



ICP-OES with the CETAC DSX-100 Preconcentration / Matrix Elimination System

Problems: Saturated solutions of NaCl need to be analyzed for trace metal content in the chlor-alkali industry. The high dissolved solids content seriously degrades ICP-OES sensitivity and fouls sample introduction hardware.

Solution: Use the CETAC DSX-100 Discrete Sample Treatment System with chelating suspended particulate reagent (SPR) to preconcentrate trace elements and remove unwanted matrix components. Chelation chemistry used is iminodiacetate (IDA).

Advantages:

- Rapid, automated off-line sample preparation
- Preconcentration for enhanced detection
- Sodium level reduced approximately 150 fold
- Preconcentrated SPR reagent easily nebulized to the ICP

Analytes:	Cd, Cu, Ni, Pb, Zn
Matrix:	NaCl brine (30% w/v), high-purity grade
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Performance: *Matrix Elimination:* The iminodiacetate (IDA) chelation chemistry is very selective against Group I elements such as Na. The level of Na in 30% NaCl (w/v) is ~ 118,000 mg/L, and this level is reduced to ~ 800 mg/L after treatment with the DSX-100 System. (The emission line at 330.237nm was used to quantitate the remaining Na level.)

Preconcentration: Spike recoveries for the five target analytes are listed in Table 1. Spikes were added directly to 30% NaCl; recoveries range from 76% to 125%.

Spike Recoveries in 30%NaCl 10x Preconcentration with DSX-100

Element	Spike Level (µg/L)	Measured (µg/L)	Mean %Recovery
Cu	10	125 ± 8	125
Cd	10	77 ± 9	77
Ni	10	100 ± 10	100
Pb	50	380 ± 20	76
Zn	10	96 ± 9	96

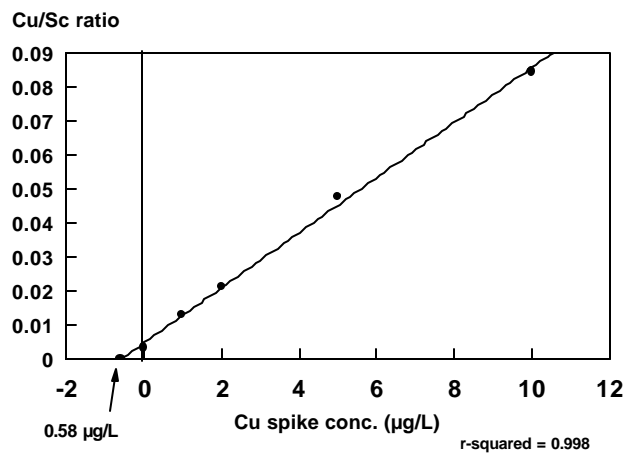
* Mean of three replicates; uncertainty is 3 sigma.

Table 1.

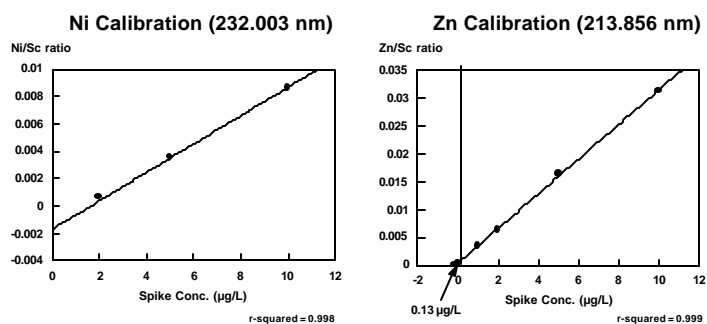
Addition Calibration: Addition calibration curves were run for each of the five target analytes spiked into 30% NaCl. All curves show excellent linearity, with detection of sub-ppb levels of Cu and Zn: 0.58 µg/L and 0.13 µg/L, respectively.

Scandium was added before sample processing to act as an internal standard. This compensates for any variations in the volume of the sample preconcentrate. Routine analysis would require only one or two sample addition points.

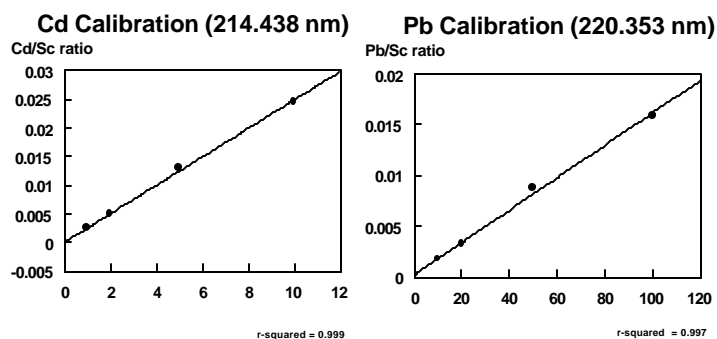
Cu Addition Calibration in 30% NaCl Cu (324.754 nm)



Addition Calibration in 30% NaCl



Addition Calibration in 30% NaCl



Instrumentation: CETAC DSX-100 Preconcentration / Matrix Elimination System

Axial ICP-OES

Operating Parameters: CETAC DSX-100

Off-line preparation times

10x preconcentration: 14 min per sample (30mL sample preconcentrated to 3mL)

SPR resin chemistry: iminodiacetate (IDA)

SPR resin aliquot: 0.01mL (10 microliters) of 10% SPR-IDA per 10mL of 30% NaCl

Sample preparation: 30% NaCl solutions acidified to < pH 2 with high-purity HNO₃

Working pH: 7.5-8.5 (adjust with 20-22% high-purity NH₄OH)

Axial ICP-OES

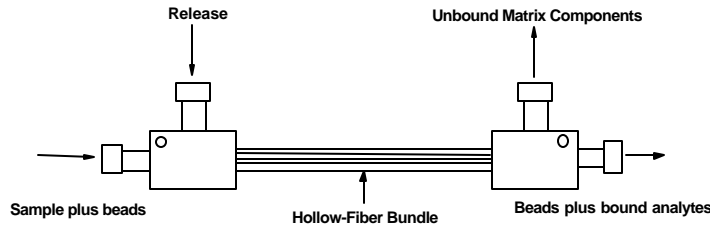
Forward power: 1300W
Plasma gas flow: 15 L/min
Auxiliary gas flow: 0.5 L/min
Nebulizer gas flow: 0.65 L/min
Sample uptake rate: 1.50 mL/min
Nebulizer style: cross-flow or babington type

Data acquisition: 5 sec integration time; 2-pt background correction
5 replicate measurements

Principal of Operation:

An aliquot of SPR polymer beads (0.2 micron diameter) is added to each sample to form a dilute suspension. After pH adjustment, analyte ions are chelated by the polymer beads and immobilized. The beads with bound analytes are retained in a hollow fiber membrane filter, while unbound matrix components pass to waste. Liquid flows into and out of the filter cartridge are automatically controlled by metal-free valves. After a wash cycle, the beads are physically released from the filter and deposited in a collection tube. The preconcentrated sample may then be nebulized directly to the ICP-OES. See the diagram of the filter cartridge below.

Preconcentration / Matrix Elimination
with a Hollow Fiber Filter Cartridge



End-on View

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