

**SPECTRO****AMETEK**

MATERIALS ANALYSIS DIVISION

AN APPLICATION REPORT FROM
SPECTRO ANALYTICAL INSTRUMENTS

ICP-90/1



SPECTRO ARCOS

Analysis of Drinking Water by ICP-OES with Axial Plasma Observation

Introduction

Clean fresh water is essential to life. Unfortunately, since the industrial revolution, waterways and coastal waters have been polluted in the process, harming human health. In the past decades, significant progress in the treatment of sewage and industrial wastes has been made. As a result, levels of most pollutants have decreased and a measurable improvement in water quality has been achieved. To protect human health from the adverse effects of any contamination of water intended for human consumption, the World Health Organization

(WHO) has defined minimum standards in its "Guidelines for drinking water quality"[1]. For the countries of the European Union, "Directive 98/83/EC on the quality of water intended for human consumption"[2] has been passed by the European Council, to define the essential quality standards. In the USA, the quality of drinking water is defined through the "Safe Drinking Water Act"[3] and the "National Primary Drinking Water Regulations"[4] setting maximum levels for contaminants[5].

2 Analysis of Drinking Water by Axial ICP-OES



The multi-element analysis of water is one of the major applications for ICP-OES.

This report demonstrates that the new SPECTRO ARCOS has the required analytical capabilities in terms of sensitivity, precision and accuracy to perform the analysis of metals and trace elements in drinking water. Instrument parameters and line selection are described. Excellent recoveries were found for the standard reference material NIST SRM 1640.

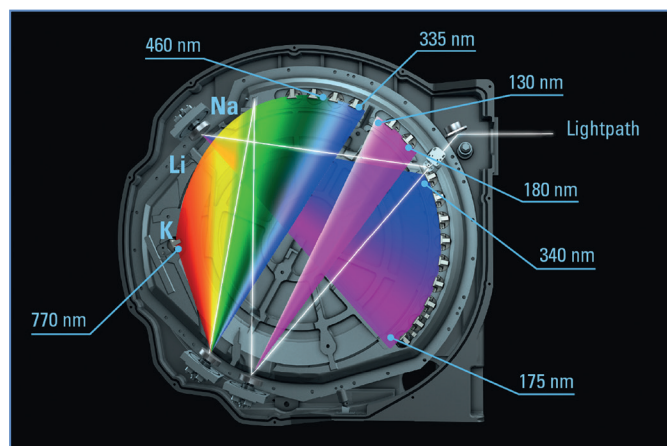
Due to its multi-element determination capability, high linear dynamic range and sensitivity, inductively coupled plasma optical emission spectrometry (ICP-OES) is widely used for the analysis of drinking waters.

The application is described in several ICP-OES standard procedures, such as US-EPA Method 200.7 [6] or ISO 11885 [7]. In this report, the analysis of drinking waters using the SPECTRO ARCOS is described. The report includes line selection, detection limits and studies on precision and accuracy.

Experimental

Instrumentation

All measurements were performed with the SPECTRO ARCOS optical emission spectrometer (SPECTRO Analytical Instruments, Kleve, Germany) with axial plasma observation.



The SPECTRO ARCOS features a Paschen-Runge spectrometer mount, employing the Optimized Rowland Circle Alignment (ORCA polychromator) technique. It consists of two hollow section cast shells, optimized small volume and 32 linear CCD detectors, the wavelength range between 130 and 770 nm can be simultaneously analyzed, allowing the relevant spectrum capture within 2s. Due to the unique reprocessing capabilities of the system, a new measurement is not required even if additional elements or lines are to be determined at a later date.

The optic is hermetically sealed and filled with argon, continuously circulated through a filter, which absorbs oxygen, water vapor and other species. High optical transmission in the VUV is achieved, allowing the determination of non-metals as well as the use of prominent and interference free lines in this region.

The state of the optical system is automatically monitored by utilizing SPECTRO's "Intelligent Calibration Logic (iCAL)," which normalizes the wavelength scale.

An air-cooled, 27.12 MHz, free running type LDMOS ICP-generator is installed, which ensures excellent stability of the forward power even in the case of rapidly changing sample loads. All relevant ICP operating parameters are software controlled, allowing easy selection of the optimum operating conditions. For sample introduction a cyclonic spray chamber and a Seaspay nebulizer was used. The ICP operating parameters were applied as given in Table 1.

Table 1: Typical ICP Operating Conditions

Power	1250 W
Coolant flow	13 L/min
Auxiliary flow	0.7 L/min
Nebulizer flow	0.8 L/min
Plasma Torch	Quartz, demountable, 2.0 mm Injector tube
Spray Chamber	Cyclonic
Nebulizer	Seaspay
Sample aspiration rate	2.0 mL/min
Replicate read time	50 sec/replicate



4 Analysis of Drinking Water by Axial ICP-OES

Calibration Standards

Commercially available standard solutions were used for calibration. The concentrations of the elements are given in Table 2. The QC of the method was performed using NIST standard reference material 1640 "Trace Elements in Natural Water." All solutions were acidified with 1% HNO₃, suprapure quality.

Table 2: Calibration Standards

Element	Std.1 [mg/L]	Std.2 [mg/L]	Std.3 [mg/L]	Std.4 [mg/L]
Ag	0	0.025	0.125	0.25
Al	0	0.1	0.5	1
As	0	0.1	0.5	1
B	0	0.1	0.5	1
Ba	0	0.1	0.5	1
Be	0	0.1	0.5	1
Ca	0	0.1	0.5	1
Cd	0	0.1	0.5	1
Co	0	0.1	0.5	1
Cr	0	0.1	0.5	1
Cu	0	0.1	0.5	1
Fe	0	0.1	0.5	1
Hg	0	0.1	0.5	1
K	0	0.1	0.5	5
Li	0	0.1	0.5	1

Element	Std.1 [mg/L]	Std.2 [mg/L]	Std.3 [mg/L]	Std.4 [mg/L]
Mg	0	0.1	0.5	1
Mn	0	0.1	0.5	1
Mo	0	0.1	0.5	1
Na	0	0.1	0.5	1
Ni	0	0.1	0.5	1
P	0	0.5	2.5	5
Pb	0	0.1	0.5	1
Sb	0	0.1	0.5	1
Se	0	0.1	0.5	1
Si	0	0.1	0.5	1
Sn	0	0.1	0.5	1
Sr	0	0.1	0.5	1
Tl	0	0.1	0.5	1
V	0	0.1	0.5	1
Zn	0	0.1	0.5	1

Results and Discussion

Table 3 shows the selected wavelengths and the limits of detection (LOD) achieved. The LODs were calculated according to the equation[8]:

$$\text{LOD} = 3 \text{ RSD}_b \text{ c}/100 \text{ SBR}$$

Where:

RSD_b - relative standard deviation of 10 replicates of the blank

c - concentration of the standard

SBR - signal to background ratio

Table 3: Limits of detection (LOD) for the selected lines

Element	λ [nm]	LOD (3σ) [$\mu\text{g/L}$]
Ag	328.068	0.22
Al	167.078	0.03
As	189.042	0.98
B	249.773	0.33
Ba	455.404	0.03
Be	313.042	0.06
Ca	396.847	0.02
Cd	214.438	0.05
Cd	226.502	0.1
Co	228.615	0.18
Cr	267.716	0.18
Cu	324.754	0.24
Fe	259.941	0.19
Hg	184.950	0.58
K	766.491	0.90
K	769.896	1.3
Li	670.784	0.03
Mg	279.553	0.015
Mg	279.078	3.4
Mn	257.610	0.02
Mo	202.030	0.32
Na	589.592	0.24
Ni	221.648	0.19
Ni	231.604	0.26
P	177.495	0.81
Pb	168.215	0.95
Pb	220.351	0.84
Sb	206.833	1.1
Se	196.090	1.3
Si	251.612	1.7
Sn	189.991	0.39
Sr	407.771	0.02
Tl	190.864	0.79
V	311.071	0.3
Zn	213.856	0.053

Accuracy

The accuracy and precision of the method were investigated analyzing the standard reference material SRM 1640 (Trace Elements in Natural Water). As shown in Table 4 the measured values and the certified values are in excellent agreement for all elements.

Table 4: Comparison of certified and measured concentrations for NIST SRM 1640

	Certified Conc. [$\mu\text{g/L}$]	Confidence Range [$\mu\text{g/L}$]	Meas. Conc. [$\mu\text{g/L}$]	Recovery [%]
Al	52	± 1.5	53.4	102.7
Ag	7.62	± 0.25	7.71	101.2
As	26.67	± 0.41	26.43	99.1
B	301.1	± 6.1	302.5	100.5
Ba	148.0	± 2.2	150.4	101.6
Be	34.94	± 0.41	36.0	103
Cd	22.792	± 0.96	22.9	100.5
Co	20.28	± 0.31	20.6	101.6
Cr	38.6	± 1.6	36.7	95.1
Cu*	85.2	± 1.2	86.5	101.5
Fe	34.3	± 1.6	32.6	95.0
K*	994	± 27	1099	110.6
Li*	50.7	± 1.4	52.6	103.7
Mn	121.5	± 1.1	118.3	97.4
Mo	46.75	± 0.26	45.8	98.0
Ni*	27.4	± 0.8	30.1	109.8
Pb	27.89	± 0.14	27.3	97.9
Sb	13.79	± 0.42	13.3	96.4
Se	21.96	± 0.51	23.1	105.2
Sr	124.2	± 0.7	128.9	103.8
V	12.99	± 0.97	13.3	102.4
Zn*	53.2	± 1.1	54.1	101.7

* non-certified concentrations (for reference only)

Conclusions

The SPECTRO ARCOS with axial plasma observation offers a simple, fast, accurate, precise and cost-efficient method for the analysis of drinking water. Excellent recoveries were determined for NIST SRM 1640. In conjunction with an autosampler, the SPECTRO ARCOS can be fully automated for water analysis. Independent from the number of lines and elements, an analysis (including 3 replicates pre-flush, and method rinse) can be performed in less than 3 minutes.

References

- [1] World Health Organization 1998, Guidelines for drinking water quality, Second Edition
- [2] Council Directive 98/83/EC of 3rd November 1998 on the quality of water for human consumption
- [3] Safe Drinking Water Act, 42 USC Sec. 300f
- [4] 40 CFR 141 National Primary Drinking Water Regulations
- [5] EPA 816-F-03-016 - List of Contaminants and their Maximum Contaminant Levels (MCL)
- [6] Method 200.7 Determination of metals and trace elements in water and wastes by inductively coupled plasma atomic emission spectroscopy Rev. 4.4
- [7] ISO 11885 (2009)
- [8] P. W. J. M. Boumans, Spectrochim. Acta 46B, 431 (1991)



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