

**ThermoFisher**  
S C I E N T I F I C

*The world leader in serving science*

**Which Instrument is Best  
for My Application?**

# An Overview of Elemental Analysis Tools

Liquid Phase



FAAS



GFAAS

Single Element

Multi-Element



ICP

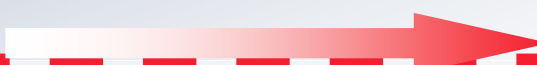


ICP-QMS



ICP-SFMS

Bulk Concentration



Ultra-trace Concentration

Solid Phase



Spark-OES



X-Ray



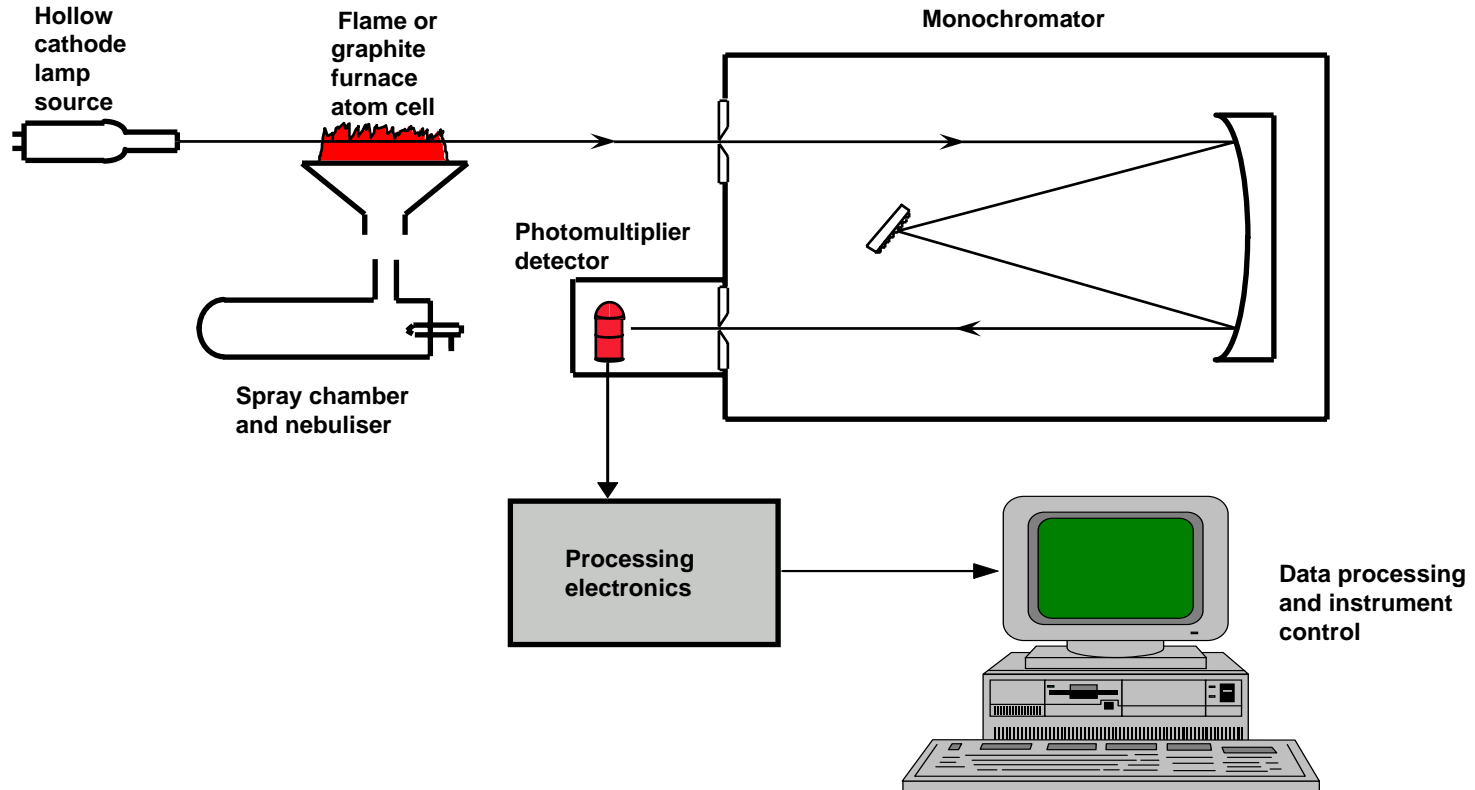
GD-MS

# Liquid Phase Trace Element Analysis Tools...

Technique	Invented	Characteristics	Thermo Scientific
AAS	~1955	Single element analysis, ppb-ppm levels with flame or furnace, good for As, Se and Hg with vapour generation	M & S Series
ICP-OES	~1972	Fast multi-element analysis, ppb-% levels	iCAP 6000 Series
ICP-QMS	~1981	Fast multi-element analysis, ppt-ppm levels	XSERIES 2
ICP-HRMS	~1987	Fast multi-element analysis, ppq-ppm levels, without interference	ELEMENT 2

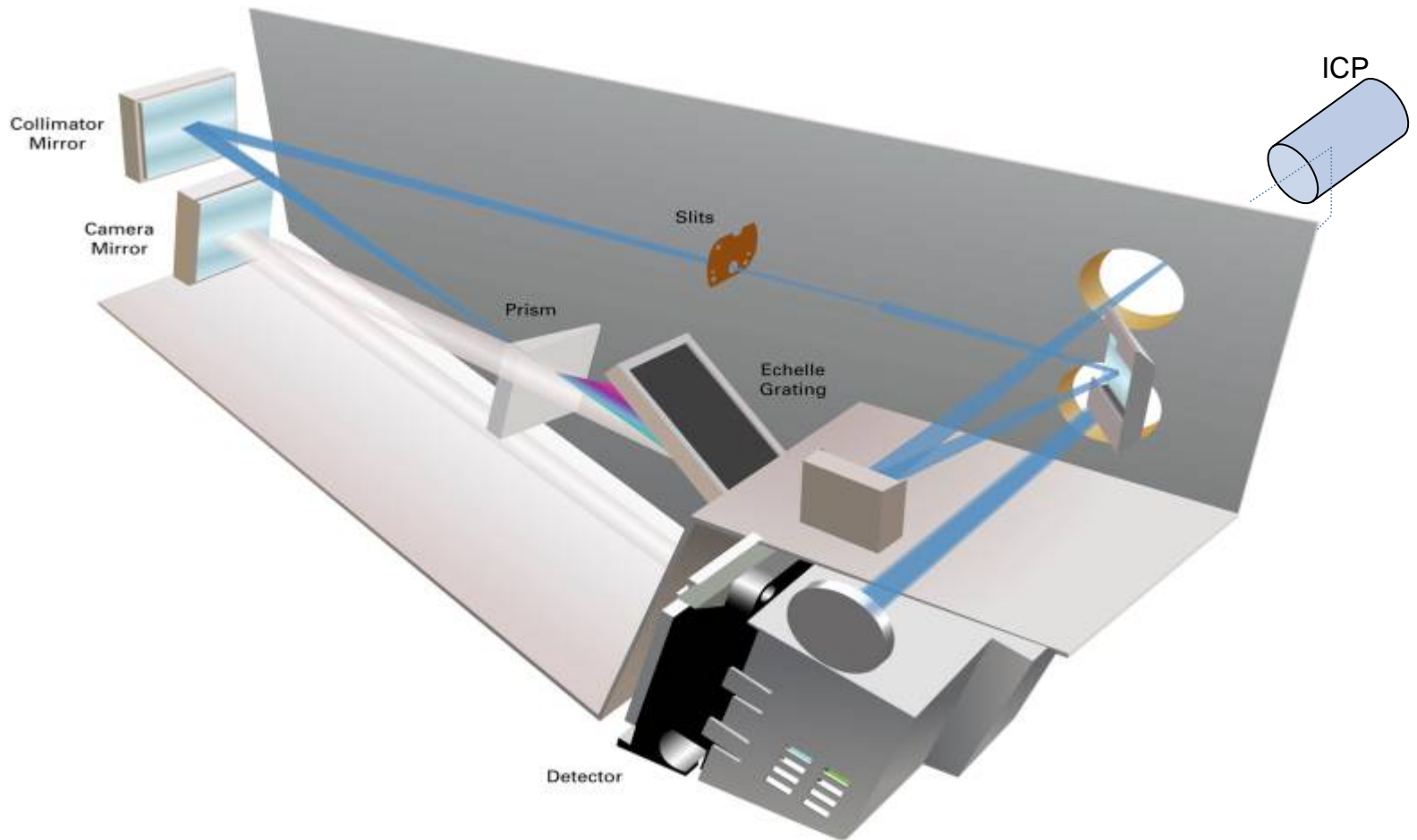
# How do they Work?

## - 1) AAS



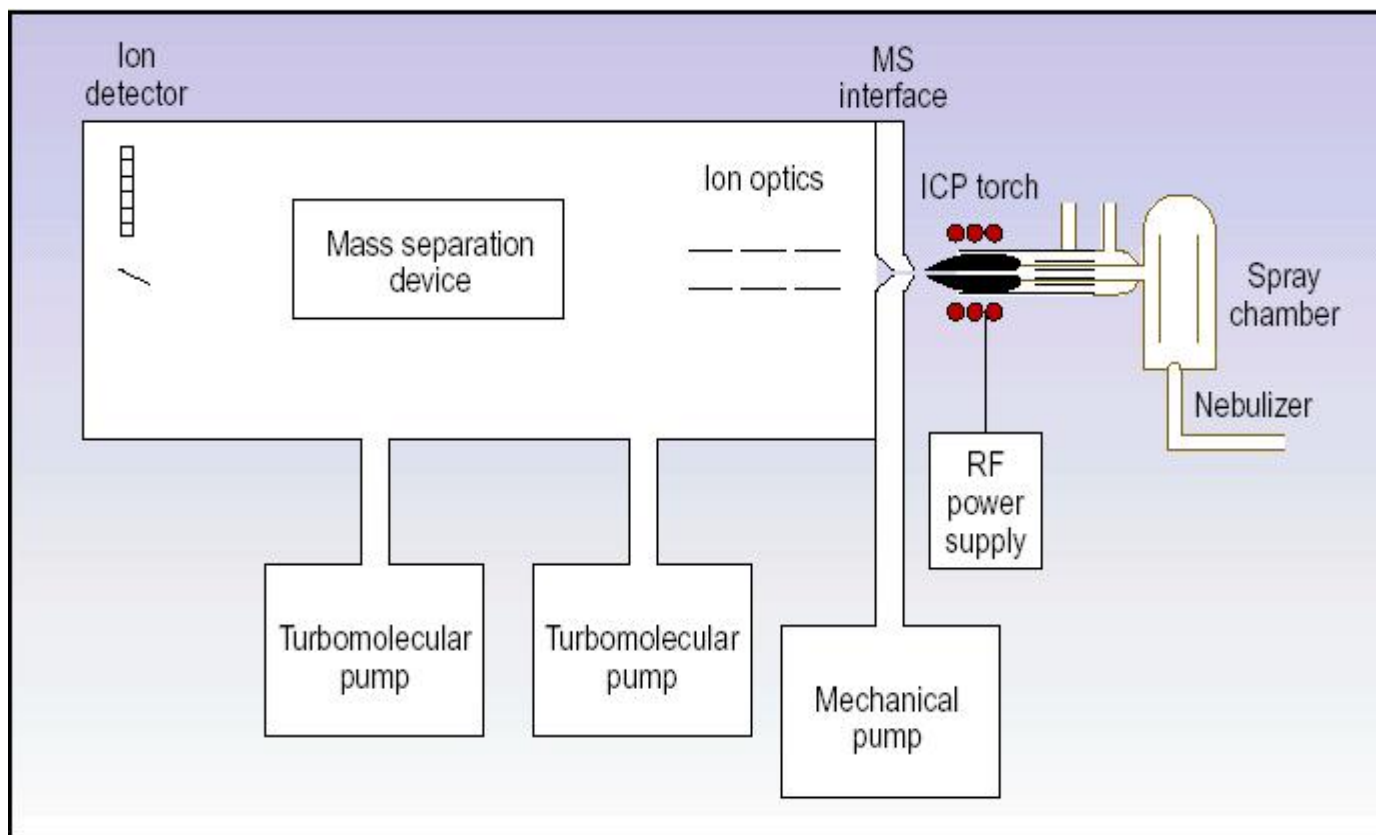
# How do they Work?

## - 2) ICP-OES

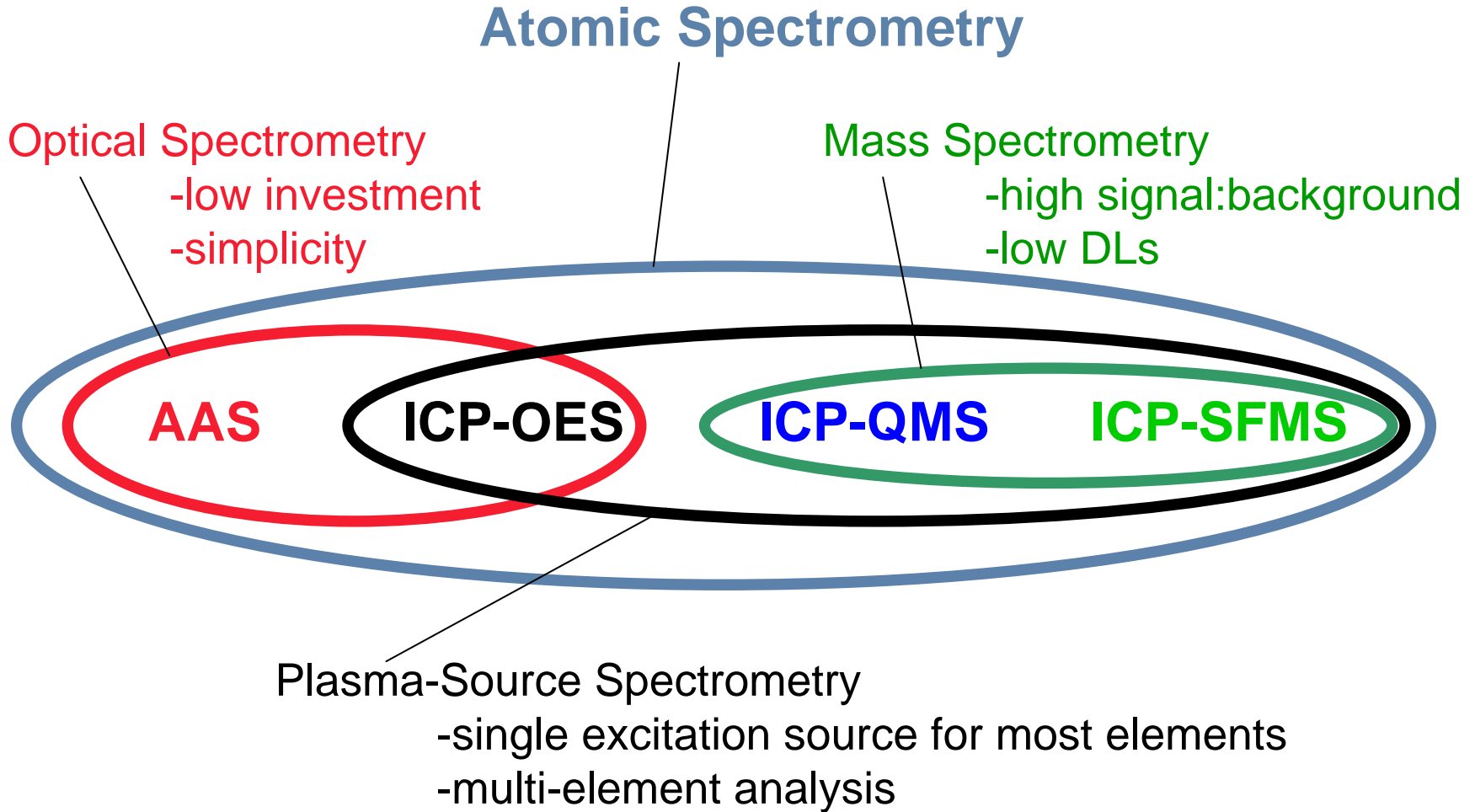


# How do they Work?

## - 3) ICP-MS



# Relationships Between the Technologies



# Flame AA Coverage (Typical)

Air-Acetylene Flame

Nitrous Oxide-Acetylene Flame

H																	He
Li	Be											B	C	N	O	F	Ne
Na	Mg											Al	Si	P	S	Cl	Ar
K	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br	Kr
Rb	Sr	Y	Zr	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Te	I	Xe
Cs	Ba	La	Hf	Ta	W	Re	Os	Ir	Pt	Au	Hg	Tl	Pb	Bi	Po	At	Rn
Fr	Ra	Ac	Rf	Db	Sg	Bh	Hs	Mt	Ds	Rg	Uub	Uut	Uuq	Uup	Uuh		

Ce	Pr	Nd	Pm	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu
Th	Pa	U	Np	Pu	Am	Cm	Bk	Cf	Es	Fm	Md	No	Lr



# Furnace AA Coverage (Typical)

H																	He																												
Li	Be											B	C	N	O	F	Ne																												
Na	Mg											Al	Si	P	S	Cl	Ar																												
K	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br	Kr																												
Rb	Sr	Y	Zr	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Te	I	Xe																												
Cs	Ba	La	Hf	Ta	W	Re	Os	Ir	Pt	Au	Hg	Tl	Pb	Bi	Po	At	Rn																												
Fr	Ra	Ac	Rf	Db	Sg	Bh	Hs	Mt	Ds	Rg	Uub	Uut	Uuq	Uup	Uuh																														
<table border="1"> <tbody> <tr> <td>Ce</td> <td>Pr</td> <td>Nd</td> <td>Pm</td> <td>Sm</td> <td>Eu</td> <td>Gd</td> <td>Tb</td> <td>Dy</td> <td>Ho</td> <td>Er</td> <td>Tm</td> <td>Yb</td> <td>Lu</td> </tr> <tr> <td>Th</td> <td>Pa</td> <td>U</td> <td>Np</td> <td>Pu</td> <td>Am</td> <td>Cm</td> <td>Bk</td> <td>Cf</td> <td>Es</td> <td>Fm</td> <td>Md</td> <td>No</td> <td>Lr</td> </tr> </tbody> </table>																		Ce	Pr	Nd	Pm	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu	Th	Pa	U	Np	Pu	Am	Cm	Bk	Cf	Es	Fm	Md	No	Lr
Ce	Pr	Nd	Pm	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu																																
Th	Pa	U	Np	Pu	Am	Cm	Bk	Cf	Es	Fm	Md	No	Lr																																

# ICP-OES Coverage

H																	He
Li	Be											B	C	N	O	F	Ne
Na	Mg											Al	Si	P	S	Cl	Ar
K	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br	Kr
Rb	Sr	Y	Zr	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Te	I	Xe
Cs	Ba	La	Hf	Ta	W	Re	Os	Ir	Pt	Au	Hg	Tl	Pb	Bi	Po	At	Rn
Fr	Ra	Ac	Rf	Db	Sg	Bh	Hs	Mt	Ds	Rg	Uub	Uut	Uuq	Uup	Uuh		

Ce	Pr	Nd	Pm	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu
Th	Pa	U	Np	Pu	Am	Cm	Bk	Cf	Es	Fm	Md	No	Lr

# ICP-MS Coverage

H																	He
Li	Be											B	C	N	O	F	Ne
Na	Mg											Al	Si	P	S	Cl	Ar
K	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br	Kr
Rb	Sr	Y	Zr	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Te	I	Xe
Cs	Ba	La	Hf	Ta	W	Re	Os	Ir	Pt	Au	Hg	Tl	Pb	Bi	Po	At	Rn
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Ce	Pr	Nd	Pm	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu
Th	Pa	U	Np	Pu	Am	Cm	Bk	Cf	Es	Fm	Md	No	Lr

# Advantages/Disadvantages

## - 1) Elements and Levels

### FAAS

- Good for Gp I&II elements, 1<sup>st</sup> row transition elements
- Not so good for refractory elements
- Very fast for single element
- \$

### GF-AAS, VG-AAS

- **GF** good for heavy metals
- **VG** good for Hg, As, Se, Te
- Limited elemental applicability
- Low ppb DLs
- \$\$

### ICP-OES

- Good for most elements
- DLs for Pb, As, Se, Hg problematic
- Fast for multi-element work
- \$\$\$

### ICP-MS

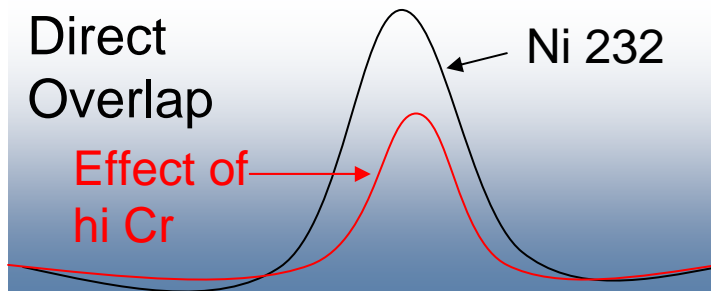
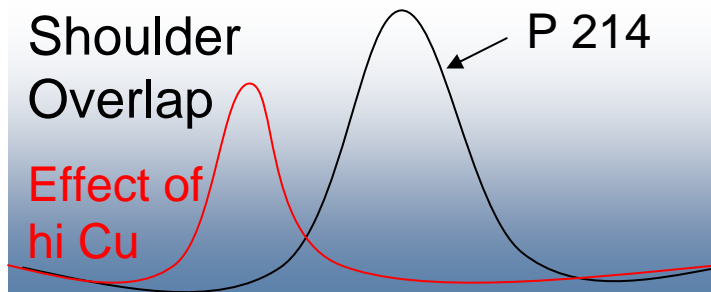
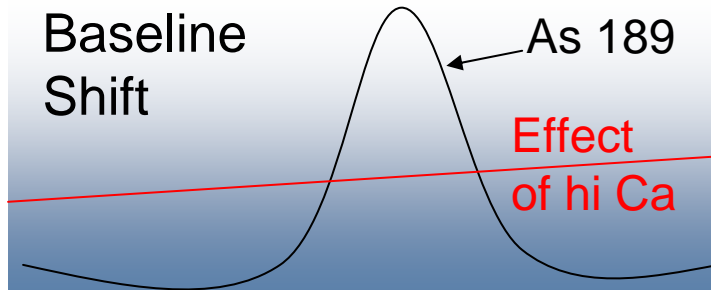
- Great for low-level heavy metals
- Range problematic for Gp I&II
- Some interferences on 1<sup>st</sup> row transitions
- \$\$\$\$

# Comparison of Working Analytical Range

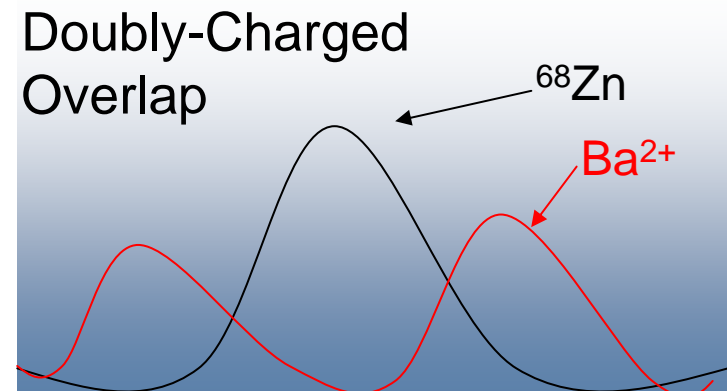
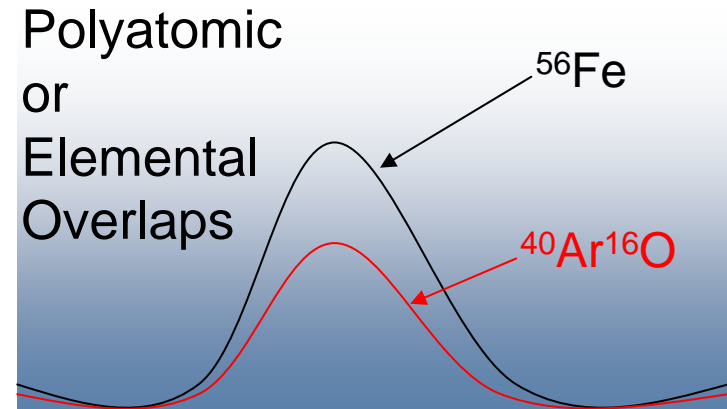


# Comparing ICP and ICP-MS: Spectral Interferences

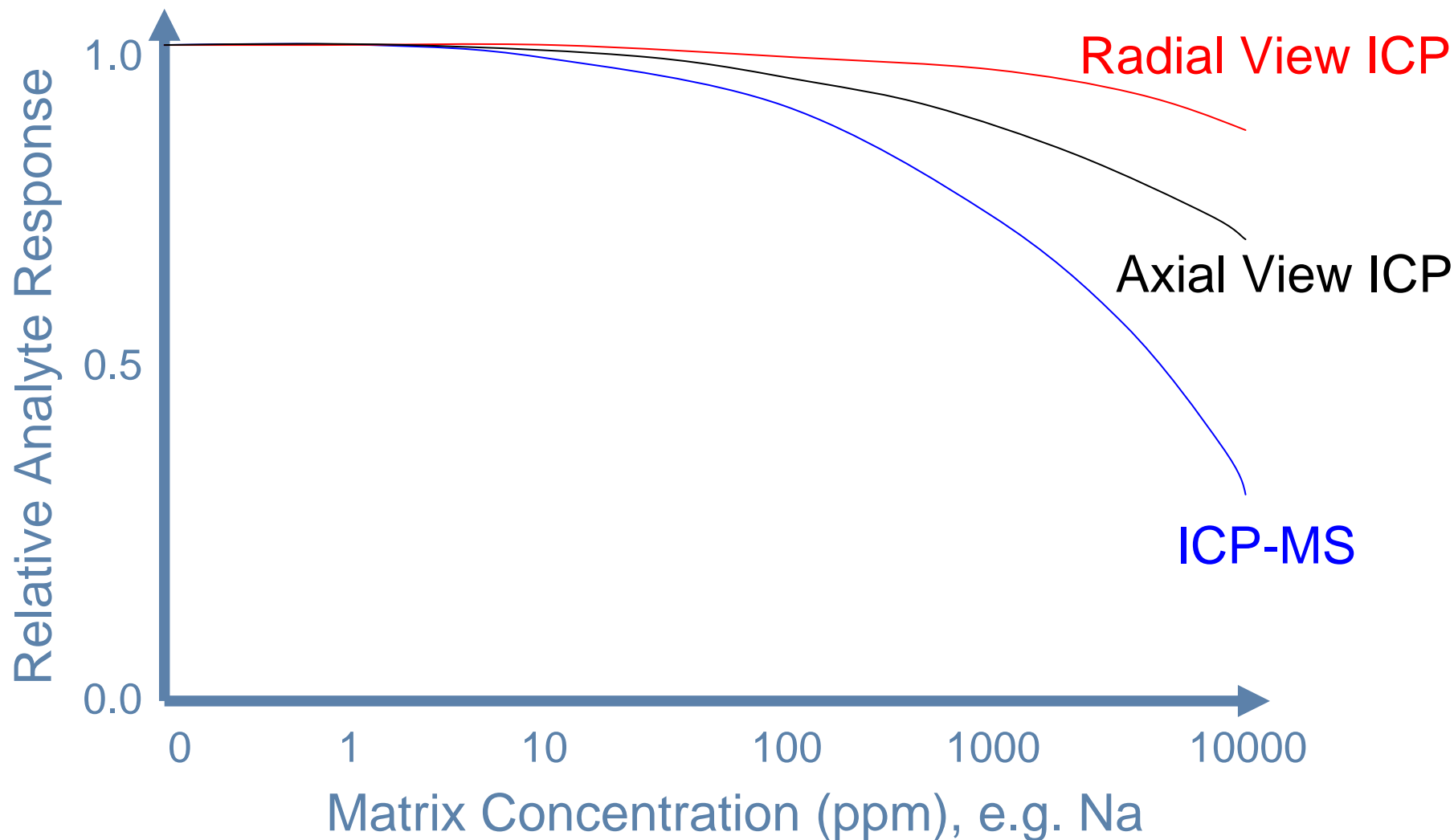
## ICP-OES



## ICP-MS

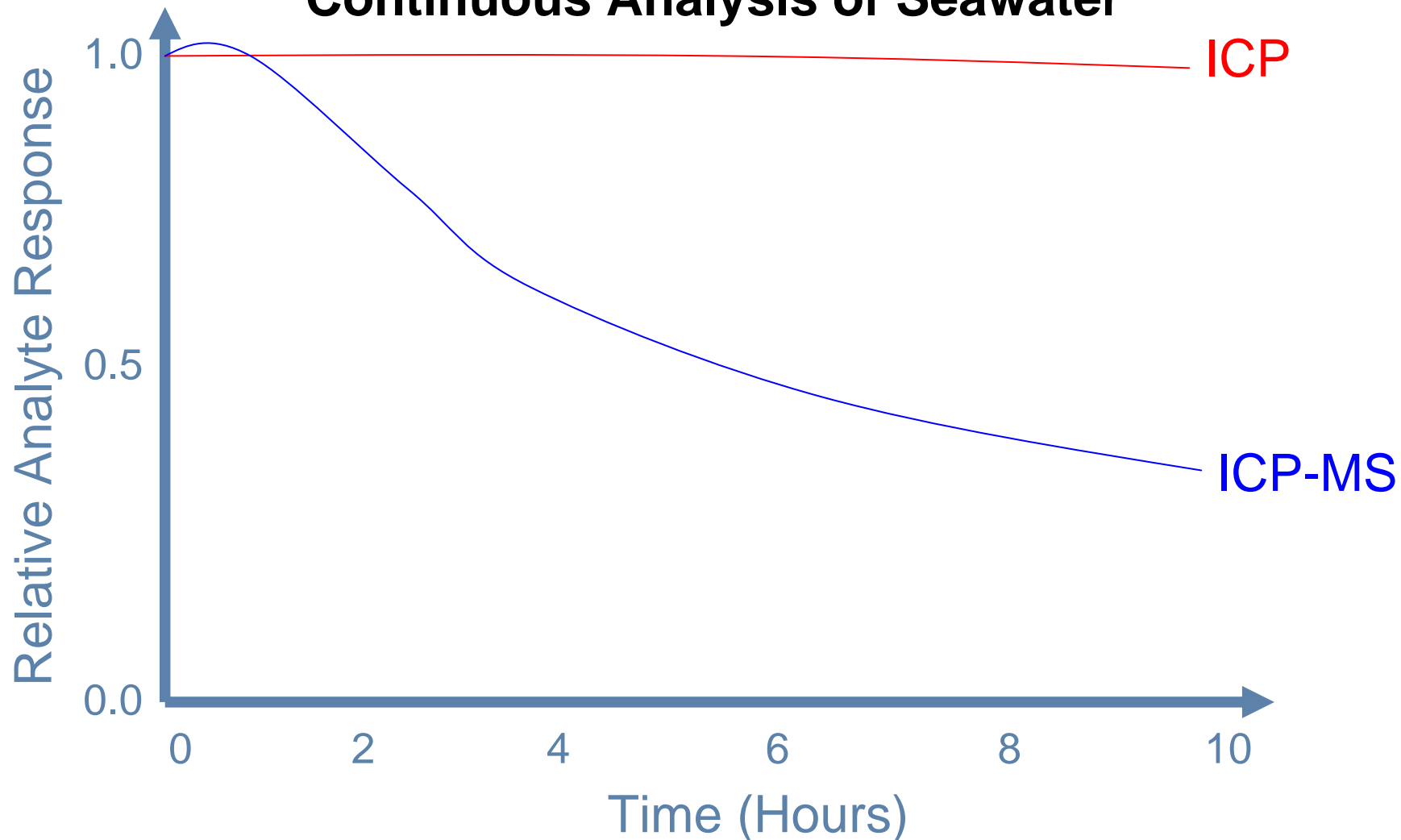


# Comparing ICP and ICP-MS: Physical Interferences



# Comparing ICP and ICP-MS: Physical Interferences

## Continuous Analysis of Seawater



# Why ICP-MS drifts when running high matrix samples

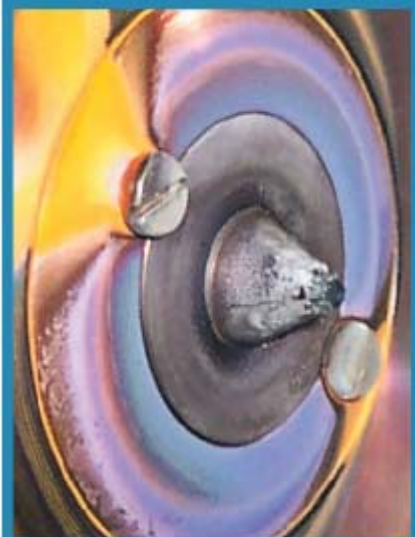
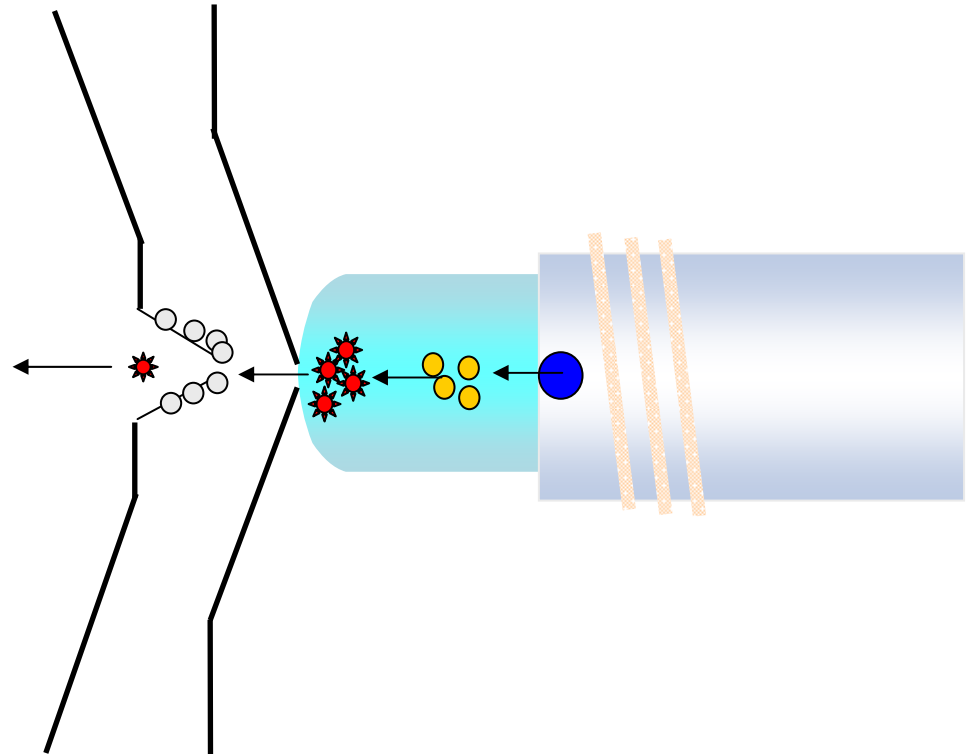


FIGURE 1. Xi skimmer after matrix run:  
Cu mount and adaptor



# The XSeries<sup>II</sup> Solution – Xt and Xs Interface

- User selectable Xt or Xs interface options

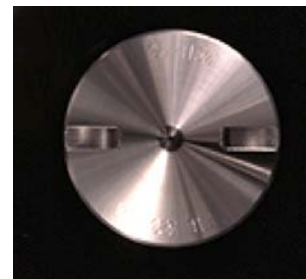
*tailored mass response for differing applications*

- *Xt interface standard*
- *Xs interface option*

- Patented sample & skimmer cone design

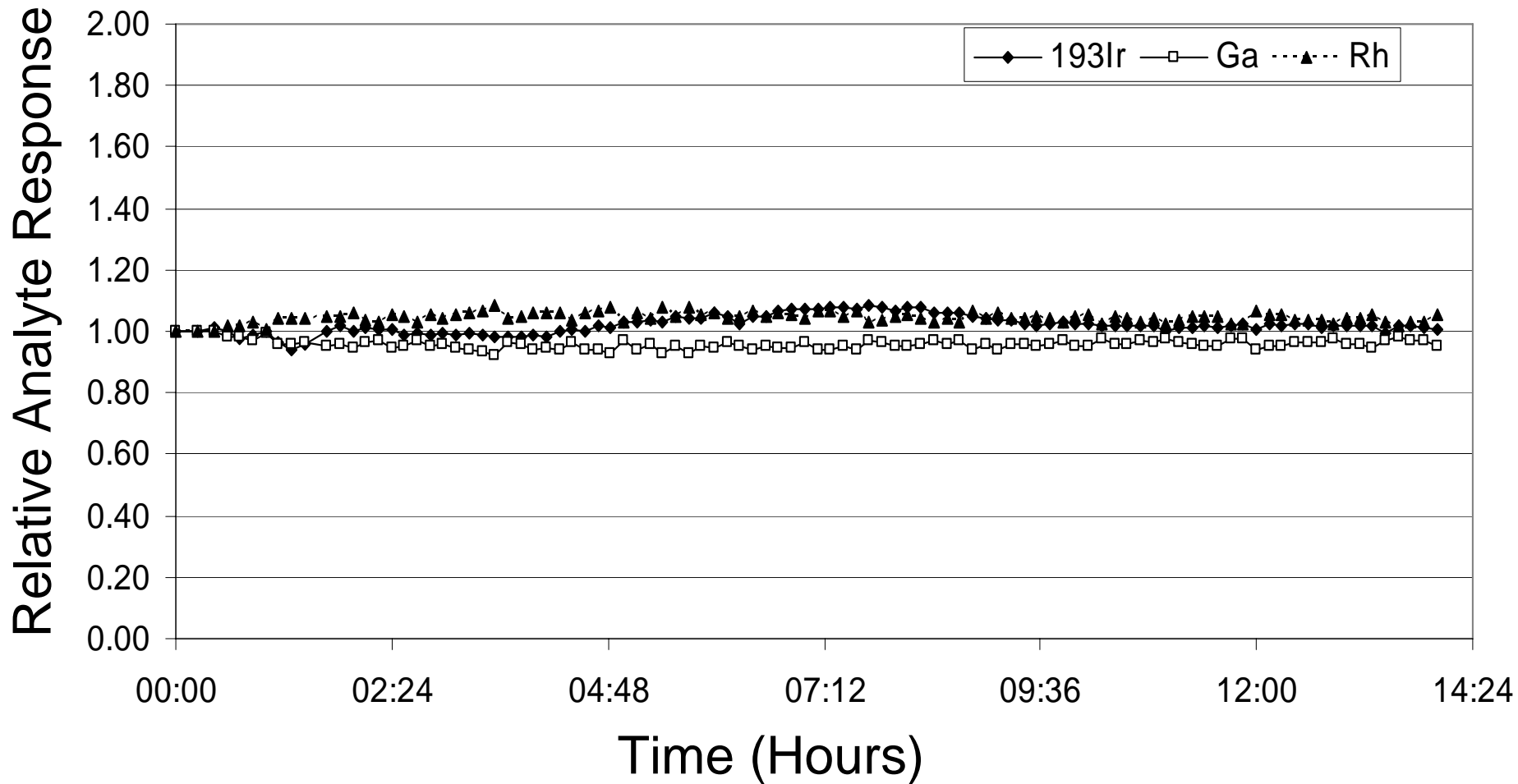
- *Large orifices 1.1mm & 0.75mm*
- *Highly resistant to matrix deposition*
- *Low frequency of cleaning*

- Quick release cone configuration
  - *Requires skimmer change only*



# The XSERIES 2 Solution – Xt and Xs Interfaces

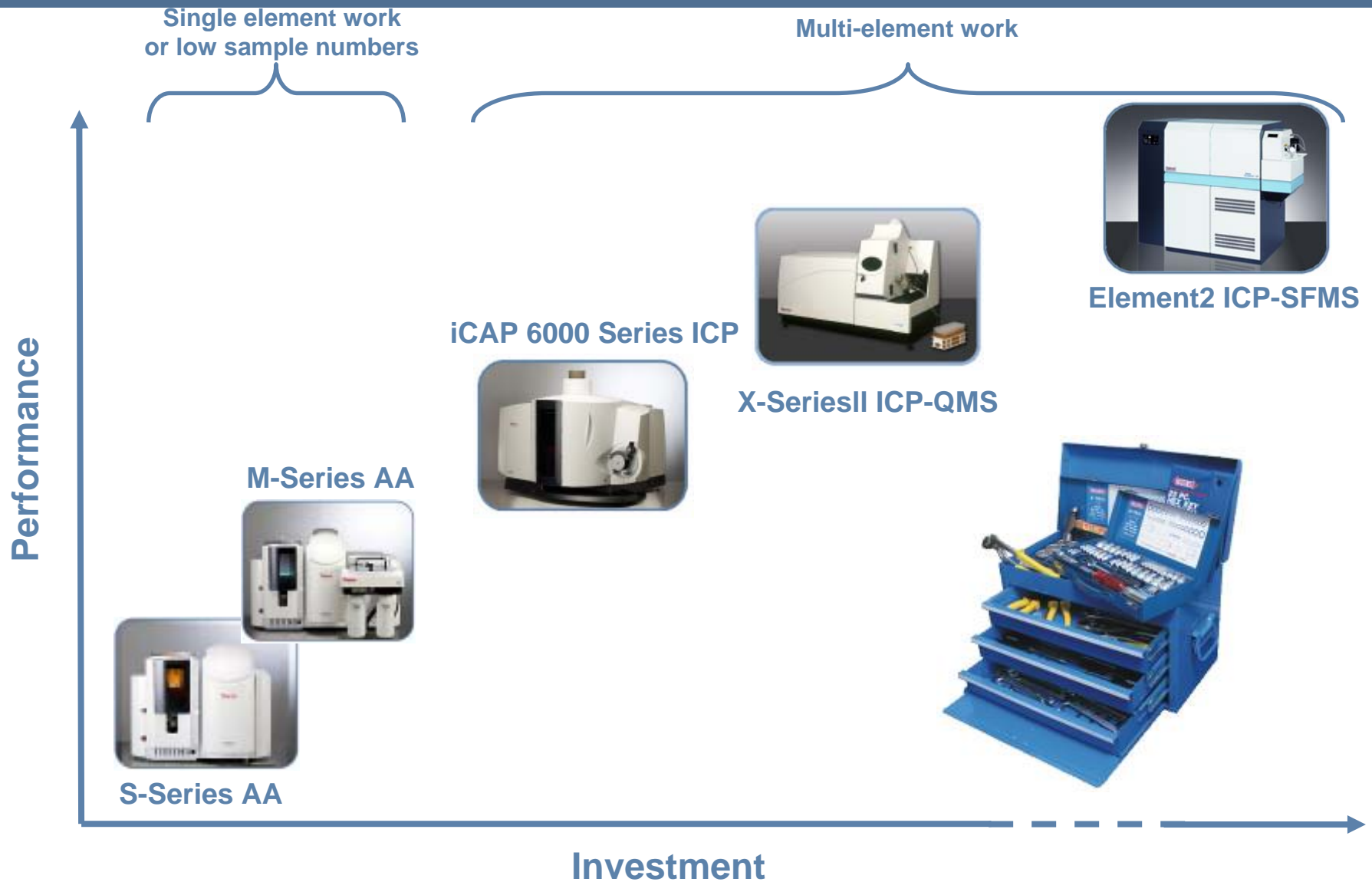
## Continuous Analysis of 1:10 Seawater (Xs+ Mode)



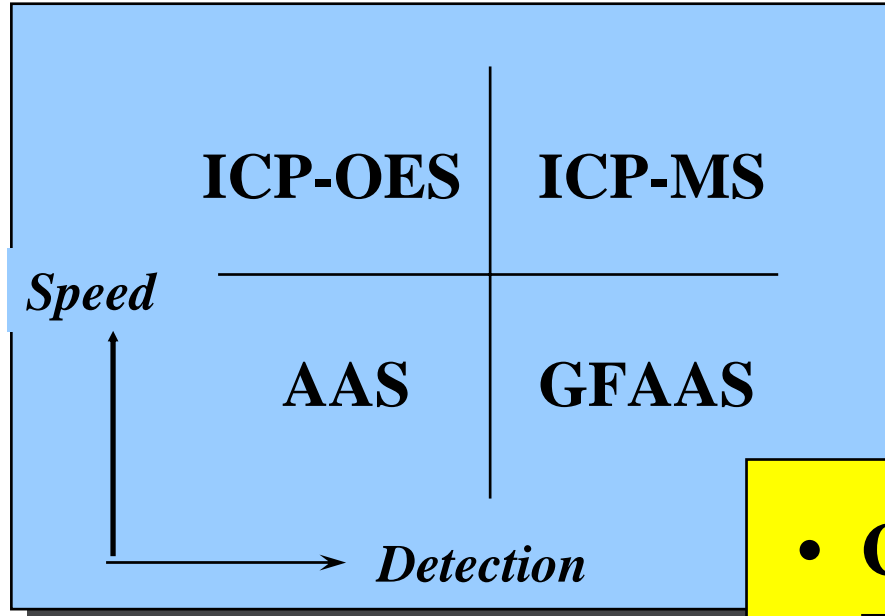
# The right tool for the job?



# Only Thermo Fisher Scientific offer a full toolkit!



# Which technique to choose?



- **Cost**
- **Detection**
- **Analysis Type**
- **Methodology**

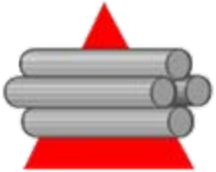
# What are the questions to answer??



- What is the application?
- What are the elements to be measured?
- What is the anticipated detection levels?



- What is the matrix-aqueous, organic, acids?
- Is the sample clean or dirty?
- How many samples per day/per week?



- What are your future requirements?
- What is your funding?

# Which technique?

## Sample throughput (Productivity)

Overall sample speeds are typically as follows:

- ICP-MS: All elements in 2-5 minutes
- ICP-OES: All elements in 2-5 minutes (Simultaneous)
- ICP-OES: 5-6 elements per minute (Sequential)
- FAAS: 4 seconds per element
- GFAAS: 2-3 minutes per element

# Which technique and why

	Primary	Secondary	Reason 1	Reason 2
Enviro water	ICP-MS	ICP/AA Fur	Detection	Speed
Pharmaceutical	AA FI/furn	ICP-MS	Cost	Samples no. small
Wear oils	ICP-OES	AA Flame	Elements range	Rugged
Metallurgy	ICP-OES	AA FI/furnace	Elements	Cost
Clinical	AA FI/Furn	ICP-MS	Samples	Cost
Geology	ICP-MS	AA Flame	Samples	Detection
Food analysis	AA FI/furn	ICP-OES	Cost	No of samples
Forensic	LA-ICP-MS	AA Furnace	Sample size	Detection

# Case Study 1 - Scenario

- Clinical chemistry lab analyzing 50 blood lead samples per day
- Level of interest 1-100 ug Pb per L of blood
- Single element analysis: **AA**
- Low concentration levels: **Furnace atomization**



# Case Study 1 - Analysis

- Matrix modifier: 0.1% v/v nitric acid, 0.2% m/v ammonium dihydrogen phosphate and 0.5% m/v Triton X-100
- 100 $\mu$ L of blood mixed with 750 $\mu$ L of matrix modifier
- Working standards prepared in low lead blood samples
- QuadLine background correction

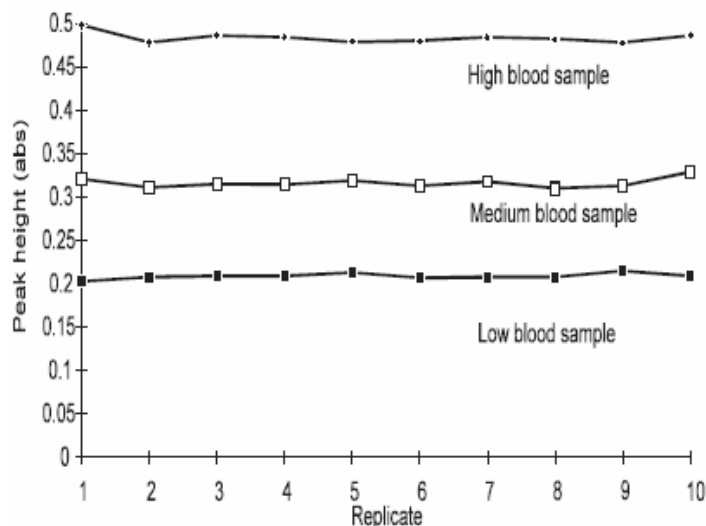
## M Series AA

(M5 & MQZ)

3-s DL =  
0.8  $\mu$ g/L



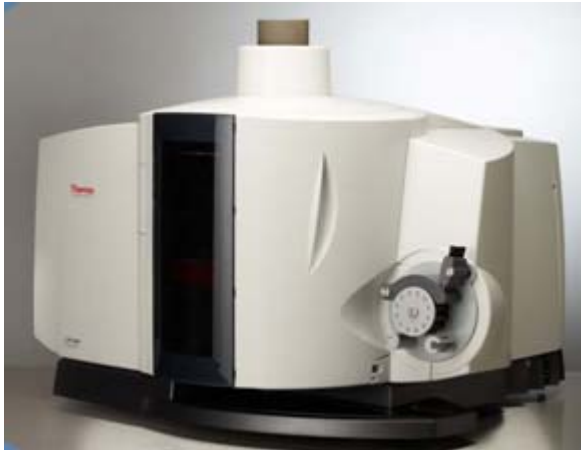
Sample	Blood 1	Blood 2	Blood 3
Pb level ( $\mu$ g/L)	5.01	13.53	30.63
Pb added ( $\mu$ g/L)	12.00	12.00	12.00
Pb expected ( $\mu$ g/L)	17.01	25.53	42.63
Pb found ( $\mu$ g/L)	17.49	26.06	43.35
Recovery	102%	102%	101%



Reference material	Certified value ( $\mu$ g/L)	Method value ( $\mu$ g/L)
BioRad 63801	5.2 $\pm$ 1.1	5.37 $\pm$ 0.27
BioRad 63802	24.5 $\pm$ 3.7	23.46 $\pm$ 0.48

# Case Study 2 - Scenario

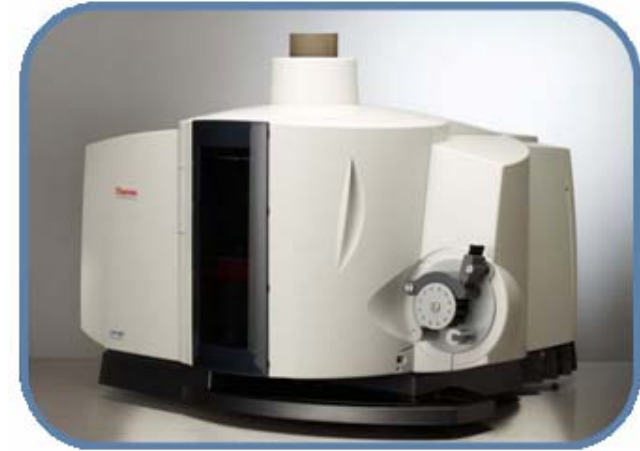
- Metallurgy lab analyzing 30 steel digest samples per day for 7 elements
- Level of interest 0.01 – 2 % levels in the solid
- Multi-element element analysis: **ICP or ICP-MS**
- Moderate to high concentration levels and high matrix concentration: **ICP**



# Case Study 2 - Analysis

- 0.5 g sample digested with 10 mL HNO<sub>3</sub> + 2 mL HCl in a high pressure microwave digestion system
- Solutions made to 100 mL
- Concentric nebulizer and cyclonic spray chamber
- The radial plasma instrument was used to reduce matrix interferences
- Simultaneous background correction

**iCAP  
6500  
Radial**



ELEMENT	3 $\sigma$ METHOD DETECTION LIMITS (ppb)				
		ZRM 476-3 MEASURED %	ZRM 476-3 EXPECTED %	GBW 01323 MEASURED %	GBW 01323 EXPECTED %
Cr 205.552 nm	1.3	0.0649	0.0648 $\pm$ 0.0012	0.368	0.389 $\pm$ 0.006
Cu 327.396 nm	4.3	0.2349	0.2445 $\pm$ 0.0025	0.276	0.277 $\pm$ 0.009
Mn 279.482 nm	11.1	1.009	0.987 $\pm$ 0.008	1.46	1.44 $\pm$ 0.02
Ni 231.604 nm	1.7	0.0576	0.0549 $\pm$ 0.0014	0.161	0.166 $\pm$ 0.004
P 178.284 nm	6	0.0901	0.0908 $\pm$ 0.0023	0.011	0.013 $\pm$ 0.001
Ti 334.941 nm	0.5	0.0202	0.0222 $\pm$ 0.0005	0.268	0.285 $\pm$ 0.006
V 268.796 nm	4	0.0101	0.0115 $\pm$ 0.0002	0.148	0.158 $\pm$ 0.005

# Case Study 3 - Scenario

- Environmental lab analyzing 100 clean water samples per day for ~20 elements
- Level of interest 0.0005 – 300 mg/L levels
- Multi-element element analysis: **ICP or ICP-MS**
- Moderate to low concentration levels and low to moderate matrix concentration: **ICP-QMS**



# Case Study 3 - Analysis

- Samples acidified to 1% with HNO<sub>3</sub>
- Internal standards: <sup>6</sup>Li, <sup>45</sup>Sc, <sup>89</sup>Y, <sup>103</sup>Rh, <sup>115</sup>In, <sup>159</sup>Tb, <sup>165</sup>Ho, <sup>175</sup>Lu, and <sup>209</sup>Bi added via a mixing tee
- Concentric nebulizer, conical impact bead spray chamber with Peltier cooling, Xt interface, CCT not required
- Sample analysis time: 2m45s



**XSERIES 2**

ANALYTE	MDL (ppb)	SLRS-4				NIST 1640			
		X	SD	CERTIFIED	RECOVERY %	X	SD	CERTIFIED	RECOVERY %
Ag	0.01	0.02	0.01			7.39	0.06	7.62	97
Al	0.02	52	1	54	97	53	1	52	101
As	0.06	0.79	0.09	0.68	116	27.7	0.6	26.67	104
Ba	0.07	12.9	0.3	12.2	106	145	3	148	98
Be	0.04	<MDL	0.002	0.007		34	1	34.94	98
Cd	0.02	<MDL	0.001	0.012		23.6	0.4	22.79	104
Co	0.01	0.031	0.005	0.033	94	19.5	0.1	20.28	96
Cr	0.02	0.28	0.02	0.33	84	38.0	0.6	38.6	99
Cu	0.02	1.46	0.06	1.81	81	81	2	85.2	96
Mn	0.01	3.38	0.09	3.37	100	118	2	121.5	97
Mo	0.03	0.262	0.009	0.21	125	43.8	0.5	46.75	94
Ni	0.03	0.62	0.03	0.67	93	25	1	27.4	92
Pb	0.006	0.080	0.003	0.086	93	26.0	0.2	27.89	93
Sb	0.03	0.27	0.01	0.23	118	13.8	0.3	13.79	100
Se	0.2	<MDL	0.1			23.2	0.3	21.96	106
Th	0.01	0.011	0.007			0.08	0.01		
Tl	0.005	<MDL	0.005			0.023	0.004		
U	0.007	0.041	0.006	0.05	81	0.78	0.02		
V	0.02	0.35	0.04	0.32	109	12.5	0.1	12.99	96
Zn	0.08	1.04	0.05	0.93	112	55.6	0.7	53.2	104
Ca*	4	5.63	0.08	6.2	91	6.9	0.1	7.045	98
Fe*	3	93	2	103	90	32.5	0.5	34.3	95
K*	2	0.63	0.02	0.68	93	0.93	0.02	0.994	94
Mg*	1	1.57	0.02	1.6	98	5.64	0.09	5.189	109
Na*	3	2.20	0.01	2.4	92	28.7	0.3	29.35	98

\* in ppm (except MDL)

# Summary

Low sample numbers or single element work	AA
Moderate-high sample numbers for multiple elements	ICP or ICP-MS
Lowest detection limits	ICP-MS > ICP
Freedom from spectral interferences	ICP-MS = ICP
Freedom from physical interferences	Radial ICP > Axial ICP > ICP-MS
'Dirty' samples with higher levels of contamination	ICP or AA
Solid digests with moderate analyte levels and high matrix	ICP or ICP-MS
'Clean' samples for low levels	ICP-MS
Challenging interfering matrices with very low analyte concentrations	ICP-SFMS (or CRC)