

ULTRAPURE WATER FOR DETERMINATION OF TOXIC ELEMENTS IN ENVIRONMENTAL ANALYSES

In this paper the importance of reagent water quality for toxic element environmental analyses is discussed, and the suitability of fresh ultrapure water produced using Merck water purification systems for ICP-OES and ICP-MS trace element analyses in environmental laboratories is demonstrated.

Introduction and Water Quality Requirements

Dramatic improvement in the sensitivity of analytical instruments over the last decades has changed our understanding of environmental contamination and hazardous effects of metals such as Be, Cr, Mn, Fe, Ni, Cu, Zn, As, Cd, Sb, Ba, Hg, Tl, and Pb. This has resulted in a number of regulations and guidelines that establish the maximum acceptable or recommendable concentrations of toxic metals in drinking water,¹ marine water,² and wastewater.³ The requirements instituted by authorities consequently have resulted in a growing need for toxic metal monitoring in environmental laboratories where spectrometry techniques are standard instrumentation recommended for the determination of trace elements.^{4,5} The preponderant role of ICP-MS and ICP-OES in the detection of traces of toxic metallic elements in environmental analyses of water and soil has led to higher quality requirements for ultrapure water, which is the most frequently used reagent in ICP-MS and ICP-OES analyses. In particular, ultrapure water is used as the reagent blank, for sample and standard preparation, and for instrument and sample container cleaning (Figure 1). Therefore, the ultrapure water must be free of metals to preserve analytical instruments from contamination and to avoid interferences with analyzed elements, in order to ensure the accuracy and precision of measurements.

Results and Discussion

To benefit fully from modern ICP-OES and ICP-MS instrumentation, ultrapure water of very good quality is required. Indeed, any contamination coming from laboratory reagents will increase background equivalent concentration (BEC) and detection limit, resulting in poorer performance of the technique. Therefore, the suitability of reagent water used in all steps of ICP-MS or ICP-OES analyses is defined by the general rule that the measured element should not be detectable in the blank, or if it is detected, its BEC should be negligible relative to the desired analytical range. In environmental analyses, elements in water samples are usually analyzed at $\mu\text{g/L}$ (ppb) analytical range⁶ and in soil samples, at mg/L (ppm) range.⁷ To ensure the success of experiments in the ppb-ppm range, it is desirable that BEC values of target elements do not exceed ppt or sub-ppt range.

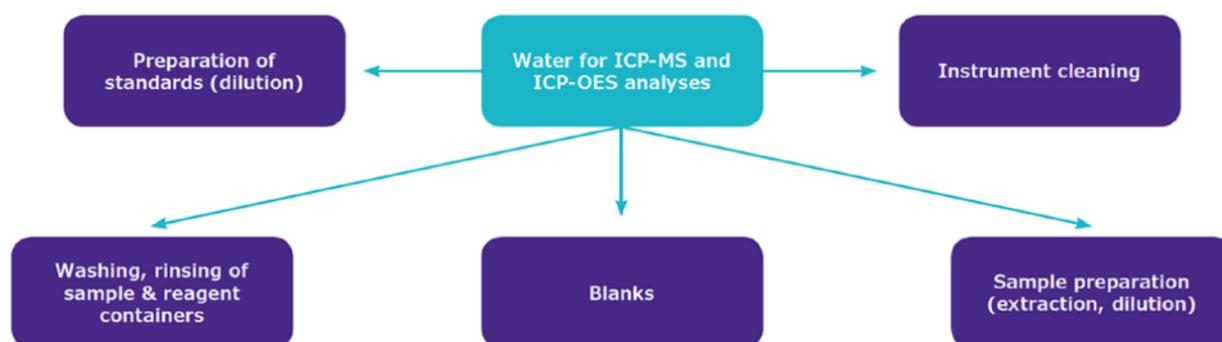


Figure 1. Different types of uses of ultrapure water in ICP-MS and ICP-OES analyses

Moreover, as LOD (Limit of Detection) is separately specified in certain analyses,¹ in addition to a negligible level of contamination, the usage of ultrapure water of consistent quality is critical.

To evaluate the suitability of reagent water necessary for ICP-MS and ICP-OES environmental analyses, the measurements of toxic elements in freshly produced ultrapure water from a Milli-Q® water purification system have been performed. The resulting BEC of reagent water, as well as the detection limits in ng/L level, are presented in Table 1. The results from Table 1 show that when using Milli-Q® water, BEC levels for the majority of analyzed elements are in the sub-ppt or low ppt range (experiments are done under normal laboratory conditions, not in a cleanroom). In case there is a need to achieve significantly lower levels of elements, it is reasonable to perform analyses in a cleanroom or metal-free laboratory environment⁸ and to use an additional polishing step such as a Q-POD® Element unit, which makes it possible to obtain BECs at sub-ppt and ppq level.^{9,10}

Experimental

Ultrapure water samples from a Merck Milli-Q® Advantage A10 water purification system, equipped with Q-Gard® and Quantum® TEX cartridges, Millipak® final filter, and fed by an Elix® Essential 5 water purification system, were analyzed for the levels of Be, Cr, Mn, Fe, Ni, Cu, Zn, As, Cd, Sb, Ba, Tl, and Pb using an Agilent® 7700s instrument, and for the level of Zn, using an Agilent® 7500s instrument.

Ultrapure water samples from a Merck Milli-Q® Direct water purification system, equipped with a QPAK® TIX cartridge and Millipak® final filter, were analyzed for the Hg level using an Agilent® 7500s ICP-MS instrument. All experiments were performed under regular laboratory conditions (not in a cleanroom).

The instrumental details and parameters for the Agilent® 7700s: PFA (perfluoroalkoxy)-50 nebulizer, PFA spray chamber, sapphire inert torch, quartz 2.5 mm i.d. torch injector, platinum sample and skimmer cone, RF power 600 / 1600 W, sampling position 12 / 8 mm, carrier gas flow 0.90 L/min, makeup gas flow 0.32 / 0.51 L/min, auto detector mode, calibration through 1, 5, 10, 50 ng/L . The instrumental details and parameters for the Agilent® 7500s: quartz nebulizer, quartz spray chamber, quartz 2.5 mm i.d. torch injector, nickel sample and skimmer cone, RF power 1300 / 1550 W, sampling position 8 mm, carrier gas flow 0.96 L/min, makeup gas flow 0.23 L/min, auto detector mode, calibration through 1, 20, 50, 100 ng/L .

The calibration standards used in experiments with the Agilent® 7700s were a mixture of Agilent® and SPEXCertiPrep®, and with the Agilent® 7500s, ROMIL PrimAg®-xtra; containers were all PFA pre-cleaned with ultrapure water. All Milli-Q® ultrapure water samples (resistivity of 18.2 $\text{M}\Omega\text{-cm}$ and TOC below 5 ppb) from the Merck water purification system were analyzed immediately after water collection.

Conclusion

The importance of reagent water quality for toxic element analyses was discussed and low levels of elements in ultrapure water produced by a Milli-Q® water purification system were demonstrated. Laboratories performing trace element analysis can rely on Milli-Q® ultrapure water purification systems to meet their stringent requirements for the highest purity water for their experiments. Choosing ultrapure water from a Milli-Q® system for trace element analyses will help to ensure the generation of high quality data.

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Table 1. The levels of elements in ng/L in freshly produced ultrapure water measured under normal laboratory conditions (not in clean room).

Element	Milli-Q® water BECs (ppt)	ICP-MS DL (ppt)
⁹ Be	0.24	0.28
⁵² Cr	2.10	0.37
⁵⁵ Mn	2.64	0.14
⁵⁶ Fe	0.60	0.19
⁵⁸ Ni	0.76	0.18
⁶³ Cu	0.19	0.11
⁶⁶ Zn	3.20	1.17
⁷⁵ As	3.10	0.72
¹¹¹ Cd	0.06	0.20
¹²¹ Sb	0.06	0.11
¹³⁷ Ba	3.90	0.50
²⁰² Hg	0.46	0.61
²⁰⁵ Tl	0.50	0.21
²⁰⁸ Pb	1.37	0.33

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