ENVIRONMENTAL ANALYSIS

DETERMINATION OF ELEMENTS IN DRINKING WATER AS PER BUREAU OF INDIAN STANDARDS 10500, 14543 & 13428 USING THE AGILENT 5110 ICP-OES

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Authors

Gaurav Kapadnis

Vinay Jain

Samir Vyas

Agilent Technologies India
Centre of Excellence (CoE),
Manesar, India

ABSTRACT

This solution note highlights a total solution for the quantitation of trace and toxic elements in drinking water, packaged drinking water and natural mineral water using the Agilent 5110 ICP-OES, following the Bureau of Indian Standards (BIS) Drinking Water Specifications IS 10500, IS 14543 and IS 13428 respectively. In order to meet the required detection limits for As, Se, Sb and Hg, a specific sample introduction system, the Multimode Sample Introduction System (MSIS) was used. Water standard reference material SRM 1643d was analyzed and showed excellent recoveries.

INTRODUCTION

Due to an increased awareness in food and drinking water safety, regulatory authorities have stringent regulations in place. There are a wide range of elements that have the potential to cause adverse effects on human health if present in drinking water. Heavy metals have become of particular interest in recent years within the framework of environmental investigation. The Bureau of Indian Standards (BIS) sets regulatory limits for the amounts of contaminants in drinking water (IS 10500), packaged drinking water (IS 14543) and natural mineral water (IS 13428).

Currently in India, laboratories are performing elemental analysis of drinking water from low µg/L to high mg/L concentrations using a range of analytical techniques, such as Flame (FAAS) or Graphite Furnace Atomic Absorption Spectrometry (GFAAS) for the lower concentration trace elements such as Cd and Pb, while Hydride Generation and Cold Vapor AAS are used for elements such as As, Se, Sb and Hg in the µg/L range. FAAS can require different burners (Air-Acetylene / Nitrous Oxide-Acetylene) for the different elements. Some labs are even performing water pre-concentration to achieve the desired detection limits. There is now a need for a more simplified routine approach with a high confidence in the results. Agilent's 5110 ICP-OES offers the complete package allowing high throughput analysis, with excellent repeatability and precision, that meets the BIS requirements, without sample pre-concentration, enabling laboratories to analyze more samples per day. A list of elements with their maximum allowable concentrations as per the different BIS regulations is shown in Table 1.

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Table 1. Bureau of Indian Standards Regulations

Aluminium	Aluminium	IS 13428	IS 14543	IS 10500
		mg/L		
Aluminium	**Al**	NA	0.03	0.03
Antimony	**Sb**	0.005	0.005	NA
Arsenic	**As**	0.05	0.05	0.01
Barium	**Ba**	1.0	1.0	0.7
Boron	**B**	5.0	5.0	0.5
Cadmium	**Cd**	0.003	0.01	0.003
Calcium	**Ca**	100	75	75
Chromium	**Cr**	0.05	0.05	0.05
Copper	**Cu**	1.0	0.05	0.05
Iron	**Fe**	NA	0.1	0.3
Lead	**Pb**	0.01	0.01	0.01
Magnesium	**Mg**	50	30	30
Manganese	**Mn**	2.0	0.1	0.1
Mercury	**Hg**	0.001	0.001	0.001
Molybdenum	**Mo**	NA	NA	0.07
Nickel	**Ni**	0.02	0.02	0.02
Selenium	**Se**	0.05	0.01	0.01
Silver	**Ag**	0.01	0.01	0.1
Sodium	**Na**	150	200	NA
Zinc	**Zn**	5.0	5.0	5.0

NA : Not applicable as per BIS

ANALYTICAL TECHNIQUE

Instrumentation:

Analysis was performed using an Agilent 5110 Synchronous Vertical Dual View (SVDV) ICP-OES. The SVDV configuration has the capability to run in axial, radial, vertical dual view and synchronous vertical dual view modes for full flexibility in the determination of major, minor and trace elements. Unique Dichroic Spectral Combiner (DSC) technology delivers the fastest analyses and the lowest gas consumption per sample. The sample introduction system used was a single-pass glass cyclonic spray chamber, a seaspray nebulizer, white-white peristaltic pump tubing and a standard 1.8 mm injector torch. A Multimode Sample Introduction System (MSIS) was used for the analysis of hydride forming elements (As, Se, Hg and Sb). An SPS 4 autosampler was used in both setups. The instrument uses a solid state RF (SSRF) system operating at 27 MHz to deliver robust plasma capable of excellent long term analytical stability. The Agilent 5110 ICP-OES uses the VistaChip II detector. This is a high speed (1MHz) CCD detector that enables fast warmup, high throughput, high sensitivity, has a large dynamic range of up to 8 orders of magnitude and provides full wavelength coverage from 167–785 nm from a single entrance slit. The instrument operating conditions used are listed in Table 2.

Table 2. Agilent 5110 SVDV ICP-OES operating parameters

Parameter	Setting Value
Read time (s)	5
Replicates	3
Sample uptake delay (s)	25
Stabilization time (s)	10
Rinse time (s)	15
Pump Speed (rpm)	12
Fast pump (rpm)	80
RF power (kW)	1.20
Aux flow (L/min)	1.0
Plasma flow (L/min)	12.0
Nebulizer flow (L/min)	0.7
Viewing height (mm)	8
Background Correction	Fitted

Sample and Standard Preparation:

The Calibration standards for As, Hg, Se and Sb were prepared using single element NIST traceable standards. The samples were prepared with conc. HCl and reduction was carried out using 0.6% $NaBH_4$ and 0.5% NaOH. They were analyzed on the Agilent 5110 ICP-OES using the Multimode Sample Introduction System (MSIS). MSIS is a simple plug and play sample introduction system that replaces the existing spray chamber and has the ability to nebulize liquid sample and create volatile hydrides simultaneously. It can be used in one of three modes, as shown in Figure 1. For this work, the four hydride elements were analyzed simultaneously using the MSIS but separately from the other elements. Peristaltic pump tubing for sample and reagent, Black-Black (Agilent catalog # 3710027200), peristaltic pump tubing for drain, Black-White (Agilent catalog # 3710068900).

A. Liquid Nebulization Only

B. Hydride Generation Only

C. Simultaneous Liquid Nebulization and Hydride Generation

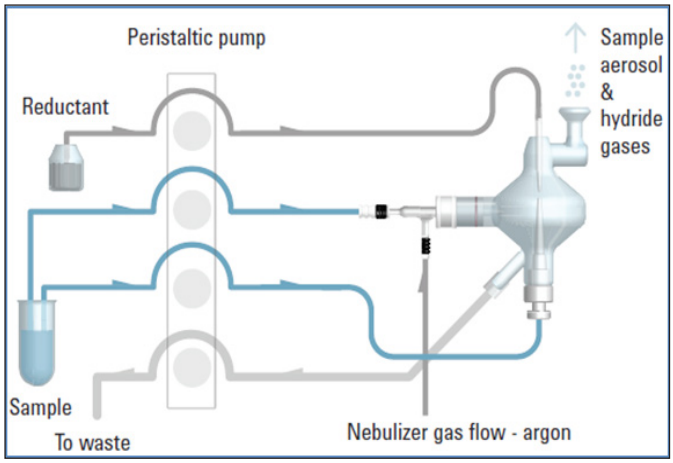

Figure 1. The Multimode Sample Introduction System (MSIS) operation

For the other elements, standards were prepared from multi element NIST traceable standards and diluted with 5% HNO3. They were introduced onto the system using the single-pass glass cyclonic spray chamber fitted with the seaspray nebulizer.

The accuracy and precision of the method was assessed by analyzing the trace elements in water Standard Reference Material 1643d (Traceable to NIST). The water analysis workflow is depicted in Figure 2. Calibration curves for As, Cd, Pb and Hg are shown in Figure 3.

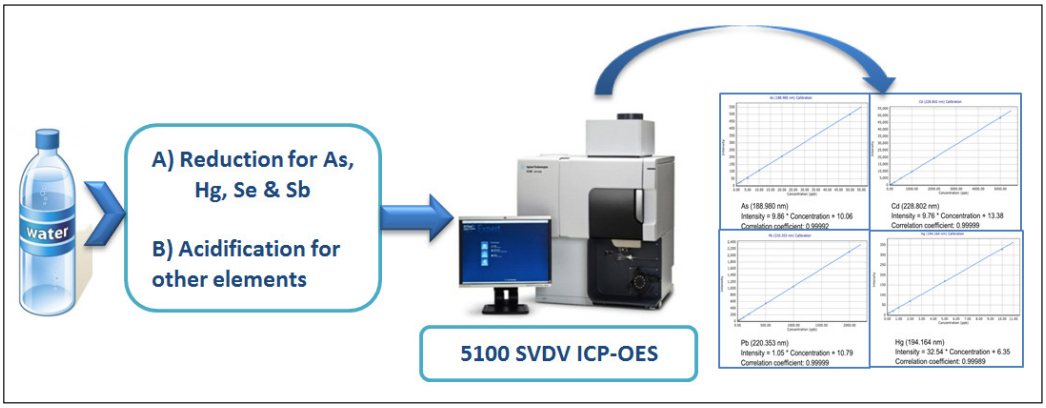

Figure 2. Schematic of water analysis workflow using the Agilent 5110 ICP-OES

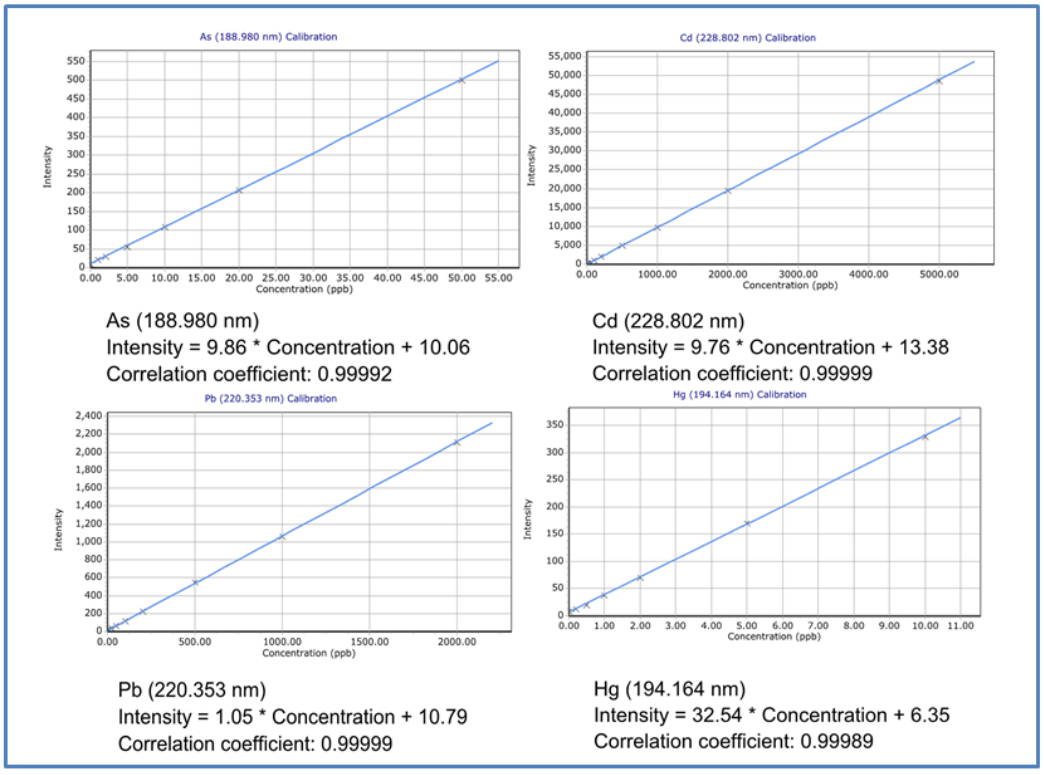

Figure 3. Calibration Curves for As, Cd, Pb and Hg

RESULTS AND DISCUSSIONS

Method Detection limits (MDL):

The Method Detection Limits (MDL) for each element are shown in Table 3. Elements were measured simultaneously in the axial and radial views by the DSC and excellent detection limits were obtained. Method detection limits (MDLs) were established by analyzing eight replicate injections of the calibration blank and multiplying the obtained standard deviation by three.

Table 3. Method Detection limits (µg/L)

Element	Wavelength (nm)	MDL (µg/L)
Aluminum	396.152 nm	1.2
Antimony*	206.834 nm	1.7
Arsenic*	188.980 nm	0.5
Barium	455.403 nm	0.1
Boron	249.772 nm	3.0
Cadmium	228.802 nm	0.4
Calcium	422.673 nm	4.0
Chromium	267.716 nm	0.7
Copper	327.395 nm	0.4
Iron	238.204 nm	0.4
Lead	220.353 nm	3.0
Magnesium	285.213 nm	5.1
Manganese	257.610 nm	0.1
Mercury*	194.164 nm	0.2
Molybdenum	202.032 nm	1.2
Nickel	231.604 nm	3.1
Selenium*	196.026 nm	0.1
Silver	328.068 nm	6.0
Sodium	589.592 nm	10.1
Zinc	213.857 nm	1.1

* Using MSIS

Standard Reference Material Recoveries

The results for the analysis of the standard reference material SRM 1643d are given in table 4. Excellent recoveries for all elements were obtained, demonstrating the method repeatability and capability using the Agilent 5110 SVDV ICP-OES to analyze trace as well as major elements in drinking water

Table 4. Recoveries for various elements in water SRM 1643d

Element	Certified Value (µg/L)	Measured n=3 (µg/L)	SD (µg/L)	Recovery (%)
Al	127.6	129.0	0.89	101.1
Sb	54.10	54.49	9.84	100.7
As	56.02	56.19	1.73	100.3
Ba	506.5	512.3	2.70	101.1
B	144.8	144.0	1.70	99.4
Cd	6.47	6.42	0.44	99.2
Ca	31040	29650	326	95.5
Cr	18.53	19.29	0.72	104.1
Cu	20.50	21.50	0.99	104.9
Fe	91.20	89.1	1.10	97.7
Pb	18.15	17.76	1.86	97.9
Mg	7989	8420	97.2	105.4
Mn	37.66	37.74	0.54	100.2
Mo	112.9	113.3	1.10	100.4
Ni	58.10	57.12	2.75	98.3
Se	11.43	11.22	2.72	98.2
Ag	1.27	1.19	2.79	93.7
Na	22070	23500	243	106.5
Zn	72.48	75.37	0.75	104.0

CONCLUSIONS

The Agilent 5110 SVDV ICP-OES with DSC combines the sensitivity benefits of axial plasma with the robust qualities of radial plasma into a single platform so that all wavelengths can be detected simultaneously in a single measurement. This gives greater precision, faster analysis times and reduced argon gas consumption.

For drinking water analysis, the Agilent 5110 SVDV ICP-OES in combination with the MSIS, offers a total solution that replaces multiple analytical approaches with a simpler, faster, more precise and cost effective approach. It is capable of measuring all required elements in drinking water, whilst meeting the required BIS detection limits of all elements. Sample preparation times are reduced and less sample is required for each analysis. Excellent method detection limits in the µg/L range were obtained for all elements and good recovery results for all elements in SRM 1643d were achieved

REFERENCES

1. Bureau of Indian Standards, IS 10500 (2012): Drinking Water [FAD 25: Drinking Water], with latest amendments.

2. Bureau of Indian Standards, IS 14543 (2004): Packaged Drinking Water (Other than Packaged Natural Mineral Water) [FAD 14: Drinks and Carbonated Beverages], with latest amendments.

3. Bureau of Indian Standards, IS 13428 (2005): Packaged Natural Mineral Water [FAD 14: Drinks and Carbonated Beverages], with latest amendments.

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