



Improved Detection of Trace Elements in 45% KOH by ICP-OES

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

Problems: Concentrated potassium hydroxide (KOH), used for a variety of industrial applications, needs to be analyzed for trace metal content. The high dissolved solids level 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
- Potassium level reduced approximately 200 fold
- Preconcentrated SPR reagent easily nebulized to the ICP

Analytes:	Co, Cu, Fe, Ni, Ti
Matrix:	11.25% KOH (45% KOH diluted 1:4)

Performance: *Matrix Elimination:* The iminodiacetate (IDA) chelation chemistry is very selective against Group I elements such as K. The level of K in 11.25% KOH is ~ 74,000 mg/L, and this level is reduced to ~ 350 mg/L after treatment with the DSX-100 System. (The emission line at 766.491nm was used to quantitate the remaining K level.)

Preconcentration: Spike recoveries for the five target analytes are listed in Table 1. Spikes were added directly to the diluted 45% KOH; recoveries range from 74% to 87%.

**Spike Recoveries in Diluted* 45% KOH
10x Preconcentration with DSX-100**

Element	Spike Level (µg/L)	Measured (µg/L)	Mean % Recovery
Co	20	158 ± 11	79
Cu	20	173 ± 9	87
Fe	20	148 ± 39	74
Ni	20	172 ± 13	86
Ti	20	159 ± 10	80

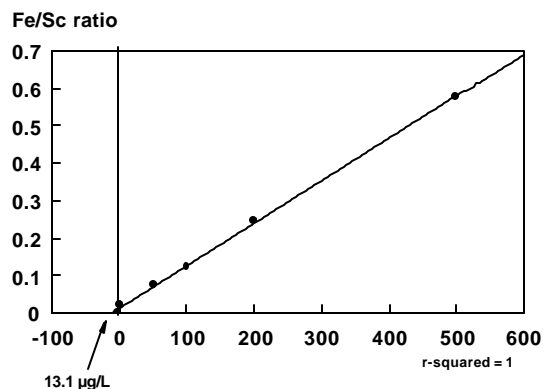
* 25.0g 45% KOH diluted to 100mL volume.
Measured values are mean of 3 replicates; uncertainty is 3 sigma.

Table 1

Addition Calibration: Addition calibration curves were run for each of the five target analytes spiked into diluted 45% KOH. All curves show excellent linearity, with detection of Fe and Ti at concentrations of 13.1 µg/L and 3.1 µg/L, respectively.

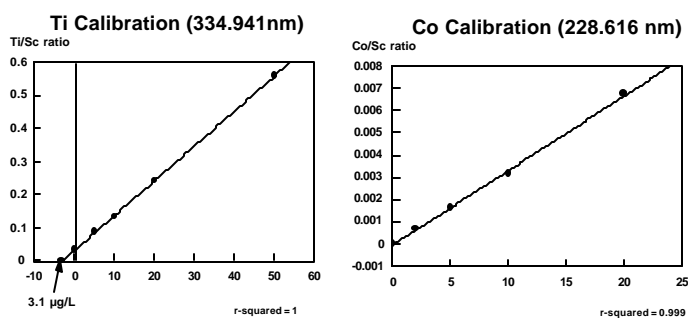
Scandium was added before sample processing to act as an internal standard. This compensates for any variation in the volume of the sample preconcentrate. Routine analysis would require only one or two sample addition points.

**Fe Addition Calibration in Diluted* 45% KOH
Fe (238.204 nm)**



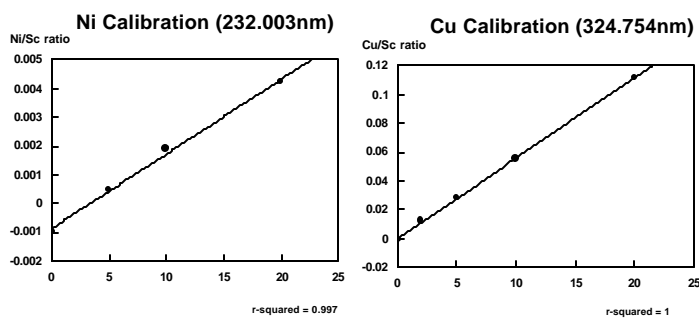
* 25.0g 45% KOH diluted to 100mL.

Addition Calibration in Diluted* 45% KOH



* 25.0g of 45% KOH diluted to 100mL

Addition Calibration in Diluted* 45% KOH



* 25.0g of 45% KOH diluted to 100mL

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: 45% KOH solution was diluted 1 to 4 with deionized water. Resulting solution was acidified to < pH 2 with high-purity HNO₃.

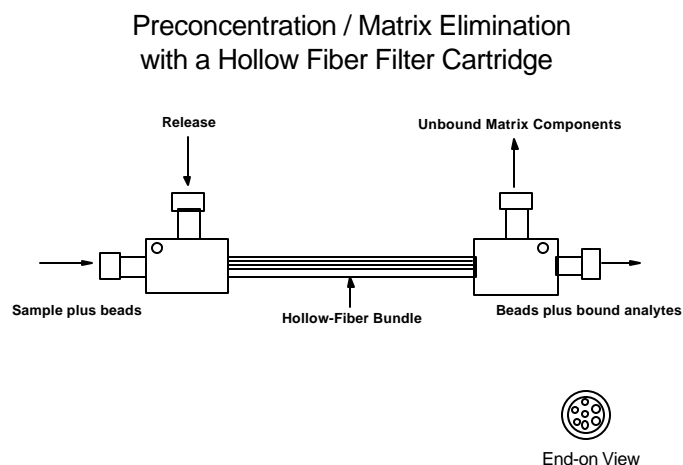
Working pH: 7.5-8.5 (adjust with 20-22% high-purity NH₄OH)

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.



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