Author: Nathan Saetveit

Complete Automation for Ultratrace Metals Determination in High Matrix Samples: Autocalibration, Auto Matrix Matching, Preconcentration and Matrix Removal, & Hydride Generation

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Abstract

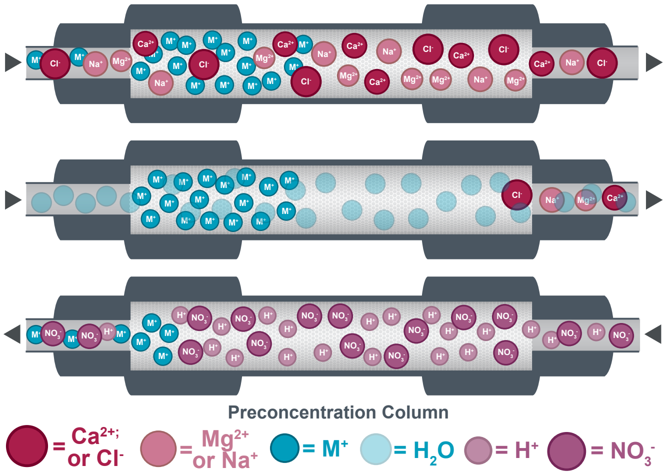
An automated analysis system for ultratrace determination of low and sub-ppt metals in high matrix samples is described. Simple external autocalibration from a single multielement stock standard eliminates common sources of contamination for ultratrace metals determination. Inline, automatic matrix matching provides exceptional accuracy. Metals may be measured in up to three modes: preconcentration and matrix removal, direct analysis with inline dilution, and hydride generation.

Introduction

Matrix effects from samples with high total dissolved solids (TDS) can cause severe problems for accurate determination of many elements by ICPMS. High dilution factors attenuate the matrix effects but are undesirable when excellent detection limits are required. Historically, a variety of advanced techniques have been used to accurately determine trace elements in high TDS samples. One common approach is preconcentration and matrix removal, which offers superb results for elements whose chemistry is compatible with the chosen chelation resin. For other elements, manually matching the matrix of calibration blanks and standards to that of the diluted samples can achieve very accurate results in high TDS samples. Lastly, hydride generation improves quantification for elements such as As and Se that are otherwise difficult to determine.

The sea*FAST* SP3 analyzes samples using one or more advanced analytical techniques: preconcentration and matrix removal, direct analysis with inline dilution, and hydride generation. For improved accuracy the sea*FAST* performs automatic inline matrix matching for calibration blanks, standard, and QC samples, as well as autodiluted samples.

Results



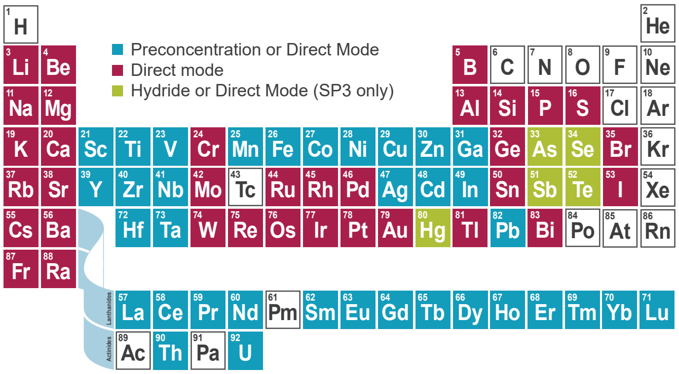


Figure 1. At a pH ~6, many metals are chelated on the preconcentration column, while matrix elements are flushed from the column. Metals are eluted directly to the ICPMS with nitric acid.

sea*FAST* Preconcentration Column

Preconcentration Mode

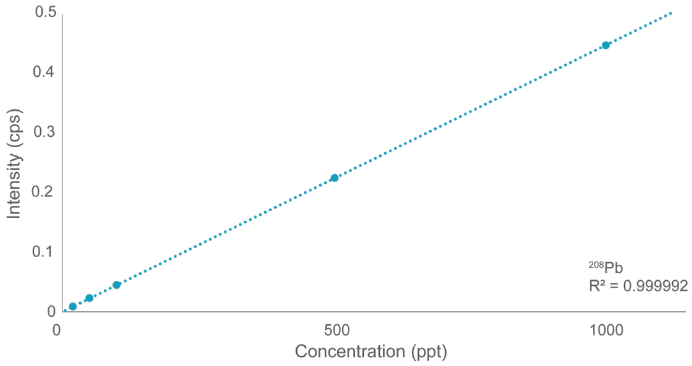


Figure 2. Preconcentration mode: Autocalibration from a single 1000 ppt calibration standard. Calibration curve was matrix matched to ~3% TDS.

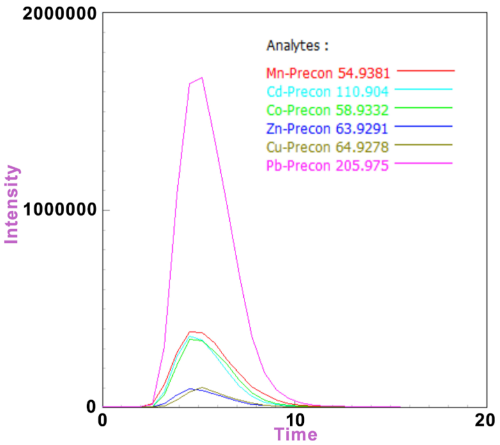


Figure 3. Simultaneous elution of 6 elements in Preconcentration mode at 1 ppb.

Results *(Continued)*

Hydride Mode

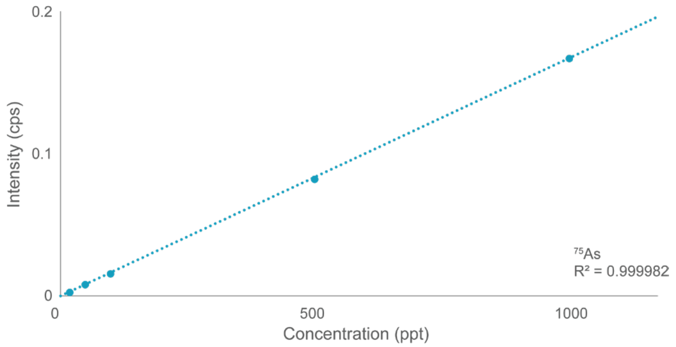
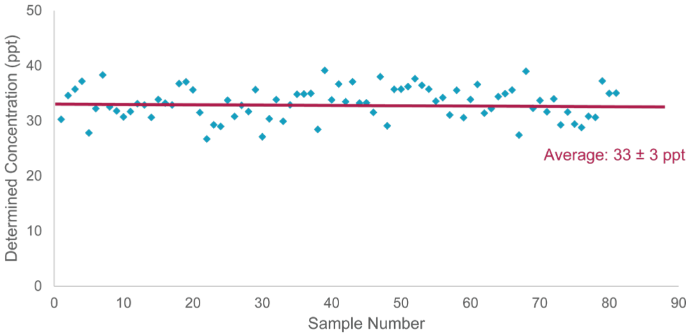


Figure 4. Hydride Mode: Autocalibration from a single 1000 ppt calibration standard. Calibration curve was matrix matched to ~3% TDS.

Direct Mode



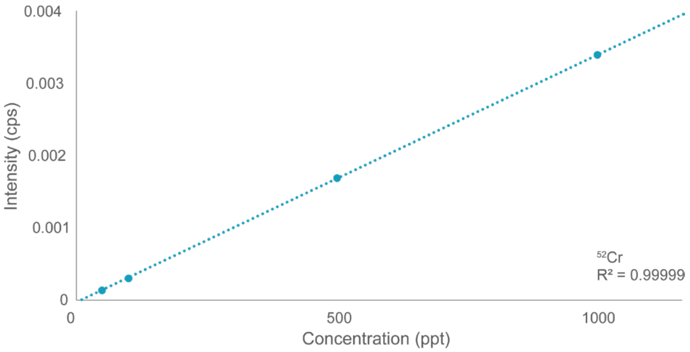


Figure 5. Long term stability of Cr in synthetic seawater shows >14 hours of repeated determinations with internal standard correction. Direct mode automatically diluted the sample inline by 10x. The determined concentration in the synthetic seawater was 33±3 ppt.

Figure 6. Direct mode with 10x inline dilution: Autocalibration from a single 1000 ppt calibration standard. Calibration curve was matrix matched to ~3% TDS.

Results *(Continued)*

Long Term Stability

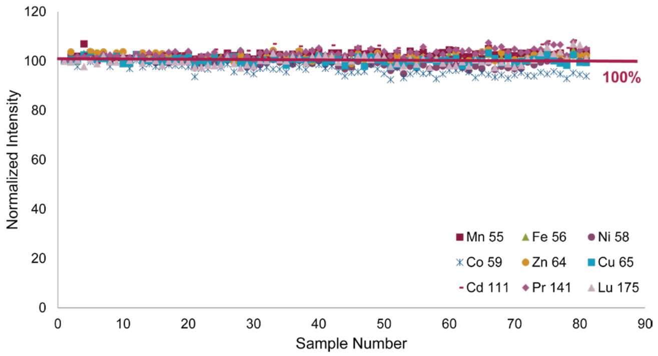


Figure 7. Common transition metals and selected rare earth elements (REEs) were determined in synthetic seawater consecutively for over 14 hours (n=81). Auto-matrix-matched blanks and QC standards are not shown, for clarity. Average precision over 14 hours was better than 2% with internal standard correction. REEs were spiked at 5 ppt.

Table 1. Long term stability of elements analyzed by sea*FAST* SP3.

Analyte	**Determined Concentration PPT (n=81)**		
^{55}Mn	84	±	1
^{56}Fe	1933	±	30
^{58}Ni	563	±	14
^{59}Co	16.4	±	0.5
^{64}Zn	61	±	1
^{65}Cu	154	±	3
^{111}Cd	42.7	±	0.7
^{141}Pr	6.02	±	0.07
^{175}Lu	5.5	±	0.1

Accuracy

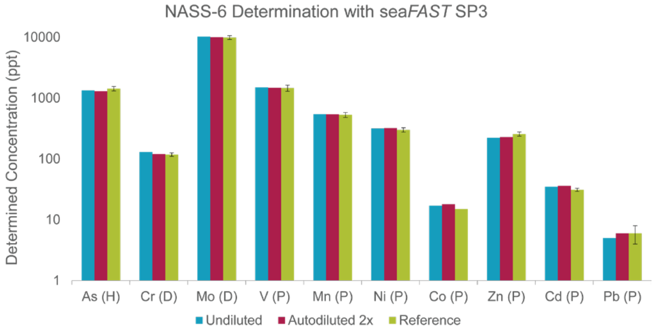


Figure 8. NASS-6 was analyzed in all three modes, Preconcentration (P), Direct with fixed 10x inline dilution (D) and Hydride (H). NASS-6 was run undiluted and with a 2x autodilution using automatic, inline matrix matching with seaBlank ultrapure sodium chloride solution. The sea*FAST* achieves excellent accuracy in all three modes, with and without autodilution.

sea*FAST*