

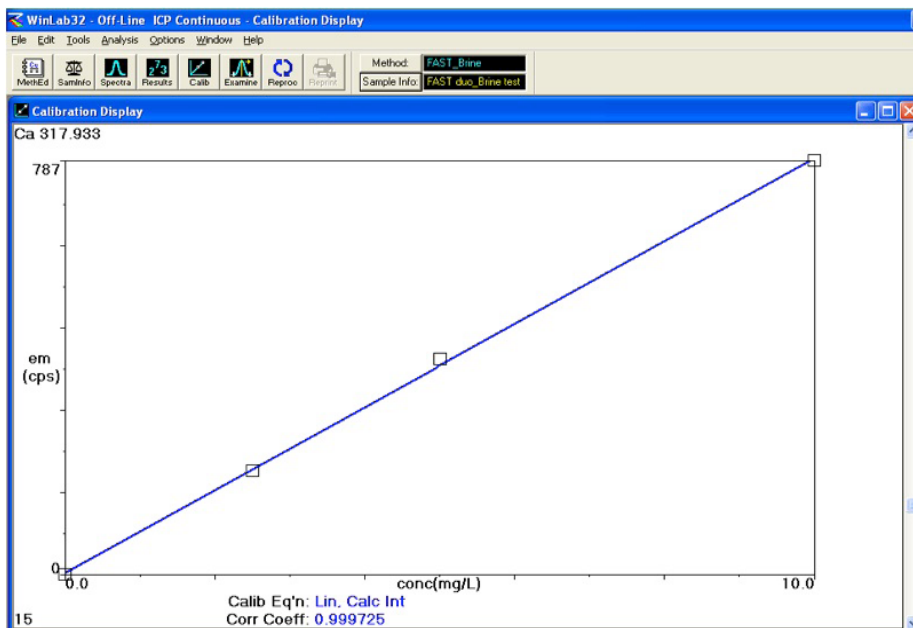
Analysis of Brine Samples — Trace Impurity Preconcentration Improving Chlor-Alkali Process Control

Introduction

Improved detection limits for alkaline earth and transition metals in high-purity brines could lead to a corresponding improvement in chlor-alkali plant process control, potentially reducing contamination and extending the life of the fluoropolymer membrane cell.

The ESI brineFAST™ is a fully-automated chelation preconcentration sample introduction system for ICPOES, which improves detection limits for critical trace elements in brine samples by more than an order of magnitude.

The brineFAST system concentrates elements such as Ca, Mg, Fe, Ba, Sr, Mn, and others on a chelation column while allowing the NaCl matrix to pass through. The concentrated metals are then eluted into a nebulizer and determined using a PerkinElmer Optima ICPOES instrument.



Calibration of Ca in Undiluted High-Purity Brine. Calibration spikes are 0, 2.5, 5, and 10 ppb.

Analyte	Traditional (ppb)	brineFAST (ppb)	Improvement Factor
Ca	8.0	0.3	20
Mg	1.7	0.2	9
Fe	6.3	0.3	21
Ba	1.4	0.4	3
Sr	1.1	0.1	10
Mn	1.2	0.2	6
Average MDL Improvement Factor			11.5

Table 1. Method Detection Limits Comparison.



Experimental

Sample Introduction

The brineFAST System is a fully-automated, high-purity autosampler with dual flowing rinse stations, two high-purity fluoropolymer injection valves, a chelation column, a high-efficiency nebulizer, an inert o-ring-free spray chamber, and a sapphire injector attached to the ICP.

Instrumentation – ICPOES

A PerkinElmer Optima ICPOES measured all samples in axial mode. Read delay on the ICP instrument is set to synchronize data acquisition with the elution event. The ICP instrument software prepares the calibration curve and calculates concentrations as it would for a normal, continuous flow analysis.

Software Settings

The brineFAST system is fully-integrated with the Optima ICP software for complete automation and control of parameters necessary for automated chelation/preconcentration of metal contaminants in brine and seawater samples.

Spike Recovery Comparison (%)		
Analyte	Traditional Introduction (spiked at 100 ppb)	brineFAST (spiked at 5 ppb)
Ca	132	100.3
Mg	64	105.1
Fe	104	96.4
Ba	106	109.6
Sr	75	107.9
Mn	111	101.6

Table 2. Comparison of spike recoveries for direct analysis and brineFAST IDA chelation technique. Note that the spike levels are 20x lower with the brineFAST than with the original introduction system.

Steps	Time (min)
Load brine sample onto preconcentration column	1
Wash column remove NaCl matrix	3
Acid elute concentrated metals from column	2
Autoclean column prior to next sample	3
TOTAL TIME PER BRINE SAMPLE	~ 10 min

Table 3. Steps in Automated IDA Chelation for Brine Analysis.

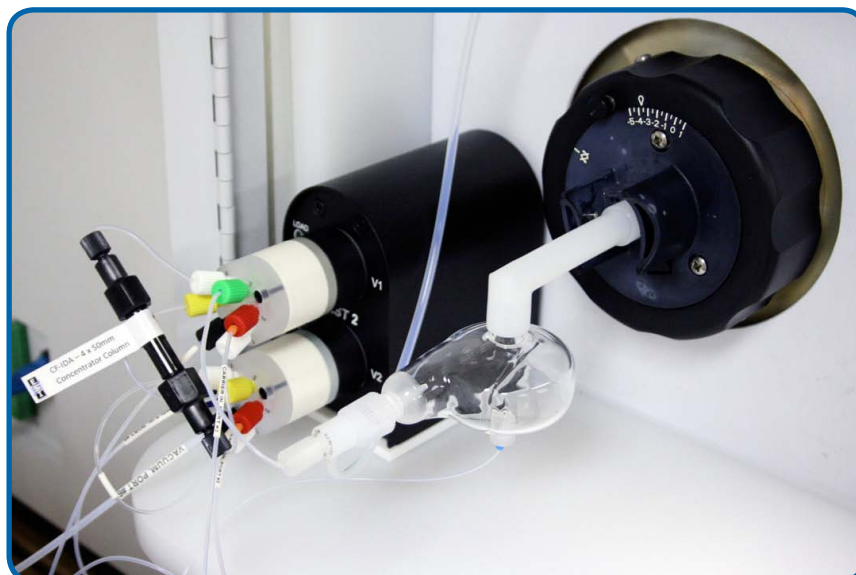
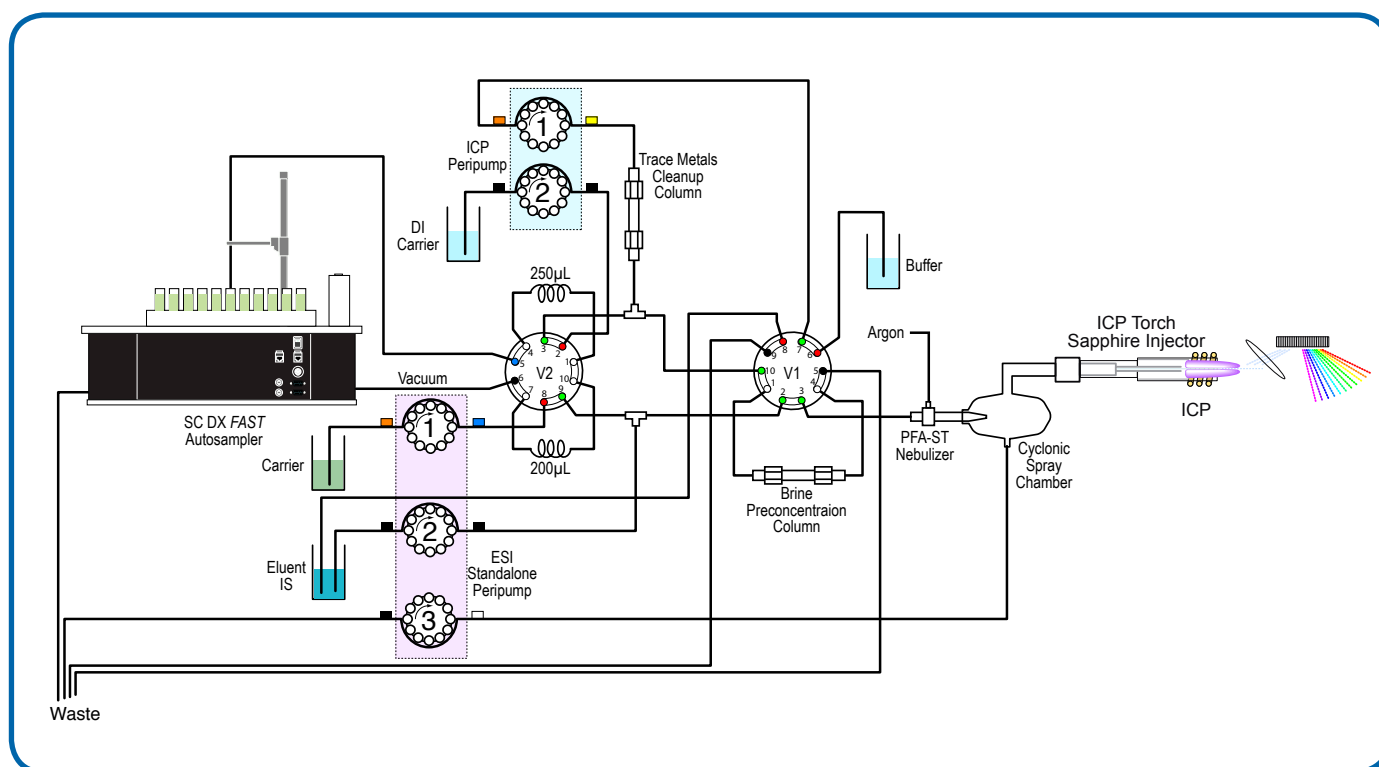


Figure 1. Close-up brineFAST valve system/PE Optima for automated determinations of Ca, Mg, Fe, Mn, Sr, Ba, Ti in 30% NaCl Brine.

Power: 1500 W
Nebulizer Gas: 0.65 L/min
Aux Gas: 0.5 L/min
Plasma: 16 L/min
Pump: 0.5 mL/min
Sample Volume: 0.1 ml

Table 3. Operating Parameters and Settings



brineFAST Automated Brine Matrix Removal System Diagram