Challenge
Reliable and robust determination of traces of As, Hg, Sb and Se in water

Solution
Coupling of the PlasmaQuant 9200 Elite with the HS PQ Pro hydride system for matrix-independent determination of environmentally relevant elements down to the sub-µg/L range

Intended audience
Environmental scientists, water quality control professionals, regulatory agencies, water treatment industry professionals, academics/ students in related fields

Analysis of hydride forming elements in water by HR-Array ICP-OES with hydride system HS PQ Pro

Introduction

Clean and fresh drinking water is crucial for sustaining life. To ensure the protection of human health, various authorities, such as the U.S. Government and the European Union, have established standards for drinking water quality. These standards define maximum allowable levels of contaminants. The evaluation of drinking water quality is performed using standard procedures like US-EPA Method 200.7 and ISO 11885.

Inductively coupled plasma -optical emission spectrometry (ICP-OES) is a widely utilized technique for the quality control of drinking water due to its ability to analyze multiple elements, its high dynamic linear range, and its sensitivity. However, conventional sample introduction setups (including nebulizers and spray chambers) often struggle to achieve the necessary analytical performance for toxic elements such as arsenic, antimony, mercury, and selenium.

To enhance the detectability of these elements, a hydride generation sample introduction system can be employed. This system utilizes reducing agents like sodium borohydride to react with the target elements, enabling them to form volatile gaseous hydrides. The use of this hydride generation technique greatly improves the sensitivity, precision, and accuracy of arsenic, antimony, mercury, and selenium determinations.

In this context, we present the capabilities of the HR-Array ICP-OES PlasmaQuant 9200 Elite, coupled with the hydride system HS PQ Pro, for the determination of hydride-forming elements.

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Materials and Methods

Sample preparation

Three sample pre-treatment strategies were employed as the yield of hydride formed varies with the oxidation state of the element. As(III) and Sb (III) were determined after incubation (room temperature (RT), 45 min) with potassium iodide and ascorbic acid in hydrochloric acid (HCl, Suprapur®). For analysis of Hg(II), the samples were stabilized with HCl and nitric acid (HNO_3 , Suprapur®). Se (IV) was detected after addition of HCl (Suprapur®) and incubation (90 °C, 45 min). Calibration standards were prepared accordingly using single-element standards (1000 mg/L, Sigma-Aldrich) and limits of detection (LOD) were obtained from aqueous matrix blanks.

Table 1: Pre-reduction procedures used for sample preparation

Procedure	Element (oxidation state)	Reagents	Conditions
1	As(III)	KI, ascorbic acid, HCl	RT, 45 min
	Sb(III)		
2	Hg(II)	stabilization with HCl/HNO_3	RT
3	Se(IV)	HCl	90 °C, 45 min

Instrument settings and method parameters

For the measurements an ICP-OES system PlasmaQuant 9200 Elite was used. The instrument was equipped with the hydride system HS PQ Pro. The basic unit of the HS PQ Pro accommodates the two mixing and reaction units (Figure 1). The acidified sample and the reducing agent are pumped to the first unit and are mixed there. In the subsequent reaction loop, the sample is reduced and gaseous metal hydride or atomic mercury vapor is released. In the second unit, argon is added to the gas/liquid mixture, which is flushed into the spray chamber.

Table 2: Instrument Settings

Parameter	Specifications 9100 Elite	Specifications 9200 Elite
Plasma power	1350 W	1400 W
Plasma gas flow	14 L/min	10 L/min
Auxillary gas flow	0.5 L/min	
Nebulizer gas flow	0.5 L/min	
Outer tube/Inner tube	Quartz/Quartz	
Injector	Quartz, ID: 2 mm	
Pump tubing	PVC (black, black)	
Sample pump rate	4.0 mL/min	
Delay time	60 s	
Torch position	0 mm	

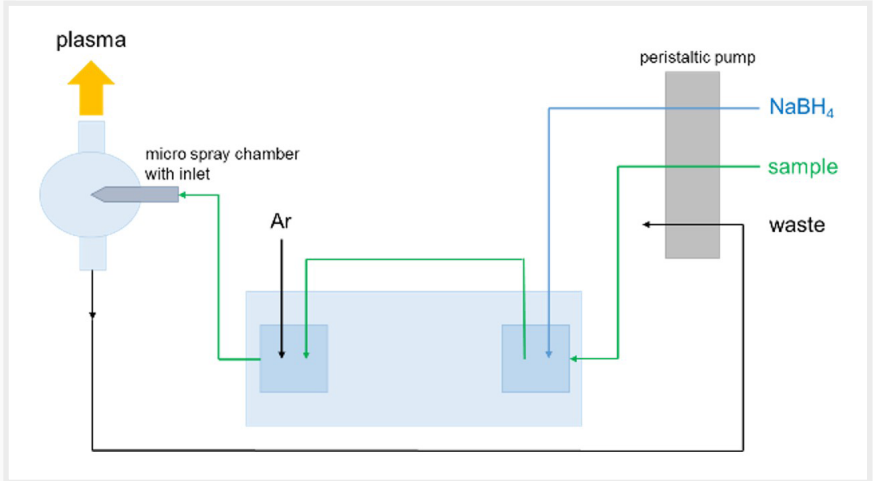

Figure 1: Schematic of the hydride system HS PQ Pro

Table 3: Method parameters

Element	Line [nm]	Plasma view	Integration mode	Read time [s]	Evaluation			
					No. of pixel	Baseline fit	Polyn. degree	Correction
As	188.979	axial	Peak	10	3	static	auto	-
Hg	184.886	axial	Peak	10	3	ABC[1]	auto	-
Sb	217.581	axial	Peak	10	3	static	auto	-
Se	196.028	axial	Peak	10	3	static	auto	-

[1] **A**utomated **B**aseline **C**orrection

Calibration

Table 4: Concentration of calibration standards for the analysis

Element	Unit	Cal. 0	Std. 1	Std. 2	Std. 3	Std. 4	Std. 5
As, Hg, Sb, Se	µg/L	0	0.5	1.0	2.0	5.0	10

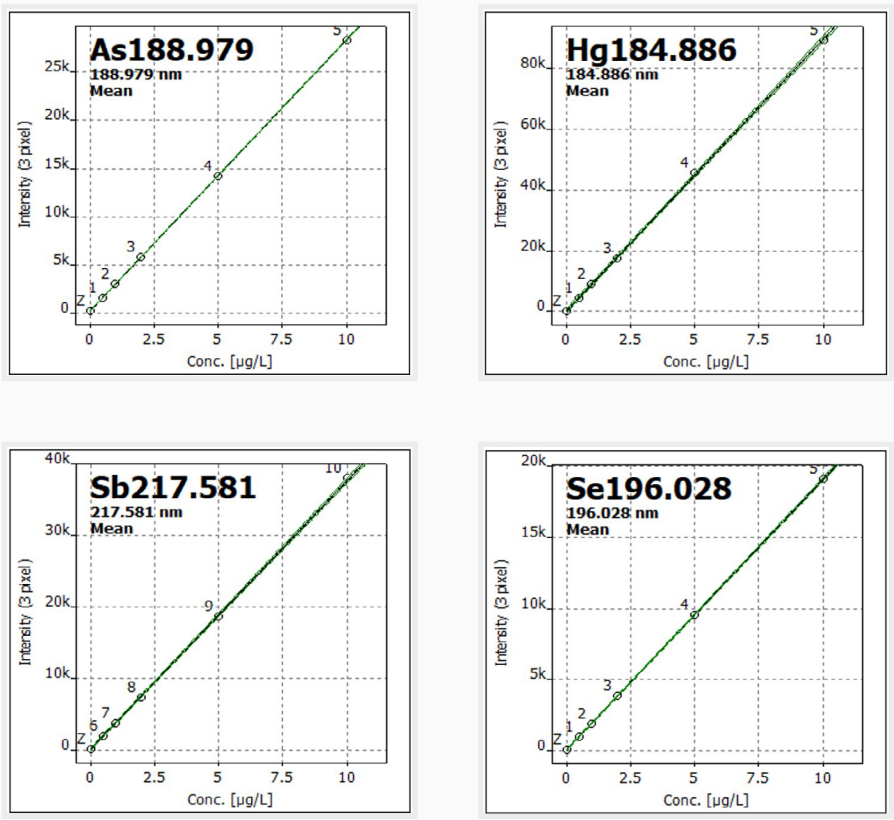

Figure 2: Calibration curves of As, Hg, Sb, and Se

Results and Discussion

Table 5: Limits of detection (LOD) for hydride elements achieved by using the HS PQ Pro in combination with PQ 9200 Elite. Recovery rates of a 1 µg/L spike to a tap water sample are presented.

Element/Line	LOD [µg/L]	Measured in tap water samples[µg/L]	Measured value after spike [µg/L]	Recovery [%]
As188	0.033	0.086	1.07	98
Hg184	0.007	0.031	0.946	92
Sb217	0.035	0.049	1.01	96
Se196	0.034	0.051	1.04	99

Table 6: Quantitative results for standard reference materials (SRMs) NIST1640a and 1643f

Element/ Line	NIST 1640a				NIST 1643f			
	Certified [µg/L]	Measured [µg/L]	Recovery [%]	RSD [%]	Certified [µg/L]	Measured [µg/L]	Recovery [%]	RSD[1] [%]
As188	8.075	7.954	99	0.5	57.42	56.96	99	0.2
Hg184	-	-	-	-	-	-	-	
Sb217	5.105	5.244	103	0.4	55.45	54.27	98	0.5
Se196	20.13	19.24	96	0.3	11.70	11.30	97	1.0

[1] Relative standard deviation

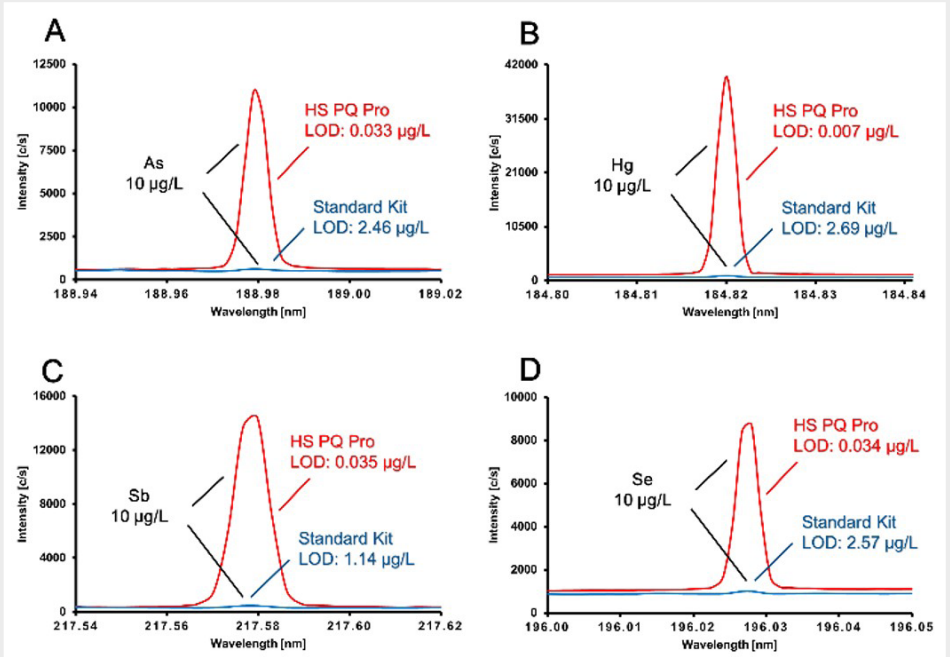

Figure 3: Comparison of signal intensities and limits of detection (LOD) achieved by either using a regular sample introduction (blue, conical nebulizer and cyclonic spray chamber) or hydride system HS PQ Pro (red) of As188 (A), Hg184 (B), Sb 217 (C) and Se196 (D)

Summary

With the simple addition of the HS PQ Pro, the unique sensitivity of the HR-Array ICP-OES PlasmaQuant 9200 Elite can be further improved achieving detection limits lower than 40 ng/L for the hydride elements antimony, arsenic, mercury and selenium. This provides an easy to implement solution for laboratories which require improved detection limits for hydride forming elements in order to ensure the quality of water.

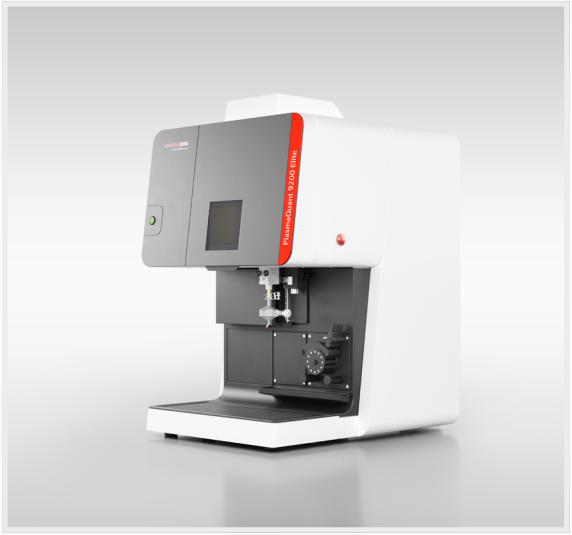

Figure 4: PlasmaQuant 9200 Elite

Recommended device configuration

Table 7: Overview of devices, accessories, and consumables

Article	Order number	Description
PlasmaQuant 9200 Elite	818-09201-2	High resolution ICP-OES
Standard Kit	810-88040-0	Sample introduction system recommended for general samples
Hydride system HS PQ Pro for PlasmaQuant 9x00 Series	810-88032-0	Hydride system for ICP-OES with continuous addition of reducing agent for the determination of hydride-forming elements and other elements from the same run (without interchanging of the sample introduction system)

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