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Analyzing Drinking Water by ICP-MS for Heavy Metal Detection

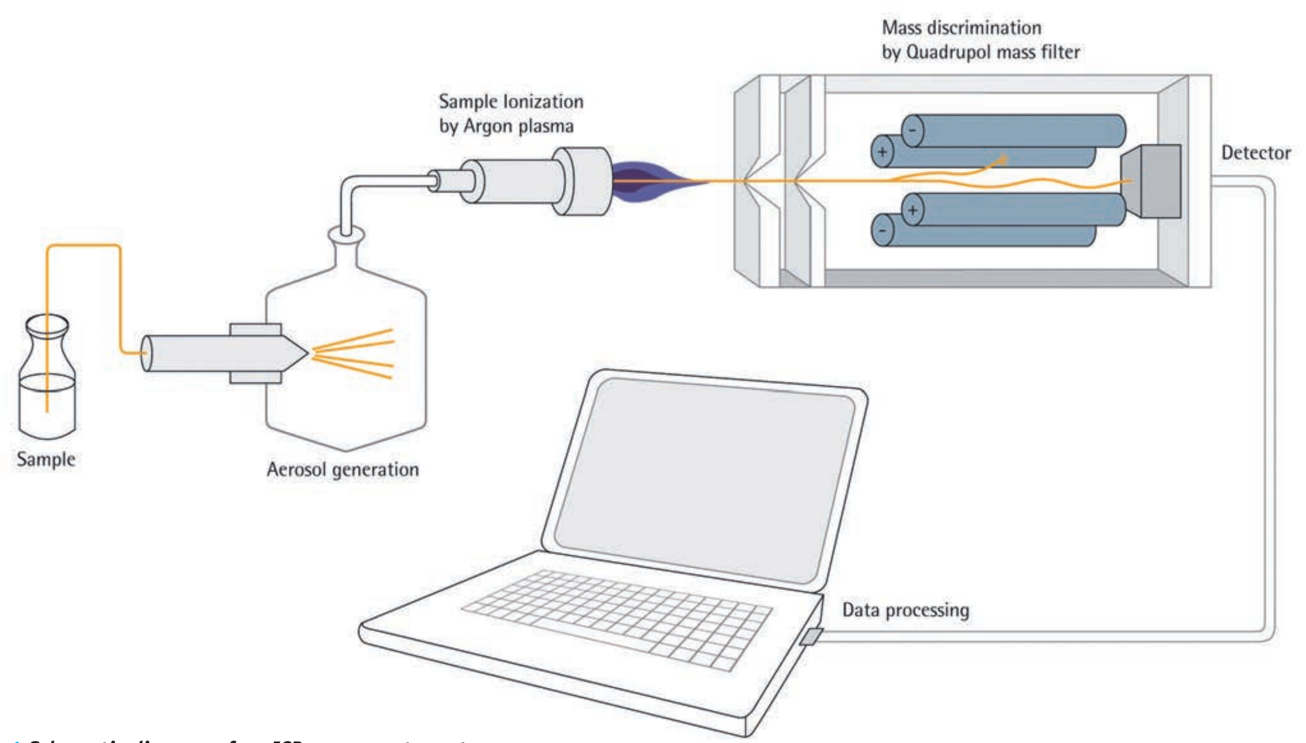
Syringe filter units used in sample preparation

Extremely sensitive analytical instruments capable of assaying analytes in the femtogram range have meanwhile become standard equipment in many laboratories. But their accuracy in analysis of drinking water is decisively influenced by the right choice of syringe filter unit, as described in this article. KLAUS SCHÖNE*

Drinking water is a nutrient essentially required for life. Therefore, its purity is the basic prerequisite for healthy nutrition. Depending on their concentration, individual substances dissolved in water can be toxic and even irreversibly impair people's health. Very slight concentrations are all it takes for a number of heavy metals to have a toxic effect. For instance, when ingested and absorbed by the blood in concentrations as little as 100 µg/L, lead, a common heavy metal, can result in neurophysiological disorders in children, such as persistent intelligence deficits as well as motoric and mental problems [1]. Legislators and health organizations, such as the WHO [2], have therefore issued recommendations and limits for the presence of metals in water. For Europe, the limits given by the Drinking Water Directive 98/83/EC [3] apply.

Regular monitoring of drinking water required

Water treated for use as drinking water is regularly analyzed to monitor the limits established for metals. A basic step in drinking water analysis is preparation of samples during which undissolved constituents are removed from each sample by filtration through a 0.45 µm filter [4]. For this purpose, syringe filter units are used. To prevent distorting the analytical results, such filters may not release any quantities of metal ions relevant for drinking water analysis into the samples to be tested. Afterwards, the metal elements are quantitatively assayed by ICP-AES or by the more sensitive method of ICP-MS. Sampled drinking water is con-



1 Schematic diagram of an ICP mass spectrometer

Sources: Sartorius AG

sidered safe for consumption if its metal values are below the legal limits.

The analytical results obtained in this study are intended to demonstrate that the Minisart syringe filter units tested have a very high degree of purity and the metal ions extracted from them are below the detection limit or considerably lower than the limits established by regulatory requirements. This study is also designed to determine whether Minisart syringe filter units are suitable for use in analysis of drinking water. In this study, ICP-MS technology was selected as the method for analysis of metal ions. Inductively coupled plasma mass spectroscopy (ICP-MS) is a highly sophisticated multi-element analytical technology that is increasingly being used in the pharmaceutical industry, the food and beverage sector and in environmental protection for analysis of trace elements. This technology enables analyses to be performed down into the sub-ppt detection limit range (parts per trillion = one particle per trillion or µg/L).

How does ICP-MS technology work?

ICP-MS technology is based on the principles of atomic emission spectroscopy. In high-temperature argon plasma, elements present in the sample are dissociated into positively charged ions and detected based on their mass-to-charge ratios as they subsequently pass through a mass spectrometer. In principle, ICP-MS consists of the following steps: sample preparation and introduction; aerosol generation; ionization by an argon plasma source; mass discrimination; and identification by the detection system, including data analysis (based on Worley and Kvech [5]). Figure 1 shows a schematic diagram of the steps in the ICP-MS process.

Syringe filter units for sample preparation

The Minisart syringe filter holders used in our study are filtration units designed for a single use and contain a microporous filter membrane between two housing parts made of highly pure plastic. This membrane is thermally sealed inside the housing without any addition of glue. Filter membranes of different materials, such as polyethersulfone (PES), cellulose acetate (CA) and regenerated cellulose (RC) can be used for analysis of metals in drinking water. Common international standards recommend a pore size of 0.45 µm, which is used to remove undissolved solids [4, 6]. In addition to PES and CA with a pore size of 0.45 µm, RC filter material (pore size of 0.2 µm) was also tested in this study. For the housing material, either polypropylene or an acrylic-based multi-polymer mixture is used. During manufacture in compliance with DIN EN ISO 9001, all syringe filter units are automatically integrity-tested. Subsequently, the quality assurance department tests the pressure-hold characteristics and flow rate performance, among other properties, of each lot manufactured.

2 Minisart PES 0.45 µm syringe filter

Test method for extraction of metals

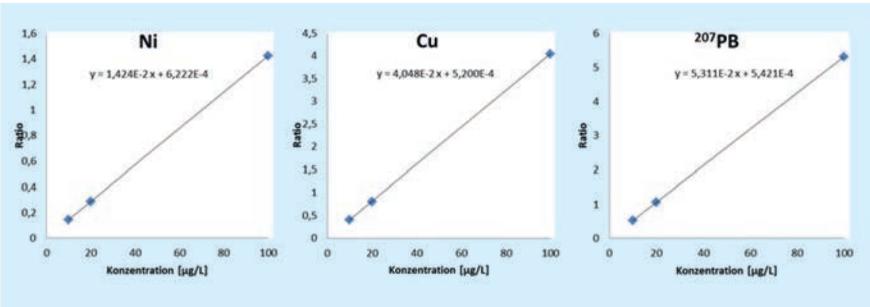
To extract metals, 0.5 mL of ultrapure Type 1 water/cm² of filter area was filtered through each group of three interconnected units of the same Minisart filter type by pressing in the syringe plunger. A 10 mL disposable B. Braun syringe was used per filtration run. Table 1 lists the volumes and eluate quantities used.

ICP-MS technology was employed to analyze the eluate obtained from the syringe filter holders for a total of 38 chemical elements [7]. The instrument used was an Agilent ICP-MS system 7500. The results section of this paper lists only the results of the elements determined according to the requirements of the WHO, as such elements are of special significance for testing drinking water.

Each eluate was collected in a 30 mL Sarstedt vial and acidified using nitric acid and hydrochloric acid for complete mineralization. For the blank, 5 mL of ultrapure water were drawn into a 10 mL single-use syringe and transferred into a Sarstedt vial. Nitric acid and hydrochloric acid were also added to this vial. To prepare for analysis using ICP-MS, rhenium

Table 1: Filter area, water quantity used per filter unit for extraction of metal ions, and eluate quantity per type of Minisart tested

Type of filter	Filter area	Water quantity used per syringe filter unit	Eluate quantity of 3 interconnected filters of each Minisart type
Minisart RC 25, 0.2 µm, type 17764-ACK #41182103	4.9 cm²	2.4 mL	4 mL
Minisart High Flow, PES, 0.45 µm, type 16537-K #41073103	6.1 cm²	3.1 mL	7 mL
Minisart NML, CA, 0.45 µm, type 16555-K #41076103	6.1 cm²	3.1 mL	7 mL



3 Calibration curves of nickel, copper and 207Pb. The y-axis shows the concentration of the respective element in µg/L. The x-axis depicts the ratio of the signals of the respective element to rhenium used as the internal standard (10 µg/L).

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was used as an internal standard at a concentration of 10 µg/L, and the eluate was filled up to a volume of 10 mL.

Calibration of the ICP-MS measurements was performed for all elements examined. Standard solutions of the respective elements together with the instrument blanks (zero values) were injected into the Agilent ICP-MS 7500 system, and the calibration curves were recorded. Figure 3 shows examples a, b and c of the calibration slopes of the elements Ni, Cu and 207Pb as a function of the ratio (corresponds to the signal of the particular element to the signal of rhenium 10 µg/L used as an internal standard) to the concentration of the element expressed in µg/L. The concentration of each element of the tested samples was calculated from the respective calibration curves and is listed in Table 2.

Table 2: ICP-MS values for the Minisart syringe filters tested, as well as limits according to the WHO

	Blank [µg/L]	Minisart High Flow 0.45 µm PES 16537-K [µg/L]	Minisart NML 0.45 µm CA 16555-K [µg/L]	Minisart RC25 0.2 µm RC 17764-K [µg/L]	WHO [µg/L]
Aluminum	0.69	<0.1	<0.1	<0.1	200
Antimony	<0.1	<0.1	<0.1	<0.1	5
Arsenic	<0.1	<0.1	<0.1	<0.1	10
Cadmium	<0.1	<0.1	<0.1	<0.1	3
Chromium	<0.1	<0.1	<0.1	<0.1	50
Copper	<0.1	<0.1	<0.1	0.13	2,000
Iron	0.58	<0.1	1.2	<0.1	–
Lead	<0.1	<0.1	<0.1	<0.1	10
Manganese	<0.1	<0.1	<0.1	<0.1	–
Mercury	<0.1	<0.1	<0.1	<0.1	6
Nickel	<0.1	<0.1	<0.1	0.33	70
Sodium	16	37	69	48	–

Results and discussion of the drinking water samples

The relevant parameters for drinking water analysis are shown in Table 2. The detection limit is 0.1 µg/L, and the recovery of the internal standard was 100% (10 µg/L). For seven out of twelve elements, the values were below this limit, and metal ions could not be detected in the solutions. For five out of twelve elements, metal quantities above the detection limit were found. This applies to the elements Al, Cu, Fe, Ni and Na. Sodium, in particular, consistently showed the highest values (16 µg/L to 69 µg/L) for all samples including the blank. For iron, a blank of 0.58 µg/L was determined, and for Minisart NML, a value of 1.2 µg/L was obtained. Aluminum was detected only in the blank (0.69 µg/L). Copper and nickel were detected for Minisart RC, with values of 0.13 µg/L and 0.33 µg/L, respectively.

The test results clearly show that the concentrations in µg/L (ppt) of the elements examined in the eluate of the various Minisart types are considerably below the limit values required by the WHO, and are also below the detection limit of 0.1 µg/L for many elements. For individual samples, particularly sodium, values above the detection limit were measured. However, these values are several powers of ten below the limits for drinking water and do not add any significant quantities of metal.

PLUS

DIGITAL: You can find more on this topic under the keyword "Sartorius syringe filter units" at www.laborpraxis-worldwide.com. Please visit also: www.sartorius.co.uk/sartoriusUK/en/GBP/products/laboratory/lab-filtration/device-filtration.

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Contamination by syringe filter holders negligible

The results obtained significantly underscore that the Sartorius Minisart syringe filters used do not release any significant quantities of metal ions into samples and are excellently suited for preparing samples, especially for removing undissolved constituents, for the detection of metals in drinking water by ICP-MS and ICP-AES.

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LP-TIP ■ on analysis of heavy metals

A syringe filter unit consists of a porous membrane and a plastic filter housing. For filtration, this unit is attached to a syringe into which liquid is drawn and pressed through the filter by pushing in the plunger. In the process, users should keep several aspects in mind:

- *The filter size needs to be selected according to the volume to be filtered; for example, a 4 mm syringe filter unit should be loaded with less than 1 mL of sample.*
- *For sample preparation, avoid using glass vials as these can leach metals into the sample. Vials made of PFA or HDPE are better suited.*

Literature

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