine (I) and Boron (B) will require appropriate

atomic ion	potential interference
$^{39}\text{K}^+$	$^{38}\text{Ar}^1\text{H}^+$
$^{40}\text{Ca}^+$	$^{40}\text{Ar}^+$
$^{56}\text{Fe}^+$	$^{40}\text{Ar}^{16}\text{O}^+$
$^{63}\text{Cu}^+$	$^{62}\text{Ni}^1\text{H}^+$
$^{63}\text{Ni}^+$ (radioactive isotope)	$^{63}\text{Cu}^+$
$^{74}\text{Ge}^+$	$^{74}\text{Se}^+$
$^{75}\text{As}^+$	$^{40}\text{Ar}^{35}\text{Cl}^+$
$^{89}\text{Y}^+$	$^{40}\text{Ar}^{49}\text{Ti}^+$
$^{99}\text{Tc}^+$ (radioactive isotope)	$^{99}\text{Ru}^+$
$^{106}\text{Pd}^+$	$^{106}\text{Cd}^+$
$^{121}\text{Sb}^+$	$^{105}\text{Pd}^{16}\text{O}^+$
$^{127}\text{I}^+$	$^{110}\text{Cd}^{16}\text{O}^1\text{H}^+$
$^{114}\text{Cd}^+$	$^{114}\text{Sn}^+$
$^{129}\text{I}^+$ (radioactive isotope)	$^{129}\text{Xe}^+$
$^{133}\text{Cs}^+$	$^{116}\text{Cd}^{16}\text{O}^1\text{H}^+$
$^{138}\text{Ba}^+$	$^{124}\text{Te}^{14}\text{N}^+$
$^{139}\text{La}^+$	$^{122}\text{Sn}^{16}\text{O}^1\text{H}^+$
$^{141}\text{Pr}^+$	$^{124}\text{Sn}^{16}\text{O}^1\text{H}^+$
$^{187}\text{Re}^+$	$^{187}\text{Os}^+$
$^{238}\text{Pu}^+$ (radioactive isotope)	$^{238}\text{U}^+$

Table 1: Some atomic ions and potential interference

rinsing solutions. In addition to these instrumental limitations, the way the experiment is performed, the quality of the reagents used and the environmental conditions while performing the analysis are of key importance.

### The Water Purification System

Blank levels of a given element are affected by such factors as the purity of sample-treating solutions, container purity and the general analytical environment. Among the various reagents used during the key steps of blank, standards and sample preparation, is ultrapure water. Investigations are often performed on the background obtained while aspirating air, pure acid or ultrapure water. Ultrapure water such as that produced by a Milli-Q® system, causes less spectral interference than high quality nitric acid. Although this acid is purified by sub-boiling, the trace element concentrations are still above those of ultrapure water<sup>15</sup>. It is clear that 18.2MΩ.cm is no longer a "quality certification" value. Studies concerning ultratrace analysis show that blank optimization is only achievable when ultrapure water with sub-ppt level contamination for most of the elements is used. Contamination risks are greatly increased when the ultrapure water is stored. Results clearly indicate that the quality of high purity water degrades with storage time<sup>16</sup>. In addition to clean air, the laboratory needs an ultrapure water

supply, and the analytical instrument, or at least the sample preparation station, should be located close to the water outlet.

### The Pretreatment System

Production of high quality ultrapure water requires a combination of technologies to process tap water to ultrapure water. Water is first purified through a system including reverse osmosis and electrodeionization (EDI). This EDI technology is a key step in the production of ion-free water. The EDI module, where a direct current voltage is applied to the resin-containing cells, maintains consistent water quality with no significant fluctuations due to changes in feed water ion concentration. The high resistivity water produced, presents a low ionic challenge to ultrapure polishing resins. The hydrolysis and ion removal in the EDI module result in a steady-state operation of the resin without exhaustion or the need for regeneration. A more complete description of this RO/EDI treatment, performed in a system called Elix®, has been previously published<sup>17</sup>. This purified water is then processed in a polishing system where additional technologies produce the ultrapure water suited to ultratrace analysis. Elix water is stored in an intermediate reservoir to provide an adequate flow rate to feed ultrapure water systems. Numerous tests were performed to select the right materials of construction, to define the reservoir design and finally to limit water degradation during storage. As a result of these tests, polyethylene with low extractable levels was selected for the container; a blowmolding process was chosen to ensure smooth and regular inner surfaces for the conical reservoir and a vent filter containing activated carbon and soda lime was developed<sup>18</sup>.

### The Ultrapure Water Polishing System

The ultrapure water system uses high quality ion-exchange mixed bed resin in a pure natural polypropylene housing selected for its low leaching characteristics.

The best ultrapure water to optimize blanks and prepare standards is obtained by adding UV photo-oxidation technology within the water system. A 185/254 nm UV lamp placed upstream of the polishing packs ensures the destruction of organics, including those with trapped metals. The released elements can then be retained by the ion exchange resins. A resin for the specific removal of boron is included in the first purification pack.

Additionally, an accurate resistivity meter is placed

upstream of the final polishing cartridge that contains mixed-bed resins (Quantum IX) in order to monitor the very first ionic release from the first purification pack (Q-Gard B1). Final filtration is ensured through a 0.1 µm filter, containing a membrane specifically designed for critical ultratrace applications. This membrane is made of ultra high molecular weight polyethylene (UHMWPE filter). The specific membrane charged structure removes colloidal traces .

The flow schematic of this water purification system called Milli-Q Element is shown in figure 1.

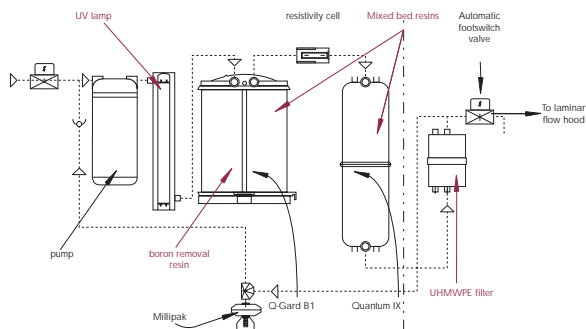


Figure 1: Flow schematic of the Milli-Q Element water purification system for ultratrace analysis.

To be able to collect the ultrapure water directly under a laminar hood, with ease and limited contamination risks, a three-meter distance can separate the main system cabinet and the point of use. The delivery of water is ensured via an automatic footswitch solenoid valve.

## Analytical Method

### Experimental Requirements

Sample and/or laboratory contamination can affect the accuracy of trace metal analysis. Special care must be taken with the experimental environment, this includes the quality of the reagent used<sup>19</sup>. Moreover, most of the contamination can result from anything that comes into contact with the sample, including glassware, the laboratory environment, the air, and anything used during sample preparation. Even cleanroom gloves can present significant metal contamination<sup>20</sup>.

Precise washing protocols are thus established in order to remove any contamination coming from the various containers used to prepare samples and standards. High quality plastic bottles, mainly polyethylene (PE), perfluoroalkoxy (PFA) or fluorinated ethylenepropylene (FEP) are used throughout all sampling and analysis procedures. Several acid and ultrapure water washing steps should be performed prior to running the

experiment in order to avoid further leaching from the vials<sup>21</sup>. The impact of contamination coming from the various vials, or sample adsorption on the walls of such containers, have led scientists performing ultratrace analysis to develop cleaning steps for materials<sup>22</sup>. The procedure followed by scientists working in the Glaciology field is given hereafter as an example<sup>23</sup>: “LDPE bottles for storage of samples and plastic tools were acid cleaned in the class 100 environment [...]. Briefly, items were cleaned as follows: rough rinse with tap water to remove dust; degrease with chloroform and rinsing with ultrapure water; immersion in a first acid bath, (HNO<sub>3</sub>/ultrapure water 1/3, 50°C, 2 weeks), and rinsing with ultrapure water; immersion in a second acid bath (HNO<sub>3</sub>/ultrapurewater 1/1000, 50°C, two weeks) and rinsing with ultrapure water; immersion in a third acid bath (HNO<sub>3</sub>/ultrapure water 1/1000, 50°C, two weeks); finally, bottles are rinsed several times with ultrapure water, filled with a diluted ultrapure HNO<sub>3</sub> fresh solution and stored inside double polyethylene acid clean bags”.

Washing steps are even more important in the Microelectronics industry, where efficient rinsing protocols should be employed after wafer-cleaning steps<sup>24</sup>.

### Sample preparation

Maximum precautions should be taken in order to avoid contamination from the environment, the reagents and the containers used. Cleanroom laboratories or laminar hoods are the most popular ways to minimize external effects.

While preparing standards and samples, it is essential to avoid contact between the solutions and the outside environment. The use of a polyethylene cover to protect the sampling bottle, prevents particulate contamination while loading the sample into the analyzer (see figure 2).

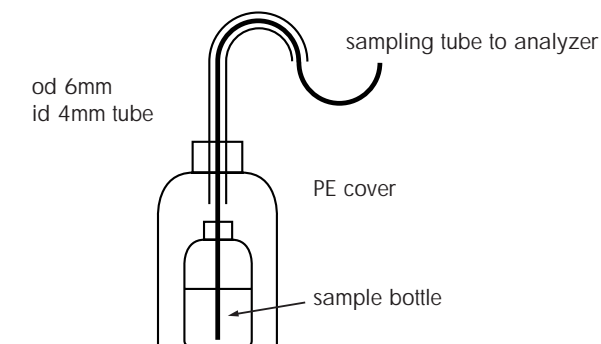


Figure 2: Schematic of “clean” sample introduction.

Standard preparations require several dilutions of commercially available solutions. Only ppm

concentration levels are available due to storage degradation of diluted solutions. Standard and sample preparations should be run simultaneously in order to get similar contamination effects, if any. Advances in analytical equipment now allow simultaneous multi-elemental analysis that in turn requires multi-elemental standard solutions. The analysis of fifteen elements at one time means the need for 15 solutions. This operation in itself entails some risks of contamination. The purity of standard solutions needs to be high, because sometimes standard solutions of a given element can be polluted by other elements, for instance, by poor handling techniques. In addition, some incorrect mixtures of one to another may cause chemical reactions that result in precipitation. Mixed standard solutions are now available and reduce such risks.

The multi-element solution used as a standard was SPEX (Cat. N° XSTC-331). It contains 28 elements and was used to perform the various calibration curves.

Acidification of the diluted standard solution, sometimes of blank water and sample water, are indispensable to stabilize the elements in the solution. Nitric acid is commonly used for this purpose and has less influence on the analyzer as far as system interference is concerned. Many grades of nitric acid are found in the catalogues of laboratory suppliers, the purity of some of the higher grade ones being certified by documents that can assist in contamination control. Because nitric acid has the ability to oxidize and dissolve chemicals, it tends to get much more polluted than standard solutions.

Ultrapure grade nitric acid (Kanto Kagaku) was used for acidification of standard solutions and dilutions.

All sampling bottles have to be free of chemical extractables. As previously described, well-established cleaning protocols are checked with a preliminary extraction test on bottles. When samples are kept acidified, the same nitric acid concentration is used to perform extractable tests. The adsorption phenomenon on the bottle walls should also be taken into account.

For this study, all sampling bottles are washed with successive ultrapure water and nitric acid baths.

#### ICP-MS conditions

Simultaneous multi-elemental analysis requires a single tuning setting that is adapted to all elements. Lower plasma power and ShieldTorch were employed to reduce the production of mass interference ions, such as Ar, ArH or ArO. To see the sensitivity and linearity

of the signal gain on each element, a preliminary calibration curve was prepared with standard additions at 20, 40 and 60 ppt (see figure 3). Each element has a different sensitivity that depends on the ionization efficiency in the plasma torch. Elements that have higher ionization potential tend to be less sensitive. In addition, lower plasma power limits ionization capacity. In some cases, this signal loss can be compensated by longer signal accumulation time on given elements.

A good linearity was obtained up to 60 ppt concentration. No signal saturation at detector was observed in this range.

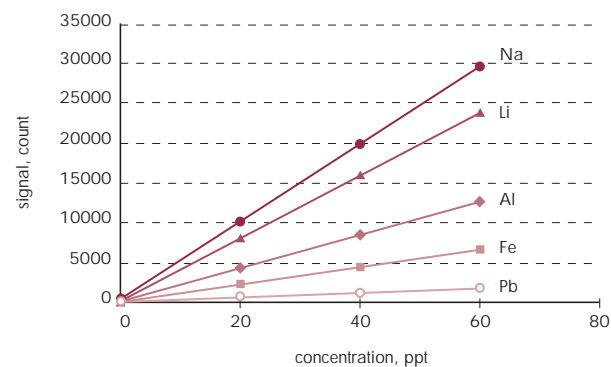


Figure 3: ICP-MS calibration curves

Data presented in the results are obtained with cold plasma conditions in order to get information on ions that are difficult to measure. The ICP-MS configuration used is indicated in table 2.

RF power (W)	600
PF matching (V)	2.3
sampling position (mm)	14.3
torch horizontal position (mm)	0
torch vertical position (mm)	0
carrier gas flow (L/min)	1.19
blend gas flow (L/min)	0.4
peristaltic pump (rpm)	0.3
spray chamber temp. (C)	0
plasma torch	quartz
spray chamber	quartz
shield plate	applied
nebulizer	quartz, co-axial type
sampling tube	PFA, id 0.15mm
drawing electrode 1 (V)	-80
drawing electrode 2 (V)	-19
einzel lens 1.3 (V)	-80
einzel lens 2 (V)	-4
omega vias (V)	-100
omega lens (+) (V)	-8
omega lens (-) (V)	3
quadrupole focus (V)	-2
alternate electrode (V)	20
AMU gain	115
AMU offset	175
mass axis gain	0.999
mass axis offset	0
plate vias (V)	-10
pole vias (V)	-10
discriminator (mV)	13
EM voltage (V)	-1700
EM final stage (V)	-278

Table 2: HP 4500 ICP-MS conditions

Preliminary Study

ICP-MS analysis with no element-specific optimization is performed on ultrapure water and counts are recorded to get an idea of the achievable blanks. The addition of 10 ppt standards of the studied elements gives information on the achievable quantification limits (see table 3).

The high counts generated for <sup>40</sup>Ca clearly show the interference due to <sup>40</sup>Ar and confirm that specific conditions must be applied to the ICP-MS instrument in order to perform sensitive and accurate calcium determinations.

Element	blank water		+ 10 ppt	
	count	%RSD	count	%RSD
<sup>7</sup> Li	96.7	4.7	31500	1.0
<sup>23</sup> Na	8390	3.1	92100	2.9
<sup>24</sup> Mg	1840	4.7	16200	1.2
<sup>27</sup> Al	710	2.7	20100	3.0
<sup>39</sup> K	11800	1.1	20500	4.8
<sup>40</sup> Ca	15200	3.9	16000	5.4
<sup>44</sup> Ca	970	5.2	1560	2.6
<sup>53</sup> Cr	293	7.1	943	5.8
<sup>55</sup> Mn	3890	2.1	15800	3.8
<sup>56</sup> Fe	1140	2.8	6310	6.3
<sup>59</sup> Co	3680	4.1	10200	1.9
<sup>60</sup> Ni	197	7.8	1820	5.8
<sup>63</sup> Cu	360	4.2	4500	4.3

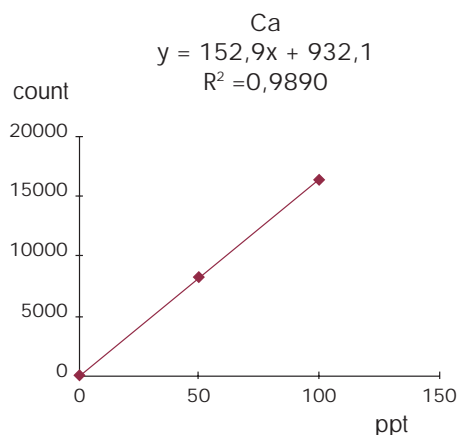
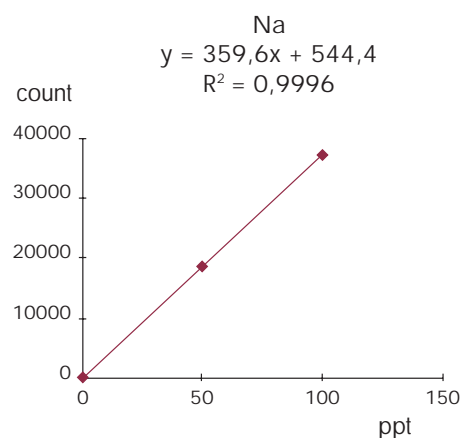
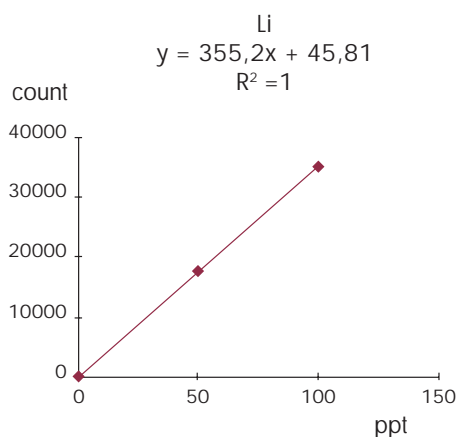
Table 3: HP4500 ICP-MS data counts for ultrapure water blanks and 10 ppt spikes with Relative Standard Deviation (RSD).

Elemental analysis of Milli-Q® Element water

A significant number of experiments were performed with a Milli-Q® Element system fed by an Elix®, system. Results, indicated in ppt, are shown in table 4. In each case, the limit of detection (DL) is taken as 3 times the standard deviation of ten replicates of a blank (Milli-Q SP ICP-MS water, Nihon Millipore Ltd., Japan), the limit of quantification (QL) being 3.33 times this limit of detection (or 10σ). Values are given for ultrapure water samples, even when these are under limits of quantification.

BEC corresponds to the Blank Equivalent Concentration calculated using linear calibration curves obtained with 0,50 and 100 ppt standard solutions for each element (figure 4). Extrapolation to the x-axis (y=0) gives the BEC value and is a good indication of the level of contamination. The standard

curve obtained for Ca clearly shows the limitation for this ion (due to instrumentation and experimental conditions). On the other hand, the good Fe results confirm the interference elimination with the chosen ICP-MS conditions as indicated in Material and Methods.



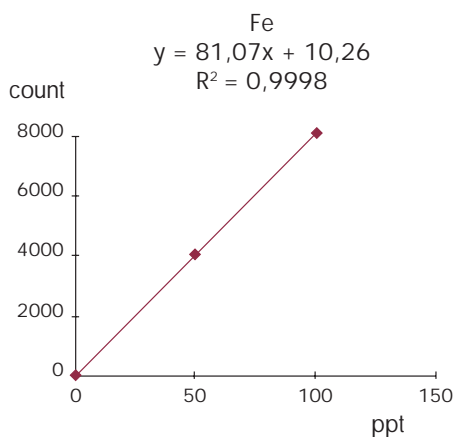


Figure 4: Some calibration curves

As shown in table 4, very good results are obtained in terms of low levels of elemental contamination. The ultrapure water produced by a system combining all the advanced water purification technologies and used in a specifically clean and controlled environment, allows sub-ppt levels to be reached for most elements.

Element	DL 3σ	QL 10σ	BEC	Elix water	Ultrapure Water
<sup>7</sup> Li	0.008	0.027	0.13	0.34	0.034
<sup>23</sup> Na	0.055	0.184	1.5	545.5	0.32
<sup>24</sup> Mg	0.213	0.709	0.34	0.99	< QL (0.34)
<sup>27</sup> Al	0.111	0.371	0.039	9.9	< QL(0.18)
<sup>39</sup> K	0.247	0.825	5.9	36.2	5.2
<sup>40</sup> Ca	1.369	4.563	6.1	12.14	6.8
<sup>52</sup> Cr	0.055	0.183	0.12	0.29	< QL(0.082)
<sup>55</sup> Mn	0.193	0.643	0.69	0.51	< QL(0.4)
<sup>56</sup> Fe	0.03	0.101	0.13	1.1	0.46
<sup>59</sup> Co	0.212	0.708	0.67	0.32	< QL(0.5)
<sup>58</sup> Ni	0.129	0.428	0.21	0.69	0.5
<sup>63</sup> Cu	0.032	0.106	0.53	1.38	< QL (0.067)
<sup>64</sup> Zn	0.722	2.407	2.4	34.58	4.4
<sup>208</sup> Pb	0.066	0.221	0.13	1.15	0.94

Table 4: ICP-MS Blank quantification of some elements with ultrapure water produced by the Milli-Q® Element water purification system (all values in ppt).

## Conclusion and perspectives

Taking the environmental field as an example, over the past 10 years, reported background dissolved trace element concentrations have declined from tens of ppb (µg/L) through single digit ppb, to the ppt (ng/L) range. This in fact does not reflect improved water quality, but rather, reduction in contamination introduced during sampling, processing and analysis.

These improved instrumental and experimental procedures highlight the impact of minute contamination. Consequently, the use of very high quality ultrapure water is required to prepare blanks and standards, perform critical cleaning and run high sensitivity analyses.

Being able to modulate the polishing cartridge

composition according to the application allows blank optimization for trace analysis of a specific element such as boron. Other developments can also be geared to specific needs, such as the focus on Silica<sup>25</sup> for example. Additional added features, such as the ability to operate the system with a footswitch, ensure minimum cross contamination by other users and facilitate operation under a laminar hood.

The combination of these different advancements in instrumental and purification technologies has resulted in a system that produces ultrapure water suitable for ultratrace analysis at the sub-ppt levels.

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