

Application Note ICP-5 SPECTRO CIROS^{CCD}

DETERMINATION OF TRACE ELEMENTS IN SULFURIC ACID

T. Brandt, K. Krengel-Rothensee,
N. Wieberneit and P. Heitland
Spectro Analytical Instruments GmbH



ABSTRACT

In the production of sulfuric acid quality control is often required. In this paper the determination of trace elements in sulfuric acid is described. Sample preparation, line selection and detection limits are discussed. The SPECTRO CIROS^{CCD} is well suited for the rapid determination of the trace elements in sulfuric acid. Low detection limits and good precision are achieved.

EXPERIMENTAL

(a) Instrumentation

The SPECTRO CIROS^{CCD} is equipped with a free-running 27,12 MHz RF-generator. Sample introduction is performed by a cross-flow nebulizer with a double pass spray chamber. Further operating conditions are listed in Table 1. Calibration was performed with a blank solution (20% (V/V) sulfuric acid) and 20 % (V/V) sulfuric acid standard solutions with element concentrations of 1 mg/L, 2 mg/L and 4 mg/L, respectively.

Table 1: ICP operating conditions

Generator	Free-running at 27,12 MHz
Power	1350 W
Sample Introduction	
Nebulizer	„Cross-flow“ (SPECTRO)
Spray chamber	Double pass spray chamber, Scott type (SPECTRO)
Sample uptake rate	2 mL/min
Gas Flows	
Outer gas	12 L/min
Intermediate gas	0,5 L/min
Nebulizer gas	0,95 L/min

(b) Sample Preparation

Concentrated sulphuric acid (98%) was diluted with deionized water in a ratio of 1+4 (V/V). The resulting 20 % (V/V) sulfuric acid can be analyzed directly.

3. RESULTS AND DISCUSSION

The line selection and the detection limits are listed in Table 2. The detection limits are calculated from the standard deviation (3σ) of the blank sample (20 % (V/V) sulfuric acid). The relative standard deviations without internal standardization are between 0,1 and 0,9 % and with the use of Sc as an internal standard in a range of 0,05-0,3 %.

Tab. 2 Line selection and limits of detection (LOD) for several elements in 98 % sulfuric acid after dilution of 1+4 (V/V) with deionized water

Element	Line (nm)	LOD ($\mu\text{g/L}$)
Al	167.078	1
Al	396.152	5
As	189.042	35
B	136.246	115
B	208.959	27
Bi	223.061	45
Cd	226.06	1
Co	238.892	5
Co	228.616	4
Cr	267.716	6
Cu	324.754	3
Hg	184.950	98
K	766.491	4
Mn	257,61	1
Mo	202.030	11
Na	589.592	3
Ni	231.60	3
Sb	206.833	90
Ti	334,941	2
Zn	213.856	2

