

Application Note ICP-6 SPECTRO CIROS^{CCD}

APPLICATION OF PROMINENT LINES IN THE SPECTRAL RANGE BETWEEN 125 NM AND 180 NM

P. Heitland, D. Ardelt, T. Brandt,
K. Krengel-Rothensee and N. Wieberneit
Spectro Analytical Instruments GmbH & Co. KG



INTRODUCTION

The use of the VUV-region between 125 nm and 180 nm in ICP-OES offers many analytical possibilities. Most of the commercially available ICP-OES instruments do not operate in this wavelength range, even though many intense lines are found here. The SPECTRO CIROS^{CCD} is the first ICP-OES instrument on the market applying CCD-technology in the VUV-region. The use of analytical lines in the VUV not only allows the determination of elements otherwise affected by significant interferences, but also enables the determination of non-metals, a field hardly applied in ICP-OES. The application of prominent emission lines in the VUV is discussed in this application note.

EXPERIMENTAL

Instrumentation

The 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 3 s.

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.

Typical operating parameters are given in table 1.

Table1: Operating parameters

Generator Power	Free-running at 27,12 MHz 1400 W
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 15 s was applied, within which the full spectrum was recorded.

RESULTS AND DISCUSSION

The detection limits calculated from the threefold standard deviation of the blank measurement are given in table 2.

Table 2: Limits of detection for elements displaying prominent lines in the VUV in aqueous solutions

Element	Line (nm)	LOD (µg/L)
Al	167.078	0.07
B	136.246	6.9
Bi	143.683	31
Bi	153.317	37
Br	148.845	68
Br	154.065	25
Br	157.480	230
Br	163.340	212
Cl	133.573	805
Cl	134.724	102
Cl	135.165	206
Cl	136.345	549
Ga	141.444	2.8
Ge	164.917	3.2
I	178.276	7
I	142.549	48
I	161.760	35
In	158.583	0.5
Ni	174.155	5.6
P	138.147	69
P	168.599	46
Pb	168.215	4.5
Pt	177.708	4.6
S	166.668	18
S	142.503	47
Si	152.672	7.4
Sn	140.045	5.6
Sn	147.516	3.7
Sn	175.790	3.4
Te	170.001	11
Tl	132.171	39

Many of the prominent analytical lines found above 180 nm are affected by severe spectral interferences. The availability of intense lines in the VUV-range below 180 nm allows the user to resort to lines free from interferences.

One impressive example is the determination of Tl. The most intense analytical Tl line at 190.864 nm is severely affected by Ti. Using the spectral line at 132.171 nm, the determination of Tl can be performed free from interferences by Ti, with little loss of performance.

A further example is the determination of I in the presence of P. An interference by P on the most sensitive analytical line of I at 178.276 nm can be avoided using the lines at 142.549 nm and 161.760 nm. This example also demonstrates the exceptional performance of the SPECTRO CIROS^{CCD} in the determination of non-metals, due to the availability of the VUV region.

CONCLUSIONS

Being the only instrument on the market applying CCD technology in the VUV region, the SPECTRO CIROS^{CCD} offers unique analytical possibilities. Not only can spectral interferences be avoided by applying lines free from interferences in the spectral range between 125 nm and 180 nm, but the system also allows the determination of non-metals, which are otherwise literally impossible to determine in the sub-ppm level with conventional ICP-OES systems. In addition, the most intense lines for Br, Cl, Ga, Ge and In are found in the VUV range leading to unmatched detection limits.

A further advantage is the fast, simultaneous scan, yielding complete sample information within a few seconds. Interferences and alternate lines are easily recognized.

