AN APPLICATION REPORT FROM
SPECTRO ANALYTICAL INSTRUMENTS

ICP-182, Rev. 0

SPECTRO **GENESIS**

Analysis of Unpolluted Aqueous Solutions by ICP-OES using Dual-Side-On Interface Plasma Observation

Introduction

Water is indispensable for life on earth, - it should be valued and kept clean. Inductively coupled plasma optical emission spectroscopy (ICP-OES) is an excellent analytical method to ensure the quality of water and examine pollution in aqueous solutions, due to its multi-element determination capability, high dynamical linear range and sensitivity.

This report describes the principle methodology for the analysis of aqueous solutions. It presents typical detection limits for a wide range of elements as well as studies on accuracy, analyzing a certified reference material.

Instrumentation

All measurements were performed with the SPECTRO GENESIS ICP optical emission spectrometer (SPECTRO Analytical Instruments, Kleve, Germany) with dual side-on plasma observation. It enables an average factor 2 enhanced sensitivity compared to single radial plasma observation, while eliminating typical axial view interferences. In addition, it offers a high matrix compatibility, linear dynamic range and precision without the need to change the plasma observation mode during analysis. The SPECTRO GENESIS features a Paschen-Runge spectrometer mount, employing the proprietary Optimized Rowland Circle Alignment (ORCA) technique. Consisting of two hollow section cast shells, optimized small volume and 15 linear CMOS detectors, the wavelength range between 175 and 770nm can be analyzed, allowing complete spectrum capture. 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 point in time.

For UV access (<200 nm), the optical system can be purged with argon. The purge rate during normal operation is 0.5 L/min.

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 SeaSpray nebulizer and a cyclonic spray chamber were used. The ICP operating conditions are given in Table 1.

Table 1: Typical ICP operating conditions for Dual Side-On Interface and Side-On Interface

Power	1000 W
Observation mode	Dual-Side-On
Coolant flow	13.0 L/min
Auxiliary flow	0.80 L/min
Nebulizer flow	0.88 L/min
Plasma torch	Quartz, fixed, 1.8 mm injector tube
Spray chamber	Cyclonic
Nebulizer	SeaSpray
Sample aspiration rate	2 mL/min
Replicate read time	49 s per replicate

Calibration

For calibration, a commercially available multi-element standard [1] and single element standards [3] were used and acidified with 1% HNO_3 (v/v) [2]. The concentrations of the resulting calibration standards are given in the following table 2.

Table 3: Calibration standards

Element	Blank [mg/L]	Std.1 [mg/L]	Std.2 [mg/L]	Std.3 [mg/L]	Std.4 [mg/L]	Std.5 [mg/L]	Std.6 [mg/L]
Ag	0	0.05	0.25	0.5			
Al	0	0.2	1	2			
As	0	0.2	1	2			
Au	0				0.2	1	2
B	0	0.2	1	2			
Ba	0	0.2	1	2			
Be	0	0.2	1	2			
Ca	0	0.2	1	2			
Cd	0	0.2	1	2			
Ce	0				0.2	1	2
Co	0	0.2	1	2			
Cr	0	0.2	1	2			
Cu	0	0.2	1	2			
Fe	0	0.2	1	2			
Ge	0				0.2	1	2
Hf	0				0.2	1	2
Hg	0	0.2	1	2			
K	0	0.2	1	2			
Li	0	0.2	1	2			
Mg	0	0.2	1	2			
Mn	0	0.2	1	2			
Mo	0	0.2	1	2			
Na	0	0.2	1	2			
Ni	0	0.2	1	2			
P	0	0.2	1	2			
Pb	0	0.2	1	2			
Pd	0				0.2	1	2
Pr	0				0.2	1	2
Pt	0				0.2	1	2
Ru	0				0.2	1	2
Sb	0	0.2	1	2			
Se	0	0.2	1	2			
Si	0	0.2	1	2			
Sn	0	0.2	1	2			
Sr	0	0.2	1	2			
Ti	0				0.2	1	2
Tl	0	0.2	1	2			
V	0	0.2	1	2			
W	0				0.2	1	2
Zn	0	0.2	1	2			
Zr	0				0.2	1	2

Results

Table 3 shows the selected wavelengths and the limits of detection (LOD) achieved. The LODs were calculated according to the equation [4]:

$$LOD = 3\ RSD_b\ c/\ 100\ SBR$$

Where:

RSD_b: - relative standard deviation of 10 replicates of the blank (in %)

c: - concentration of the standard

SBR: - signal to background ratio

Table 3: Typical Limits of Detection (LOD) for the selected lines with Dual-Side-On Interface.

Element	Wavelength [nm]	LOD (3σ) Dual-Side-On [µg/L]
Ag	328.068	1.0
Al	396.152	3.5
As	189.042	4.8
Au	242.795	1.9
B	249.773	0.7
Ba	455.404	0.07
Be	313.042	0.04
Ca	393.366	0.1
Cd	226.502	0.4
Cd	228.802	0.5
Ce	418.66	4.0
Co	228.616	0.8
Cr	205.618	0.6
Cu	324.754	0.7
Fe	259.941	0.6
Ge	265.118	4.2
Hf	264.141	1.8
Hg	184.95	1.8
Hg	194.227	1.9
K	766.491	26
Li	670.78	1.0
Mg	279.553	0.04

Element	Wavelength [nm]	LOD (3σ) Dual-Side-On [µg/L]
Mn	257.611	0.1
Mo	202.095	0.8
Na	589.592	4.0
Ni	221.648	1.1
P	177.495	3.4
P	178.287	4.6
Pb	220.353	6.5
Pd	324.27	6.6
Pr	417.939	3.7
Pt	177.708	5.3
Ru	240.272	3.2
Sb	206.833	5.2
Se	196.09	6.7
Si	251.612	2.8
Sn	189.991	2.5
Sr	407.771	0.02
Ti	334.941	0.3
Tl	190.864	4.3
V	311.071	0.8
W	207.911	3.2
Zn	213.856	0.4
Zr	339.198	0.6

Accuracy

The accuracy of the method was investigated by analyzing the standard reference material NIST 1643f. Table 4 shows measured and certified values, which are in excellent agreement for all elements. Even Ca, Mg and Na, with concentrations above the calibration range, could be determined with excellent accuracy by extrapolation due to the large linear dynamic range of the SPECTRO GENESIS.

Table 4: Comparison of certified and measured concentrations for NIST1643f

Element	Wavelength [nm]	Certified Concentrations [µg/L]	Measured Concentrations [µg/L]	Recovery [%]
Ag	328.068	0.977 ± 0.0055	<LOD	--
Al	308.215	133.8 ± 1.2	135	101%
As	189.042	57.42 ± 0.38	56.7	99%
B	249.773	152.3 ± 6.6	146	96%
Ba	455.404	518.2 ± 7.3	498	96%
Be	313.042	13.67 ± 0.12	13.2	97%
Ca	315.887	29430 ± 330	27583	94%
Cd	226.502	5.89 ± 0.13	5.59	95%
Co	228.616	25.3 ± 0.17	23.3	92%
Cr	205.618	18.5 ± 0.10	17.6	95%
Cu	324.754	21.66 ± 0.71	20.2	93%
Fe	259.941	93.44 ± 0.78	88.3	94%
K	766.491	1932.6 ± 9.4	1909	99%
Li	670.78	16.59 0.35	16.1	97%
Mg	285.213	7454 ± 60	7350	99%
Mn	257.611	37.14 ± 0.60	36.8	99%
Mo	202.095	115.3 ± 1.7	111	96%
Na	589.592	18830 ± 250	19044	101%
Ni	221.648	59.8 ± 1.4	55.1	92%
Pb	220.353	18.488 ± 0.084	17.1	92%
Sb	206.833	55.45 ± 040	50.8	92%
Se	196.09	11.583 ± 0.081	11.5	99%
Sr	407.771	314 ± 19	314	100%
Tl	190.864	6.892 ± 0.035	6.77	98%
V	311.071	36.07 ± 0.28	34.6	96%
Zn	213.856	74.4 ± 1.7	68.6	92%

Conclusion

The SPECTRO GENESIS with dual-side-on interface offers a simple, fast, accurate, precise, and cost-efficient method for the analysis of aqueous solutions. Excellent recoveries were determined for NIST 1643f. In conjunction with an autosampler, the SPECTRO GENESIS can be fully automated. Independent from the number of lines and elements, an analysis (including three replicates and pre-flush) can be performed in less than four minutes.

References

[1] Bernd Kraft GmbH Duisburg, Germany

[2] HNO_3 Suprapur®, 65%, Merck, Darmstadt, Germany

[3] Inorganic Ventures, Christiansburg, Virginia

[4] P.W.J.M. Boumans, Spectrochim. Acta Part B: Atomic Spectroscopy 46, 431-445 (1991)

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