

How to Decide Which is the Right Technique for your Application?

Evrim Kilicgedik Product Specialist, Atomic Spectroscopy

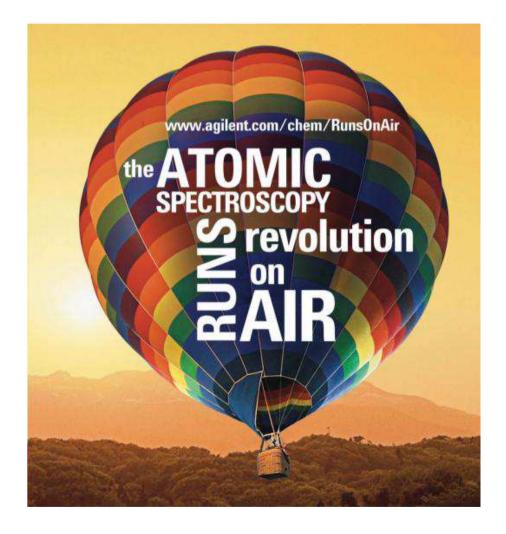
**Agilent Technologies** 

22.03.2012



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### **2011 – The Atomic Spectroscopy Revolution**



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With the 2011 launching of the Atomic Spectroscopy Revolution, Agilent now offers the most comprehensive and innovative range of inorganic (atomic) spectroscopy instruments for liquid sample analysis:

- Flame Atomic Absorption (AA);
- Graphite Furnace AA;
- Vapor Generation AA;
- Microwave Plasma-Atomic Emission (MP-AES);
- ICP-Optical Emission; as well as
- The 7700 Series ICP-MS



### **Agilent's New Atomic Spectroscopy Portfolio**



**ICP-MS** 

4100 MP-AES





Flame AAS



AAS instruments can be flame only, furnace only, or combined (switchable) Graphite Furnace AAS





### **Important Decision Making Criteria**

### Sample type

Analytical performance required

- Detection limits
- Precision

### Required throughput

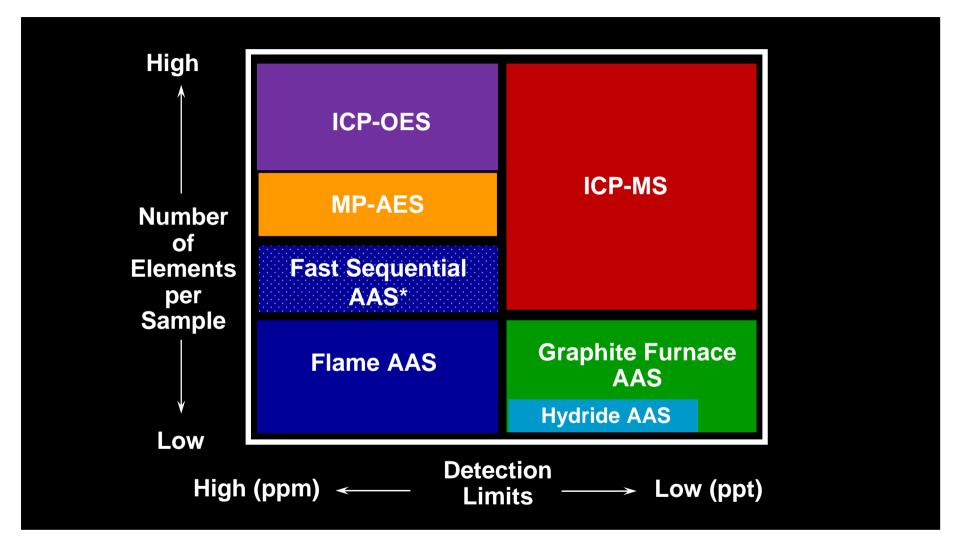
- Number of samples
- Number of analytes
- **Operator skill**
- Capital investment

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**Operating cost** 



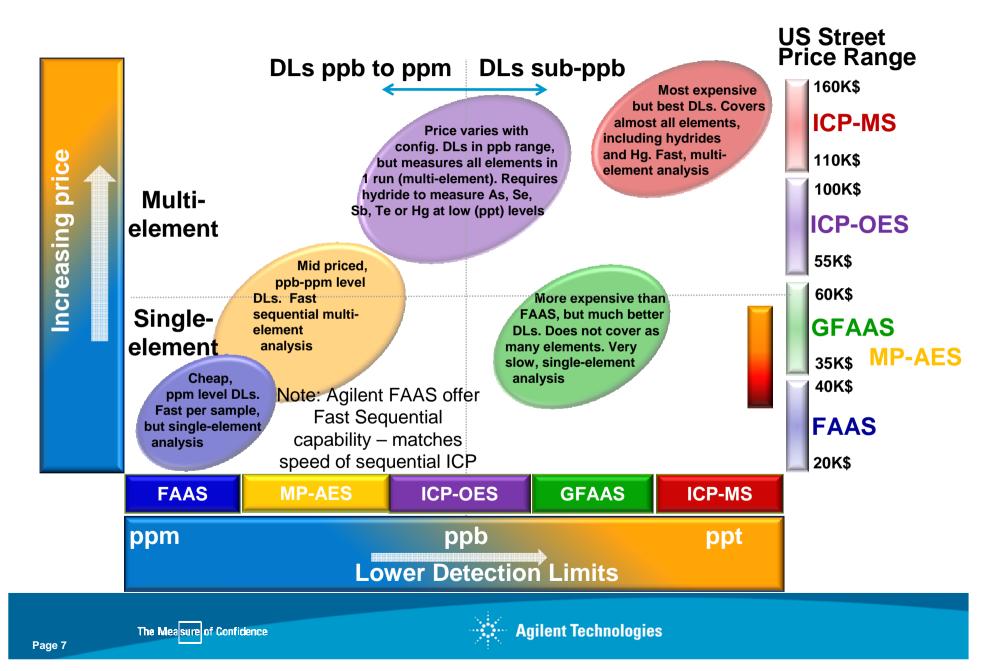
### **Number of Analytes vs Detection Limits**



\* Note: Fast Sequential AAS doubles throughput & is exclusive to Agilent Flame AA (as is MP-AES)

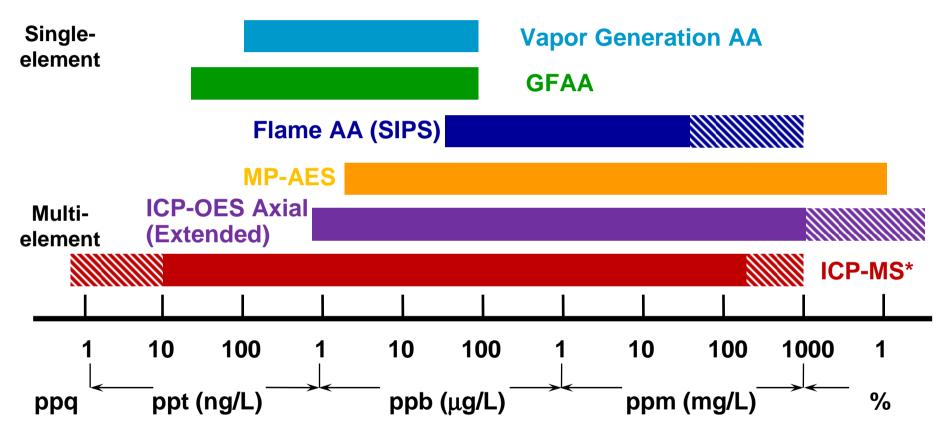


### **Agilent Inorganic Product Portfolio Positioning**



# **Example - Dynamic or Working Range Comparison**

The true working range combines the detector dynamic range, the matrix tolerance and method limitations such as calibration linearity and washout Note – this is the measurement range, not the matrix level that can be tolerated



\* Note: 9 orders detector dynamic range can be extended with HMI (standard on the 7700x). For many elements, accurate analysis at <10ppt requires a cleanroom

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# **Overview of Atomic Absorption (AA) Spectroscopy**





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### **Characteristics of Atomic Absorption**

Determine concentrations of metals in solution

67 elements

From ppb to % levels

Precision typically better than 1 % RSD

Sample preparation is simple

Instrument is easy to tune

Instrument is easy to operate

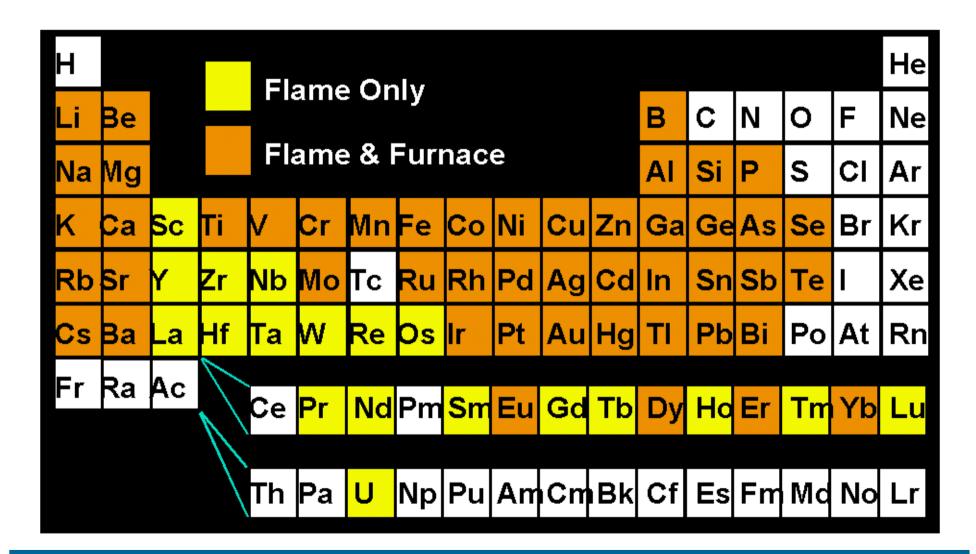
Technique is very specific

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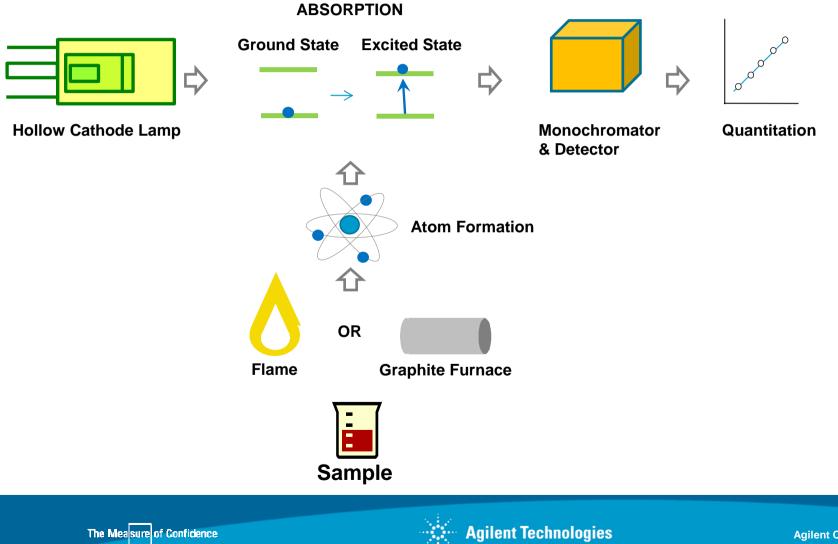
### **Elements Measurable by AA**





### **Atomic Absorption Overview**

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### **Agilent Atomic Absorption Product Range**

Entry level - SpectrAA-55B

- Stand alone system
- Keyboard control with LCD
- Optional PC control (req. for accy support)

Mid range – AA240 series

- External PC control
- Greater automation
- *"Fast Sequential"* capability (4 lamps)

#### Top end – AA280

- External PC control
- Premium positioning
- Highest performance
- Greatest automation
- *"Fast Sequential"* capability (8 lamps)

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### **Compatibility with Other Agilent Accessories**

SPS3 for automated sample presentation



SIPS-10/20 for on-line dilution with flame AA



UltrAA high intensity lamps



VGA-77 for hydride determinations





### Agilent Flame AA Systems – Benefit # 1

### Flexibility, Ease of Use & Superior Flame Performance

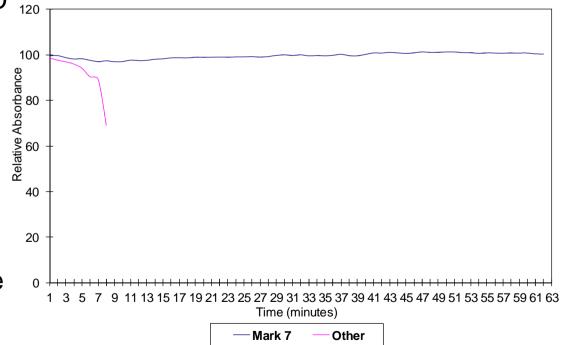
Tunable performance means...

- Highest flame sensitivity:
   > 0.9 Abs. for 5 mg/L Cu
- Best precision: < 0.5 % RSD using 10 x 5 s readings
- Extended operation with difficult samples
- No loose gas hoses and no tools required for gas connection
- Fast change-over to furnace operation (manual - < 30 s)</li>





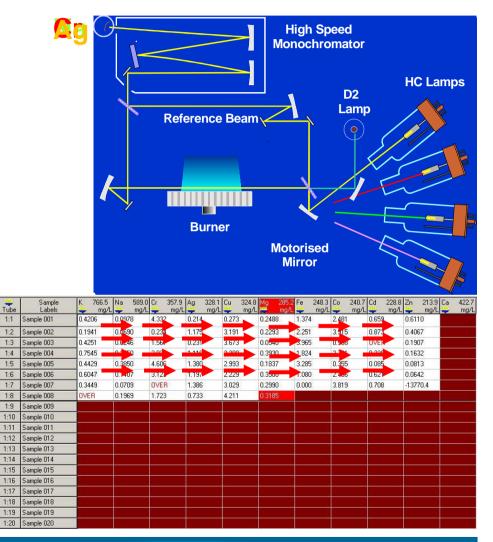
5 mg/L Cu in 7.5% NaCl



# Agilent Flame AA Systems – Benefit # 2

### **Fastest Flame AA Analysis – Double the Productivity**

- FS models provide Fast Sequential AA which means...
- All elements measured in a single reading
- Double the productivity of any other conventional AA
- Conserves sample volume
- Saves labor and reduces running costs
- Allows Internal Standard correction for improved precision and accuracy





### Simultaneous flame & furnace operation

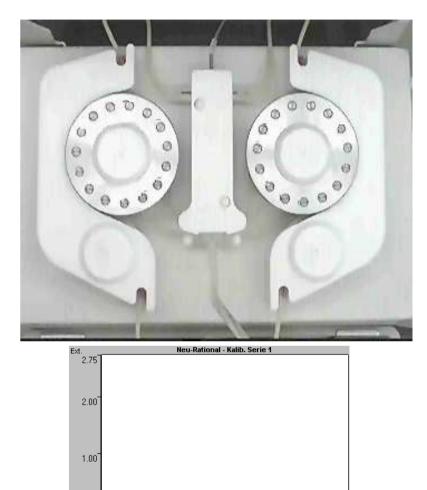






### **SIPS Intelligent Dilution System**

1.10



Preparation of a complete calibration from a single standard (max. 10 standards)

On-line addition of reagents (e.g. ionization suppressant, Lanthanum)

Fast on-line dilution of over range samples

Automatic pre-dilution of samples

Extends operating range (factor 100)

On-line preparation of Standard additions calibration from a single standard (up to 10 additions)

Automatic addition of Internal Standard



0.50

On-line addition of Cs buffer during

5 point autocalibration with SIPS-20



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0.00

### **GTA120 Graphite Tube Atomizer**





The GTA-120 graphite furnace system is suitable for use with the current range of Agilent PC controlled instruments





### Agilent Furnace AA Systems – Benefit # 3

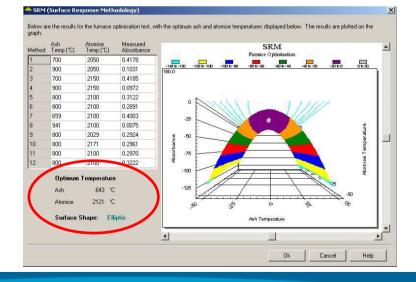
### Ease of Use

- PSD120 autosampler eliminates tedious standard preparation
  - Calibrate the AA using just 1 standard
  - Automatically dilute over range samples
  - Pre, Post or Co-inject modifiers
  - Hot Injection for reduced drying time

•Furnace viewing camera provides real time images of sample injection/drying for easy optimization

•Automated method optimization provides optimum ashing/atomization temperatures in < 30 mins (using SRM)







### **Vapor Generation Solutions**

Agilent's VGA-77 provides:

- Convenience of flame AA operation
- Better sensitivity than FIAS
- Exceptional precision of 1-2 % RSD at ppb levels
- High throughput up to 70 samples/hr
- Modular operation change modules to eliminate contamination





# **Vapor Performance Comparison (µg/L)**

<u>Element</u>	<u>Characteristic</u> Concentration	<u>Detection</u> Limit
As	0.2	0.1
Se	0.3	0.15
Hg (normal cell)	0.3	0.10
Hg (flow thru cell)	0.2	0.05
Sb	0.2	0.06
Bi	0.2	0.07
Те	0.15	0.15
Sn	0.3	0.2



# **Overview of Microwave Plasma (MP) Spectroscopy**





# Agilent 4100 Microwave Plasma-Atomic Emission Spectrometer (MP-AES)

#### New technique for elemental determination using atomic emission

- Microwave excited plasma source
- Nitrogen based plasma runs on air (using a N<sub>2</sub> generator)

#### Improved performance compared with flame AA:

- Higher sample throughput with fast sequential measurement
  - More than 2x faster than conventional flame AA
- Superior detection limits and improved dynamic range

#### Easy to use:

- New generation software featuring automated optimization and software applets that load a preset method
- One piece torch with easy torch removal and replacement no alignment

#### **Reduced operating costs:**

- Runs on air eliminates need for Acetylene, Argon, etc.
- Eliminates need for source/hollow cathode lamps
- Simple installation no chiller, 10 A supply

#### **Improved Safety:**

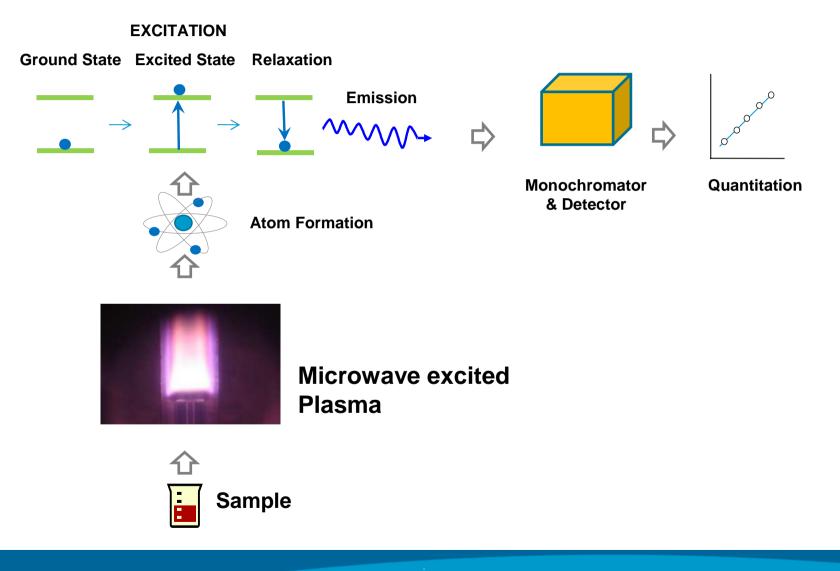
- Eliminates need for flammable gases and cylinder handling
- Safe, reliable unattended multi-element overnight operation







### **Microwave Plasma Emission Overview**



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### **Accessory Options for the 4100 MP-AES**

- Automate and simplify analysis with the SPS3 autosampler (required for unattended overnight operation)
- For organic applications, use the EGCM to bleed air into the plasma minimizing C build-up and reducing background

 also requires the OneNeb inert nebulizer (incl. with the Organics kit)

 To enable low ppb level detection of As, Se or Hg, use the Multimode Sample Introduction System (MSIS)

 also requires the 5 channel peristaltic pump option

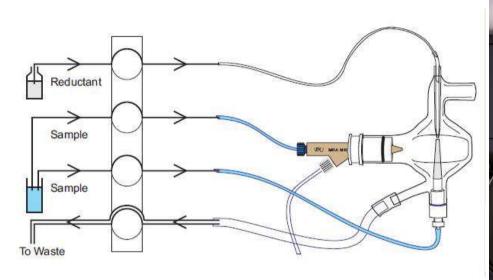




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# Improving Detection Limits for As, Se, Hg with MSIS





Analyte	Conventional Nebulization ug/L	MSIS in Simultaneous Mode ug/L
As	90	1.0
Se	126	2.0
Hg	14	0.5

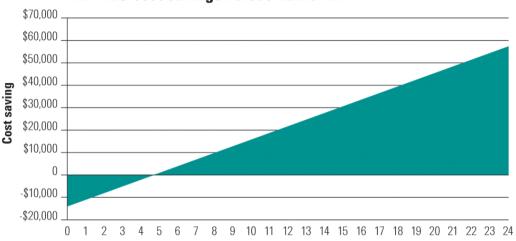
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# Agilent 4100 MP-AES

### Lowest running costs

Runs on Air (using a nitrogen generator), which means...

- Eliminates need for flammable and expensive gases e.g. acetylene
- Improved safety no need for flammable gases in the lab. and no manual cylinder handling
- Provides safe, unattended multielement overnight operation
- Greater flexibility no need for source lamps, so you can add extra elements at any time



#### **MP-AES cost savings versus flame AA**



Months



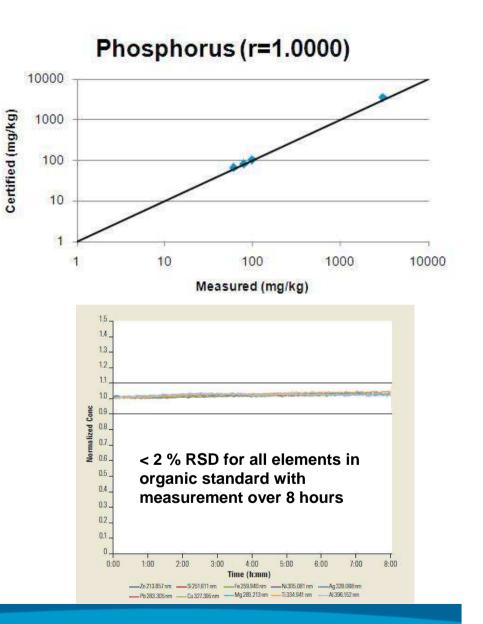
# Agilent 4100 MP-AES

### **Superior performance to Flame AA**

- MP-AES provides Fast Sequential multi-element determinations which means...
- All elements measured in a single reading
- More than double the productivity of conventional flame AA systems
- Superior detection limits
- Improved dynamic range

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 Better sensitivity - especially for refractory elements - and more elemental coverage e.g. S





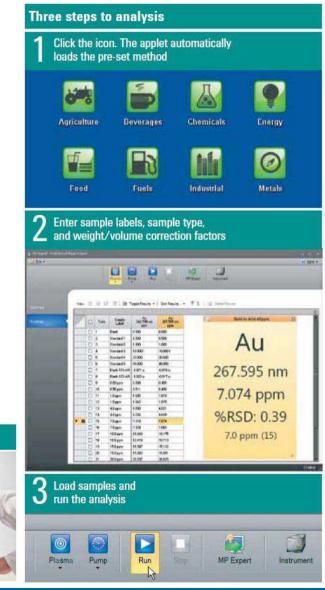
### Agilent 4100 MP-AES

### Ease of Use

Simplifies operation for all users – both novice and more experienced users

- Software applets that automatically load a pre-set method ideal for novice users
- One piece torch with simple torch loading – and no alignment
- 1 step automated method optimization

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# **Overview of ICP-OES Spectroscopy**





# **Three New ICP-OES Models!**

### Agilent 710/715-ES

- Entry level system with CCD detector
- Affordable price, uncompromised performance
- A fully featured ICP-OES for laboratories with moderate or small sample loads

### Agilent 720/725-ES

- High sample throughput
- Ultimate in performance
- Flexibility to upgrade in the future for higher productivity and performance

### Agilent 730/735-ES

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- Highest sample throughput
- Ultimate in performance
- The World's best ICP-OES!



0 = Axial View 5 = Radial View



### Agilent 720/730-ES Series ICP-OES

40 MHz free-running RF Generator proven in more than 4000 systems State-of-the-art, customdesigned CCD detector with unmatched performance

Switching Valve System improves washout for greater productivity and lower cost per analysis



Only system to provide true simultaneous measurement from ppb to % levels

Customized sample introduction system for every application ICPExpert II software provides even greater easeof-use and and security



### Agilent 720/730-ES Series ICP-OES – Optical Bench

One of the most important and unique features of this imagemapped detector is its individual pixel anti-blooming control



### Agilent 720/730-ES Series ICP-OES – Most Innovative Detector

- Full wavelength coverage
  - Wavelength coverage 167 785 nm
  - Use alternate wavelengths to extended dynamic range and confirm results
- Individually addressed arrays
  - Adaptive Integration
- True simultaneous measurement
- Duplex readout circuitry halves readout time
- Peltier cooled to -35°C

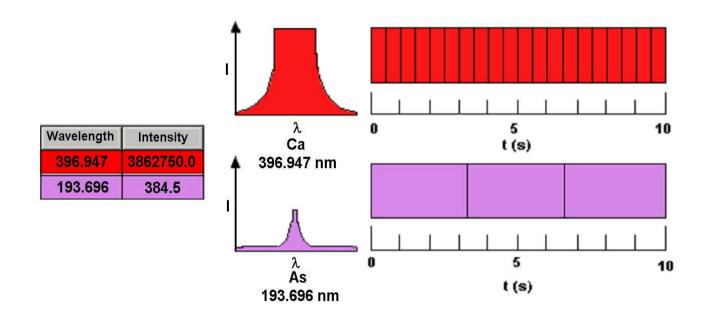
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 Ensures low noise and best detection limits





### **Adaptive Integration**<sup>™</sup>



#### Achieve outstanding results automatically with Adaptive Integration<sup>™</sup>.

Based on signal to noise ratios, multiple short readings are averaged for high signals and a smaller number of longer readings are averaged for low signals.



### Agilent 710/715-ES ICP-OES

40 MHz free-running RF Generator proven in more than 4000 systems High performance megapixel CCD detector provides excellent detection limits and wide linear dynamic range

Complete wavelength coverage from 177 to 785nm



Measure ppb to % levels from a single view with Multi**Cal** 

Start analyzing samples 10 minutes following plasma ignition

ICPExpert II software provides even greater easeof-use and and security



# Agilent 710/715-ES ICP-OES

Low cost entry level simultaneous ICP-OES

- Equipped with 3 channel pump as standard
  - On-line addition of internal standard and ionization buffer
- Optimized sample introduction system
  - 715-ES optimized for difficult samples
    - robust, inert sample introduction
  - 710-ES optimized for maximum sensitivity and lowest DL's
- Supports a range of accessories for added performance
  - AGM-1 Oxygen accessory for stable analysis of volatile organics
  - Mounted VGA provides sub-ppb measurement of Hg, As, Se, Sb
  - SPS3 Sample Preparation System



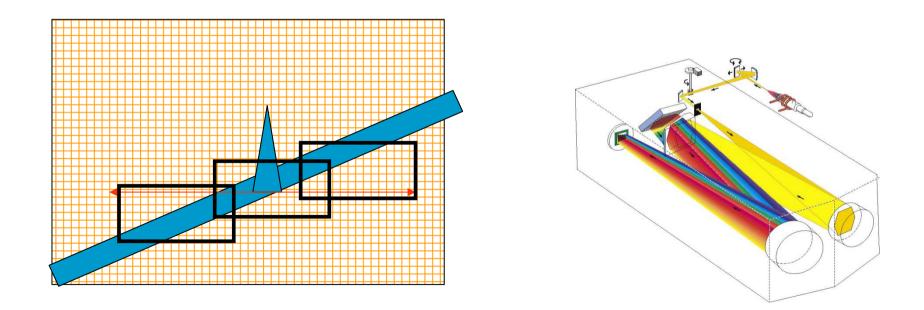
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### Agilent 710/715-ES ICP-OES - Echelle Optics

Similar optics to 720/730-ES Series

An echelle order falls across a number of detector rows



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## **ICP-OES Configuration**

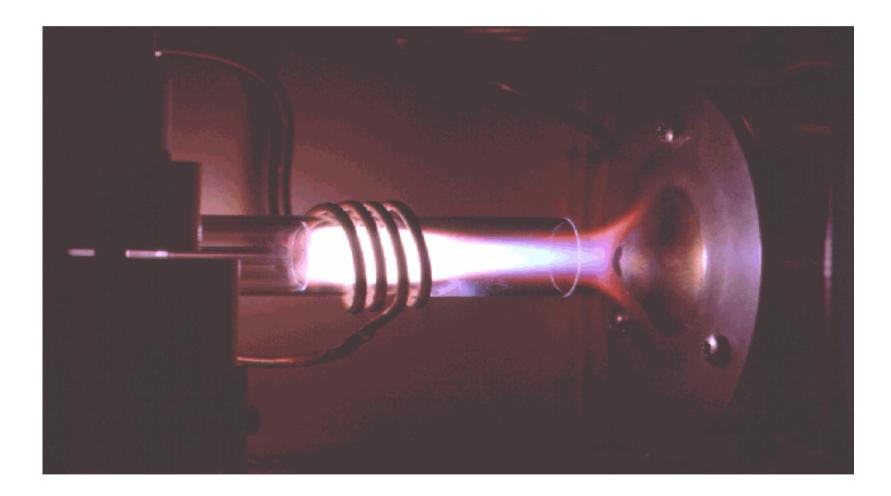
- Two configurations are available:
- Axial
  - Plasma viewed end-on
    - Lower detection limits
  - Cooled cone interface makes dual-view optics obsolete
- Radial
  - Plasma viewed side-on

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- Recommended for complex matrices
  - High Total Dissolved Solids (>20%)
  - Organic matrices. Eg..Oils



#### **Cooled Cone Interface (CCI)**



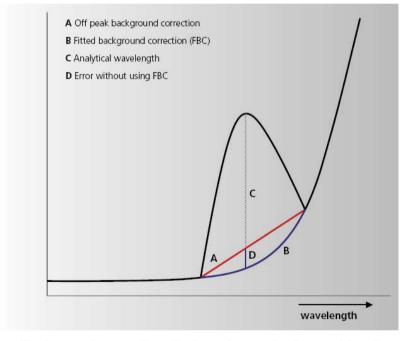


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# Agilent 700-ES Series ICP-OES - Background Correction Methods

# Traditional off-peak - User can view actual signals to select background points



Fitted background correction calculates the true background signal, improving accuracy.

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# Agilent's unique Fitted background correction

- Peak shaped functions applied to the analyte peak
- Polynomial Interpolation of background signal

#### **Advantages**

- Improved accuracy
- Requires no extra user input
- Truly simultaneous correction

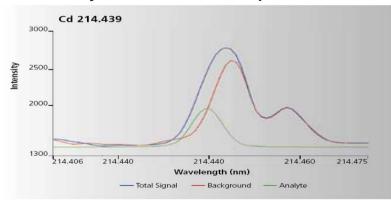


# Agilent 700-ES Series ICP-OES - Background Correction Methods

Agilent's patented FACT background correction

Fast Automated Curve-fitting Technique (FACT) for accurate removal of spectral interferences

 Peak modelling approach - Uses spectral data from analyte and interference standards to de-convolve the analyte peak from nearby interference peaks



FACT removes iron interference providing accurate determination of trace level cadmium in soils.

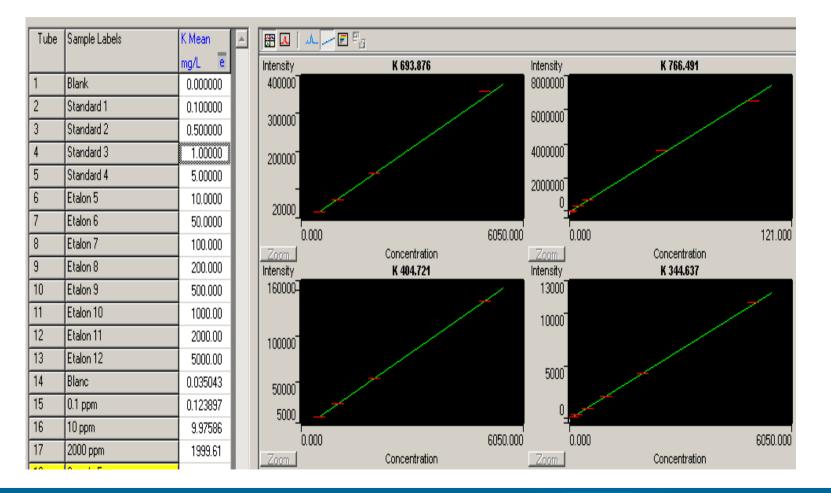
#### **Advantages**

- Resolves extremely complex spectral interferences
- Gives access to extra wavelengths for improved validation
- Allows resolution of interferences as close as 3 pm



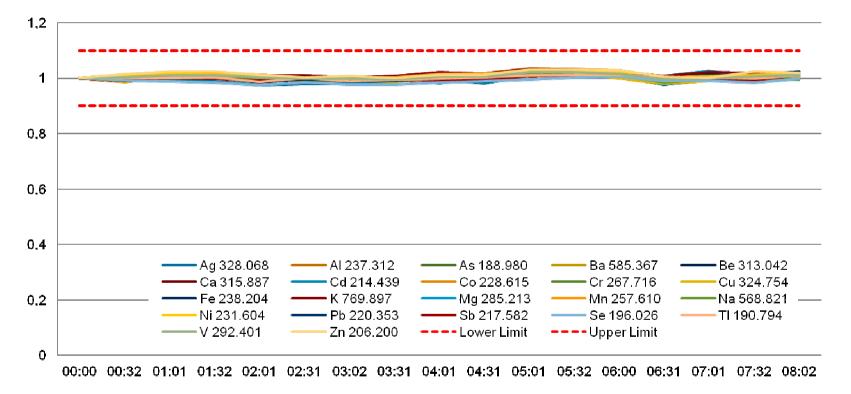
### **Continuous Wavelength Coverage**

# K measured over the concentration range from 100 ppb – 5000 ppm, using combination of sensitive and less-sensitive lines





### **Robust, Stable Plasma System**



#### Agilent 720 stability < 0.98% RSD Max.



# **Overview of ICP-MS Spectroscopy**





## Agilent 8800 ICP-QQQ

- World's first Triple Quadrupole ICP-MS (ICP-QQQ)
- New modes of operation and performance not possible with quadrupole ICP-MS
- Joins the Agilent 7700, the highest performing quadrupole ICP-MS system
- Unique capabilities, based on proven technology





#### **7700 Series – New Product Highlights**

- New ORS<sup>3</sup> Collision/Reaction Cell
  - Longer, narrower rods, higher cell pressure and frequency MUCH better performance in He mode
- New RF Generator
  - u Fast, frequency-matching 27MHz generator, for better tolerance to changing matrix (incl. organics)
- **Increased Matrix Tolerance** 
  - u High Matrix Introduction (HMI) standard on 7700x model
- Much smaller cabinet
  - u >30% smaller footprint than any other ICP-MS
- Simple software; reliable Auto-Tuning
  - MassHunter software intuitive and easy to learn. Pre-set plasma conditions and fast lens auto-tuning





### 7700x – The Three Key Performance Benefits



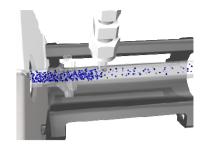
- 1. Matrix Tolerance Sample Intro/Plasma/HMI (unique to Agilent)
- 2. Interference Removal He Mode *(unique to Agilent)*
- 3. Dynamic Range 9 Orders at the Detector (unique to Agilent)



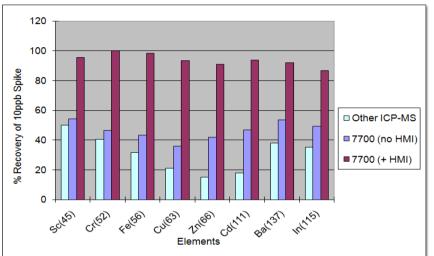
# 7700 Series ICP-MS

#### 7700 has better matrix tolerance

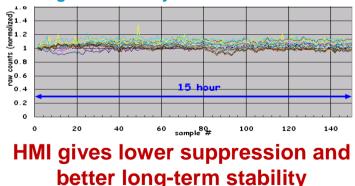
- Handles tough sample matrices better than any other ICP-MS
  - Highest plasma temperature (lowest CeO/Ce) as standard
  - AND HMI (high matrix introduction).
     HMI is still unique to Agilent and is standard on the 7700x
- Gives lower levels of interference and better long-term stability
  - Interferences are broken-down in the plasma; less material deposits on the interface cones



#### Relative signal (suppression) in undiluted seawater



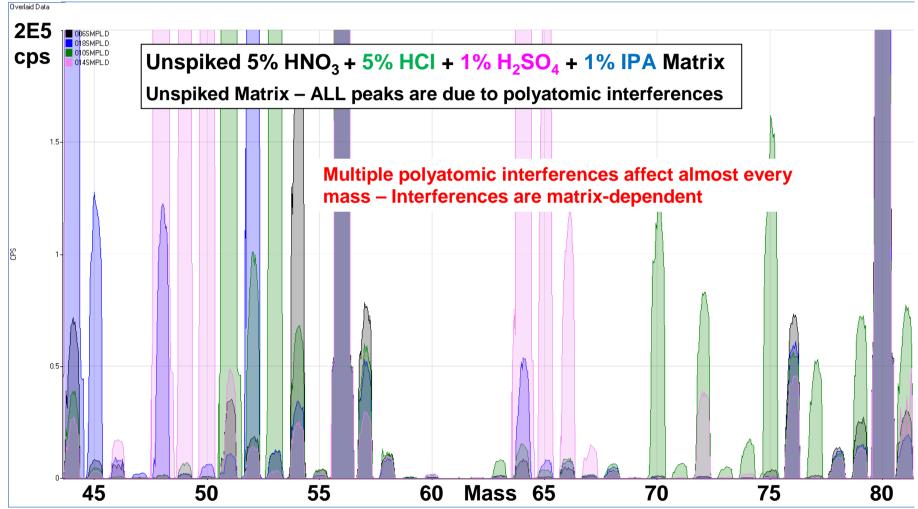
Long-term stability in undiluted seawater





#### **Blank Acid Matrices and IPA in No Gas Mode**

#### Color of spectrum indicates which matrix gave each interfering peak



No Gas Mode



#### **Blank Acid Matrices and IPA in He Mode**

#### Overlaid Data 2E5 006SMPL.D 018SMPL.D 010SMPL.D cps Unspiked 5% HNO<sub>3</sub> + 5% HCI + 1% H<sub>2</sub>SO<sub>4</sub> + 1% IPA Matrix 014SMPL.D ALL polyatomic interferences are removed in He Mode (same cell conditions) 1 F **ALL polyatomic** interferences are removed Is sensitivity still OK? in He Mode 0.5 Mass 65 70 45 50 55 60 75 80 **He Mode**

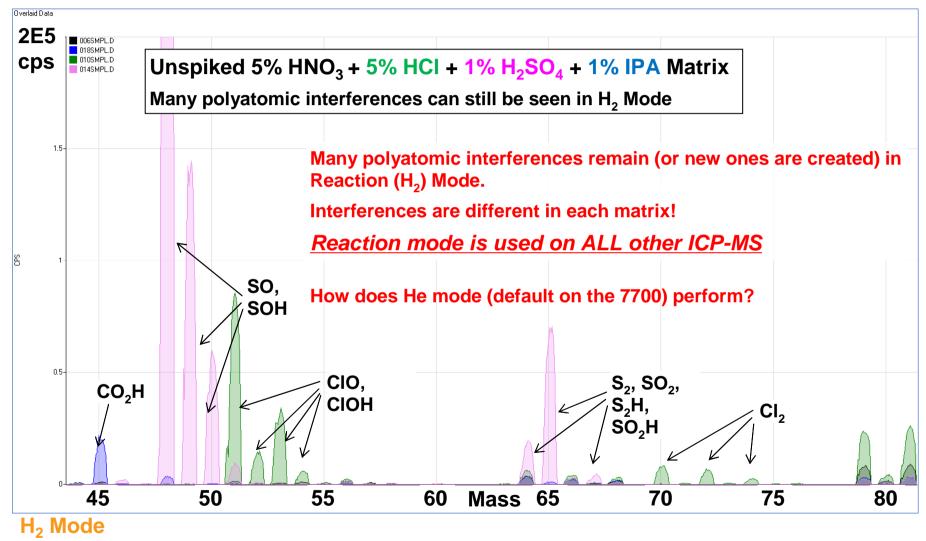
#### Color of spectrum indicates which matrix gave each interfering peak

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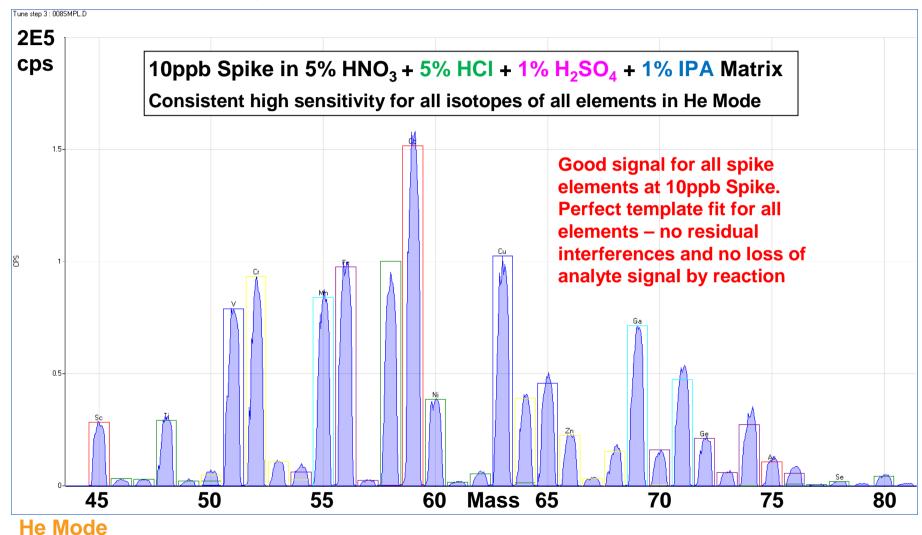
#### **Blank Acid Matrices and IPA in H<sub>2</sub> (Reaction) Mode**

#### Color of spectrum indicates which matrix gave each interfering peak





#### Matrix Mix with Spike (10ppb) in He Mode



Consistent sensitivity and perfect template match for all elements





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#### 7700x – 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

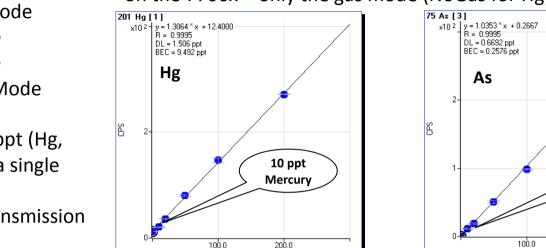
#### Na

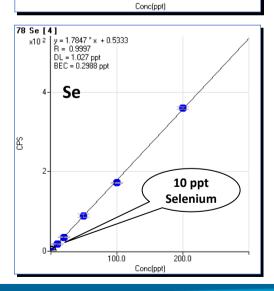
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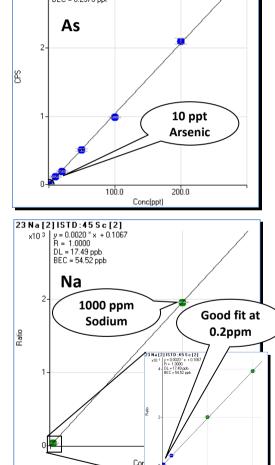
Typically, ICP-MS cannot measure above 200ppm Na without changing quad resolution or ion lens settings Hg

Hg LOD on 7700x is about 2ppt – 7700x can QUANTITATE at 10ppt!

7700x can do both of the above in the same run!







20000.0

These 4 plots were obtained under the same analytical conditions on the 7700x – only the gas mode (NoGas for Hg) changed

Technique Selection Criterie					
<b>Technique Selection Criteria</b>		Flame AA	GFAA		
	Detection limits				
Detection limits	%				
Sample throughput	ppm				
Number of elements measured per	High ppb				
sample	Low ppb		$\bigcirc$		
Matrix Interferences	ppt		$\bigcirc$		
✓Ease of use	Number of Samples				
✓Cost of Ownership	Few				
-	Several				
	Many				
ICP-MS ICP-OES		Number of Elements per S			
	Single				
MP-AES	Few				
GFAA Flame AA	Many				
		Sample	e Matrix		
\$0K \$50K \$100K \$150K \$200K	< 3%				
System Cost (\$)	3 – 10%	•			

#### MP-AES its ples er Sample 1 > 10%

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# Technique **Selection** Criteria

	Flame AA	GFAA	MP-AES	ICP-OES	ICP-MS
Criteria					
Measurement Range					
high > 10%				x	
1 - 10 %	x		x	x	
ppm	х		X	x	X
high ppb	х	х	X	x	Х
low ppb		х	X	х	Х
ppt		Х			Х
Number of samples					
Few	х	х	X		
Several	x		X	х	Х
Many				х	Х
No Elements per Sample					
Single	x	х	X	x	X
Few (2-5)	х		X	x	X
Intermediate (5-10)			X	х	x
Many				x	х
Sample Matrix					
< 3%	x	х	x	x	x
3-10 %	X	X	X	х	
> 10%		х		x	



# Technique Selection Criteria

Note: 1) with SIPS

	Flame AA	GFAA	MP-AES	ICP-OES	ICP-MS
Criteria					
Linearity			-		
up to 3 orders	x	Х	x	X	Х
5 orders	X 1)		x	x	X
6 orders				x	X
> 7 orders					х
Ease of Use			_		
Simple	x		x		
Moderate				x	X
Complex		х			x
Capital Investment					
Low	x				
Low-Med	x	Х	x		
Med-High				x	
High					x
Running Cost					
Low	x		X		
Med	x	Х		Х	x
High				x	x





#### The Market Leaders in Atomic Spectroscopy

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