Implementing EPA Method 6020 with the Agilent ICP-MS Portfolio

NEMC August 2018

Craig Jones
Applications Specialist
ICP-MS, ICP-MS/MS

Agilent
Trusted Answers

The Agilent Atomic Spectroscopy Lineup
An Instrument for Any Application!

55B AA

240FS/280FS Flame AA

240Z/280Z Furnace AA

4200 MP-AES

5110 ICP-OES

7800 ICP-MS

7900 ICP-MS

NEW! 8900 ICP-QQQ

Leading the way in atomic spectroscopy innovation

www.agilent.com/chem/atomic

Agilent's History in ICP-MS
30 Years of ICP-MS Innovation

Enabling high sensitivity metal analysis

PMS series

First computer-controlled ICP-MS

Enabling routine robust ICP-MS analysis

4500

First benchtop ICP-MS
Cool plasma

Enabling control of common interferences

7500

9 orders detector
ORS cell

Enabling ease of use and productivity

7700

HMI
ISIS-DS
MassHunter SW

A new era in ICP-MS performance

7900

UHMI
ODS detector
ISIS 3

Enabling simplified ICP-MS workflows

7800

Solution ready
Method automation

Enabling controlled reaction chemistry

8800 ICP-QQQ

World's first ICP-QQQ

Flexible, high performance MS/MS

New 8900 ICP-QQQ

Second generation
ICP-QQQ

#1 selling ICP-MS !

1987 1994 2000 2009 2012 2014 2015 **2016**

Most Compact Instruments on the Market

Agilent 7800

Agilent 7900

Agilent 8800

Agilent 8900

Some of the Benefits of ICP-MS

- Multi-elemental technique
- High sensitivity, ppq/ppt for most elements
- Short analysis time (~ 3 min to <60sec)
- Extremely linear with wide dynamic range
- Minimum number of interferences
- High Productivity

Most Important Performance Consideration for Environmental Analysis

- Sensitivity
- Interference Removal
- Matrix Tolerance
- Linear Dynamic Range

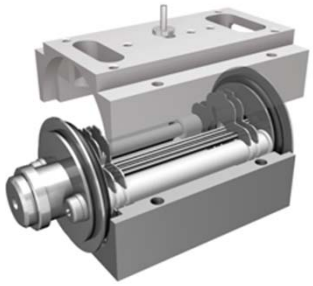
4 Key Benefits of Agilent ICP-MS

1) Matrix Tolerance

Ultra High Matrix Introduction System (UHMI)

Analyze Higher TDS samples than any other instrument on the market.

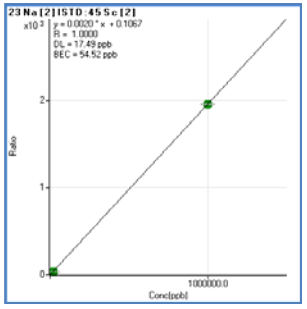
2) Interference Removal



Octopole Reaction System (ORS⁴) He Mode

Simple, effective removal of all common interferences.

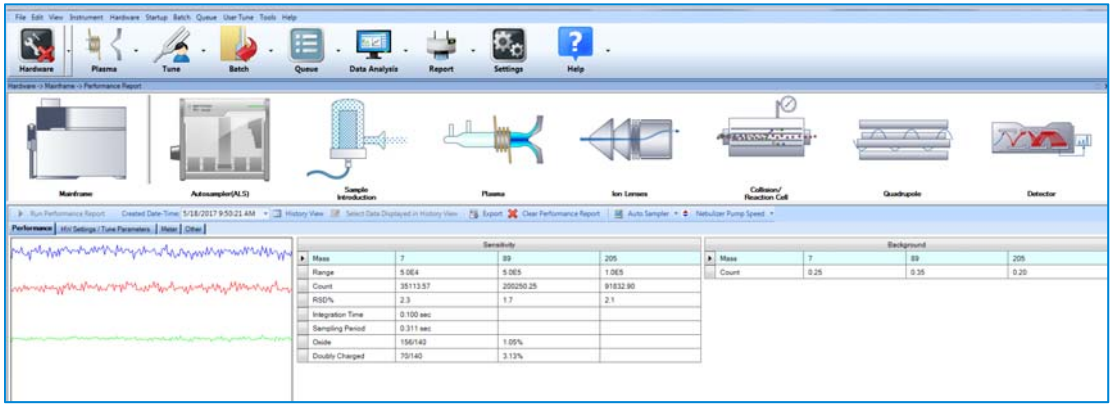
3) Dynamic Range



Widest Dynamic Range

Up to 11 orders dynamic range:
0.1ppt(DL) to 10,000ppm

4) Mass Hunter Software



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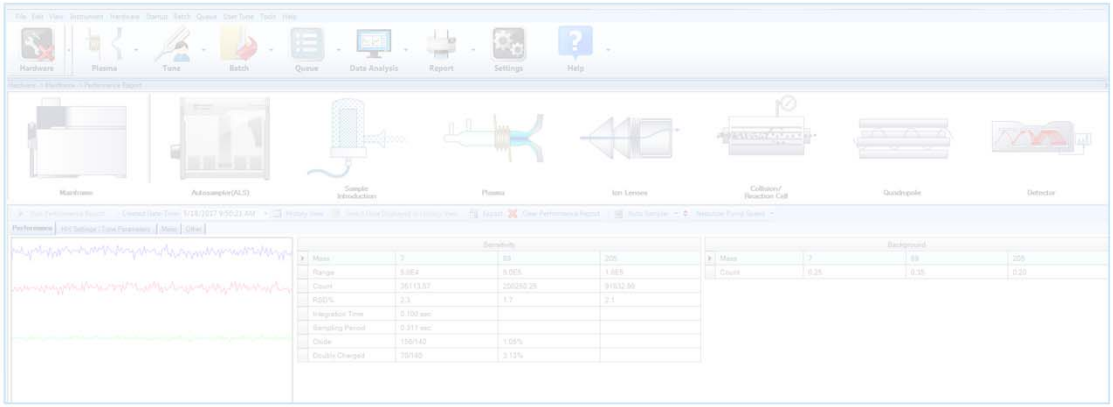

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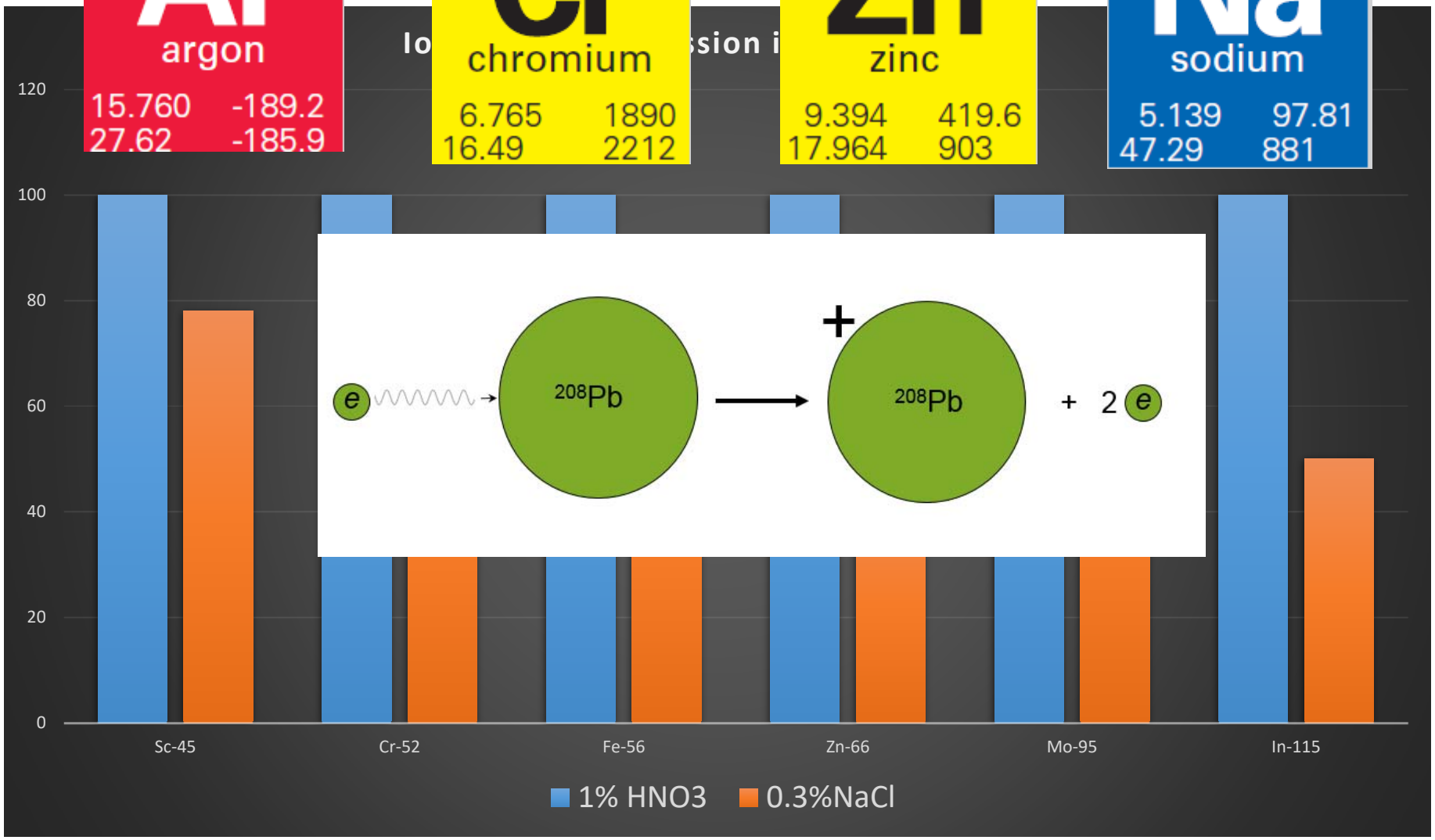
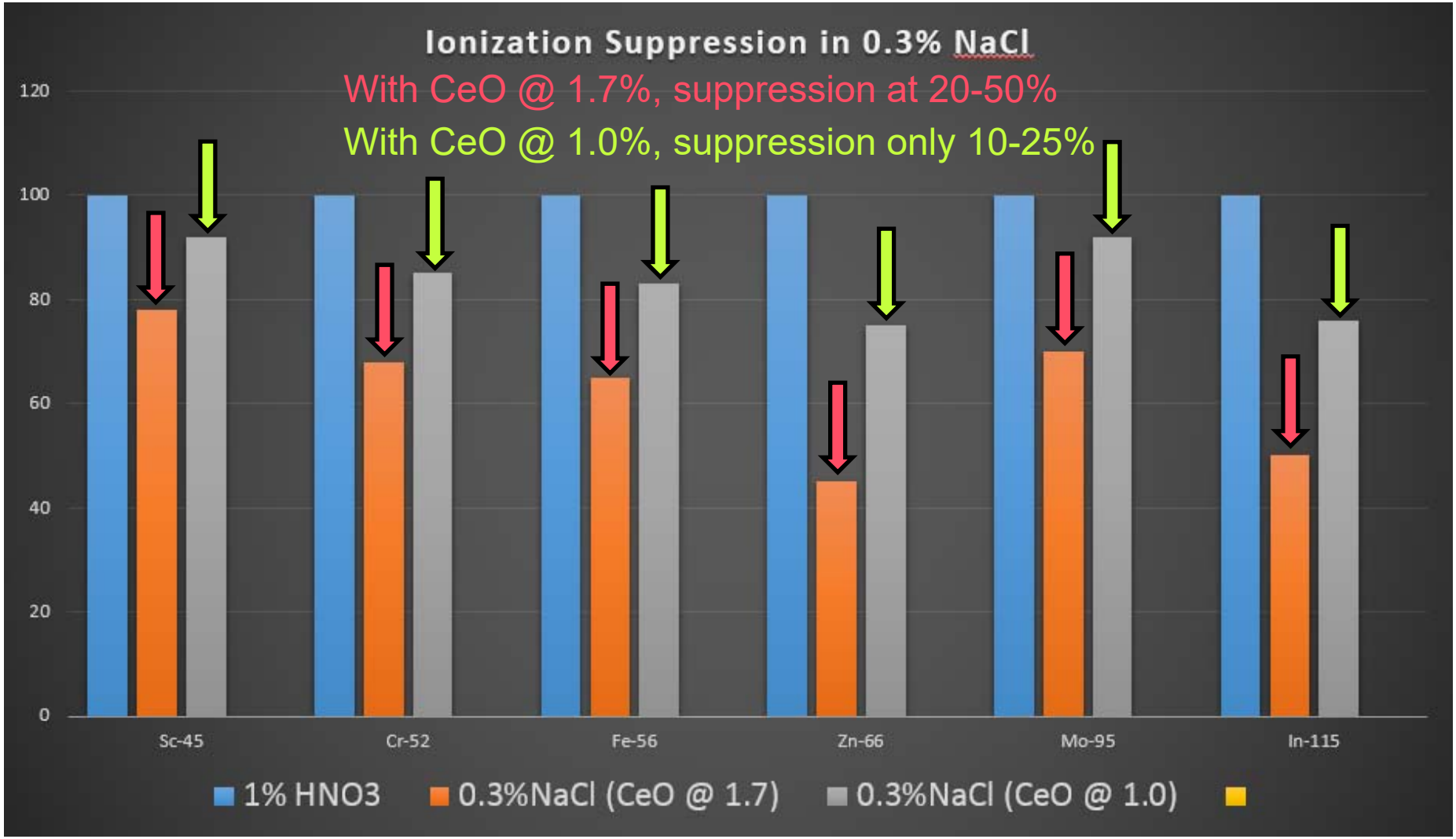
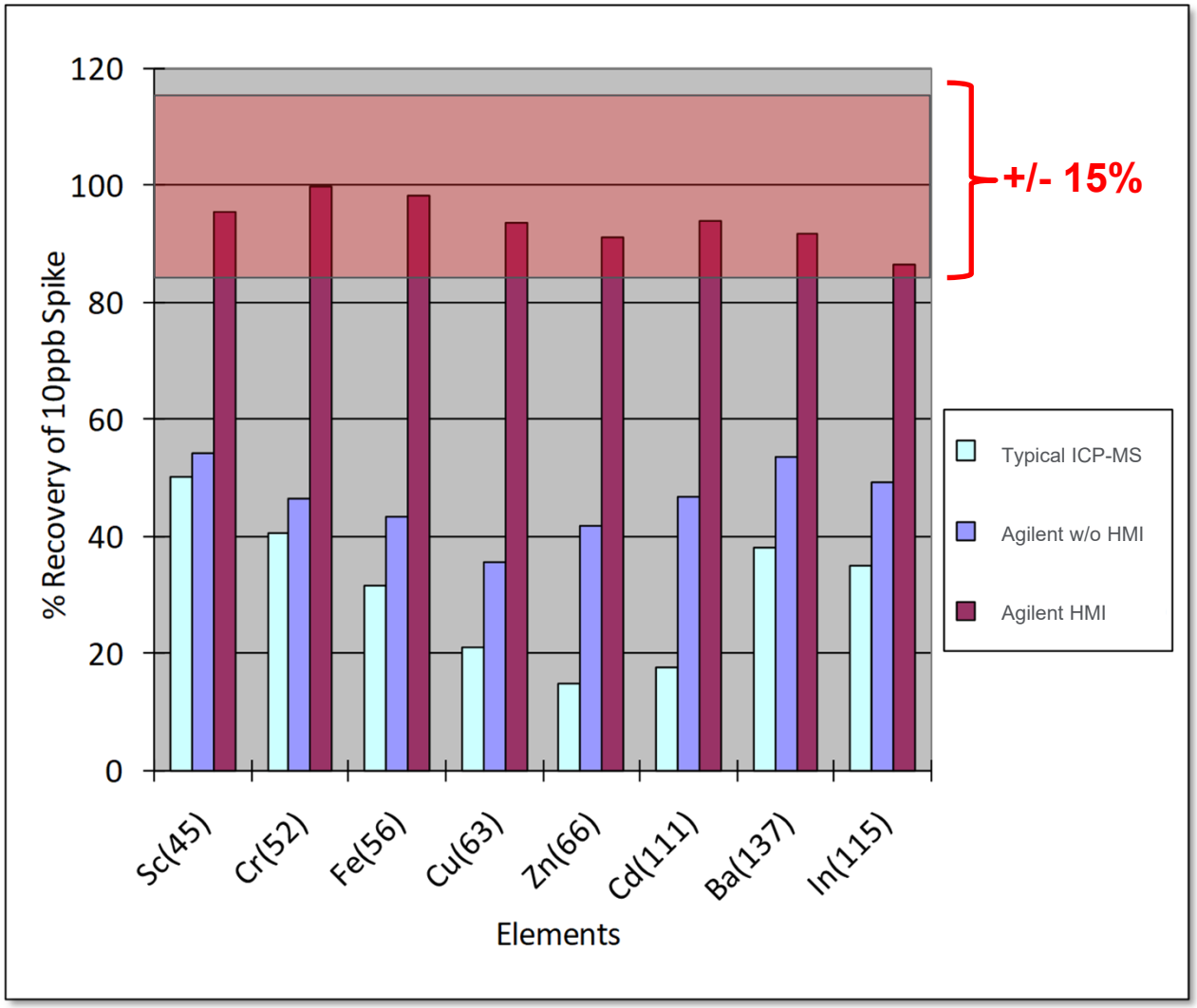

18
39.95
Ar
argon
15.760
-189.2
27.62
-185.9
24
52.00
Cr
chromium
6.765
1890
16.49
2212
30
65.39
Zn
zinc
9.394
419.6
17.964
903
11
22.99
Na
sodium
5.139
97.81
47.29
881
Io ssion i
120
100
80
60
40
20
0
e
208Pb
+
208Pb
+ 2 e
Sc-45
Cr-52
Fe-56
Zn-66
Mo-95
In-115
1% HNO3
0.3%NaCl

Ionization Suppression as a Function of Oxides



HMI – Effect on Matrix Suppression in Undiluted Seawater (~3% NaCl)



HMI dilutes aerosol density & water vapor, as well as sample matrix.

Gives much higher plasma temp; much better matrix decomposition. Matrix suppression is almost eliminated.

Plot shows % recovery in undiluted seawater vs aqueous calibrations.

With HMI, ALL results are within +/- 15% recovery (shaded area)

Without HMI, ALL results are below 60% recovery

Sample Introduction – HMI

US007671329B2

(12) **United States Patent**
Sakata et al.

(10) **Patent No.: US 7,671,329 B2**
(45) **Date of Patent: Mar. 2, 2010**

(54) **INDUCTIVELY COUPLED PLASMA MASS SPECTROMETER**

(75) Inventors: **Kenichi Sakata**, Tokyo (JP); **Noriyuki Yamada**, Tokyo (JP)

(73) Assignee: **Agilent Technologies, Inc.**, Santa Clara, CA (US)

(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 231 days.

(21) Appl. No.: **11/825,702**

(22) Filed: **Jul. 9, 2007**

(65) **Prior Publication Data**

US 2008/0035844 A1 Feb. 14, 2008

(30) **Foreign Application Priority Data**

Aug. 11, 2006 (JP) 2006-219520

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JP	11-006788	1/1999

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Primary Examiner—David A Vanore
Assistant Examiner—Michael Maskell
(74) *Attorney, Agent, or Firm*—Marc Bobys

Introducing the Ground-Breaking Agilent UHMI

- UHMI (Ultra High Matrix Interface) – much more than just a simple T-piece
- UHMI uses optimized gas mixing geometry and sophisticated plasma/gas-flow tuning algorithm to set reproducible conditions for predictable aerosol dilution rate

“Big Four” Spiked into Different Salt Matrices

NaCl Amount	75 As [25 ppb]	114 Cd [50 ppb]	208 Pb [50 ppb]	201 Hg [1 ppb]
0%	26.9	49.2	49.7	0.85
0.5%	24.2	49.0	50.1	0.99
1%	24.8	51.5	50.2	0.93
1.5%	25.5	50.0	50.5	0.88
2%	24.6	50.0	49.7	1.03
5%	25.4	48.7	50.7	0.89
10%	22.8	46.1	49.8	0.91
25%	26.2	45.4	49.0	0.96
Average	**25.1**	**48.7**	**50.0**	**0.93**
% Recovery	**100%**	**97%**	**100%**	**93%**
% RSD	**5%**	**4%**	**1%**	**6%**

Interfered Elements Spiked into Different Salt Matrices

NaCl Amount	51 V [50 ppb]	52 Cr [50 ppb]	60 Ni [50 ppb]	63 Cu [50 ppb]
0%	49.2	49.1	49.9	49.6
0.5%	47.3	50.3	48.3	48.6
1%	49.5	49.3	48.8	48.8
1.5%	50.5	50.3	49.9	49.6
2%	49.7	49.1	49.4	48.7
5%	48.9	50.3	47.2	47.7
10%	47.8	50.3	46.3	47.7
25%	48.0	48.7	50.9	50.5
Average	**48.9**	**49.7**	**48.8**	**48.9**
% Recovery	**98%**	**99%**	**98%**	**98%**
% RSD	**2%**	**1%**	**3%**	**2%**

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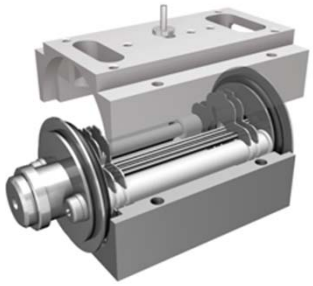
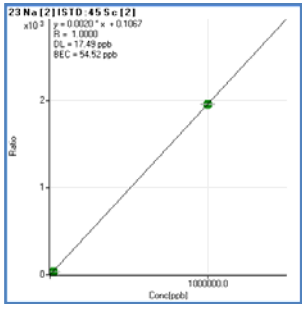


Octopole Reaction System (ORS⁴) He Mode

Simple, effective removal of all common interferences.

3) Dynamic Range



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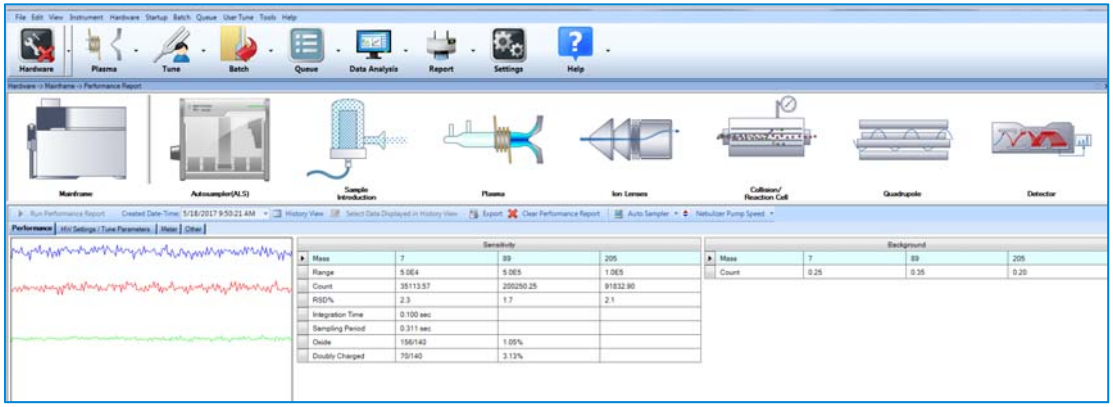

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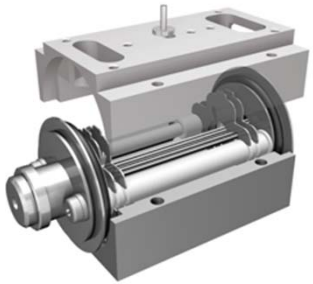

Octopole Reaction System (ORS^4) He Mode

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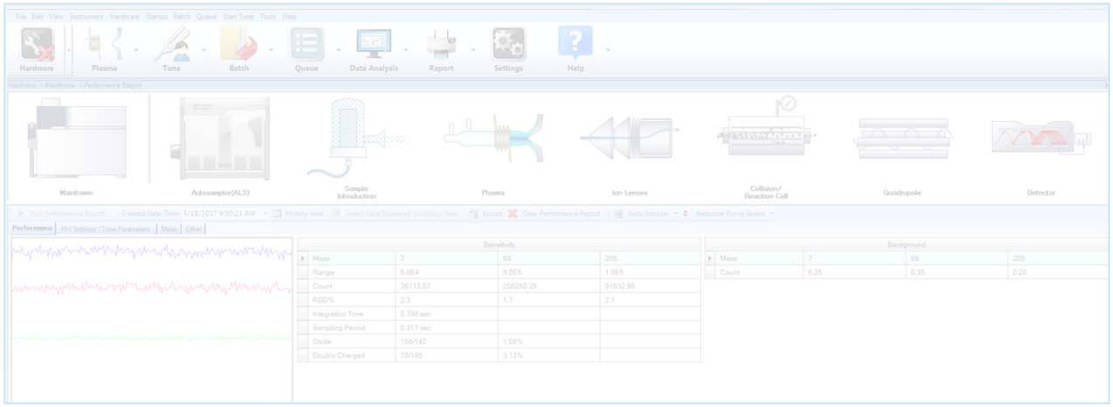
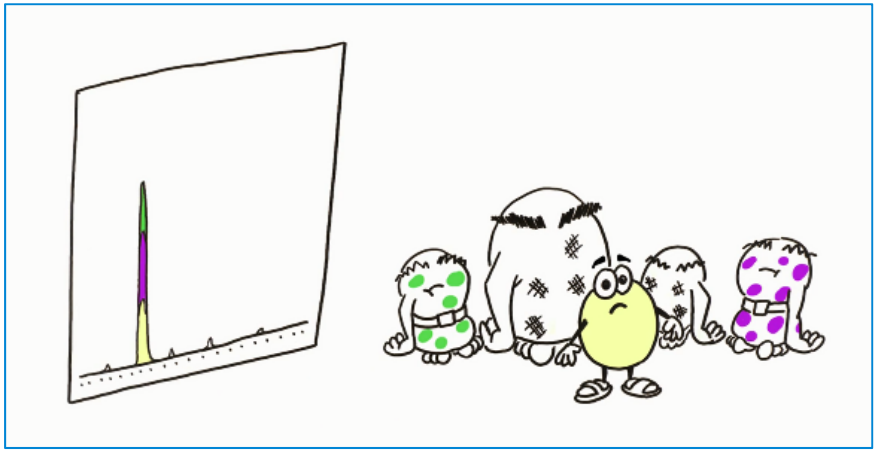
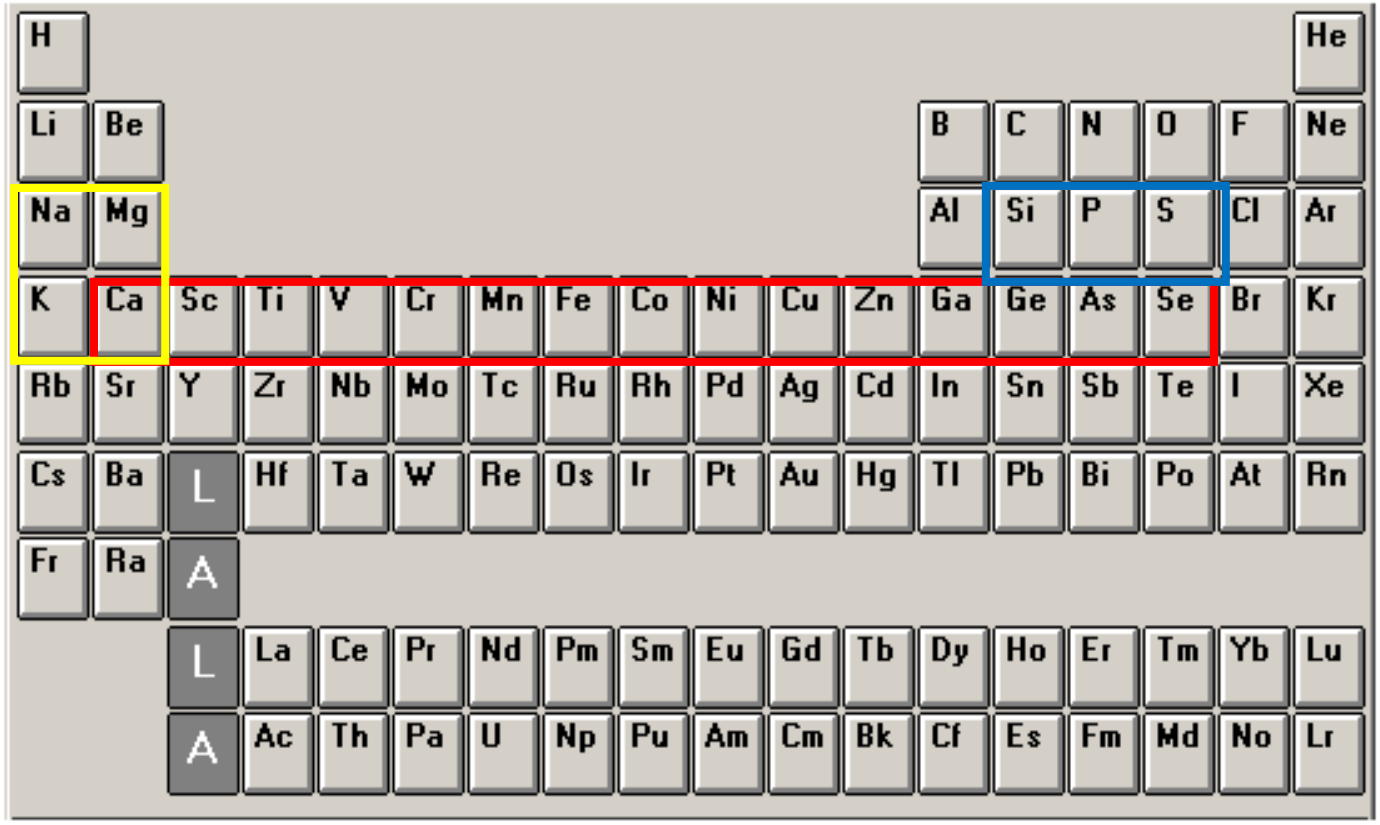


What about Interferences?

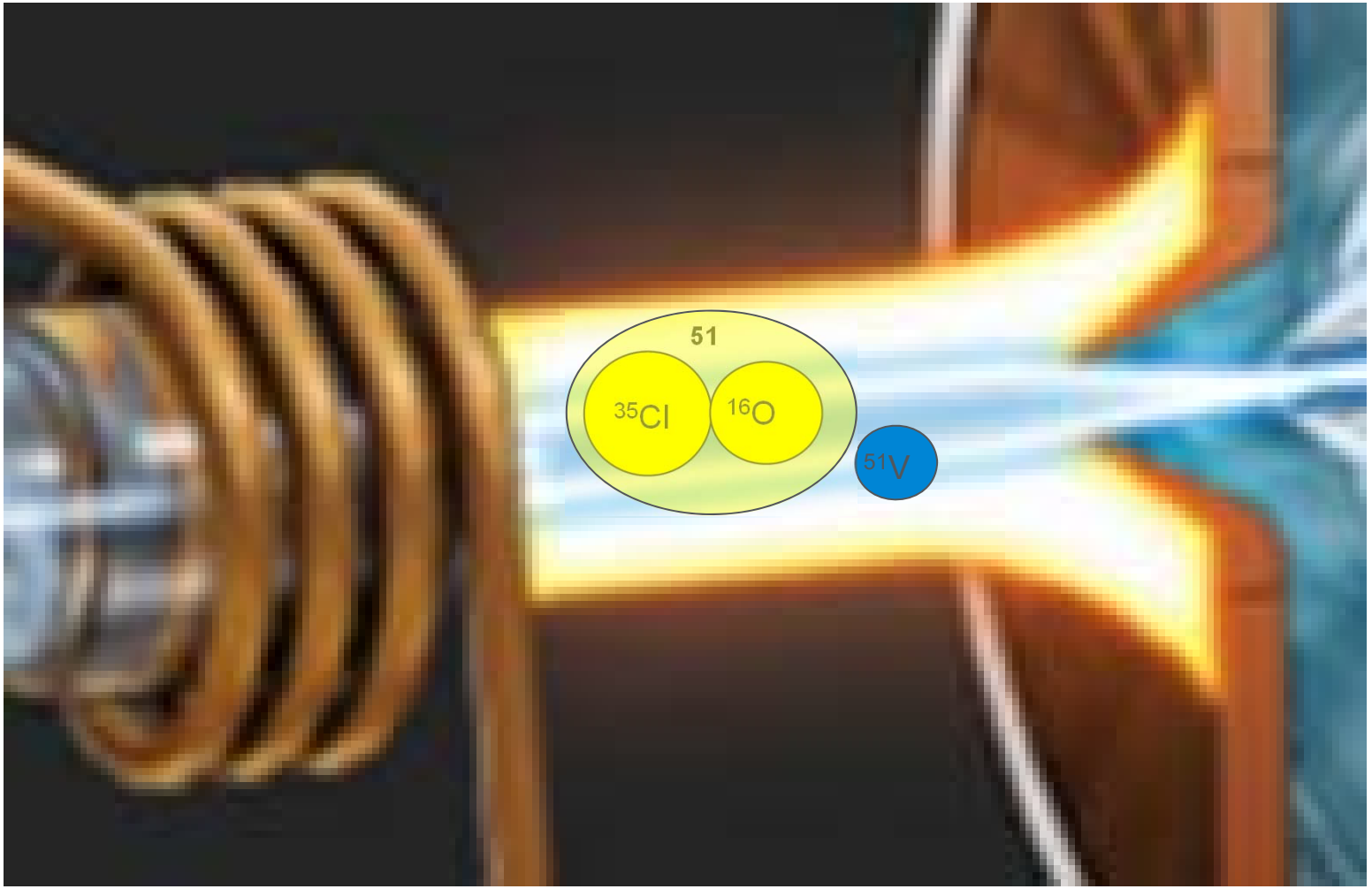
- Three main types of interferences
  - Spectroscopic
  - Physical
  - Memory
- How to control them?
  - Cell technologies
  - HMI / UHMI
  - Discrete sampling techniques



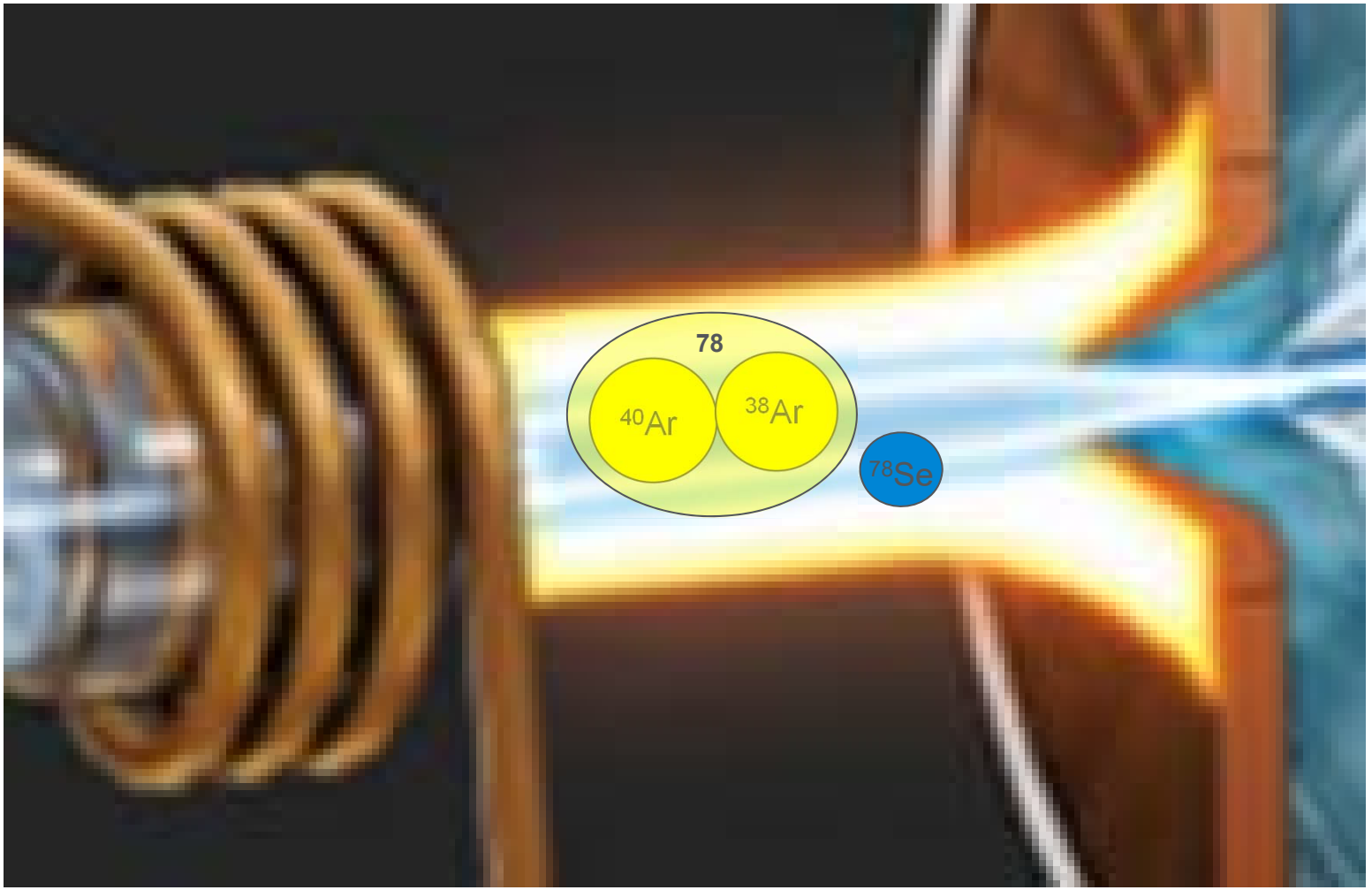
Troublesome Region of the Periodic Table



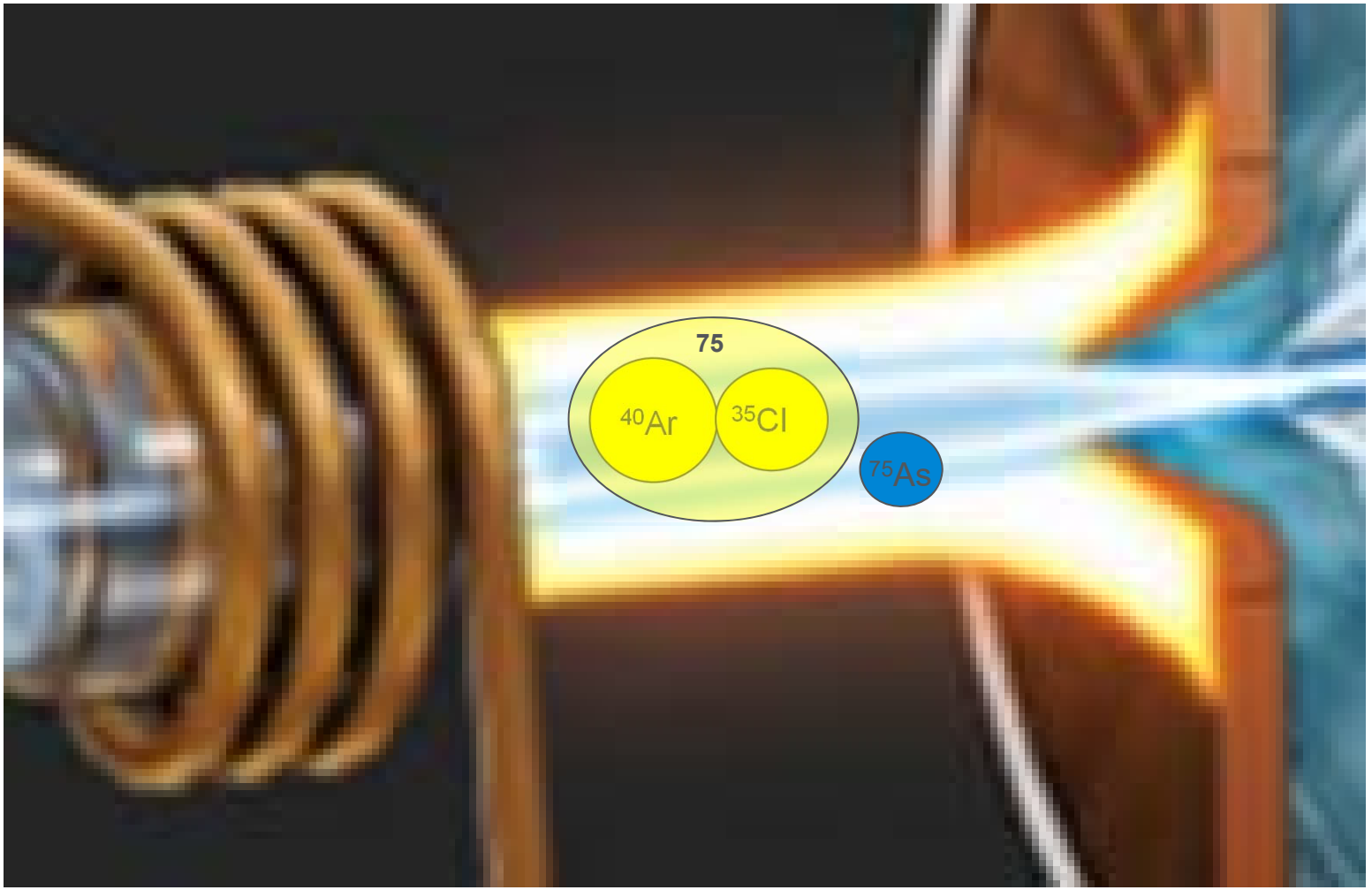
Polyatomic Interference Formation - Matrix



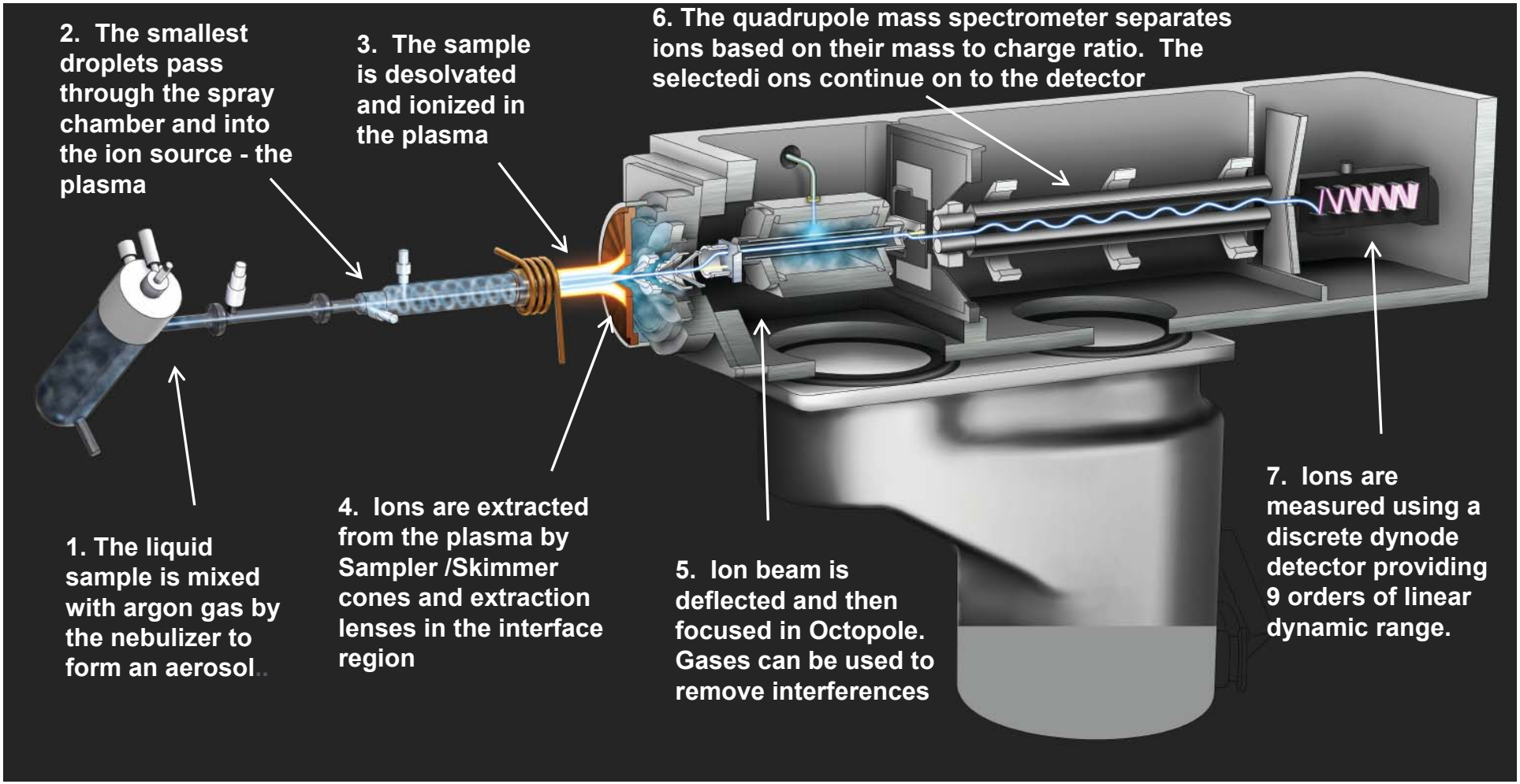
Polyatomic Interference Formation - Argon



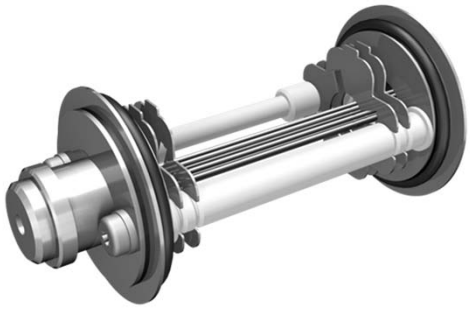
Polyatomic Interference Formation - Both



ICP-MS with ORS Technology



Processes of Interference Removal in Collision/Reaction Cell



Collisional Dissociation

- Limited in ICP-MS, as collision energy must be higher than bond dissociation energy

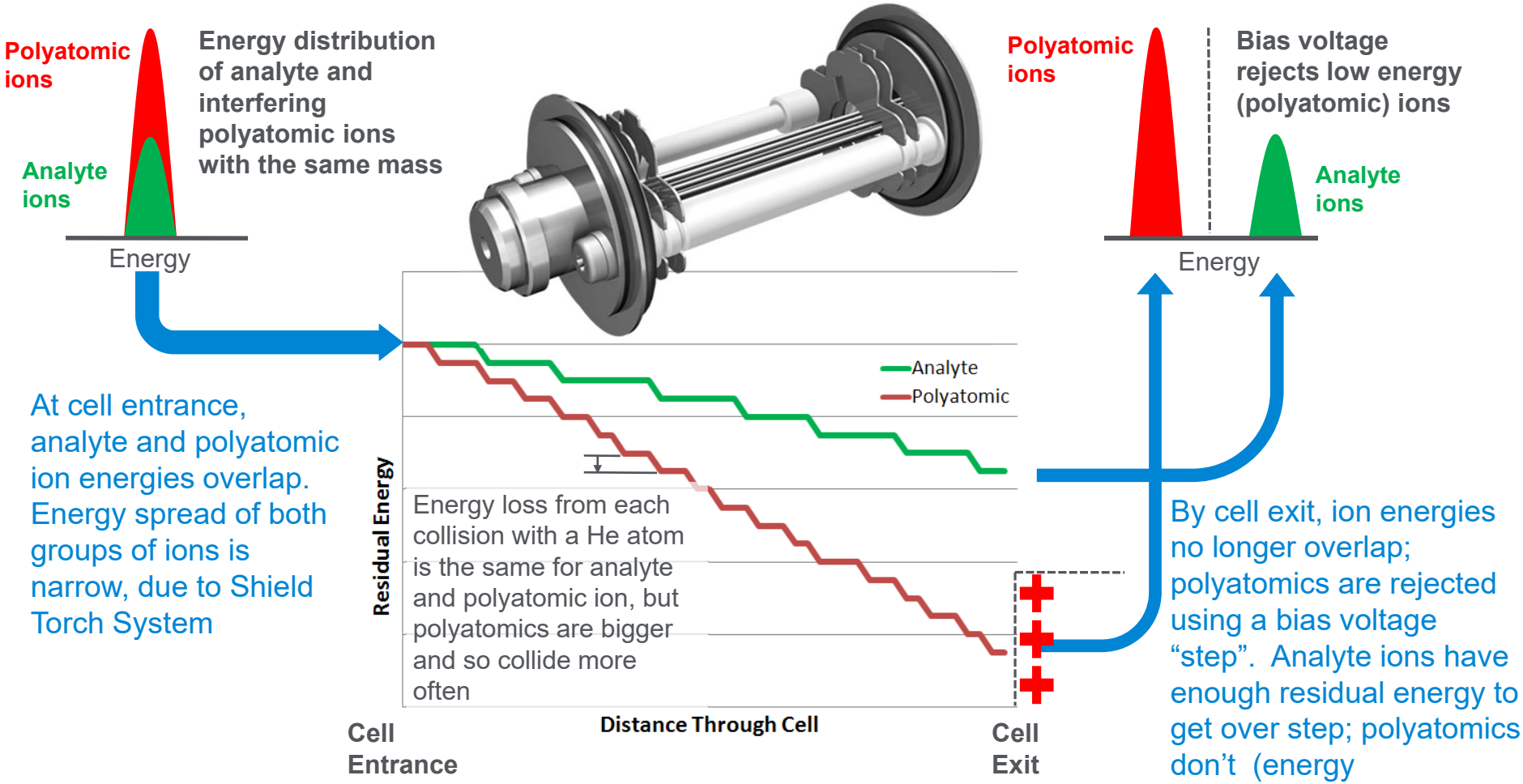
Reaction

- Can be very efficient – up to 9 orders reduction – but can also be non-selective. Highly reactive gases may react with analytes, matrix components and residual cell contamination, giving analyte loss and the formation of complex cluster ions

Energy Discrimination

- Useful in complex, variable and unknown matrices, as interference removal occurs, regardless of the level, source and chemistry of the interfering species. Can use inert cell gas, so no reaction with analytes and no formation of new cluster ions

Principle of Cell Gas Mode and KED for removing polyatomic interferences*



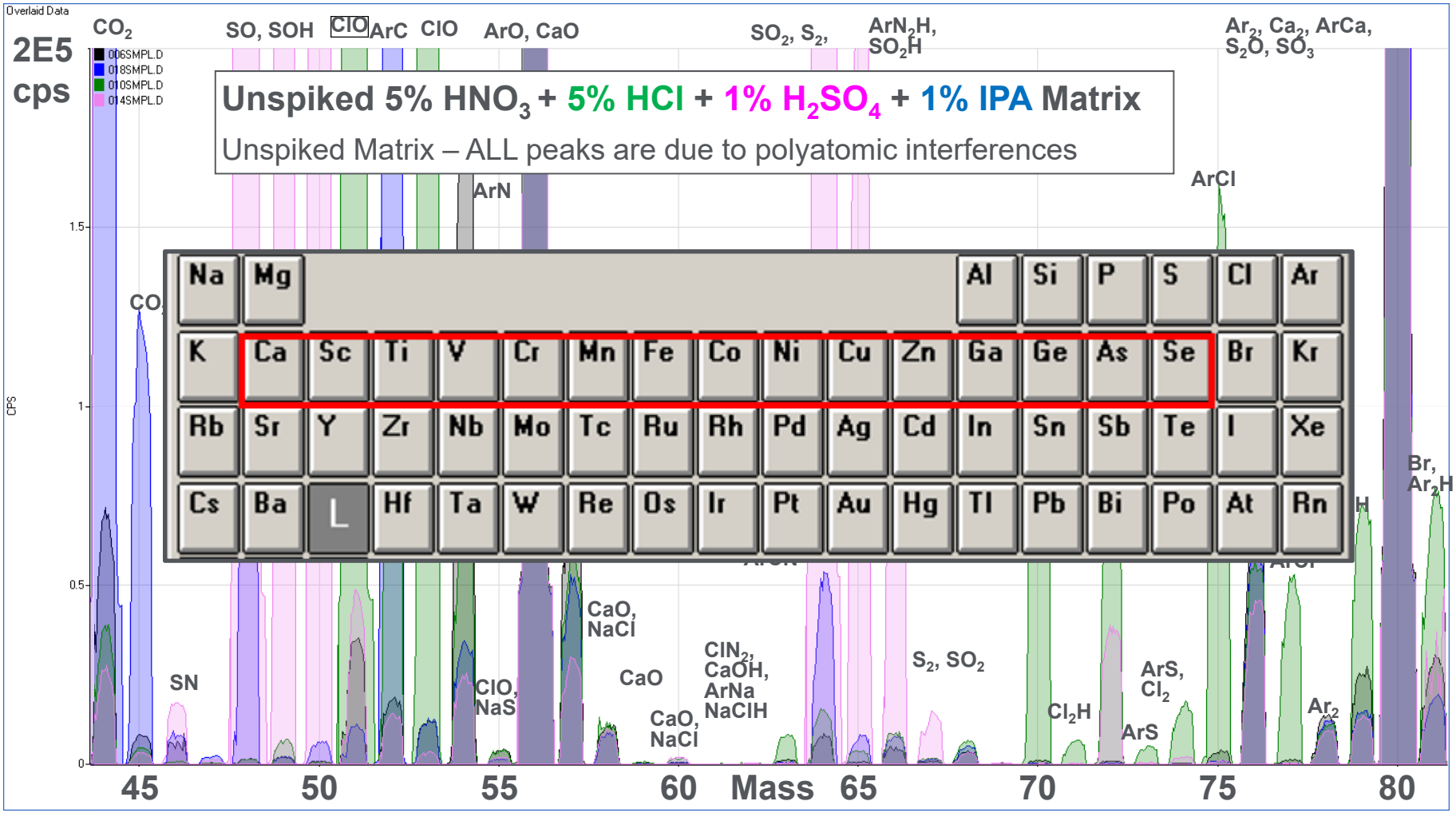
At cell entrance, analyte and polyatomic ion energies overlap. Energy spread of both groups of ions is narrow, due to Shield Torch System

By cell exit, ion energies no longer overlap; polyatomics are rejected using a bias voltage "step". Analyte ions have enough residual energy to get over step; polyatomics don't (energy discrimination)

***KED = Kinetic Energy Discrimination**

Polyatomic Interferences in No Gas Mode

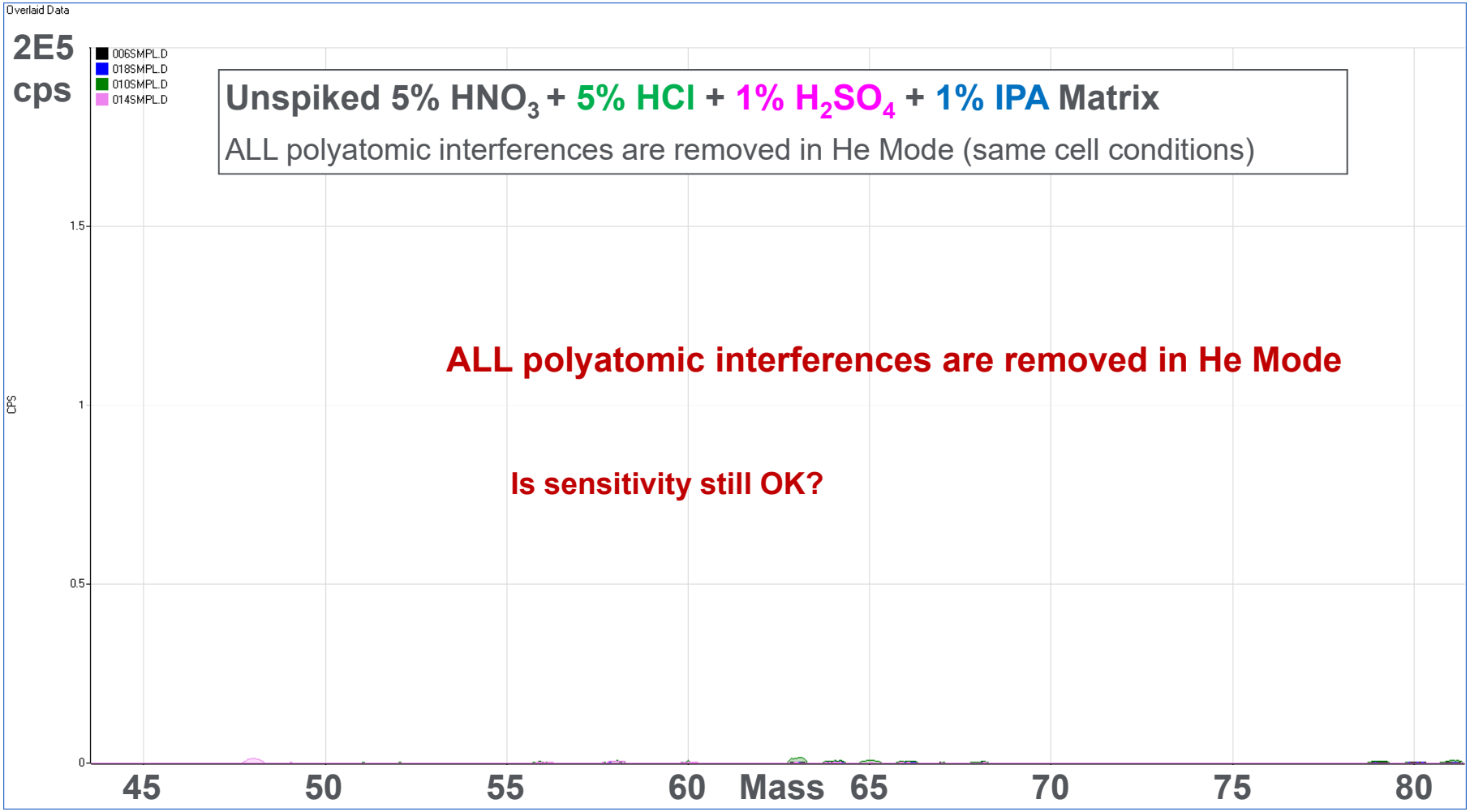
Color of spectrum indicates which matrix gave each interfering peak



No Gas Mode

Polyatomic Interferences in He Mode

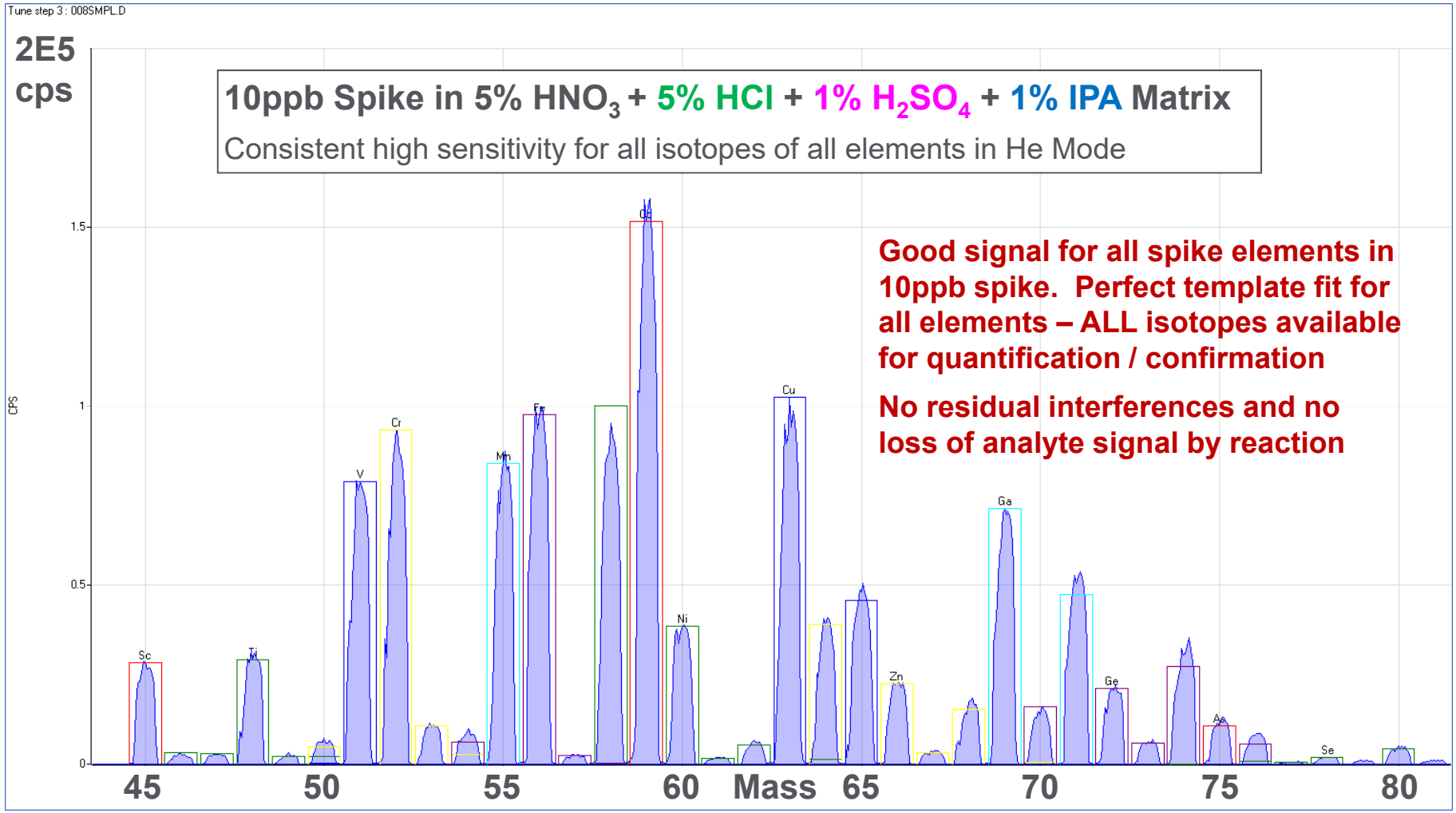
Color of spectrum indicates which matrix gave each interfering peak



He Mode

Matrix Mix with Spike (10ppb) in He Mode

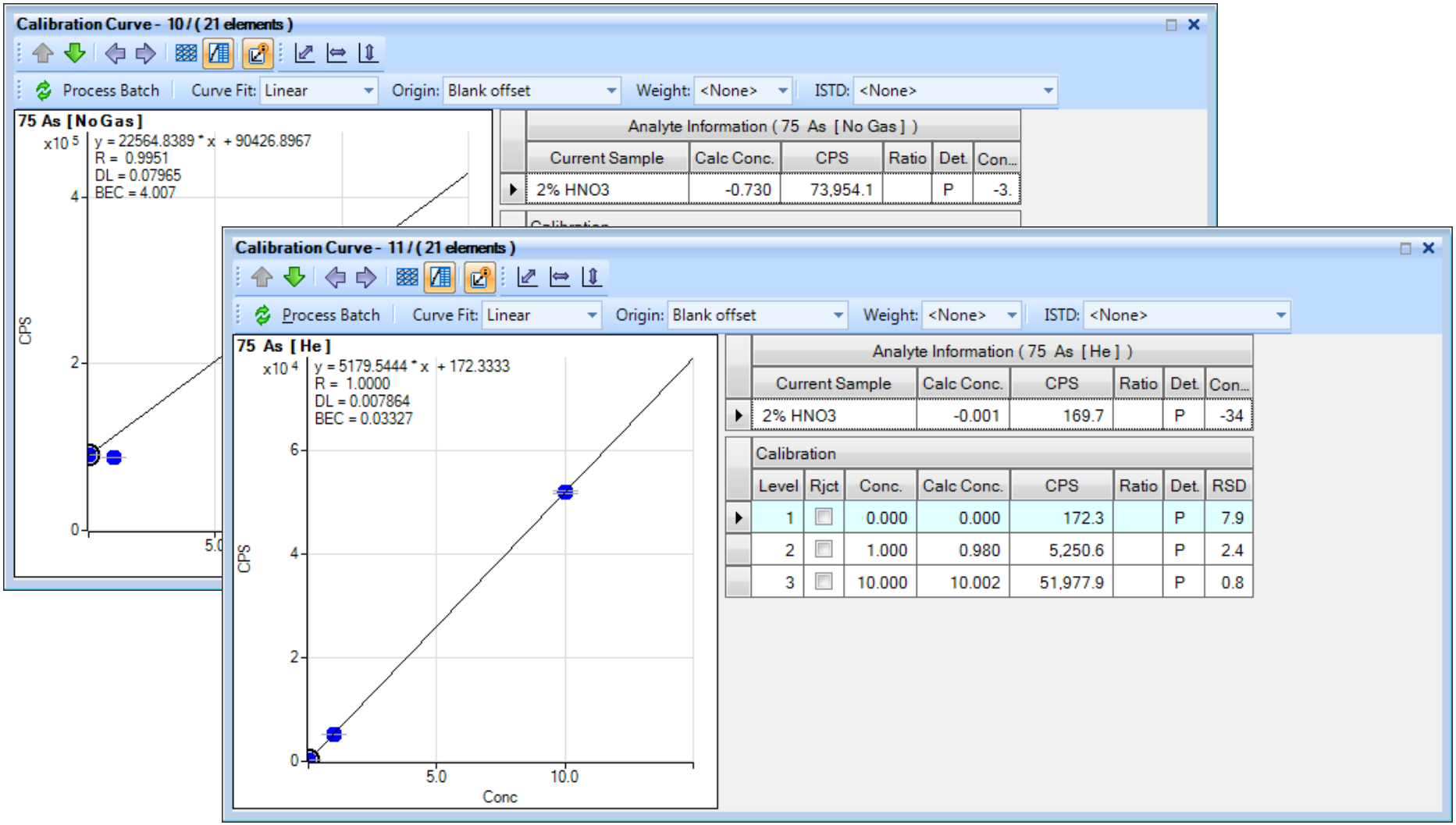
Consistent sensitivity and perfect template match for all elements



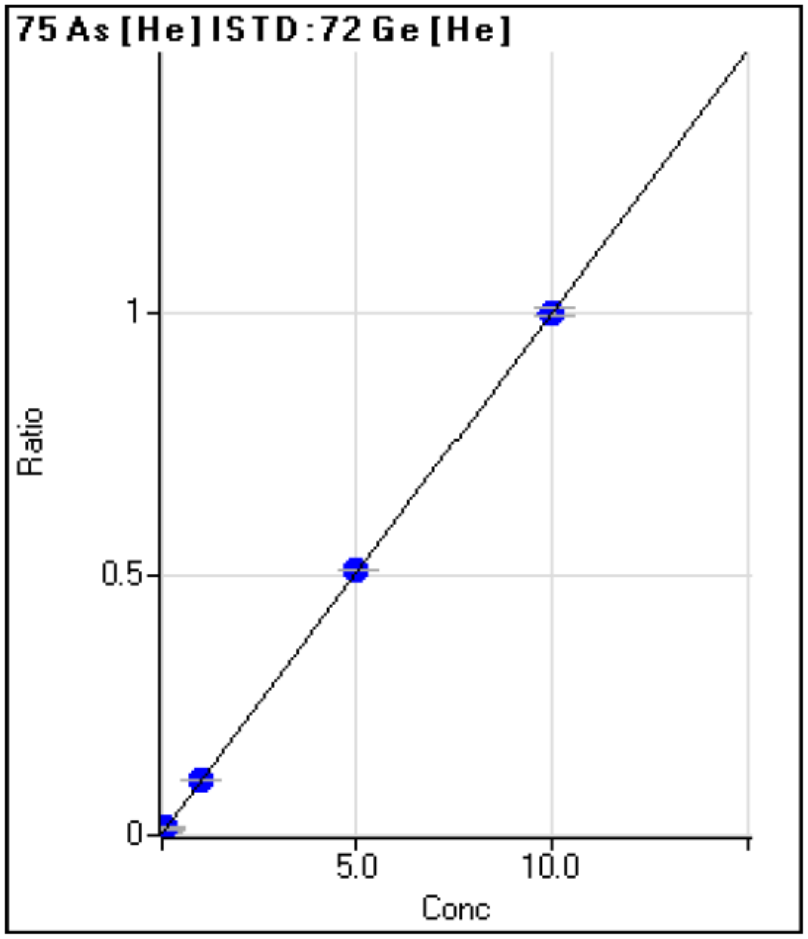
He Mode

Cal 0, 1, 10 ppb Arsenic in 1%HNO3/0.5% HCl

(ArCl interference on m/z 75)



Helium Mode Eliminates ArCl Interference on As



	Rjct	Conc.	Calc Conc.	CPS	Ratio	Det.	RSD
1	☐	0.000	0.000	48.33	0.0007	P	12.
2	☐	0.100	0.100	762.35	0.0106	P	7.
3	☐	1.000	1.017	7487.12	0.1021	P	1.
4	☐	5.000	5.041	36672.57	0.5033	P	0.
5	☐	10.000	9.978	73250.29	0.9954	P	1.
6	☐						

y = 0.0997 * x + 6.6377E-004

R = 1.0000

DL = 0.002449

BEC = 0.006658

Weight: <None>

Min Conc: <None>

Calibration standards were prepared in 2%HNO3 / 1% HCl (in order to stabilize Hg). Notice complete elimination of ArCl.

Largest Analytical Range of any ICP-MS

Calibration ranges

Hg (10 – 200ppt) – NoGas Mode
As (10 – 200 ppt) – He Mode
Se (10 – 200 ppt) – He Mode
Na (0.05 – 1000 ppm) – He Mode

Overall calibration range 10ppt (Hg, As, Se) to 1000 ppm (Na) in a single method
- *without* attenuating ion transmission to increase working range

Na

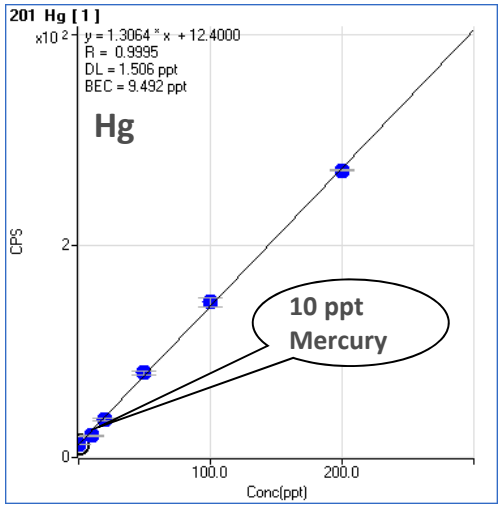
Typically, ICP-MS cannot measure above 200ppm Na without changing quad resolution or ion lens settings

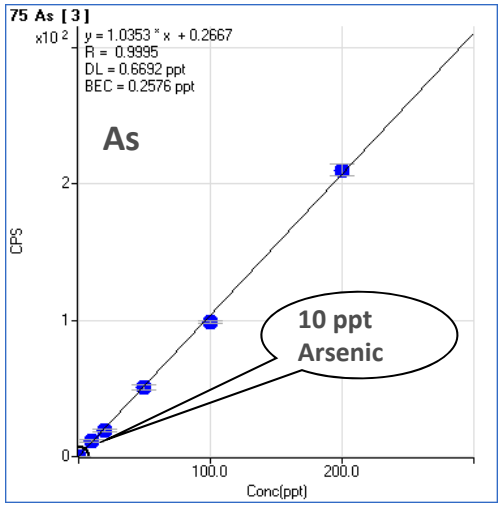
Hg

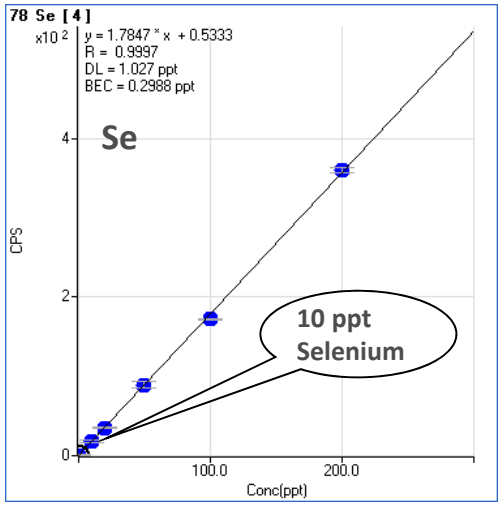
Hg LOD on 7800 is about 2ppt – 7800 can QUANTITATE at 10ppt!

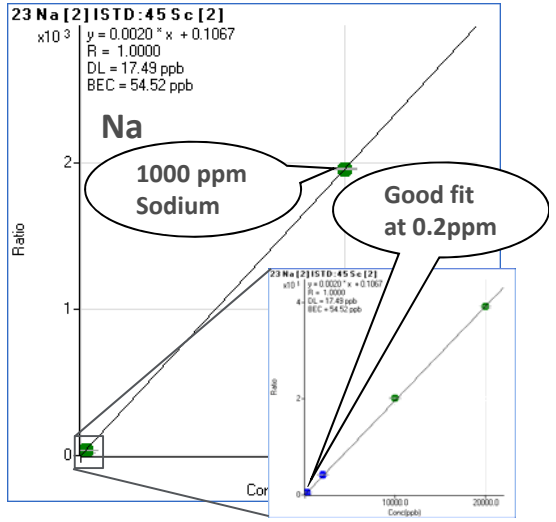
7800: 10 orders (1000's ppm)
7900: 11 orders (% Level)

These 4 plots were obtained under the same analytical conditions on the 7800 – only the gas mode (NoGas for Hg) changed



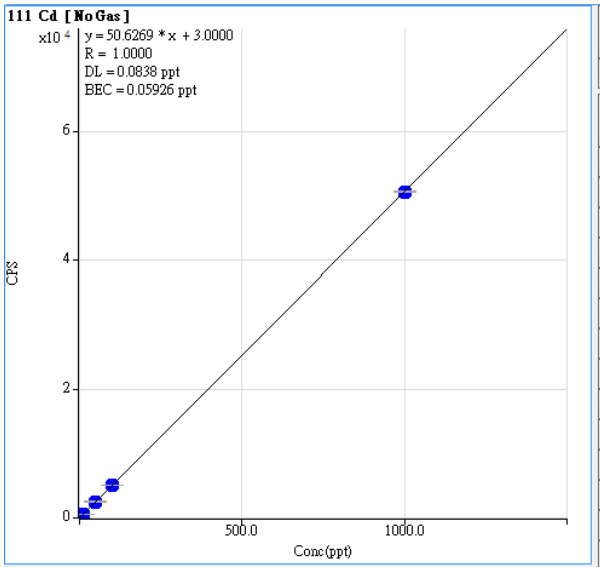






Far Wider Dynamic Range Than Any Other ICP-MS
11 orders - low and high level calibrations in one run

Cd (1ppt - 1ppb) and Na (100ppb - 10,000ppm (1%)) in the same run



Analyte Information (111 Cd [No Gas])

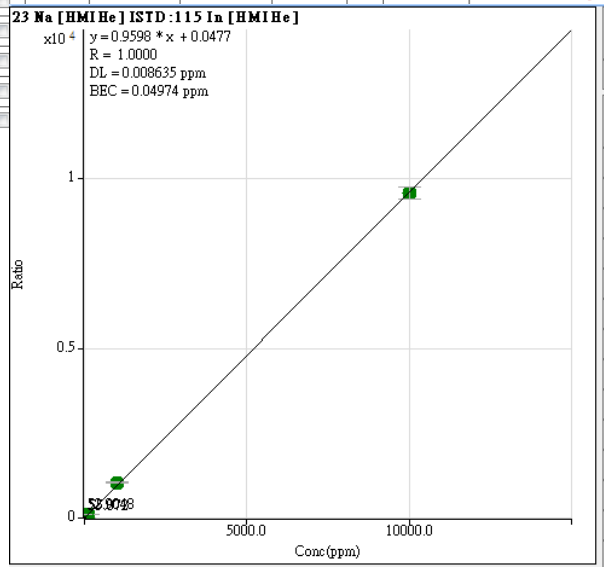
Current Sample	Calc Conc.	CPS	Ratio	Det.	Conc. RSD
1ppbBeAsCdHgPb	1000.006	50630.23		P	0.4

Calibration

Level	Rjct	Conc.	Calc Conc.	CPS	Ratio	Det.	RSD
1		0.000	0.000	3.00		P	47.1
2		1.000	0.960	51.60		P	14.9
3		10.000	10.078	513.21		P	3.8
4		50.000	49.851	2526.83		P	2.9
5		100.000	100.008	5066.11		P	2.2
6		1000.00	1000.006	50630.23		P	0.4
7							
8							
9							
10							
11							
12							
13							
14							

Both calibrations are linear.

Total **concentration range** covered from Cd blank (BEC of <0.1ppt) to Na top standard (1%) is 11 orders



Analyte Information (23 Na [HMI He] ISTD: 115 In [HMI He])

Current Sample	Calc Conc.	CPS	Ratio	Det.	Conc. RSD
rinse	55.072	3281161.01	52.9048	A	31.7

Calibration

Level	Rjct	Conc.	Calc Conc.	CPS	Ratio	Det.	RSD
1		0.000	0.000	1944.19	0.0477	P	5.8
2		0.100	0.109	6393.55	0.1528	P	2.9
3		1.000	1.049	43220.26	1.0541	P	1.3
4		10.000	10.192	421823.28	9.8296	P	3.2
5		100.000	99.124	4004216.88	95.1848	A	1.7
6		1000.00	1089.335	40455458.40	1,045.570	A	1.5
7		10000.0	9991.075	328778051.1	9,589.288	A	3.7
8							
9							
10							
11							
12							
13							
14							

Enviro: 7800 HMI / Estuarine Samples: 40-75% Seawater

~1745 ppm Na in Solution

~860 ppm Na in Solution

~430 ppm Na in Solution

Sample Name				
Acq. Date-Time				10/20/2009 5:59 PM
Sample Name	Dilution	5	10	20
Dilution				Sample C 20x
9 Be [NoGas]	9 Be [NoGas]	0.0[illegible]0	0.0[illegible]7	0.[illegible]86
23 Na [He]	23 Na [He]	8726108	8611994	8628625
24 Mg [He]	24 Mg [He]	1040282	1031898	1058318
27 Al [He]	27 Al [He]	1.6166	0.6615	3.7364
39 K [He]	39 K [He]	380963	371819	365013
44 Ca [He]	44 Ca [He]	414310	409728	404271
51 V [HEHe]	51 V [HEHe]	1.4565	1.2708	1.2975
52 Cr [He]	52 Cr [He]	0.2165	0.3066	0.3074
55 Mn [He]	55 Mn [He]	1.0943	1.1272	0.9813
56 Fe [He]	56 Fe [He]	<2.5000	<5.0000	<10.000
59 Co [He]	59 Co [He]	0.0221	0.0234	0.0266
60 Ni [He]	60 Ni [He]	0.2081	0.2032	<0.6000
63 Cu [He]	63 Cu [He]	0.2909	0.2419	0.2371
66 Zn [He]	66 Zn [He]	0.0254	0.1200	0.4770
75 As [He]	75 As [He]	1.3844	1.4452	1.4770
78 Se [HEHe]	78 Se [HEHe]	0.3[illegible]3	0.3[illegible]3	<0.4[illegible]00
95 Mo [NoGas]				5.4514
107 Ag [NoGas]				<0.1000
111 Cd [NoGas]				0.0497
121 Sb [NoGas]				0.4872
137 Ba [NoGas]				25.616
201 Hg [NoGas]				0.1662
205 Tl [NoGas]				<0.1000
208 Pb [NoGas]				<0.5000
232 Th [NoGas]				<0.0600
238 U [NoGas]				1.4673

~0.275 ppb As in Solution

~0.144 ppb As in Solution

~0.074 ppb As in Solution

7900 Heavy Matrix Analysis (HMI-25)

-Major constituents reported in ppm

-Trace elements reported in ppb

General Seawater Composition

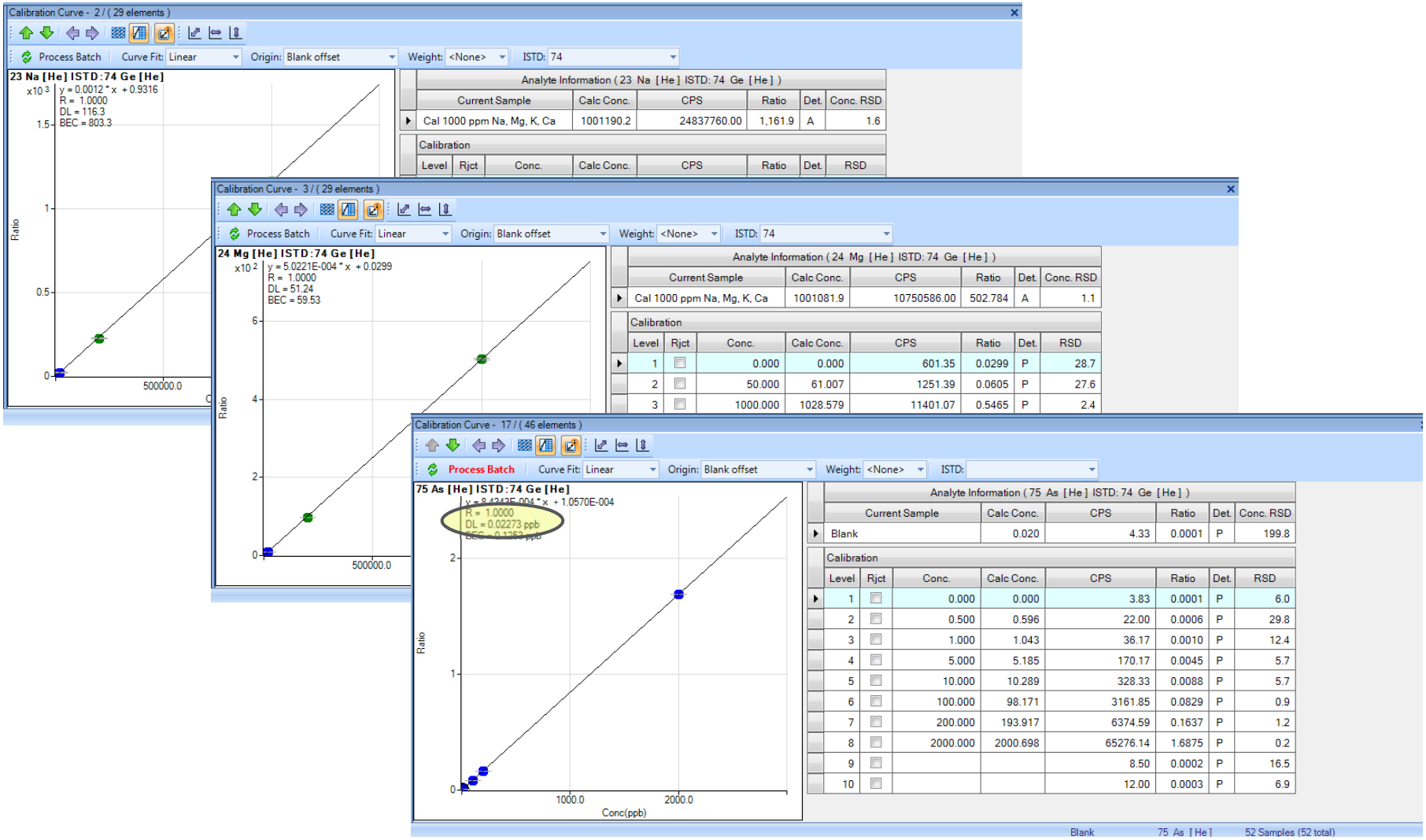
Element	At.weight	ppm
Hydrogen H2O	1.00797	110,000
Oxygen H2O	15.9994	883,000
Sodium NaCl	22.9898	10,800
Chlorine NaCl	35.453	19,400
Magnesium Mg	24.312	1,290
Sulfur S	32.064	904
Potassium K	39.102	392
Calcium Ca	10.08	411
Bromine Br	79.909	67.3

Most analytes acquired using Helium mode.

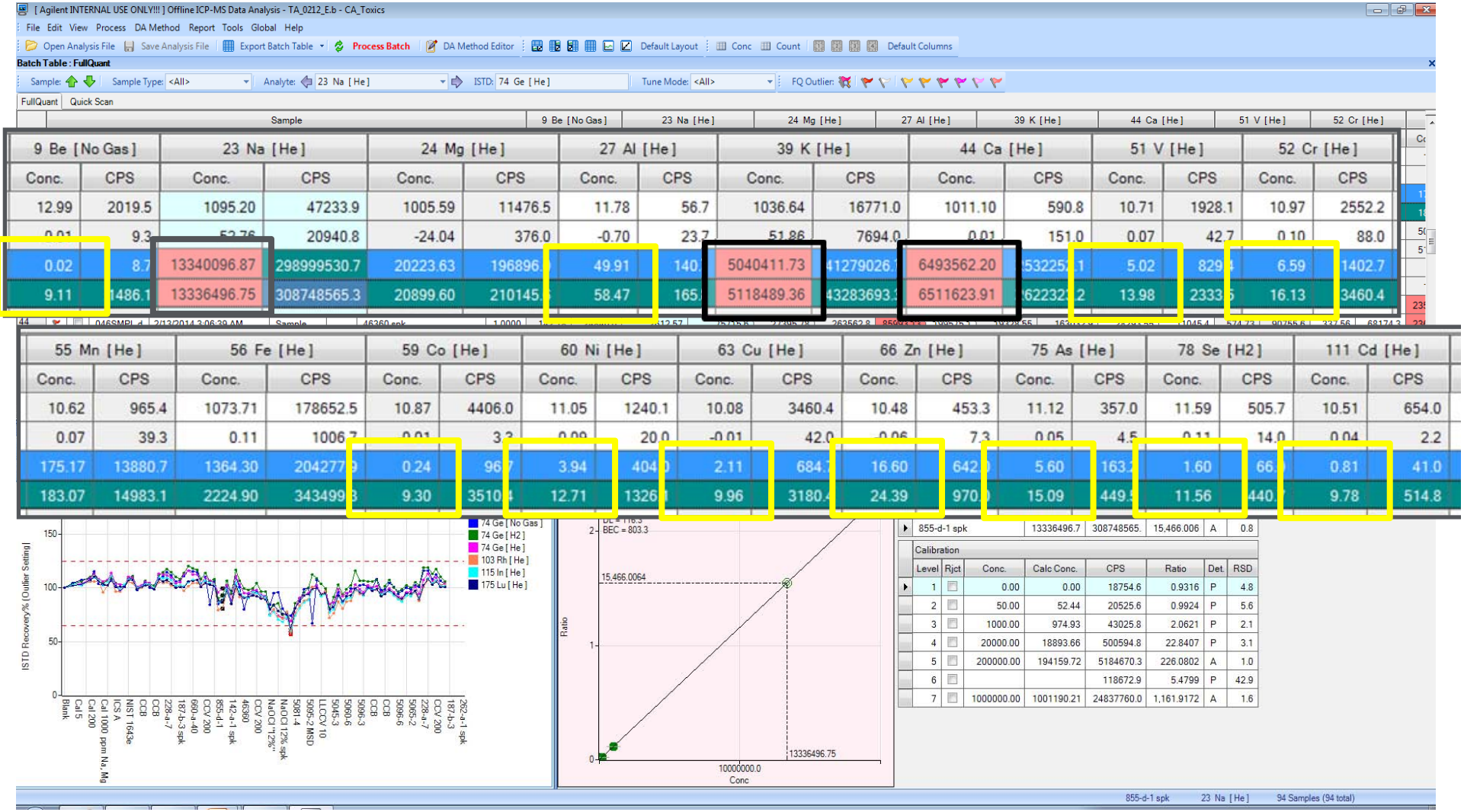
H2 Mode used for select elements to provide the best DL's (Si, Ca, Se)

		6020 Unknown Results ppb	
		(Na,Mg,S,K,Ca,Fe ppm)	
	Acq. Date-Time	9/8/2017 13:24	9/8/2017 13:27
	Type	Sample	Sample
	Level		
	Sample Name	SAMPLE A	SAMPLE B
	Total Dil.	1.11	1.11
9 Be [No Gas]	Conc. [ppb]	86.3314	83.1840
11 B [He]	Conc. [ppb]	15661.9829	16122.3051
23 Na [He]	Conc. [ppm]	1167.3130	1153.2386
24 Mg [He]	Conc. [ppm]	2812.7092	2786.6283
27 Al [He]	Conc. [ppb]	356519.5441	349253.6402
28 Si [H2]	Conc. [ppb]	48360.4536	47745.2191
34 S [He]	Conc. [ppm]	553.3986	537.7674
39 K [He]	Conc. [ppm]	61.2055	60.5476
40 Ca [H2]	Conc. [ppm]	483.6432	491.6683
51 V [He]	Conc. [ppb]	271.6009	247.0839
52 Cr [He]	Conc. [ppb]	23758.5895	22959.0748
55 Mn [He]	Conc. [ppb]	74152.6376	72835.0402
56 Fe [He]	Conc. [ppm]	544.9229	530.3951
59 Co [He]	Conc. [ppb]	1129.1326	1132.3477
60 Ni [He]	Conc. [ppb]	21512.9884	21352.7933
63 Cu [He]	Conc. [ppb]	5.3540	2.0105
66 Zn [He]	Conc. [ppb]	3902.0441	4234.4300
75 As [He]	Conc. [ppb]	13.1571	12.4169
78 Se [H2]	Conc. [ppb]	96.1427	114.5517
88 Sr [He]	Conc. [ppb]	800.8821	771.5266
95 Mo [He]	Conc. [ppb]	3.5913	2.3013
107 Ag [He]	Conc. [ppb]	0.1056	0.0166
111 Cd [He]	Conc. [ppb]	30.5723	39.1334
118 Sn [He]	Conc. [ppb]	0.3993	0.3039
121 Sb [He]	Conc. [ppb]	0.6053	0.3508
137 Ba [He]	Conc. [ppb]	11.3219	10.4092
205 Tl [He]	Conc. [ppb]	0.5324	0.6342
208 Pb [He]	Conc. [ppb]	0.1570	0.0861

Enviro: 7900 UHMI / Very High Matrix Samples (HMI)



7900 UHMI: Samples Containing ~12% TDS



*Calibration Standards in 2% HNO3/1% HCl

4 Key Benefits of Agilent ICP-MS

1) Matrix Tolerance

Ultra High Matrix Introduction System (UHMI)

Analyze Higher TDS samples than any other instrument on the market.

2) Interference Removal

Octopole Reaction System (ORS⁴) He Mode

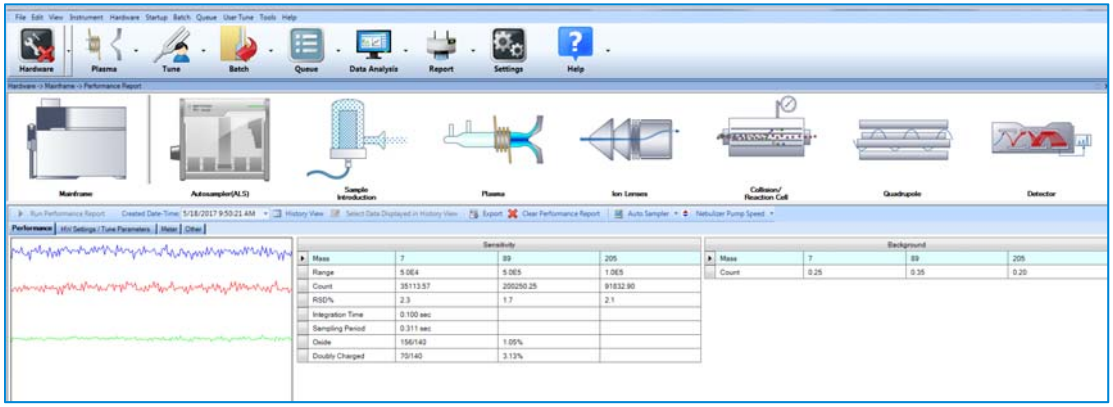
Simple, effective removal of all common interferences.

3) Dynamic Range

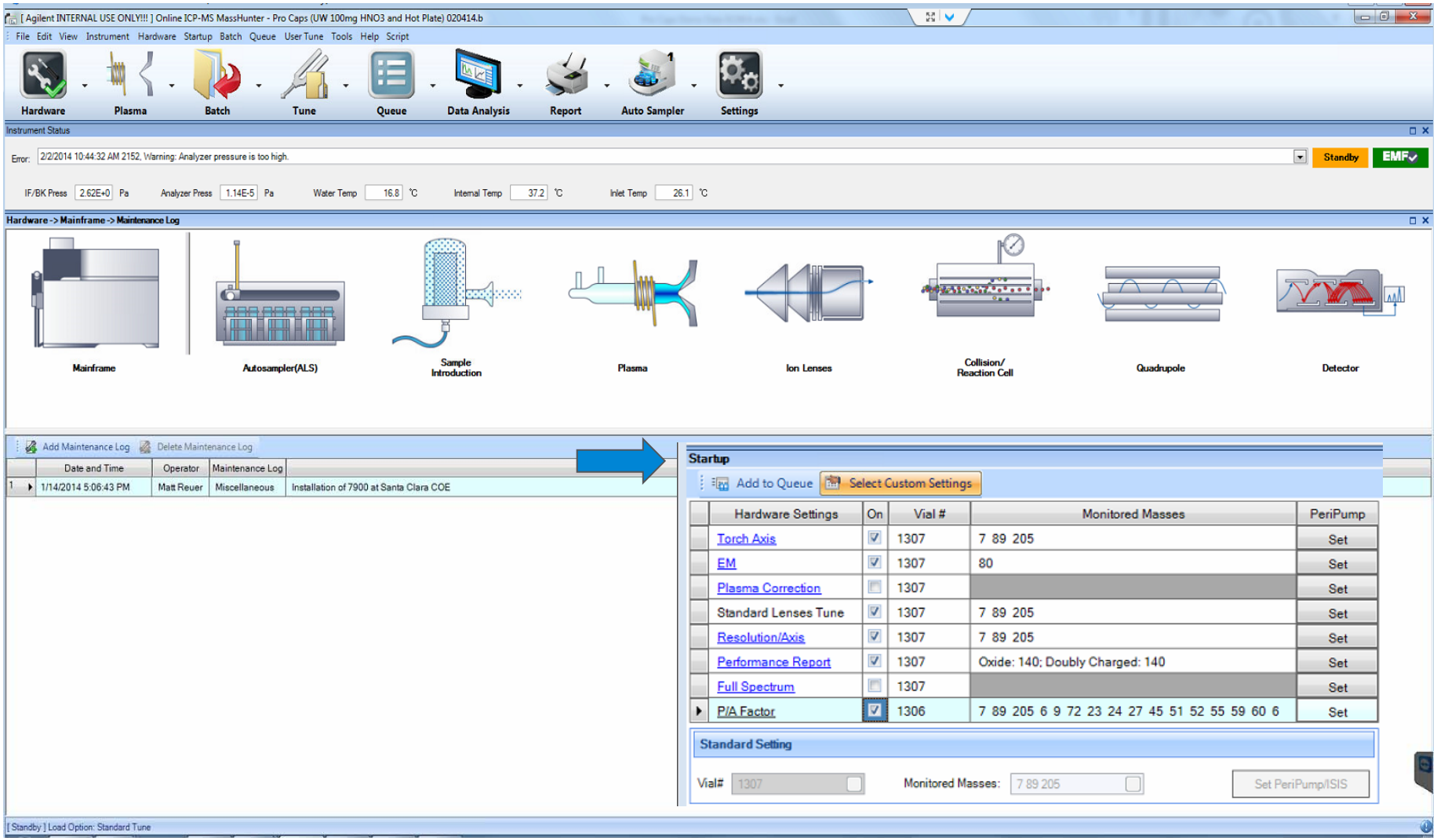
Widest Dynamic Range

Up to 11 orders dynamic range:
0.1ppt(DL) to 10,000ppm

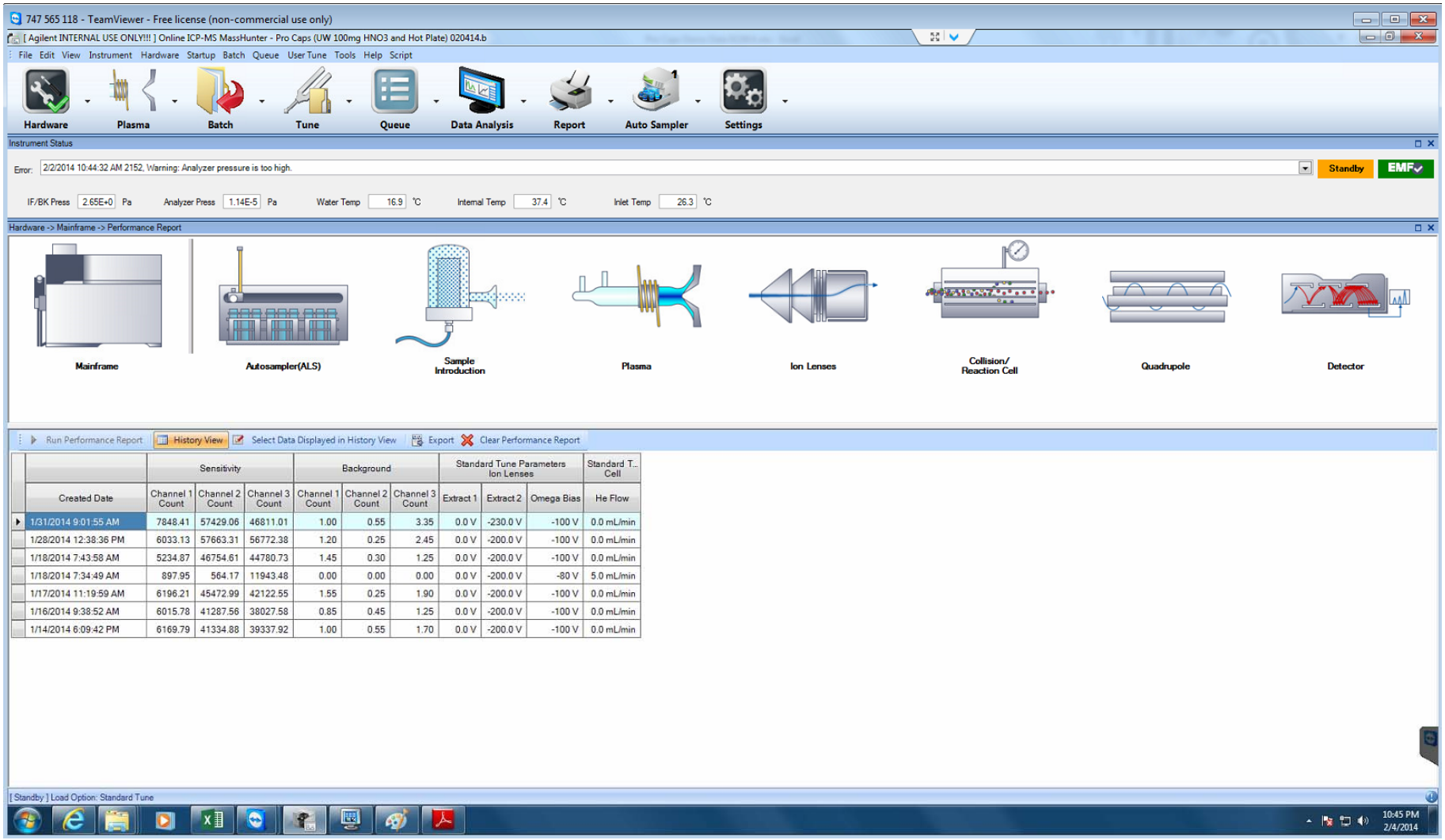
4) Mass Hunter Software



Mass Hunter 4.4: Truly Automated Software

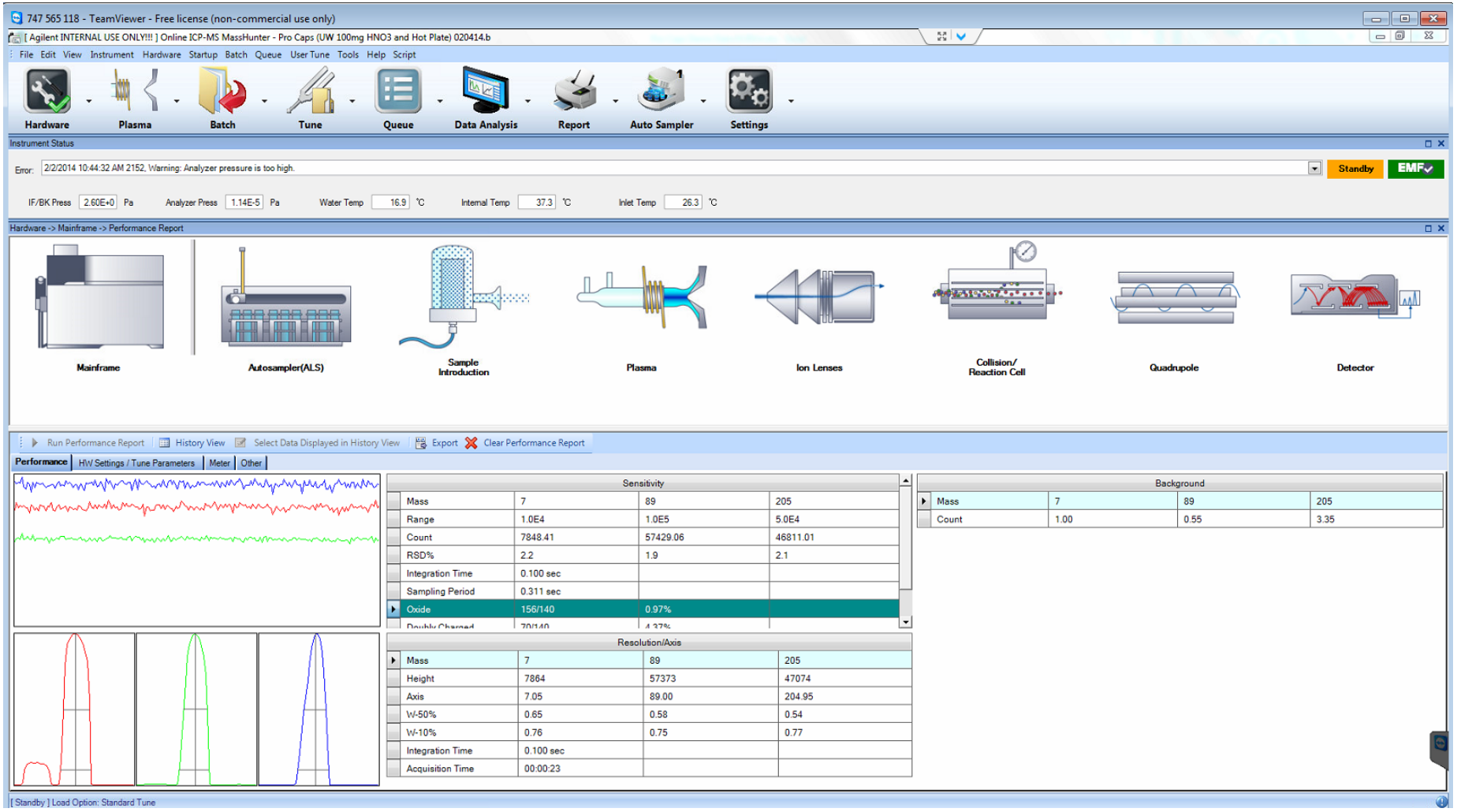


Daily Performance Log

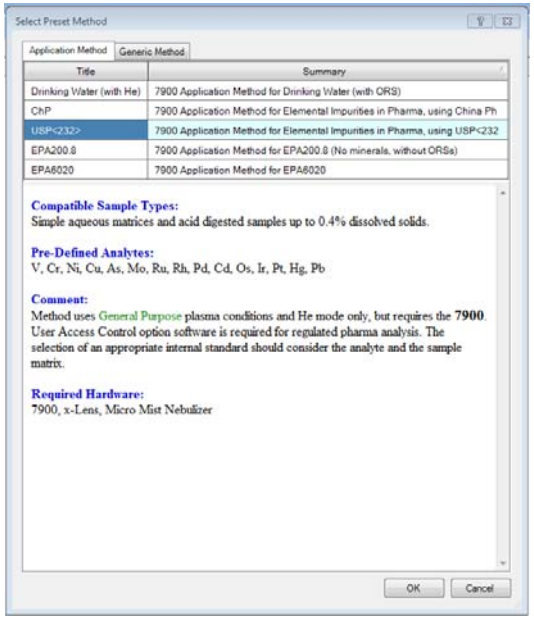


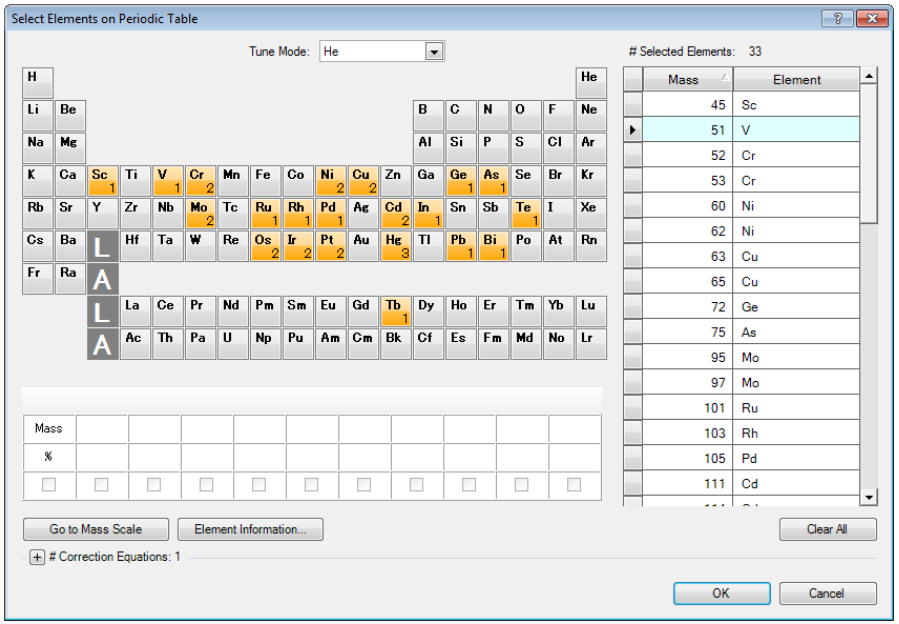
	Sensitivity			Background			Standard Tune Parameters Ion Lenses			Standard T... Cell
Created Date	Channel 1 Count	Channel 2 Count	Channel 3 Count	Channel 1 Count	Channel 2 Count	Channel 3 Count	Extract 1	Extract 2	Omega Bias	He Flow
1/31/2014 9:01:55 AM	7848.41	57429.06	46811.01	1.00	0.55	3.35	0.0 V	-230.0 V	-100 V	0.0 mL/min
1/28/2014 12:38:36 PM	6033.13	57663.31	56772.38	1.20	0.25	2.45	0.0 V	-200.0 V	-100 V	0.0 mL/min
1/18/2014 7:43:58 AM	5234.87	46754.61	44780.73	1.45	0.30	1.25	0.0 V	-200.0 V	-100 V	0.0 mL/min
1/18/2014 7:34:49 AM	897.95	564.17	11943.48	0.00	0.00	0.00	0.0 V	-200.0 V	-80 V	5.0 mL/min
1/17/2014 11:19:59 AM	6196.21	45472.99	42122.55	1.55	0.25	1.90	0.0 V	-200.0 V	-100 V	0.0 mL/min
1/16/2014 9:38:52 AM	6015.78	41287.56	38027.58	0.85	0.45	1.25	0.0 V	-200.0 V	-100 V	0.0 mL/min
1/14/2014 6:09:42 PM	6169.79	41334.88	39337.92	1.00	0.55	1.70	0.0 V	-200.0 V	-100 V	0.0 mL/min

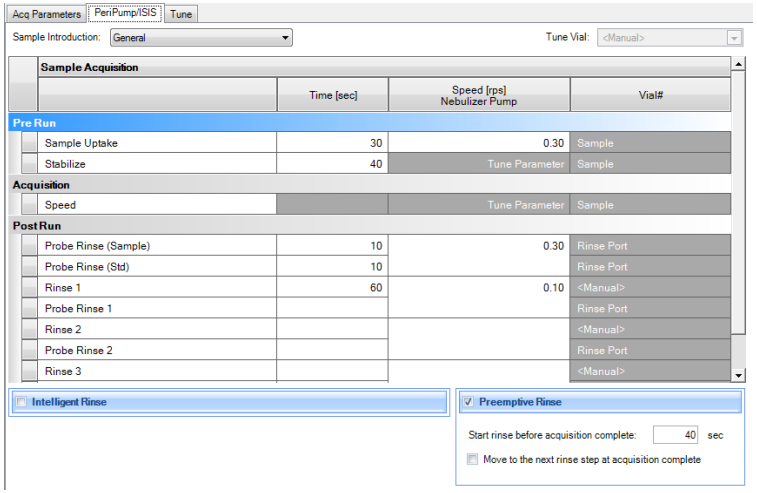
Detail View of Autotune

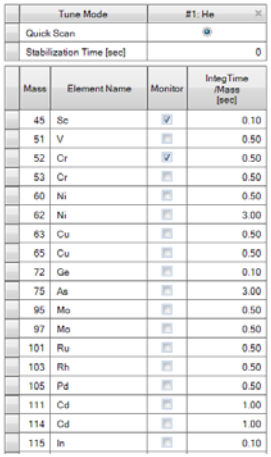


Pre-Configured Methods for Common Applications

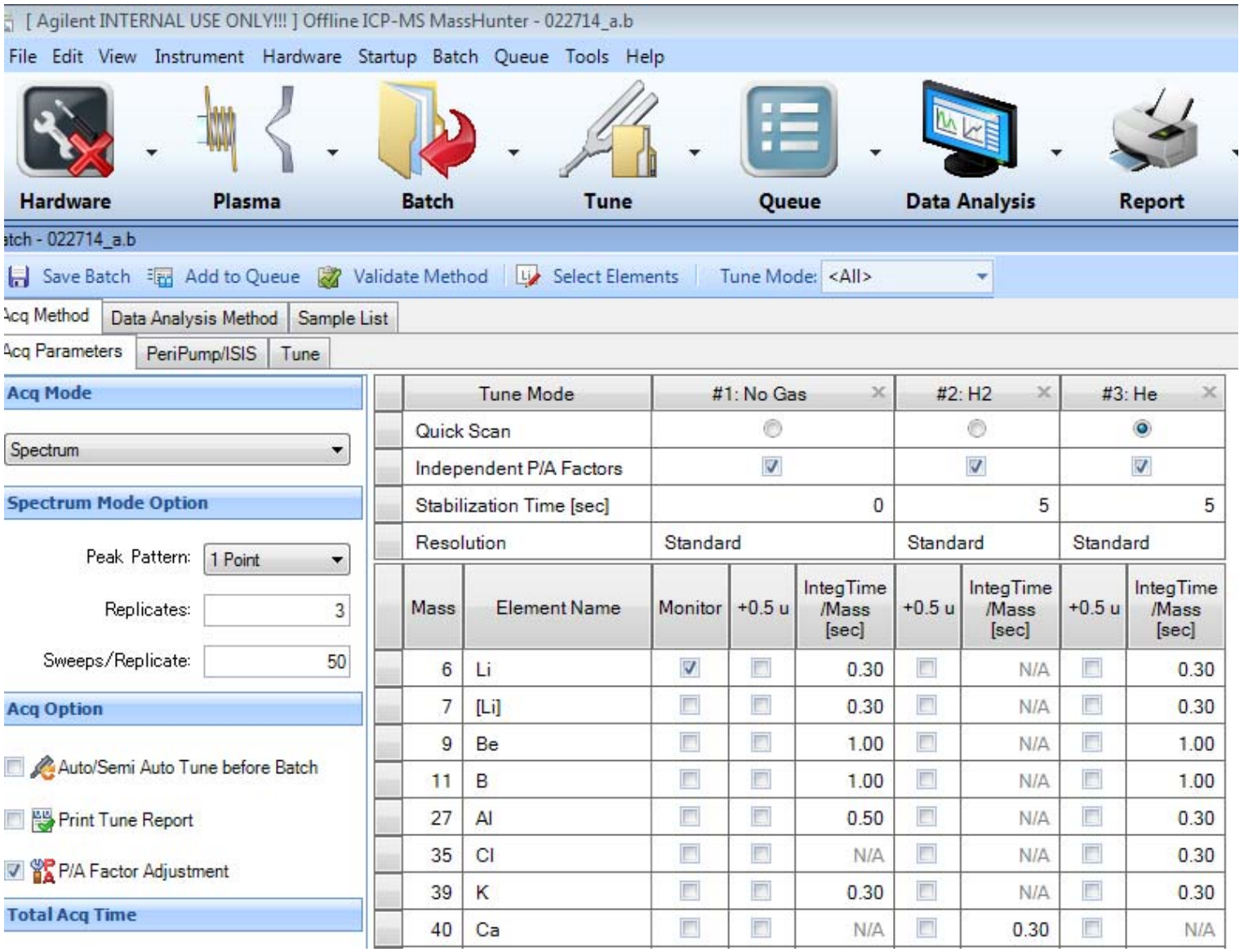




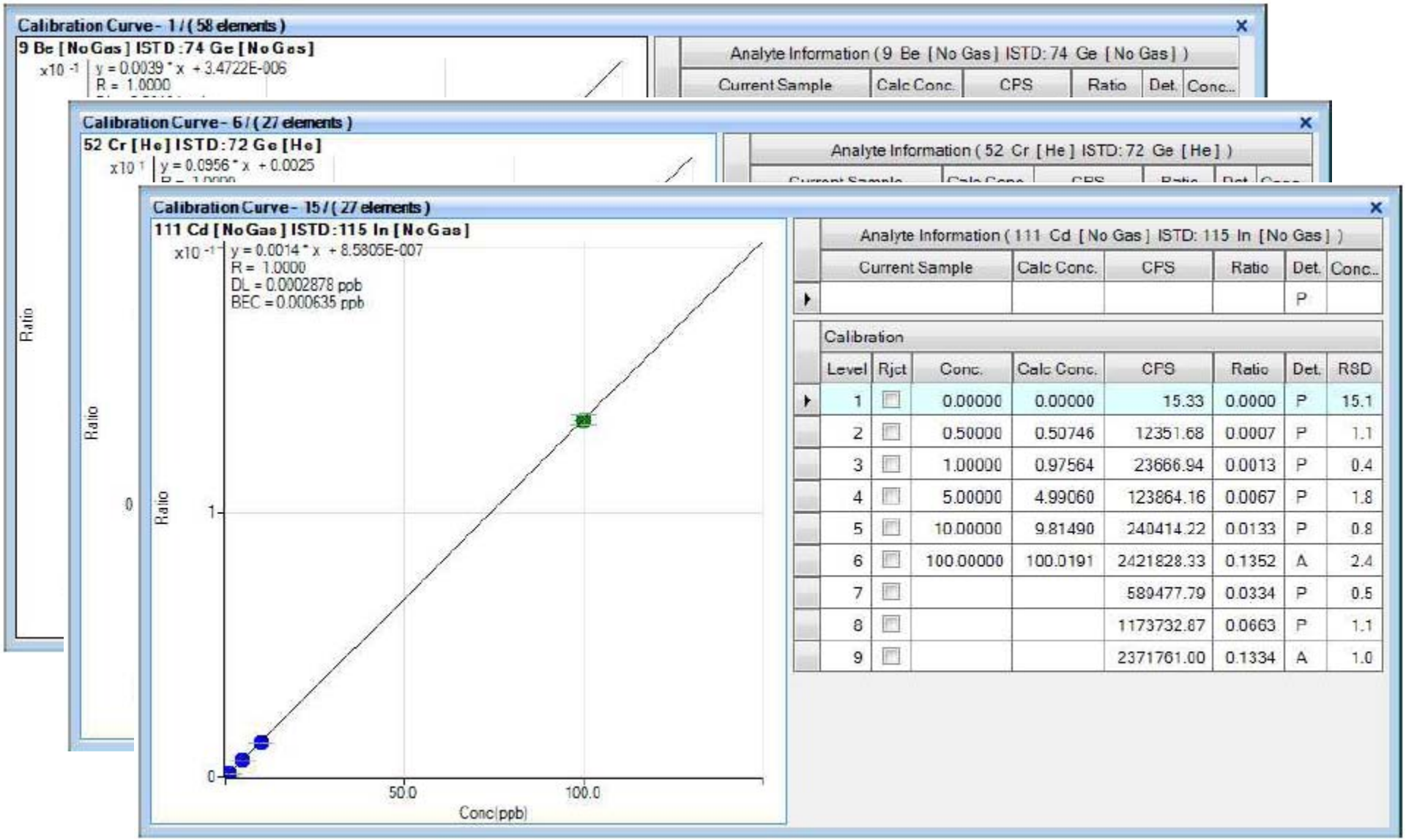




Automated P/A Calibration



Seamless Pulse to Analog Transition

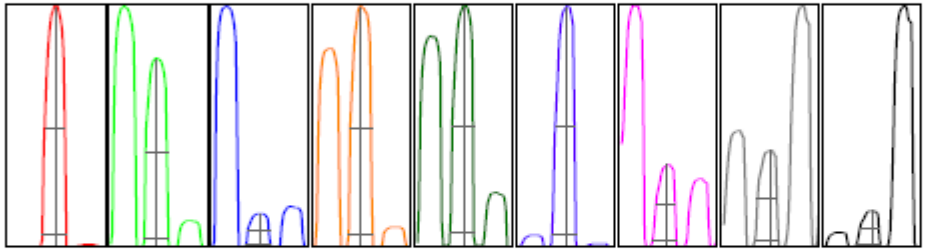


EPA Compliance Software

200.8 Tune Check Sample Report

Batch Folder C:\Agilent\ICPMH\1\DATA\epa tune check.b
Report Comment American West Tune 110923
Instrument Name G3281A JP10420651

[No Gas]



Mass	Peak Height	Axis (Actual)	Axis (Required)	Axis (Flag)	Width-X% (Actual)	Width-X% (Required)	Width-X% (Flag)
9	49907	9.00	8.9 - 9.1		0.742	0.900	
24	14962090	23.95	23.9 - 24.1		0.742	0.900	
25	2081180	25.00	24.9 - 25.1		0.769	0.900	
26	2515438	25.95	25.9 - 26.1		0.740	0.900	
59	502004	59.00	58.9 - 59.1		0.733	0.900	
115	5395881	115.00	114.9 - 115.1		0.734	0.900	
206	409162	205.95	205.9 - 206.1		0.737	0.900	
207	341574	206.95	206.9 - 207.1		0.728	0.900	
208	845273	207.95	207.9 - 208.1		0.730	0.900	

X% = 5 **Integration Time [sec] =** 0.1 **Acquisition Time [sec] =** 235 **Y Axis =** Linear

Individual Sample QC Limits (ie: ICSAB)

22	LLCCV
23	LLICV
24	ICB
25	CCB
26	AllRef
27	SpkDup
28	Dup
29	PDS
30	ICSA
31	ICSB
32	LFM
33	LFMDup
34	LRB
35	LFB
36	QCS1
37	LRS
38	Dil
39	CCV2
40	Blank
41	PB
42	LCS
43	FRB
44	SpikeRef

Analyte

	Tune Mode	Mass	Name	QC Check	Report	Main Criteria1 Lower Exp.	Lower Error Lim.	Upper Exp.	Upper Error Lim.
11	3: He	52	Cr	☑	☑	200	0.8	200	1.2
12	1: No Gas	55	Mn	☑	☑	200	0.8	200	1.2
13	3: He	55	Mn	☑	☑	200	0.8	200	1.2
14	2: H2	56	Fe	☐	☐	250000	0.8	250000	1.2
15	3: He	56	Fe	☐	☐	250000	0.8	250000	1.2
16	1: No Gas	59	Co	☑	☑	200	0.8	200	1.2
17	3: He	59	Co	☑	☑	200	0.8	200	1.2
18	1: No Gas	60	Ni	☑	☑	200	0.8	200	1.2
19	3: He	60	Ni	☑	☑	200	0.8	200	1.2
20	1: No Gas	63	Cu	☑	☑	200	0.8	200	1.2
21	3: He	63	Cu	☑	☑	200	0.8	200	1.2
22	1: No Gas	65	Cu	☑	☑	200	0.8	200	1.2
23	1: No Gas	66	Zn	☑	☑	200	0.8	200	1.2

ISTD (Method Name : IS)

	Tune Mode	Mass	Name	QC Check	Report	Main Criteria1 Lower Exp.	Lower Error Lim.	Upper Exp.	Upper Error Lim.
1	1: No Gas	45	Sc	☑	☑	100	0.6	100	1.25
2	2: H2	45	Sc	☑	☑	100	0.6	100	1.25

Each QC Sample is Customized for its' individual QC requirements

ICSAB, CRDL, ICV, CCV, CCB, etc.

Associated Sample Report may also be Customized to Display all required Information

Unique Performance of the 7800 & 7900

Better matrix tolerance than any other ICP-MS

Higher plasma temperature (lower CeO/Ce ratio) under standard conditions than any other system

- 7800 with HMI: **~2-3% TDS** & 7900 with UHMI: **~25% TDS**

Best performance with Helium cell gas – eliminates need for reaction gases in all common applications

- 7800/7900 ORS^4 – removes polyatomic interferences in complex matrices while maintaining excellent sensitivity.

Wider dynamic range than any other quadrupole ICP-MS

- 7800: 10 orders of Magnitude (~1000s ppm) (3 ms Integration)
- 7900: 11 orders of Magnitude (~Percent) (0.1 ms Integration spNP)

Maximizing Productivity for High Matrix Sample Analysis Using the Agilent 7900 ICP-MS with ISIS 3 Discrete Sampling System

Publication number: 5991-5208EN

Publication date: October 2014

Environmental

Kazuo Yamanaka[1] and Steve Wilbur[2]

[1]Agilent Technologies, Japan
[2]Agilent Technologies, USA

Introduction
Faster analysis, improved ease-of-use, superior matrix tolerance

Combing the unique benefits from each of the following provides much faster analysis, improved ease-of-use and superior matrix tolerance:

- Agilent 7900 ORS^4 He mode collision cell
  - For interference removal
- Ultra High Maxtrix Introduction (UHMI)
  - Aerosol dilution
- ISIS 3
  - Discrete sampling to achieve the best possible productivity

Experimental
Operating conditions

Table 1. Agilent 7900 ICP-MS operating conditions

ICP-MS parameters	No gas mode	He mode
RF power (W)	1600	
Carrier gas flow (L/min)	0.77	
Dilution gas flow (L/min)	0.28	
Lens tune	Autotune	Autotune
Cell gas flow (mL/min)	0.0	4.3
Energy discrimination (V)	5.0	
Number of elements	1 analyte, 1 ISTD	25 analytes, 6 ISTDs
Total acquisition time (3 reps) (seconds)	41	

Experimental
ISIS-DS operation

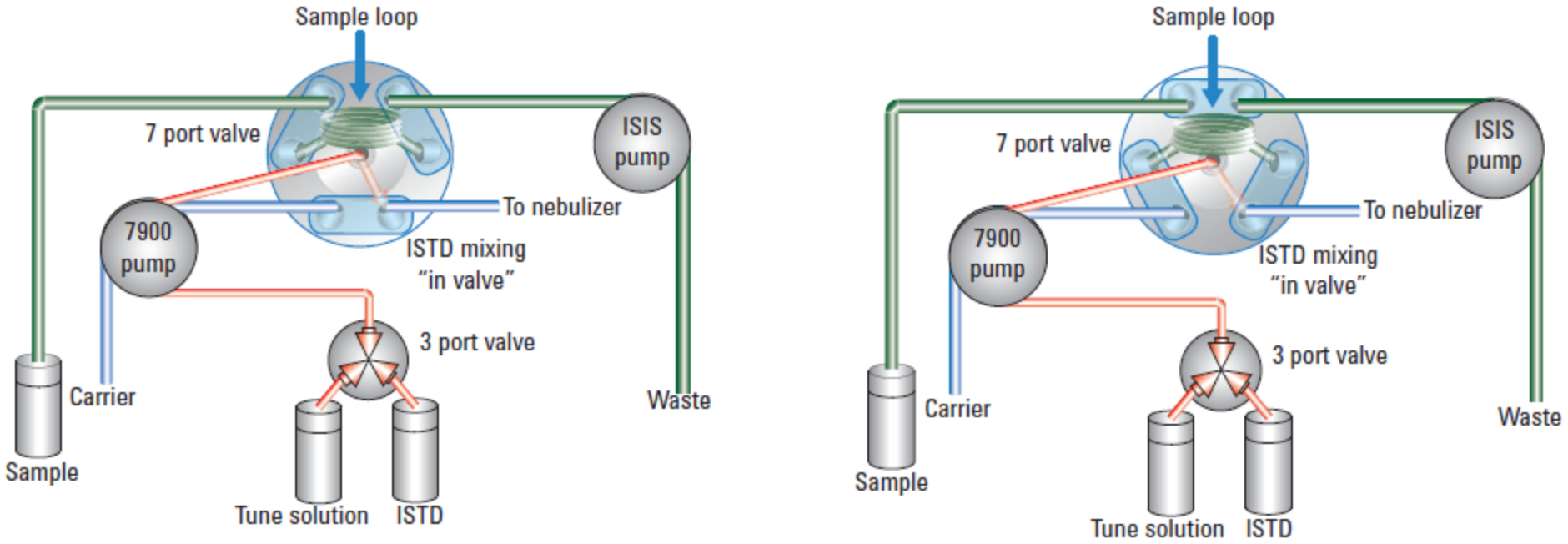


Figure 1. Overview of ISIS-DS operation. Valve in load position (upper left) and inject position (upper right). Actual analyte and internal standard signals during ISIS 3 operation are shown in lower plot with annotation (shown on next page).

Experimental
ISIS-DS operation

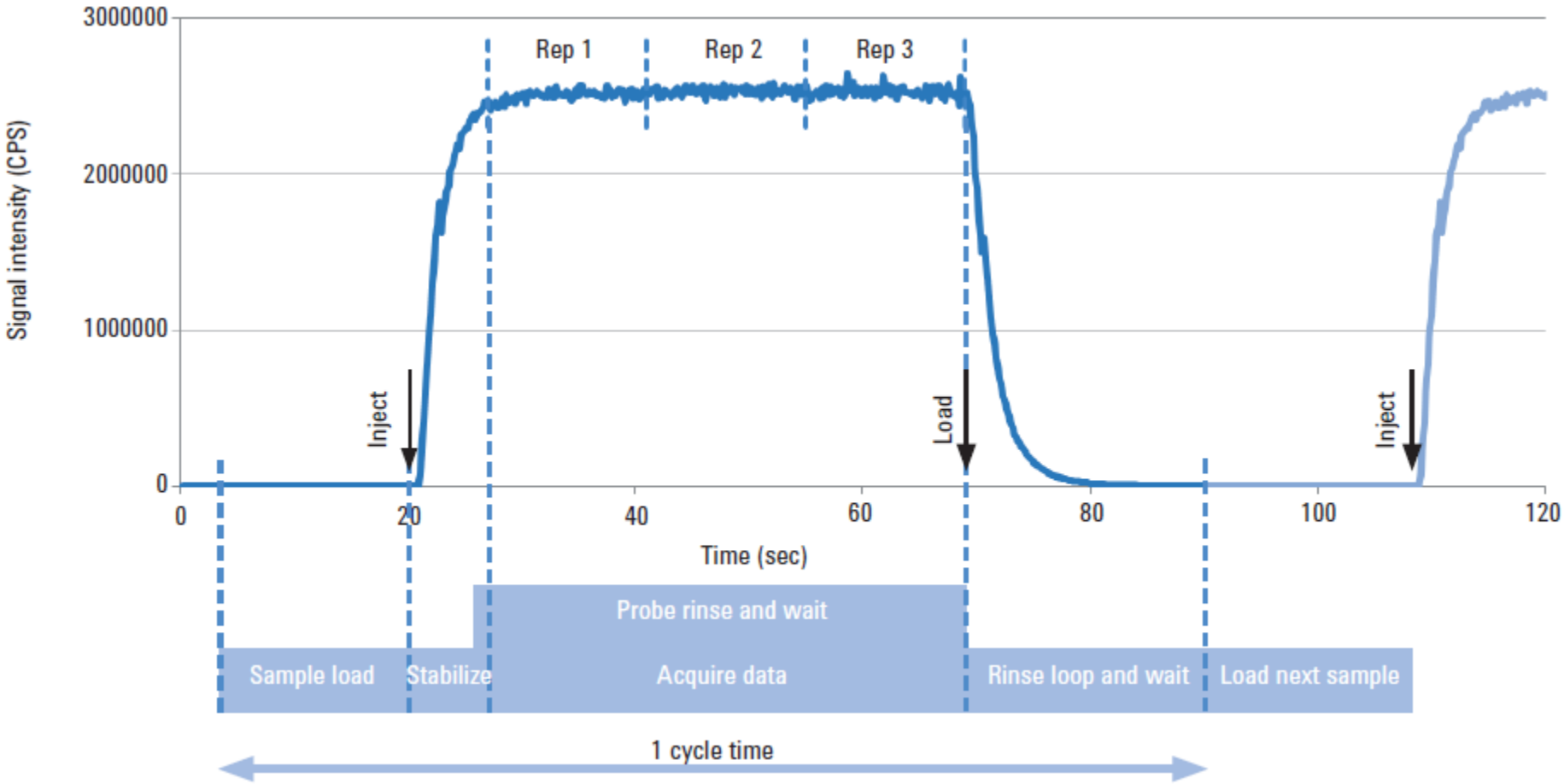


Figure 1 continued. Actual analyte and internal standard signals during ISIS 3 operation are shown with annotation.

Results

This study used He cell mode for most elements and no gas mode for low mass elements.

383 samples were measured in 9 hours and 35 minutes with a run-to-run time of 90 seconds per sample.

Results
Meeting EPA QA/QC requirements

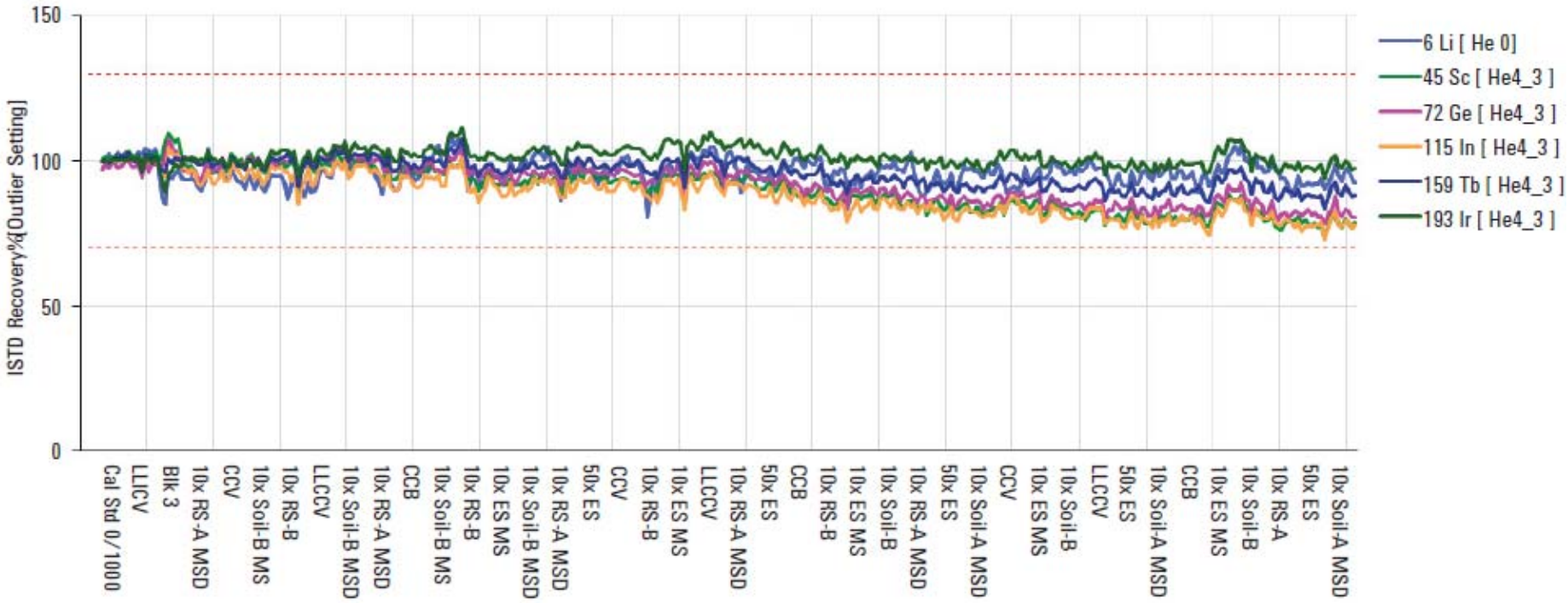


Figure 4. Internal standard recoveries normalized to the calibration blank for all samples. Due to limited space, not all sample names are shown. No internal standard failures occurred.

Results and Discussion

Recovery of certified reference values

Table 4. Mean measured values, recoveries, percent Relative Standard Deviations (%RSDs) for all certified elements in the six CRMs analyzed. Blank cells indicate no certified value.
* Concentration of 1/10 diluted solution. ND: less than detection limit

Isotope/ Element	NIST 1640a (n=7)			River Sediment A (1/10, n=14)*			River Sediment B (1/10, n=14)*		
	Mean conc. (ppb)	%RSD	Mean recovery (%)	Mean conc. (ppb)	%RSD	Mean recovery (%)	Mean conc. (ppb)	%RSD	Mean recovery (%)
9 Be	2.80	2.4	93	0.026	9.8		ND		
23 Na	3,112	2.0	107	5,326	1.8	107	5,610	1.6	112
24 Mg	1,062	1.7	101	7,375	1.8	105	13,130	1.3	109
27 Al	57.8	4.4	109	25,180	1.3	101	61,460	0.6	102
39 K	561	5.4	97	14,730	2.4	98	20,220	2.3	101
44 Ca	5,373	3.2	97	29,050	1.8	97	28,960	1.6	97
51 V	15.4	1.5	103	25.5	1.1	102	104	0.7	104
52 Cr	40.5	1.6	101	30,950	1.1	103	1,592	0.9	106
55 Mn	40.0	1.7	100	786	2.2	98	587	1.1	98
56 Fe	44.2	5.7	121	122,000	0.9	102	41,610	0.6	104
59 Co	20.7	2.1	103	12.1	1.5	121	16.3	1.3	109
60 Ni	25.7	1.8	103	52.2	1.4	104	52.8	1.4	106
63 Cu	87.9	2.0	103	101.9	1.7	102	104	1.4	104
66 Zn	56.1	1.3	102	1,454	0.9	97	493	0.8	99
75 As	7.99	1.4	100	60.2	1.3	100	20.4	1.4	102
78 Se	18.5	5.1	93	2.28	5.7	114	0.95	10.5	95
95 Mo	45.8	2.0	101	0.074	7.0		0.17	4.8	
107 Ag	8.25	2.8	103	ND			0.17	2.3	
111 Cd	3.97	1.8	100	10.2	1.1	102	3.18	2.4	106
121 Sb	5.07	1.8	100	50.7	0.7	102	4.22	0.9	106
137 Ba	147	1.1	98	49.3	0.7	99	392	0.5	98
201 Hg	ND			ND			0.023	10.9	
205 Tl	1.58	3.0	98	0.99	1.4	99	1.15	1.6	115
208 Pb	12.5	4.4	104	742	1.8	106	212	1.7	106
232 Th	0.002	40		2.04	2.6	102	9.93	2.3	99
238 U	25.2	3.8	100	1.02	3.1	102	3.02	2.9	101

Conclusions

Long sequences of high Total Dissolved Solids (TDS) samples can be analyzed with high accuracy, precision, and long term stability using the Agilent 7900 ICP-MS.

This was shown by combining the benefits of He mode for removing polyatomic interferences with the UHMI for highly robust plasma conditions, and the ISIS 3 discrete sampling system.

Questions?

Craig Jones

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