SPECTRO REPORT

Application Note ICP-4
SPECTRO CIROS^{CCD}

TRACE ANALYSIS IN WATER SAMPLES

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INTRODUCTION

One of the most frequent tasks applying ICP-OES is the analysis of water samples, in the context of various environmental and industrial applications. These include the analysis of drinking waters, surface and waste waters, but also of process and cooling water. A great number of these samples is considered unpolluted, displaying a total salt content of less than 2g/L.

The major advantages of ICP-OES are the wide linear range and the multielement capabilities, allowing the determination of major and trace components in one run. The SPECTRO CIROS^{CCD} shows excellent performance in aqueous solutions. Not only is complete sample information obtained within merely a few seconds, but the detection sensitivity ensures the precise and accurate determination of trace metals in the low µg/L level.

EXPERIMENTAL

Instrumentation

All measurements were performed with a SPECTRO CIROS^{CCD} spectrometer.

The SPECTRO CIROS^{CCD} combines proven SPECTRO ICP-technology with new innovative solutions. The spectrometer in Paschen-Runge mounting consists of a double-grating optical system with 22 CCD detectors, located in an Ar-filled chamber. The spectral range between 125 nm and 770 nm is covered, allowing complete scans within 3s.

For the first time an air-cooled ICP-generator, based on the proven, robust free-running 27.12 MHz system, has been installed. All relevant excitation parameters are now controlled by the software, such as all gas flows (by means of mass flow controllers) and positioning of the torch (using stepper motors) in front of the optical plasma interface. Optimum operating conditions are therefore easily set.

The applied operating parameters are given in table 1.

Table 1: Operating parameters

Generator	
Power	1400 W
Free-running at	27,12 MHz
Sample Introduction	
Nebulizer	Cross-flow (SPECTRO)
Spray chamber	Double pass, Scott type (SPECTRO)
Sample uptake rate	2 mL/min
Gas Flows	
Outer gas	12 L/min
Intermediate gas	0,5 L/min
Nebulizer gas	0,9 L/min

Procedure

Detection limits were determined using blank and single element standard solutions. The concentrations of the upper standard were 2 mg/L for all elements except the halogens, for which 20 mg/L standards were used. An integration time of 15s was applied, within which the full spectrum was recorded.

Detection limits

The detection limits (LOD) listed in table 2 were determined using the threefold standard deviation of the blank solution. The typical deviation of the background signal was found to be less than 1%.

Table 2: Limits of detection for selected elements

Element	Line (nm)	LOD (µg/L)
Ag	328.068	2.1
Al	167.078	0.07
As	189.042	7.4
B	249.678	1.6
Ba	455.403	0.08
Bi	223.061	6.4
Br	154.065	25
Ca	396.847	0.04
Cd	226.502	0.3
Cl	134.724	102
Co	228.616	0.7
Cr	267.716	0.78
Cu	324.754	1.5
Fe	259.94	0.8
Ga	141.444	2.8
Ge	164.917	3.2
Hg	184.95	2.1
I	142.549	48
In	158.583	0.5
K	766.491	2.3

Element	Line (nm)	LOD (µg/L)
Li	670.784	0.03
Mg	279.553	0.02
Mn	257.61	0.1
Mo	202.03	1.1
Na	589.592	0.4
Ni	231.604	0.7
P	177.495	3.6
Pb	220.353	3.1
Pt	177.708	4.6
S	180.731	7.3
Sb	231.147	0.9
Se	196.069	8.9
Si	251.611	3
Sn	189.991	1.4
Sr	407.771	0.03
Te	214.281	8.5
Tl	190.864	3.6
V	311.071	1.1
Zn	213.856	0.2

CONCLUSIONS

The SPECTRO CIROS^{CCD} is excellently suited for the determination of trace elements in aqueous solutions of low salt content. One of the major benefits is the fast simultaneous scan, allowing complete sample information within seconds. In addition, low detection limits can be achieved, in many cases taking advantage of the lines free from interferences in the VUV-region.

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