Automate
PPQ Metals

prep*FAST* PPQ

prepFAST PPQ

Automated Sample Preparation and Introduction System for PPQ Metals Determination in High Purity Chemicals

The prepFAST PPQ is the most advanced tool for analyzing ultrapure semiconductor grade chemicals with ICPMS detection. The prepFAST PPQ utilizes syringe-driven flows of UPW, semiconductor grade acids, and standard solution to automate both sample dilutions and standard curve generation. It eliminates manual handling of samples to deliver sub-ppq detection limit capabilities in concentration mode and sub-ppt detection limit capabilities in direct analysis mode.

Two High Purity Modes

Direct Analysis Mode

- Capability to analyze all semiconductor grade chemicals
- Sub-PPT detection limits for all semiconductor elements
- General purpose for any sample matrix
- Automated MSA calibration
- Automated inline dilution

Concentration Mode

- Suitable for UPW, 30% H_2O_2, and IPA
- Sub-PPQ detection limits
- Removes difficult matrices such as IPA while recovering metals
- Removes impact of ICPMS interferences and backgrounds on results
- Amplified sensitivity compared to direct analysis
- Automated MSA calibration

prepFAST PPQ
System

prepFAST PPQ Autocalibration

The prepFAST PPQ automatically prepares calibration curves for over 40 elements controlled in semiconductor manufacturing processes. Calibrations are generated by automatically diluting an enclosed multielement stock standard. Automation with the high-purity prepFAST PPQ achieves low to sub-ppt calibrations in direct analysis mode and low ppq calibrations in concentration mode.

High Purity Automation with PPT/PPQ results

Automation

- Automatic external and MSA calibrations
- Automated sample sensing
    - Accounts for viscosity and automatically adjusts timing
    - Detects and injects the sample and triggers the ICPMS
- Automated syringe-driven sample introduction
    - Sample loading
    - Sample preparation
    - Inline dilution
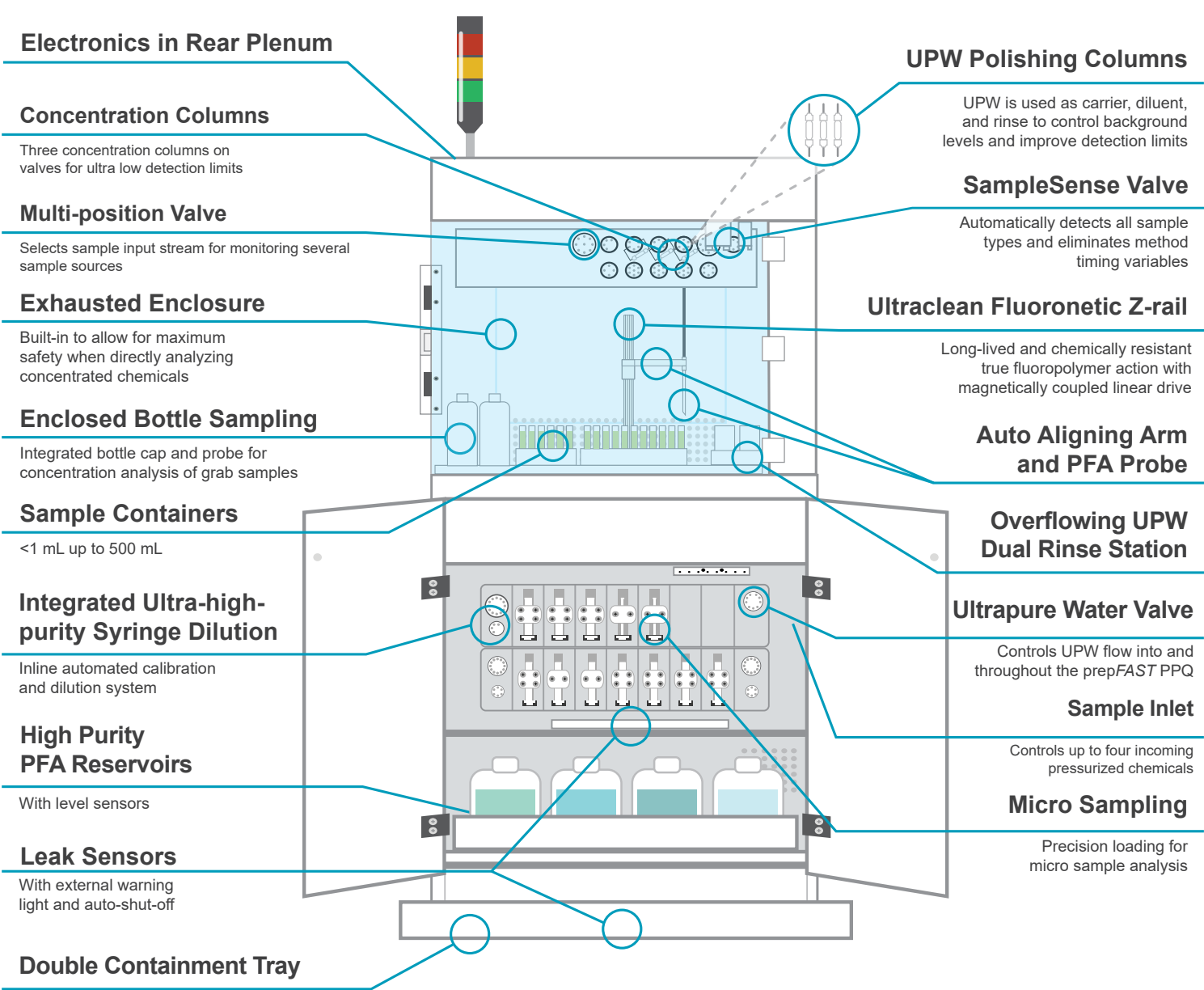   - Acid addition (direct mode only)

Ultraclean

- Ultraclean sample preparation
- Integrated ultraclean sample environment
    - Includes ultraclean air shower
    - Sample racks for PFA containers (<1 mL to 500 mL)
- Continuously-flowing high purity UPW rinse (user-supplied UPW)
- UPW polishing columns for low background

	prepFAST PPQ System					
System	Integrated Mobile Autosampler & Enclosure	Ultraclean Air Shower	Integrated FAST valve modules	PFA Nebulizer with Integrated Capillary	PFA Sample Probes	Syringe Pump S500V2
prepFAST PPQ	✓	✓	✓	✓	✓	✓

Pure Automation

prepFAST PPQ

Electronics in Rear Plenum

Concentration Columns

Three concentration columns on valves for ultra low detection limits

Multi-position Valve

Selects sample input stream for monitoring several sample sources

Exhausted Enclosure

Built-in to allow for maximum safety when directly analyzing concentrated chemicals

Enclosed Bottle Sampling

Integrated bottle cap and probe for concentration analysis of grab samples

Sample Containers

<1 mL up to 500 mL

Integrated Ultra-high-purity Syringe Dilution

Inline automated calibration and dilution system

High Purity PFA Reservoirs

With level sensors

Leak Sensors

With external warning light and auto-shut-off

Double Containment Tray

UPW Polishing Columns

UPW is used as carrier, diluent, and rinse to control background levels and improve detection limits

SampleSense Valve

Automatically detects all sample types and eliminates method timing variables

Ultraclean Fluoronetic Z-rail

Long-lived and chemically resistant true fluoropolymer action with magnetically coupled linear drive

Auto Aligning Arm and PFA Probe

Overflowing UPW Dual Rinse Station

Ultrapure Water Valve

Controls UPW flow into and throughout the prepFAST PPQ

Sample Inlet

Controls up to four incoming pressurized chemicals

Micro Sampling

Precision loading for micro sample analysis

prepFAST PPQ Inline Dilution of Semiconductor-grade Chemicals

ICPMS

9.8% H$_2$SO$_4$ with Calibration Spike Added

98% H$_2$SO$_4$

Diluent + Standard
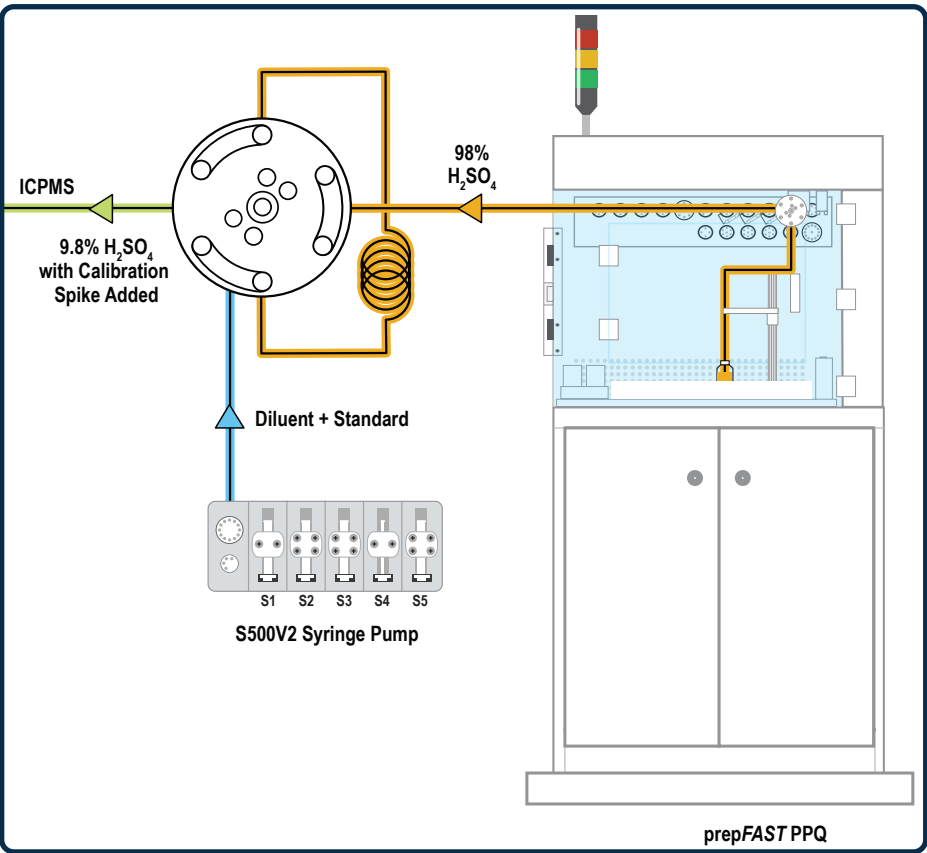
S1 S2 S3 S4 S5

S500V2 Syringe Pump

prepFAST PPQ

Diagram showing 10x inline dilution of concentrated H$_2$SO$_4$ with prepFAST PPQ.

The prepFAST PPQ allows dilution by volume or weight for IPA and H$_2$O$_2$ in concentration mode, and all semiconductor-grade chemicals in direct analysis mode. Metals are quantified using automated inline MSA or external calibration. Automated direct analysis of concentrated chemicals eliminates sample contamination caused by manual dilution into a secondary container.

Examples of Semiconductor Chemicals Analyzed at the ppt Level with prepFAST PPQ*					
Acids	98% H$_2$SO$_4$	89% H$_3$PO$_4$	70% HNO$_3$	49% HF	35% HCl	30% H$_2$O$_2$
Bases	22% NH$_4$OH	2.38% TMAH	25%TMAH	KOH		
Organics	IPA	PGMEA/PGME	Photoresist	NMP	Butyl Acetate	Cyclohexanone

*This table contains only a partial list of chemicals which can be analyzed using prepFAST PPQ in direct analysis mode.

Concentration Mode

Sampling Options in Concentration Mode

Direct sample modules

- Availability to monitor up to four pressurized sample lines
- Flushing and sampling processes are controlled by an all-fluoropolymer valve manifold

Enclosed bottle samples

- Rack with integrated probes and caps to keep bottled samples fully enclosed before and during sampling
- Can accomodate up to 2L bottles
- Automated UPW probe rinse between samples

Layout of four 1L enclosed bottle samples to the left of the autosampler.
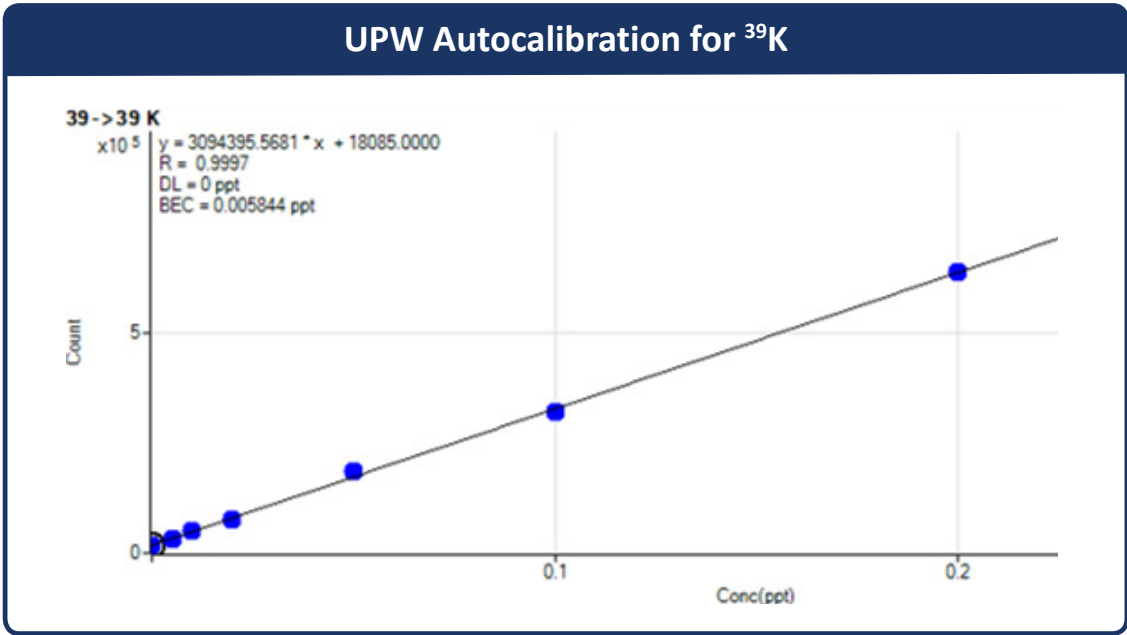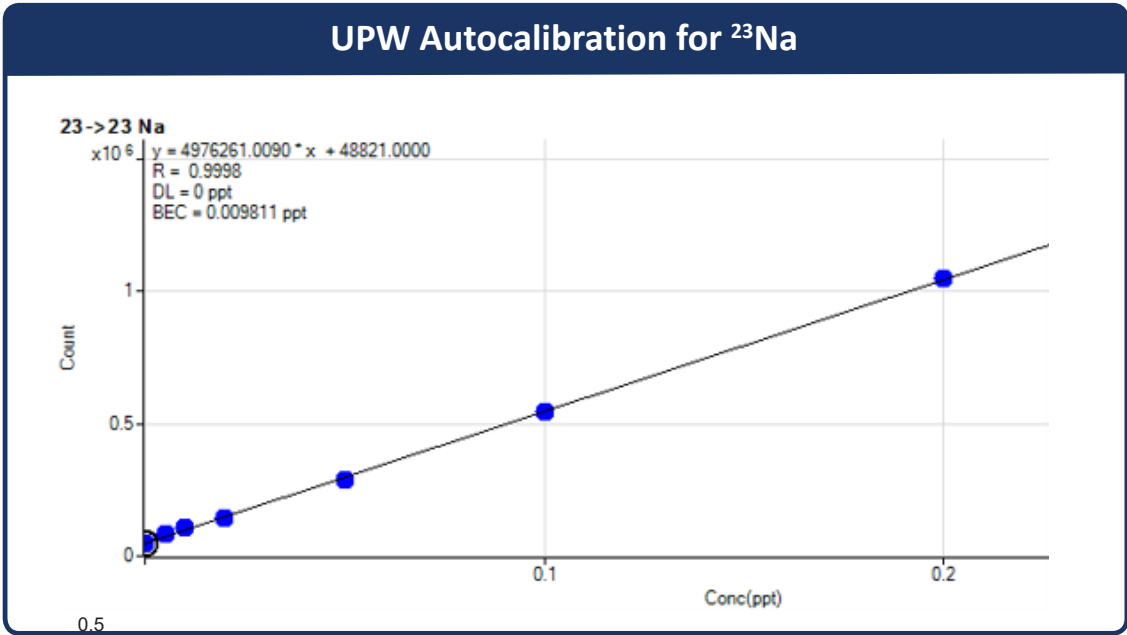
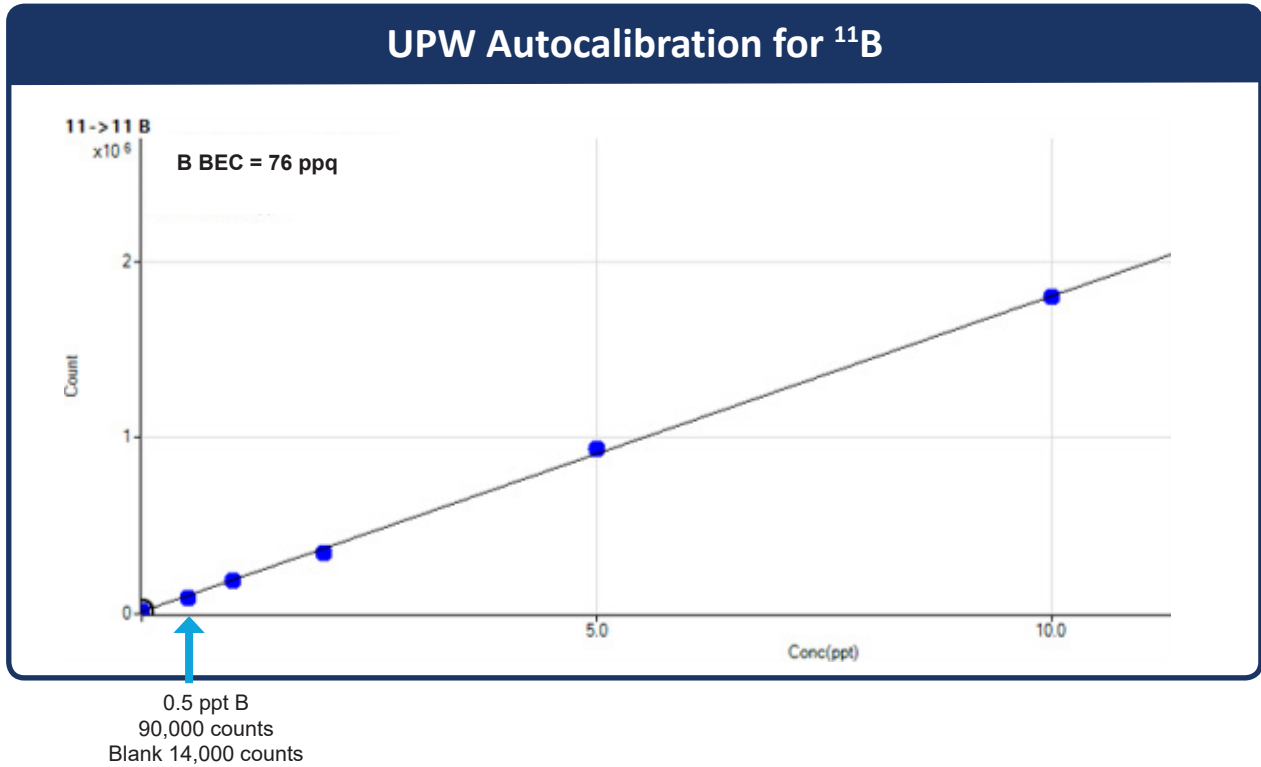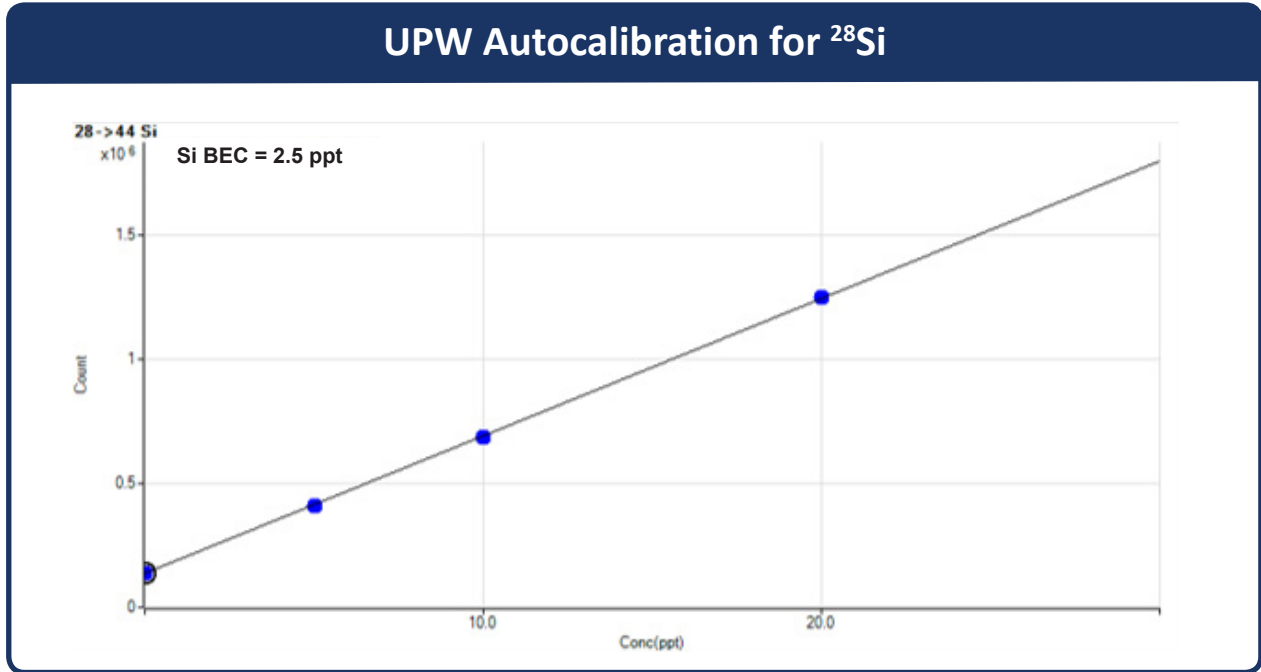| Example of Detection Limits in Non-cleanroom Environment in Concentration Mode (1000x Concentration Factor)			
Element	DL (PPQ)	Element	DL (PPQ)
^{7}Li	0.02	^{85}Rb	0.01
^{11}B	9	^{88}Sr	0.06
^{23}Na	0.6	^{90}Zr	0.1
^{24}Mg	0.2	^{93}Nb	0.2
^{27}Al	0.6	^{98}Mo	0.3
^{28}Si	240	^{75}As	0.2
^{39}K	0.4	^{107}Ag	0.05
^{40}Ca	0.3	^{114}Cd	0.06
^{42}P	2	^{115}In	0.01
^{48}Ti	0.2	^{118}Sn	0.5
^{51}V	0.7	^{121}Sb	0.4
^{52}Cr	0.4	^{133}Cs	0.007
^{55}Mn	0.07	^{138}Ba	0.04
^{56}Fe	0.3	^{180}Hf	0.2
^{58}Ni	0.07	^{181}Ta	0.3
^{59}Co	0.01	^{184}W	0.4
^{63}Cu	0.07	^{195}Pt	0.1
^{64}Zn	0.4	^{205}Tl	0.03
^{69}Ga	0.004	^{208}Pb	0.05
^{74}Ge	0.6	^{106}Pd	0.4

Concentration Mode

prep*FAST* PPQ Autocalibration

Autocalibrations for elements controlled in semiconductor manufacturing processes are generated by automatically diluting an enclosed multielement stock standard. Automation with the high-purity prep*FAST* PPQ achieves ppt to sub-ppt calibration in direct analysis mode and ppq calibrations in concentration mode.

## UPW Autocalibration in Concentration Mode

UPW Autocalibration for ^{23}Na

23->23 Na
x10^6
y = 4976261.0090 * x + 48821.0000
R = 0.9998
DL = 0 ppt
BEC = 0.009811 ppt

UPW Autocalibration for ^{39}K

39->39 K
x10^5
y = 3094395.5681 * x + 18085.0000
R = 0.9997
DL = 0 ppt
BEC = 0.005844 ppt

UPW Autocalibration in Concentration Mode

UPW Autocalibration for ^{28}Si

28->44 Si
x10^6
Si BEC = 2.5 ppt

UPW Autocalibration for ^{11}B

11->11 B
x10^6
B BEC = 76 ppq

0.5 ppt B
90,000 counts
Blank 14,000 counts

Example of Detection Limits in Non-cleanroom Environment in Direct Analysis Mode

Element	DL (ppt)	Element	DL (ppt)
^{7}Li	0.02	^{72}Ge	0.04
^{9}Be	0.007	^{75}As	0.2
^{11}B	0.9	^{85}Rb	0.008
^{23}Na	0.07	^{88}Sr	0.008
^{24}Mg	0.01	^{90}Zr	0.004
^{27}Al	0.03	^{93}Nb	0.002
^{39}K	0.06	^{95}Mo	0.5
^{40}Ca	0.4	^{111}Cd	0.07
^{48}Ti	0.02	^{115}In	0.004
^{51}V	0.1	^{118}Sn	0.05
^{52}Cr	0.1	^{121}Sb	0.05
^{55}Mn	0.009	^{137}Ba	0.04
^{56}Fe	0.04	^{178}Hf	0.003
^{58}Ni	0.01	^{181}Ta	0.01
^{59}Co	0.007	^{182}W	0.01
^{60}Ni	0.01	^{205}Tl	0.002
^{63}Cu	0.03	^{208}Pb	0.005
^{64}Zn	0.04	^{232}Th	0.000
^{71}Ga	0.002	^{238}U	0.003

Autocalibration in Direct Analysis Mode

UPW Autocalibration for ^{40}Ca

NH$_4$OH Autocalibration for ^{52}Cr
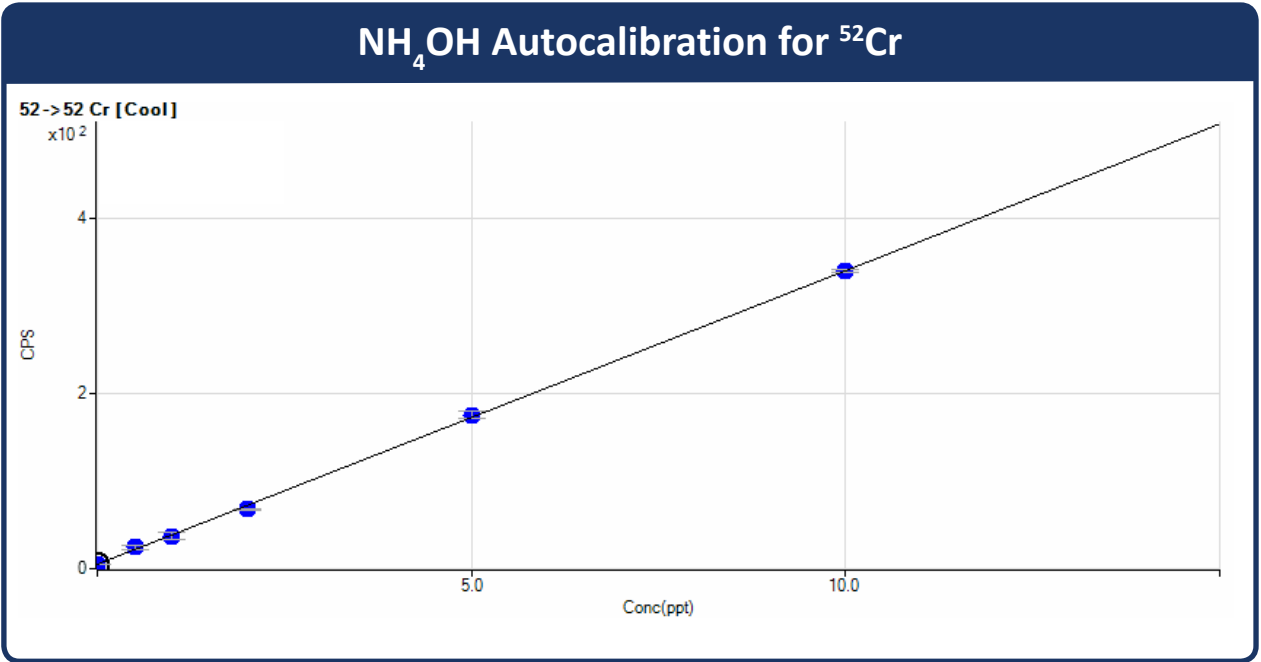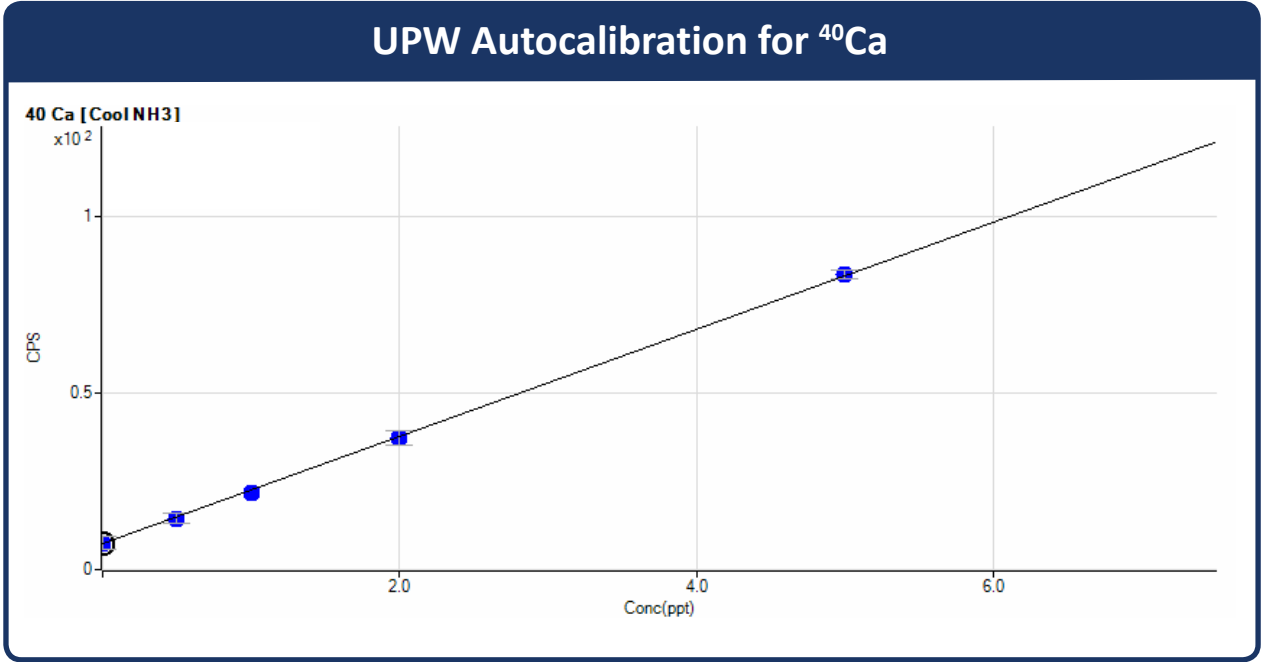
Elemental Scientific

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