Sci
Spec

Trace elemental analysis solutions for your application

June 6, 2018

- Understanding how each technique works
- Components of instrument
- Selection Criteria
- Application Fields

All these techniques can be used to measure trace metals

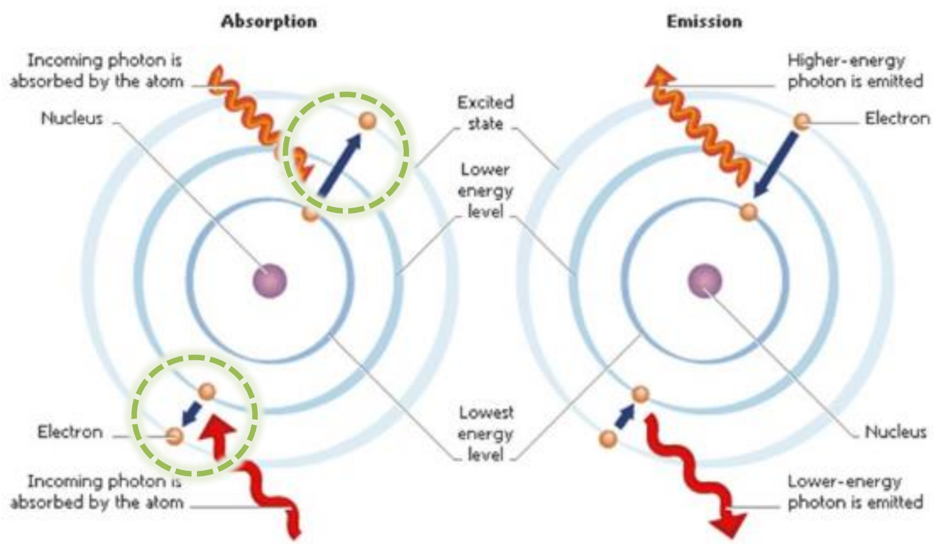
- Flame Atomic Absorption Spectroscopy (FAAS)
- Graphite Furnace Atomic Absorption Spectroscopy (GFAAS)
- Inductively Coupled Plasma Emission Spectroscopy (ICP-OES)
- Inductively Coupled Plasma Mass Spectrometry (ICP-MS)

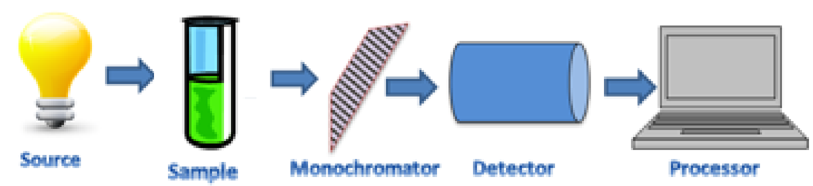
Most labs will have several techniques available.

How do you choose the best one to use for a given task ?

What is atomic absorption spectrometry? (AAs)

- The technique uses basically the principle that free atoms generated in an atomizer can absorb radiation at specific frequency
- AAs quantifies the absorption of ground state atoms in the gaseous state
- The atoms absorb UV or Vis light and make transitions to higher electronic energy levels
- The analyte concentration is determined from the amount of absorption



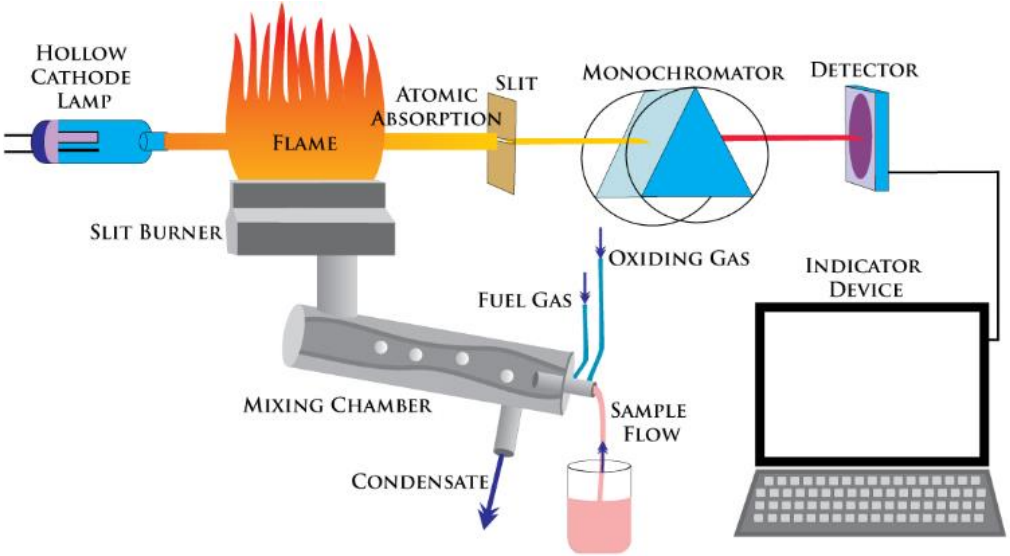


What is atomic absorption spectrometry? (AAs)

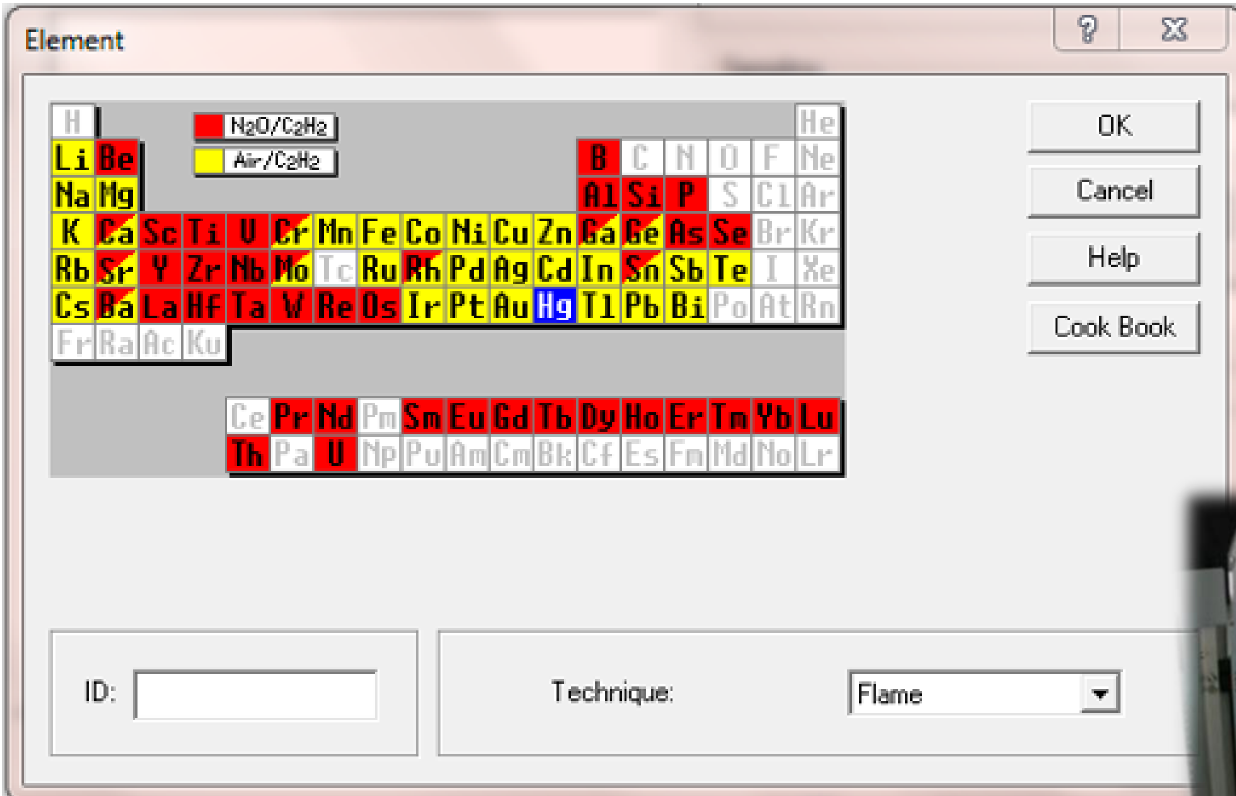
- Flame Atomic Absorption Spectroscopy (FAAS)
- Graphite Furnace Atomic Absorption Spectroscopy (GFAAS)

Air/C_2H_2

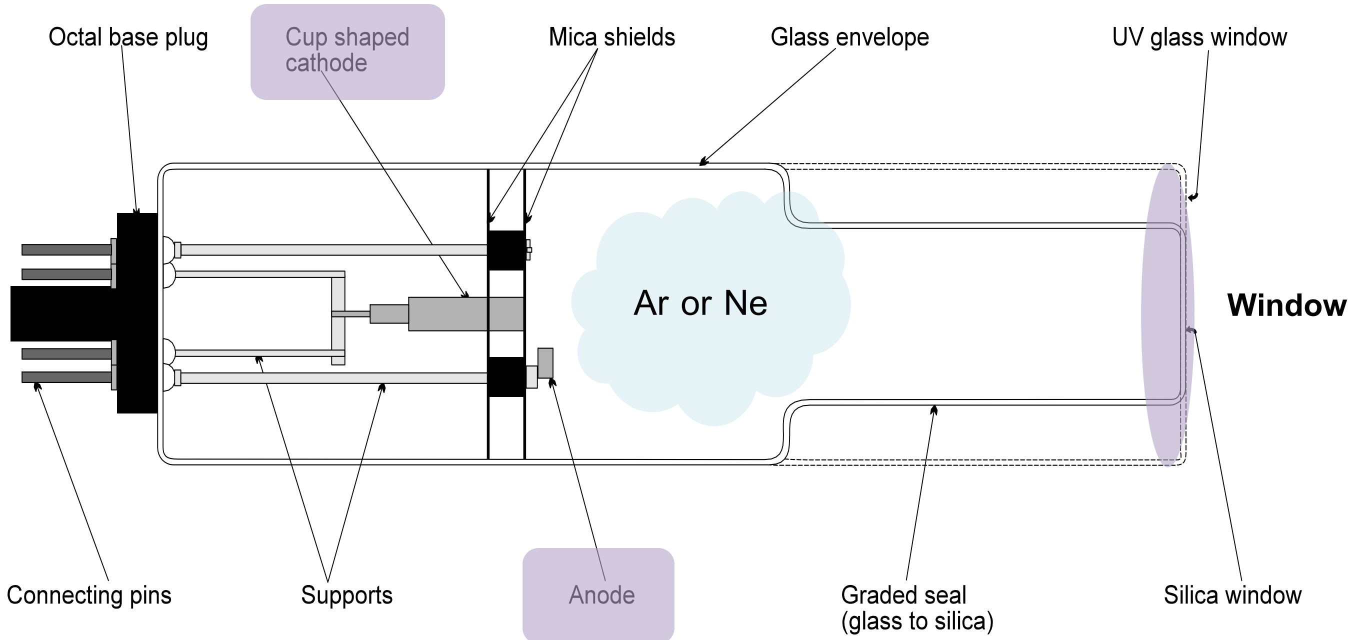
N_2O/C_2H_2



Flame Atomic Absorption Spectroscopy (FAAS)



Light source : Hollow cathode lamp (HCL)



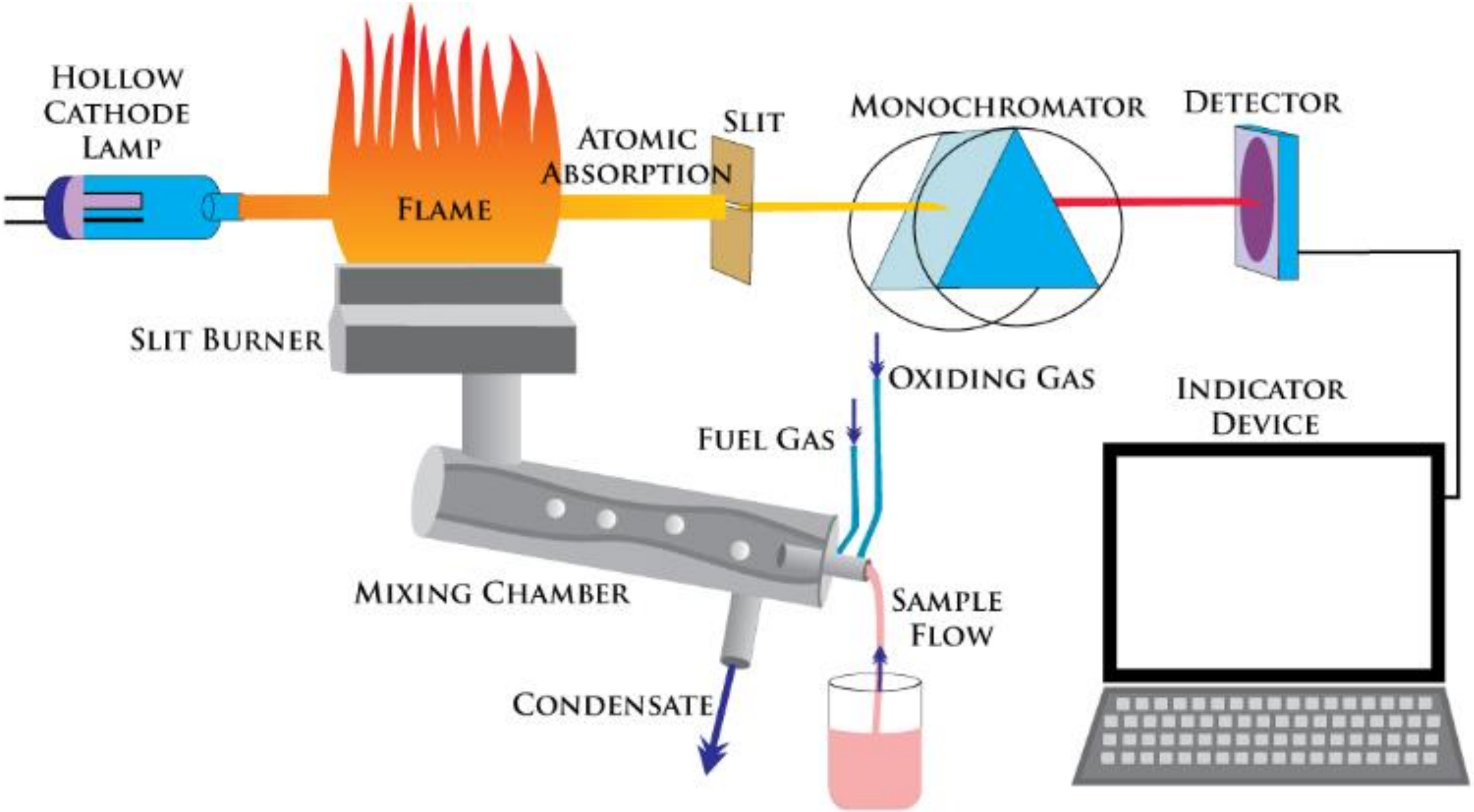
(-) Remember atoms absorb and emit light at a specific wavelength
Light is passed through the flame from a Hollow Cathode Lamp (HCL) specific to the element of analytical interest

Sputtering **Excitation** **Emission**

ɔms absorb and emit light at a specific ıvelength

ȷht is passed through the flame from a Hollow Cathode Lamp (HCL) specific to the element of analytical interest

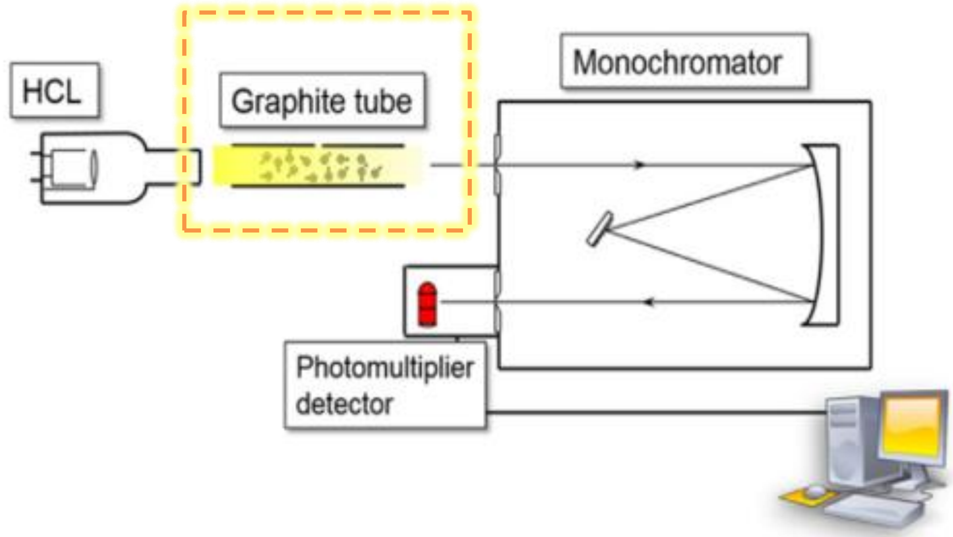
What is atomic absorption spectrometry? (AAs)



What is atomic absorption spectrometry? (AAs)

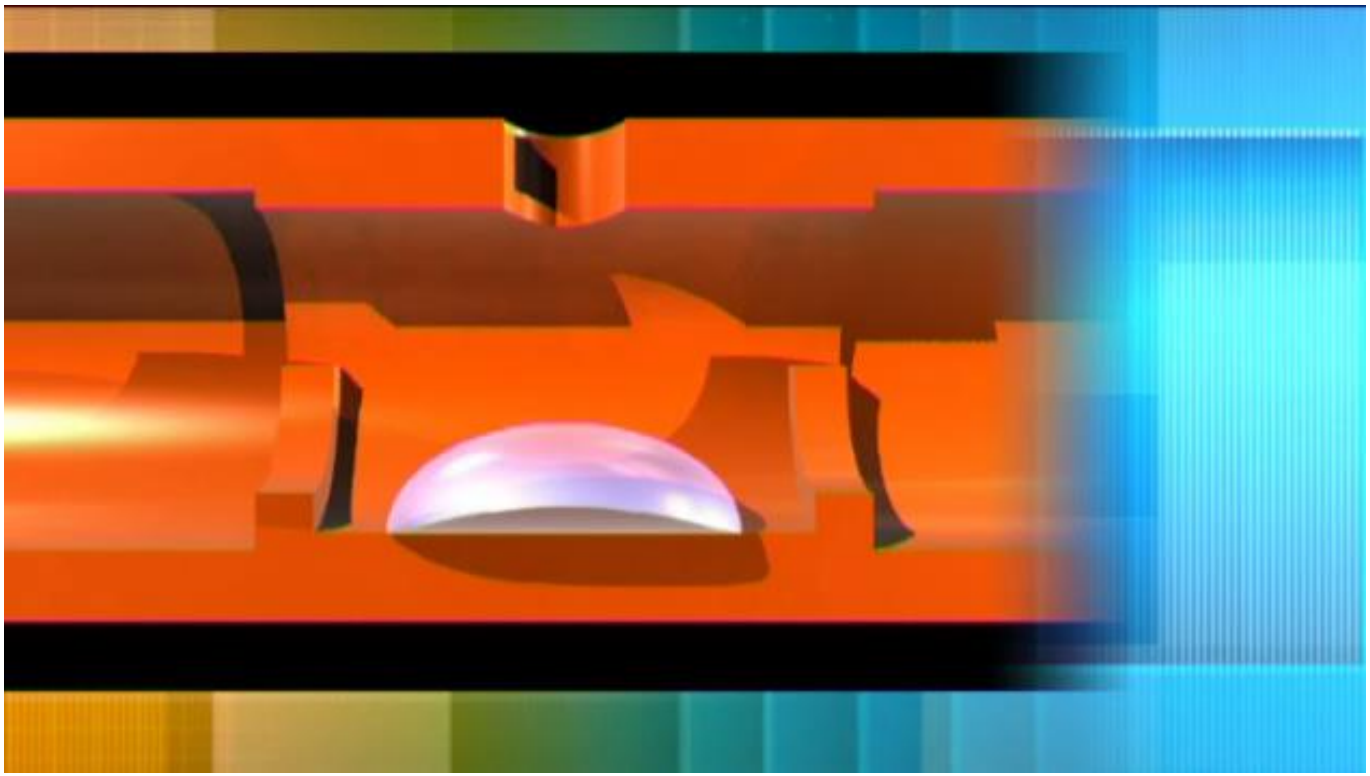
- Flame Atomic Absorption Spectroscopy (FAAS)
- Graphite Furnace Atomic Absorption Spectroscopy (GFAAS)

Graphite tube



Graphite Furnace Atomic Absorption Spectroscopy (GFAAS)

This technique is essentially the same as flame AA, except the flame is replaced by a small, electrically heated graphite tube, or cuvette, which is heated to a temperature up to 3000°C to generate the cloud of atoms. The higher atom density and longer residence time in the tube improve furnace AAS detection limits by a factor of up to 1000x compared to flame AAS, down to the *sub-ppb range*. However, because of the temperature limitation and the use of graphite cuvettes, refractory element performance is still somewhat limited.



- ✓ Superior sensitivity – detect anaytes at concentrations 10-100 times lower than flame
- ✓ High conversion efficiency of sample into free atoms.
- ✓ Low sample volumes 20 µL.
- ✓ Directs analysis of some types of liquid sample
- ✓ Low spectral interference due to generally higher temperatures.
- ✓ Is fully automated and can be left to run overnight

Atomizer	Lead (Pb)	Copper (Cu)	Arsenic (As)
Flame	0.10 mg/L	0.04 mg/L	0.40 mg/L
Furnace	0.07 µg/L	0.09 µg/L	0.26 µg/L

What is Inductively Coupled Plasma (ICP-OES)?

- A plasma will excite the atoms and ions that travel through it. When an atom or ion is excited, its electrons jump from a lower to higher energy level. Upon relaxation of these electrons to their initial 'ground' state, energy is emitted in the form of photons. The emitted photons possess wavelengths that are characteristic of their respective elements
- A detector measures the intensity of the emitted light, and calculates the concentration of that particular element in the sample
- Temperatures as high as 10,000°C, where even the most refractory elements are atomized with high efficiency. As a result, detection limits for these elements can be orders of magnitude lower with ICP than with FAAS techniques, typically at the 1-10 parts-per-billion level.

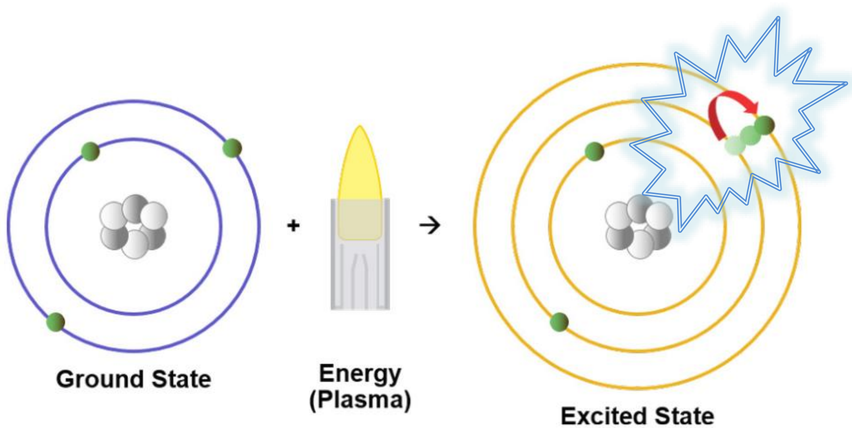
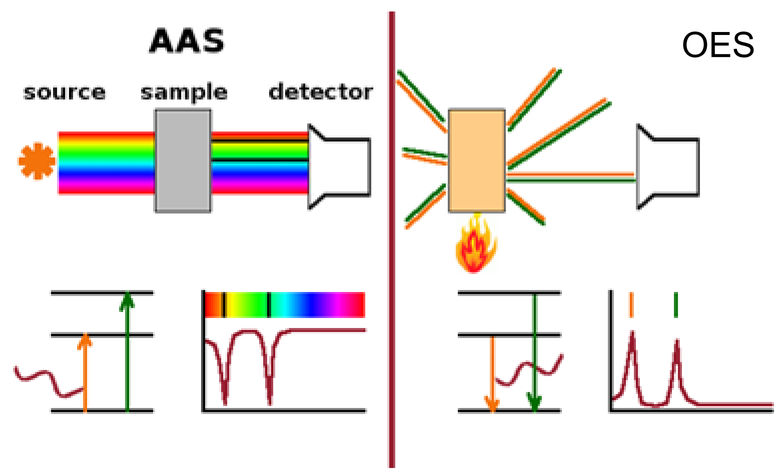
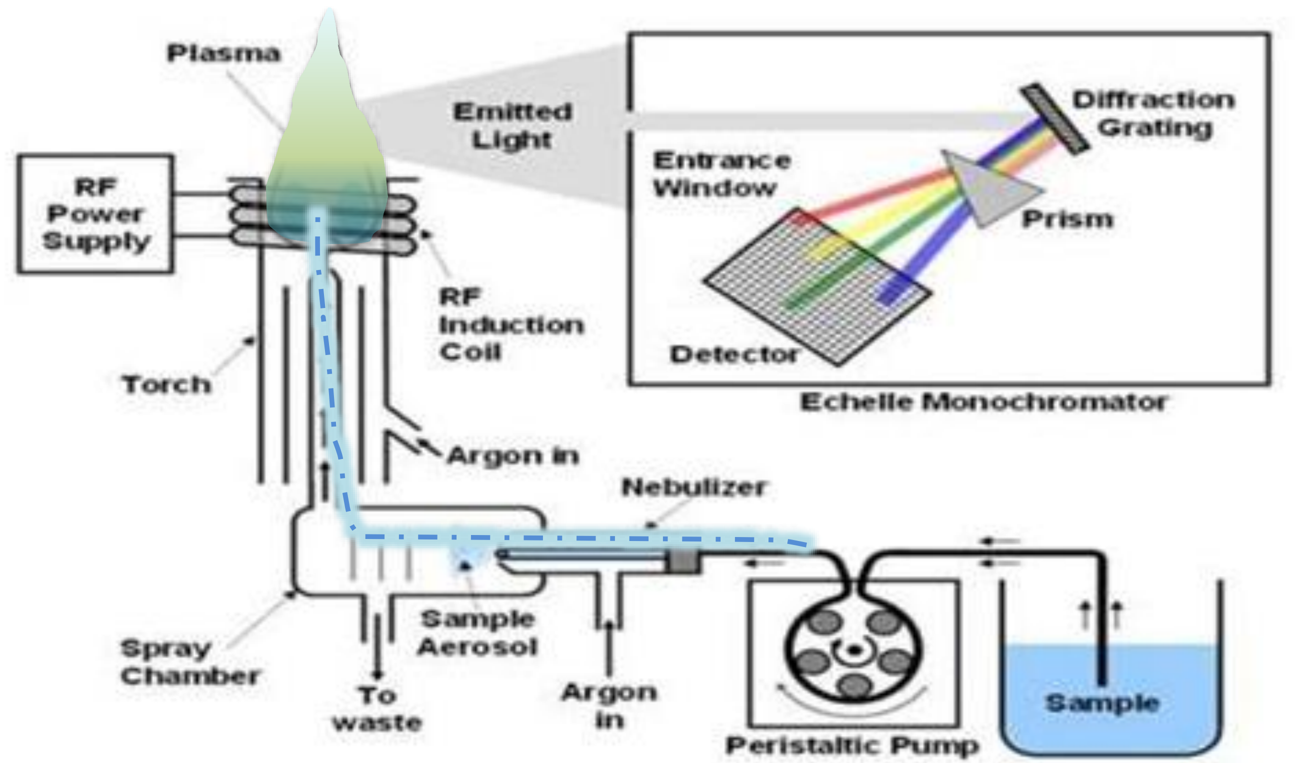


Figure 1. Excitation of an atom by a plasma.

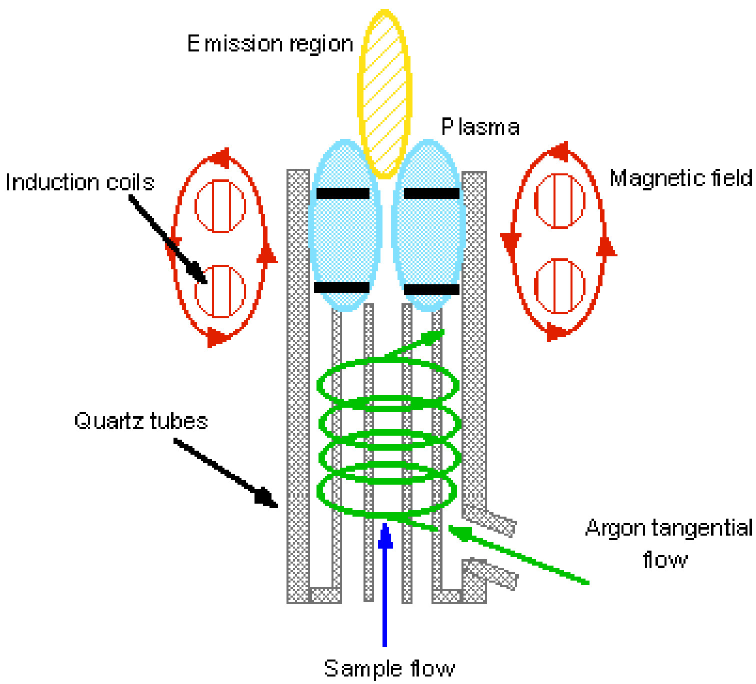


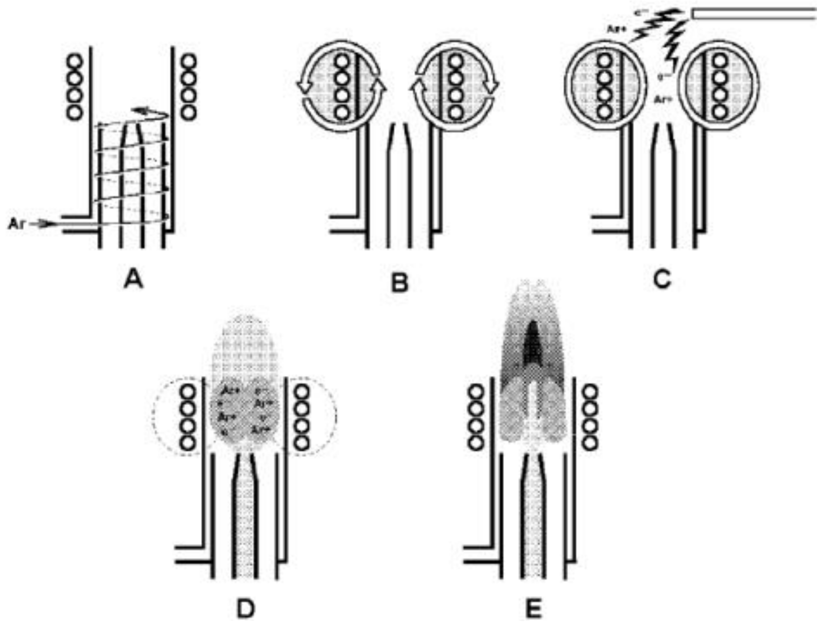
What is Inductively Coupled Plasma (ICP-OES)?



How to create the plasma?

RF power is applied to the load coil, an alternating current moves back and forth within the coil, or oscillates. This RF oscillation of the current in the coil causes RF electric and magnetic fields to be set up. With argon gas being swirled through the torch, a spark is applied to the gas causing some electrons to be stripped from their argon atoms. These electrons are then caught up in the magnetic field and accelerated by them. Adding energy to the electrons by the use of a coil in this manner is known as inductive coupling

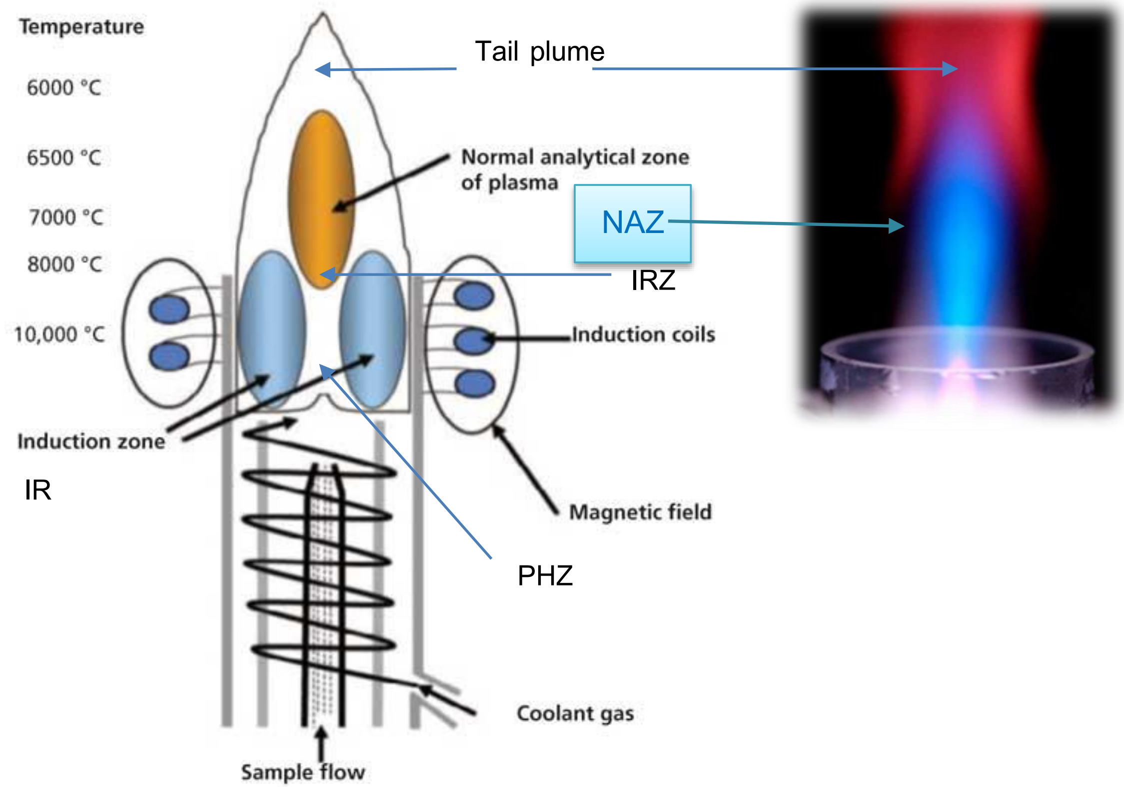




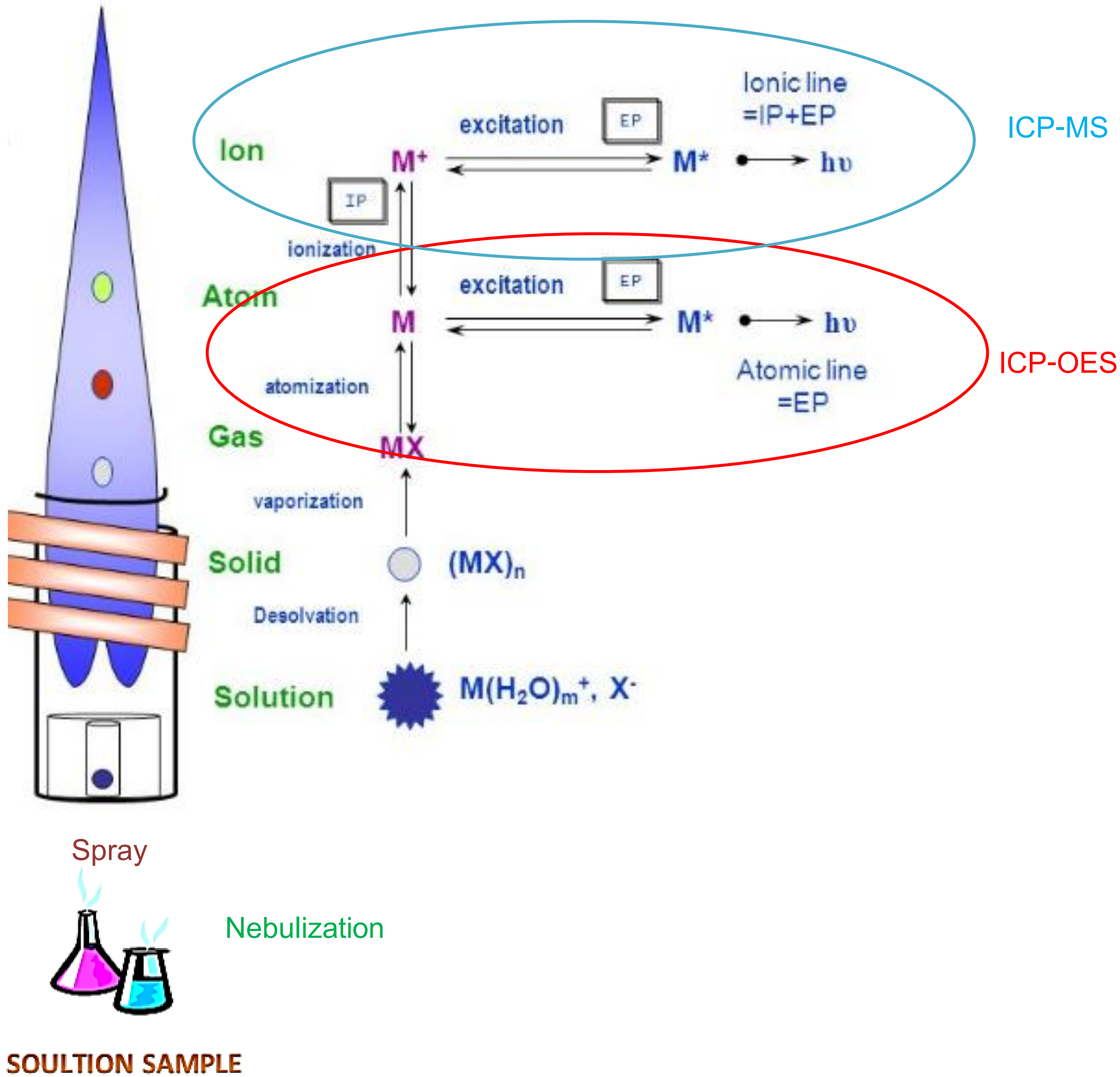
How to create the plasma?

The ICP discharge appears as a very intense, brilliant white, teardrop-shaped discharge. Which can be listed as several zone:

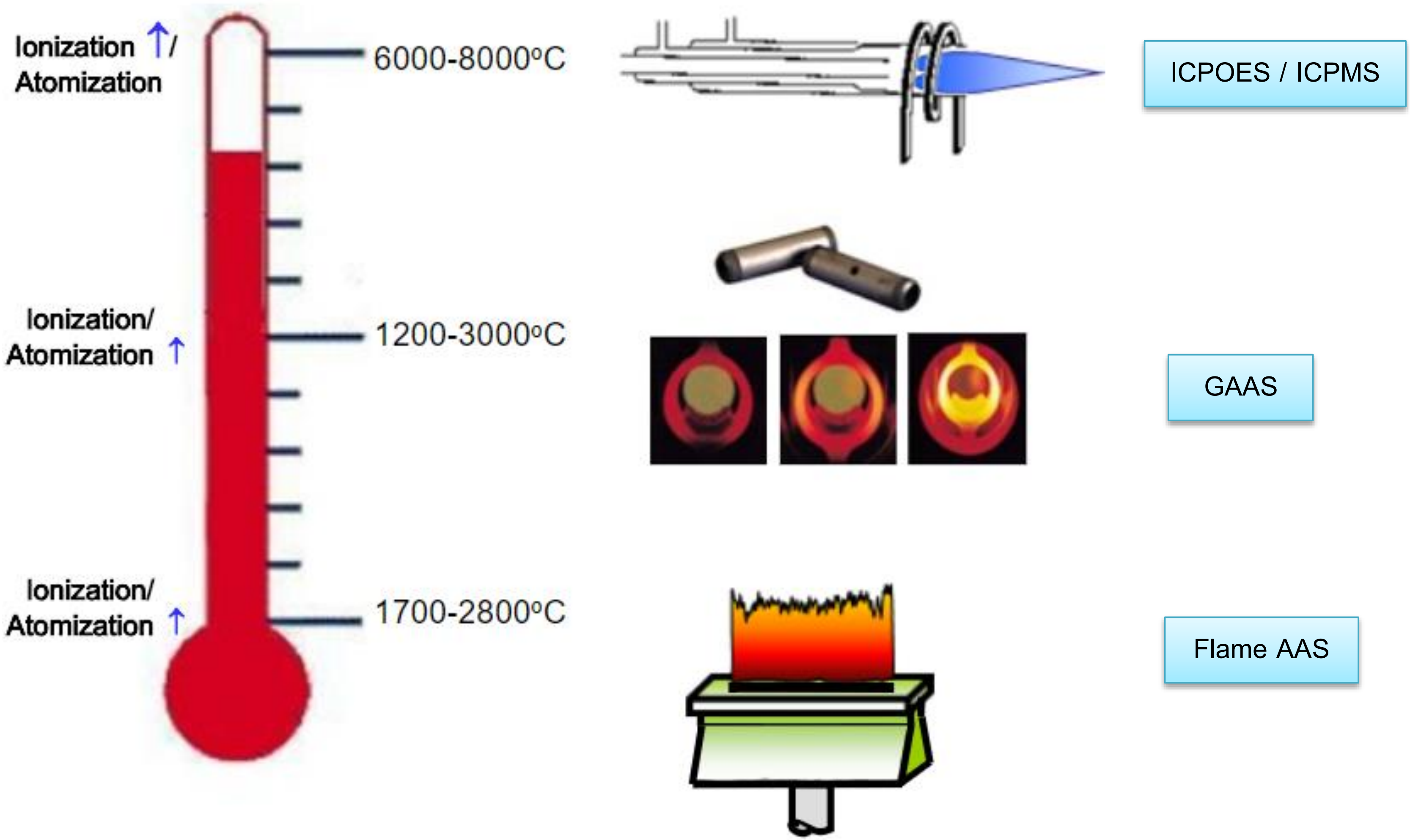
- Induction region (IR)
- Preheating zone (PHZ)
  - desolvation, vaporization, atomization
- Initial radiation zone (IRZ)
  - excitation and/or ionization
- Normal analytical zone (NAZ)
  - measurement of the emission
- Tail Plume
  - oxide formation, self absorption, molecular emission/absorption



Process occurring during Atomization and Ionization

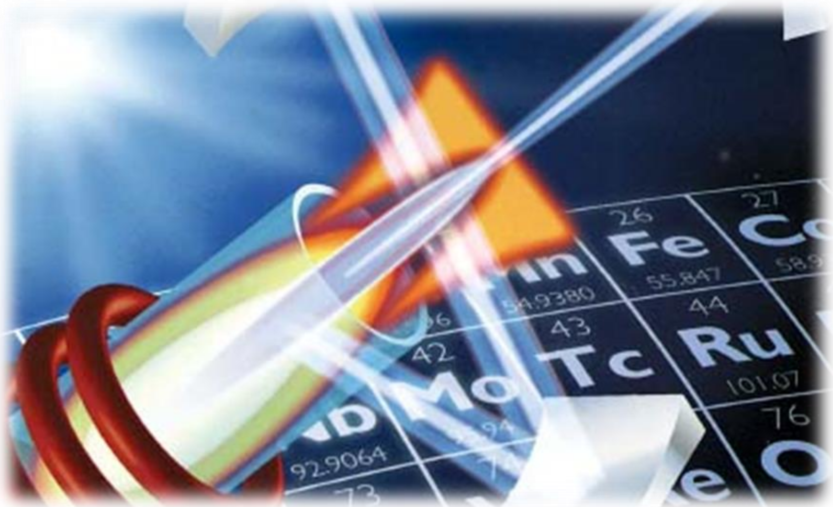


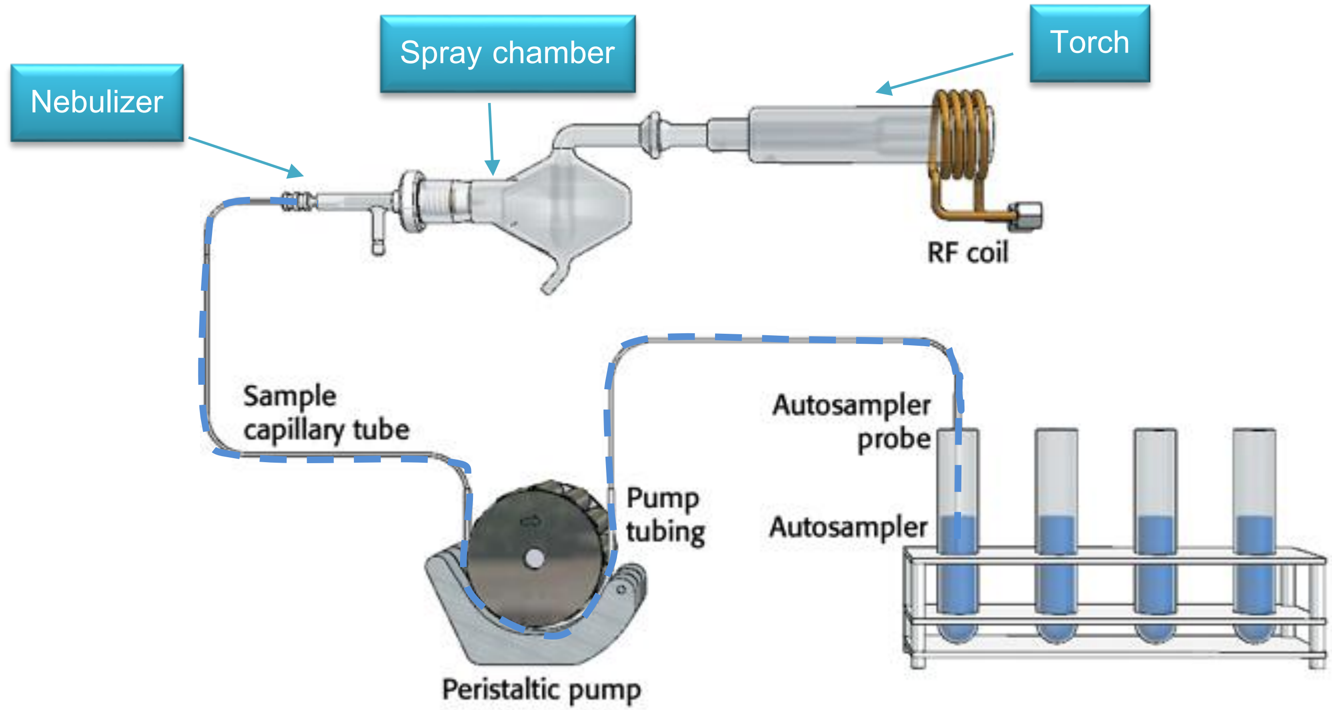
Temperature



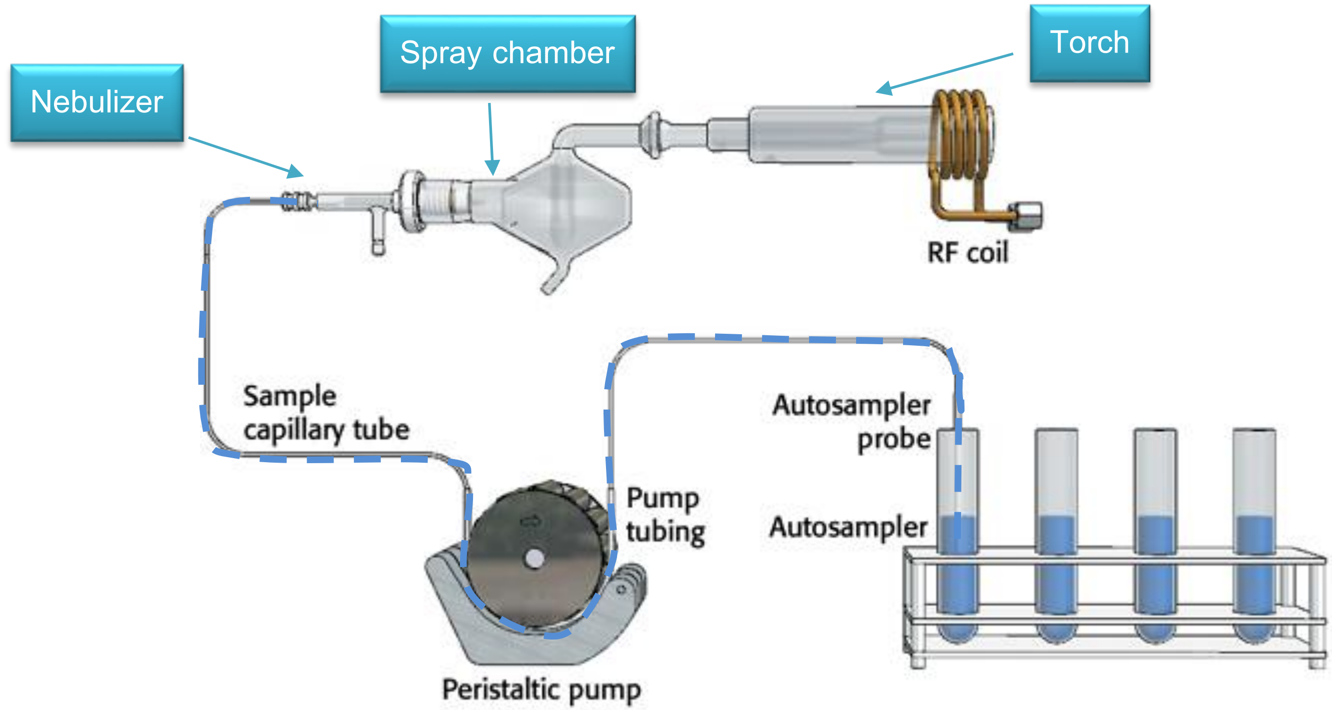
Consist of :

- Nebulizer
- Spray chamber
- Torch plasma and Viewing positions
- Monochromator
- Detector




Nebulizer
Spray chamber
Torch
RF coil
Sample
capillary tube
Autosampler
probe
Pump
tubing
Autosampler
Peristaltic pump

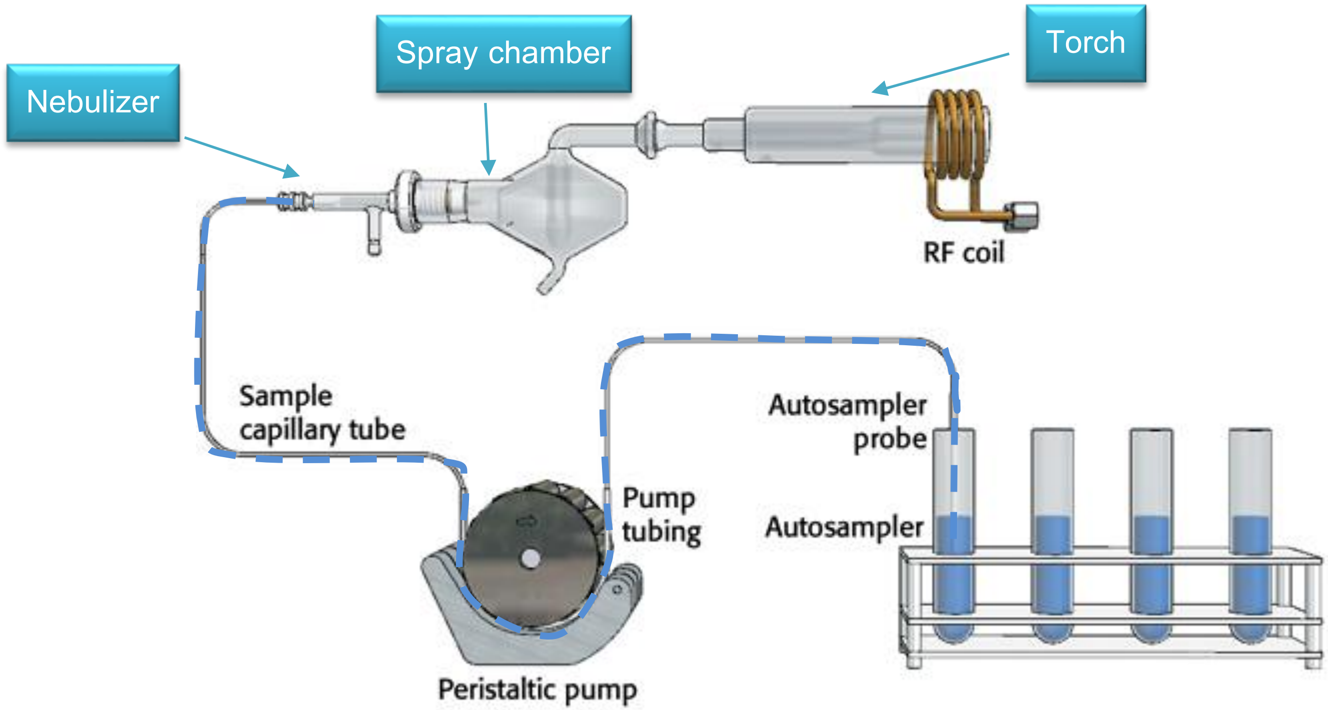
Sample Introduction - Nebulizer



Nebulizer are devices that convert a liquid into an aerosol that can be transported to the plasma.

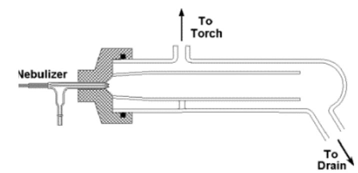
- made from glass or plastics such as PFA
- Flow rate of between 0.01 and 3 mL/min
- Concentric nebulizer for use with samples containing up to 3% TDS
- The glass construction should not be used with hydrofluoric acid or caustics such as the alkali hydroxides. Quartz construction is more resistant to chemical attack.

Sample Introduction – Spray chamber



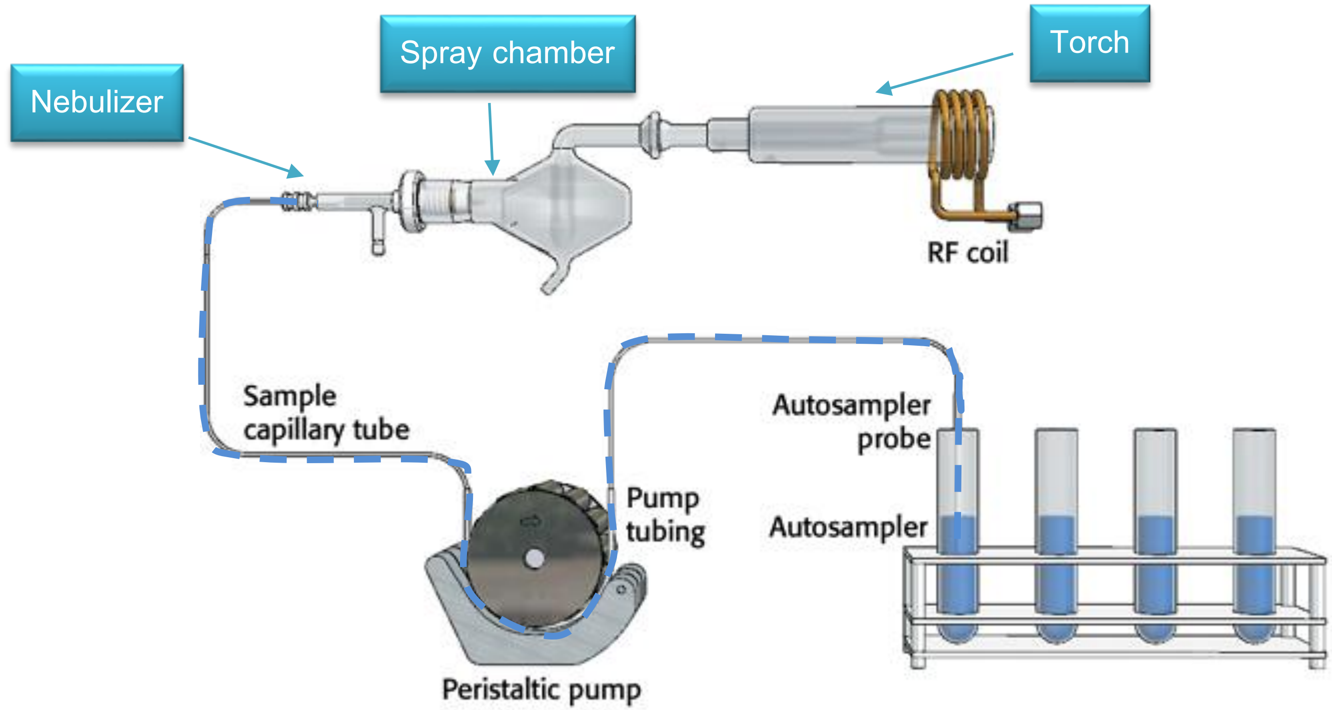
Cyclonic

Scott type



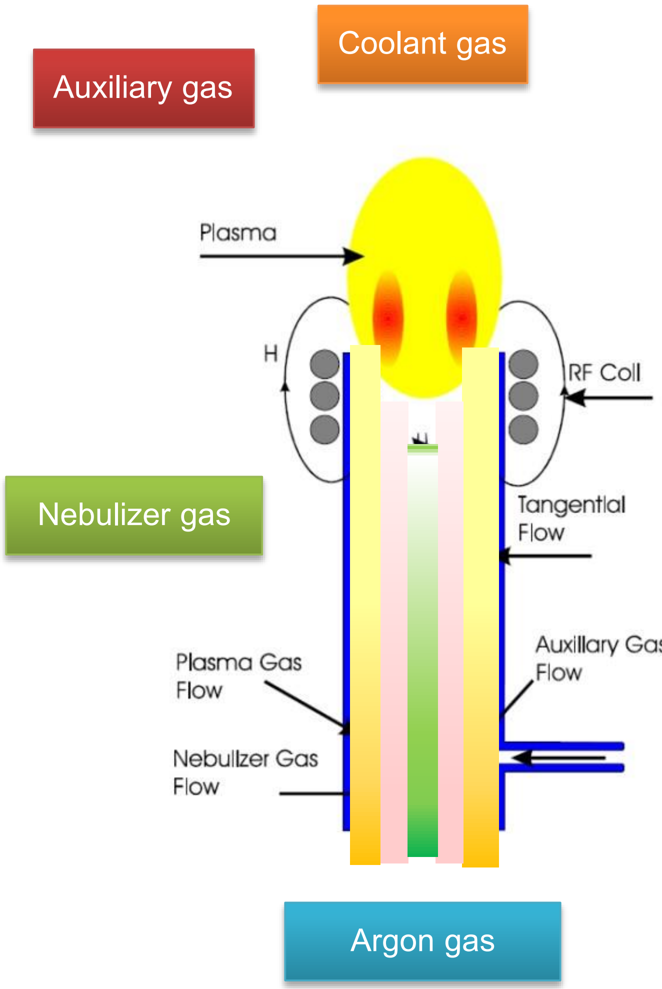
The purpose of the spray chamber is to remove droplets produced by the nebulizer that are >8 µm in diameter.

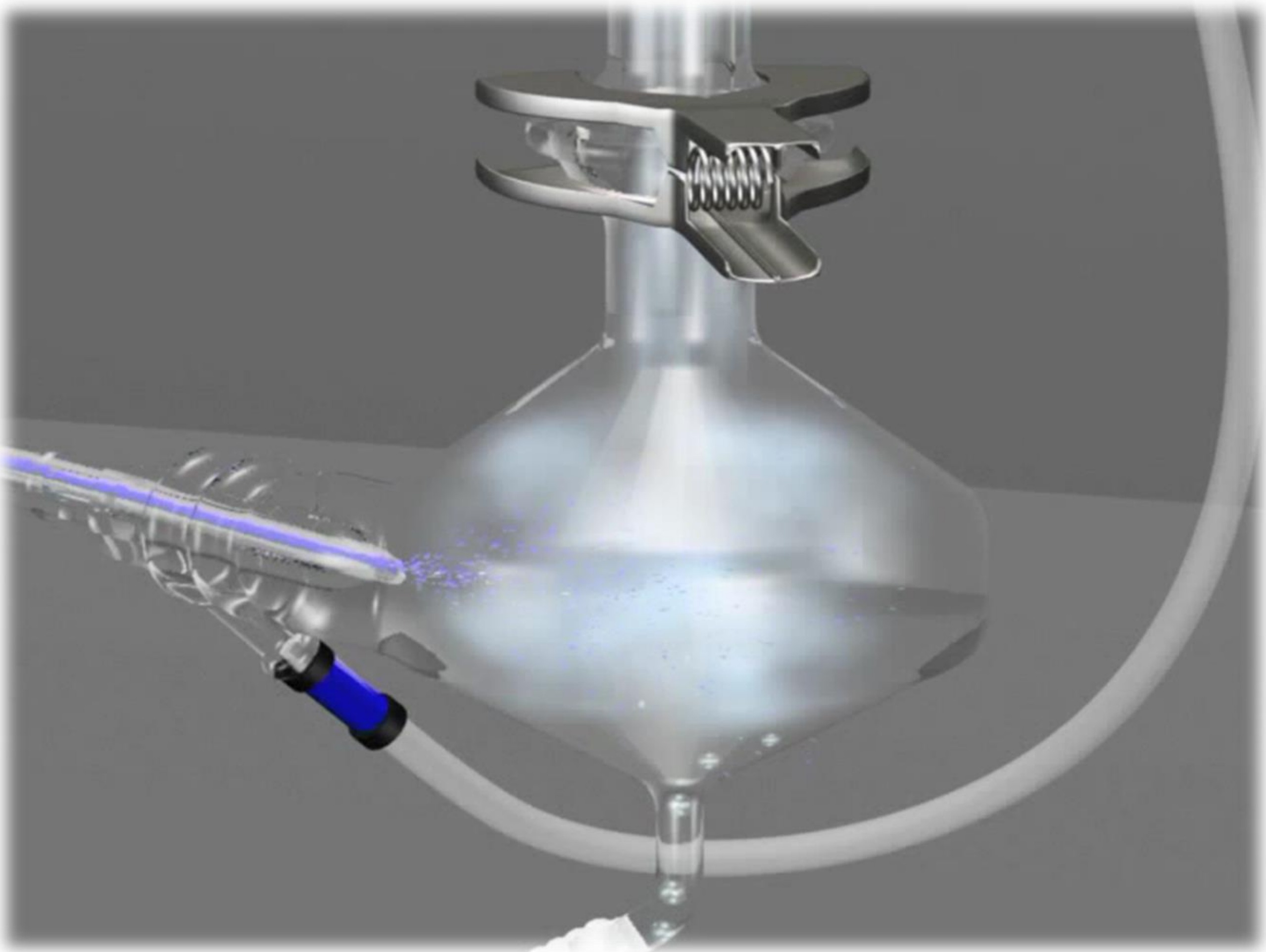
- Glass cyclonic spray chamber
- Important considerations here include the wash-in time, wash-out time, stability, and sensitivity
- Drainage process might be smooth and continuous.
- Analyst might observe faster wash-in and wash-out times with glass construction than with polymer due in part to better wettability of the glass (lack of beading).


Nebulizer
Spray chamber
Torch
RF coil
Sample
capillary tube
Autosampler
probe
Pump
tubing
Autosampler
Peristaltic pump

Sample Introduction - Torch

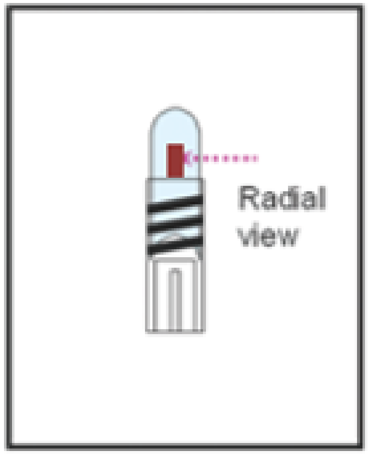
- made from Quartz
- Auto alignment of the torch in the torch box
- Ease of use for routine maintenance

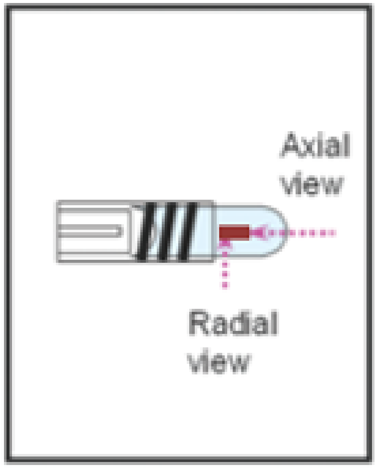


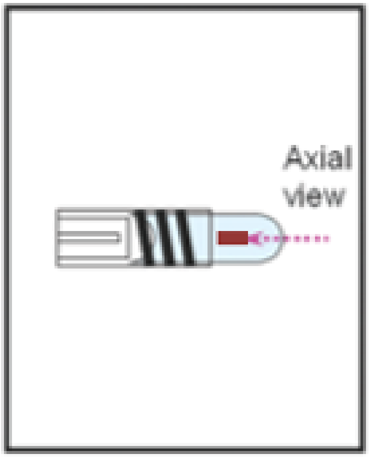


Sample Introduction

Viewing positions







Radial

Dual view (Duo)

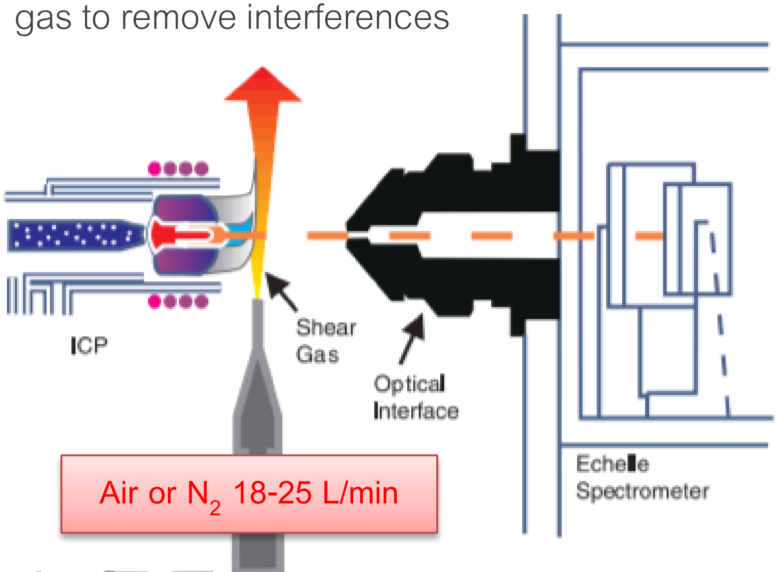
Axial

- Selectable viewing zone, Avoid matrix effects
- More suitable for hard matrices
- Alkali metal calibration is more linear
- No self absorption
- Lower sensitivity

- Longer path length,
- Higher analyte emission
- Improve sensitivity apox. 5 – 10 times
- Better detection limits
- Problems : Increase in spectral and matrix induced interference (Self-absorption effect, Reduced linear dynamic range)

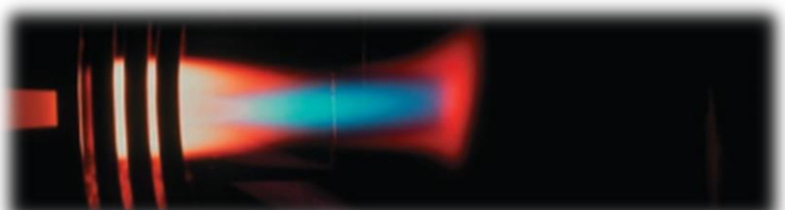
Interference : Axial viewing

Shear gas : require additional high flow rate shear gas to remove interferences

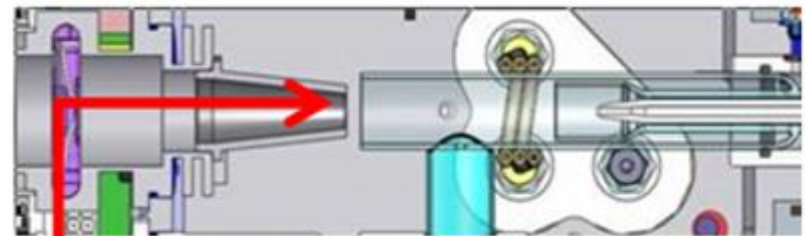


Air or N_2 18-25 L/min

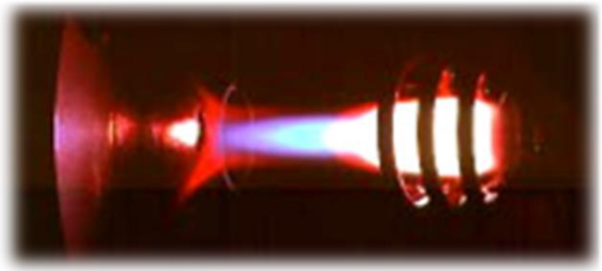
http://www.homedepot.com/



Cone interface

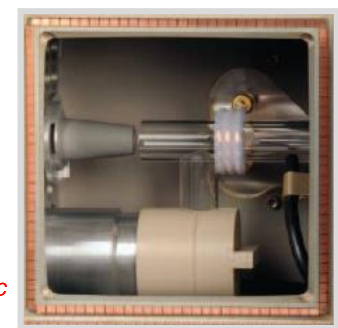


Ar or N_2 1 L/min



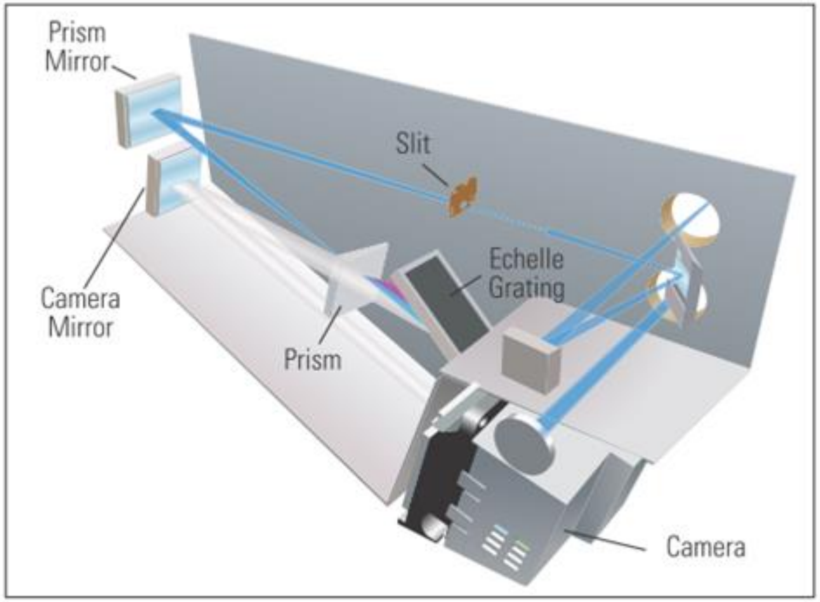
Ceramic cone

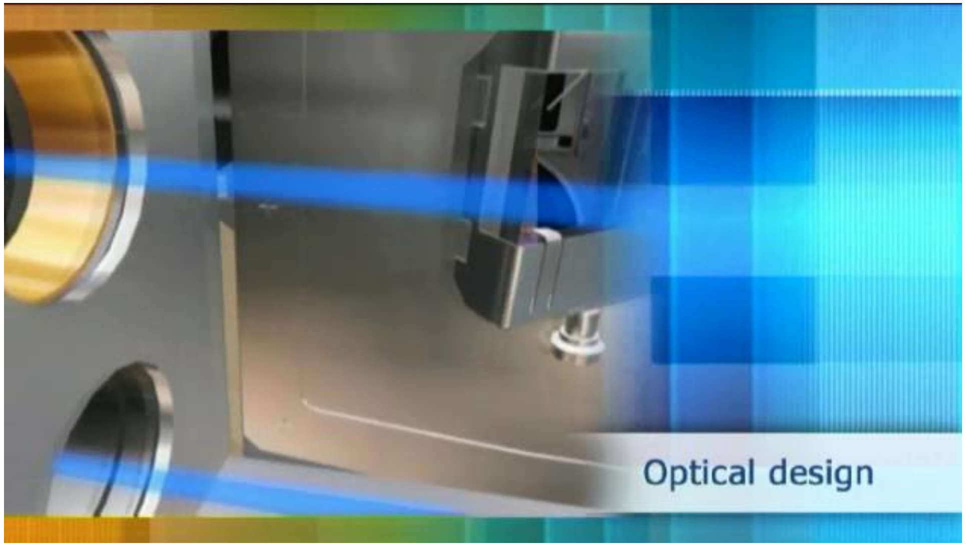
iCAP7000 plus Thermo Scientific

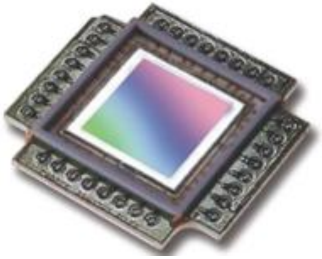


Monochromator and Detector

- Simultaneous analysis was carried out by using
- Echelle grating optics and coupled to solid state detector also know "Charge transfer device"



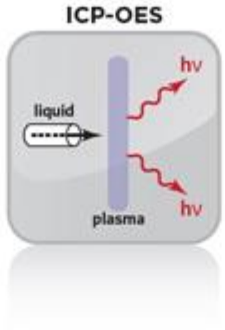


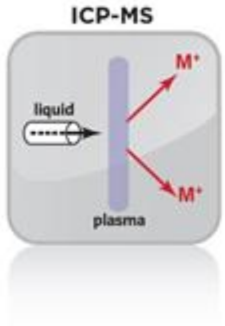


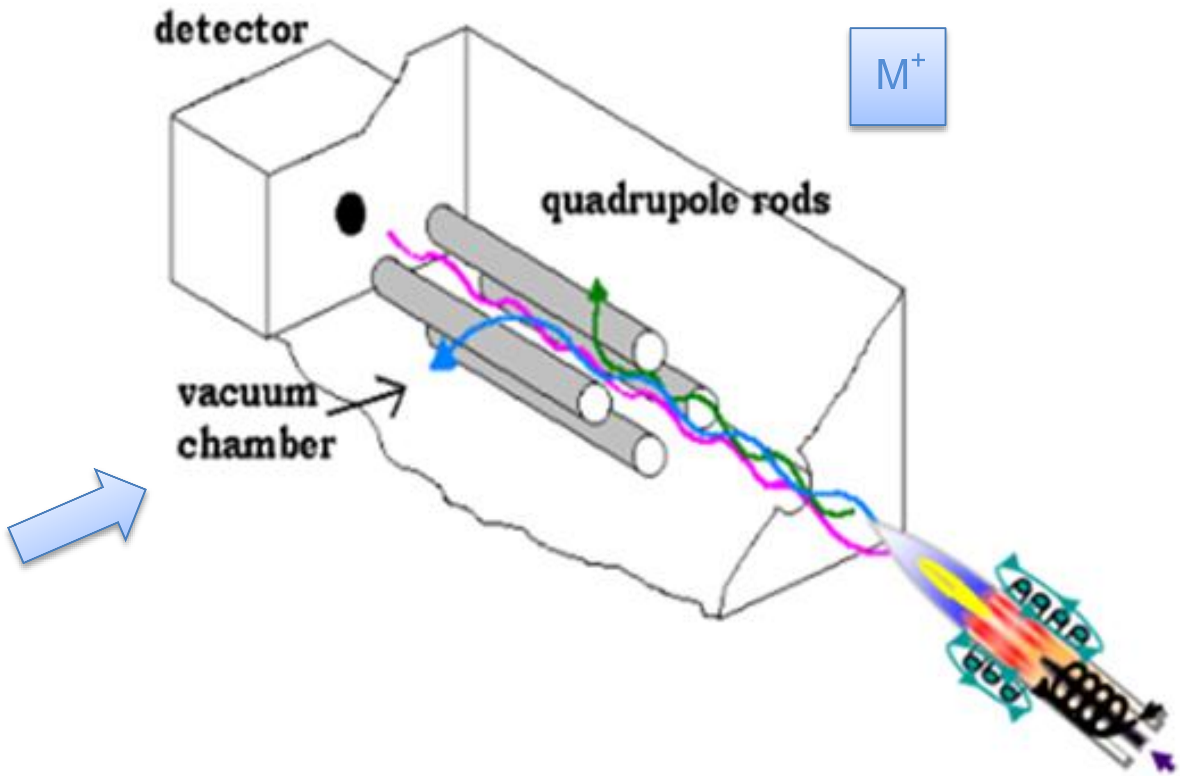
Charge Injection Devices

Inductively Coupled Plasma Mass spectrometry (ICP-MS)

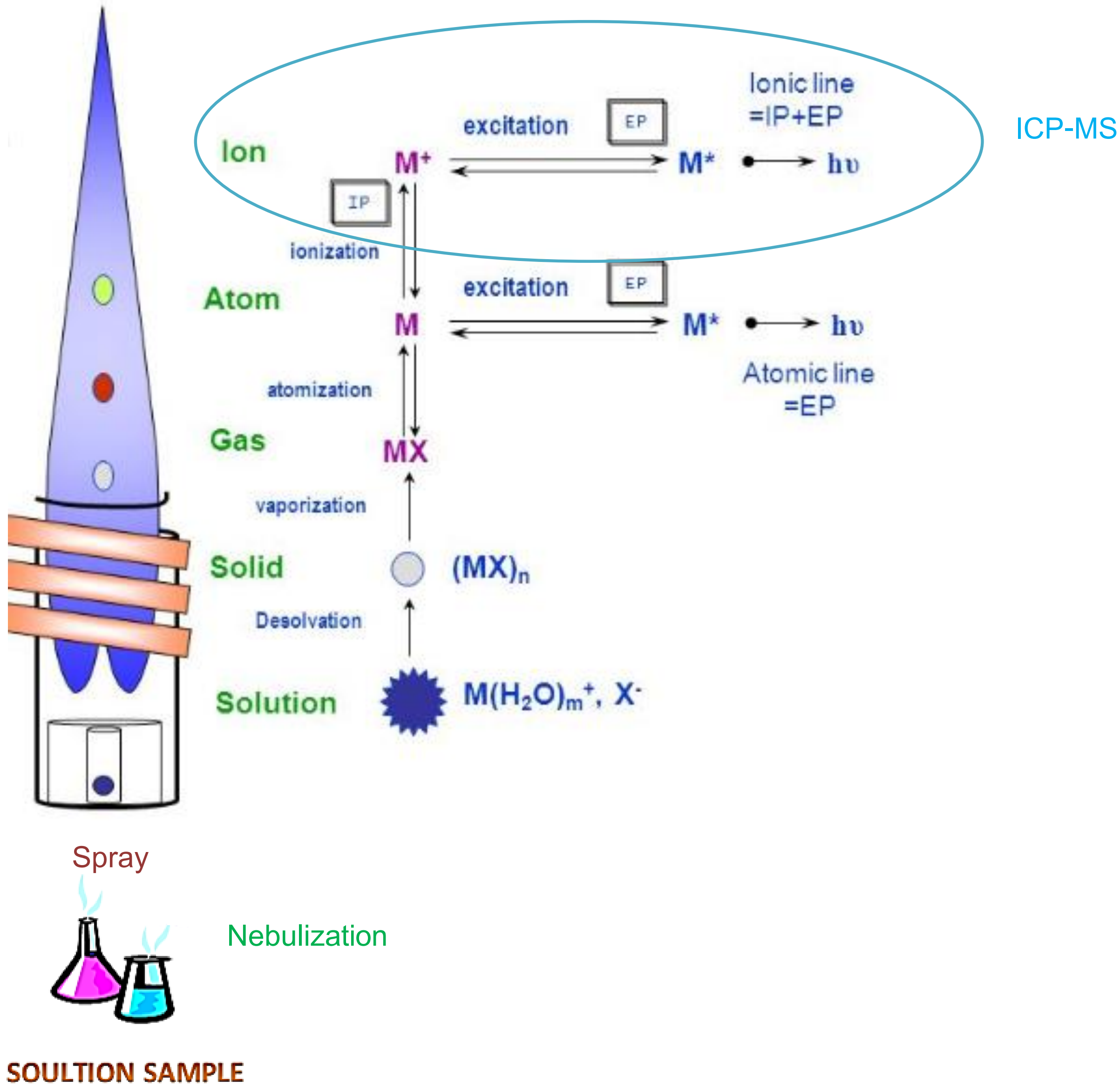
Inductively coupled plasma mass spectrometry (ICP-MS) is a type of mass spectrometry which is capable of detecting metals and several non-metals at concentrations as low as one part in 10^{12} (ppt). This is achieved by ionizing the sample with inductively coupled plasma (isotope ions) and then using a mass spectrometer to separate and quantify those ions.



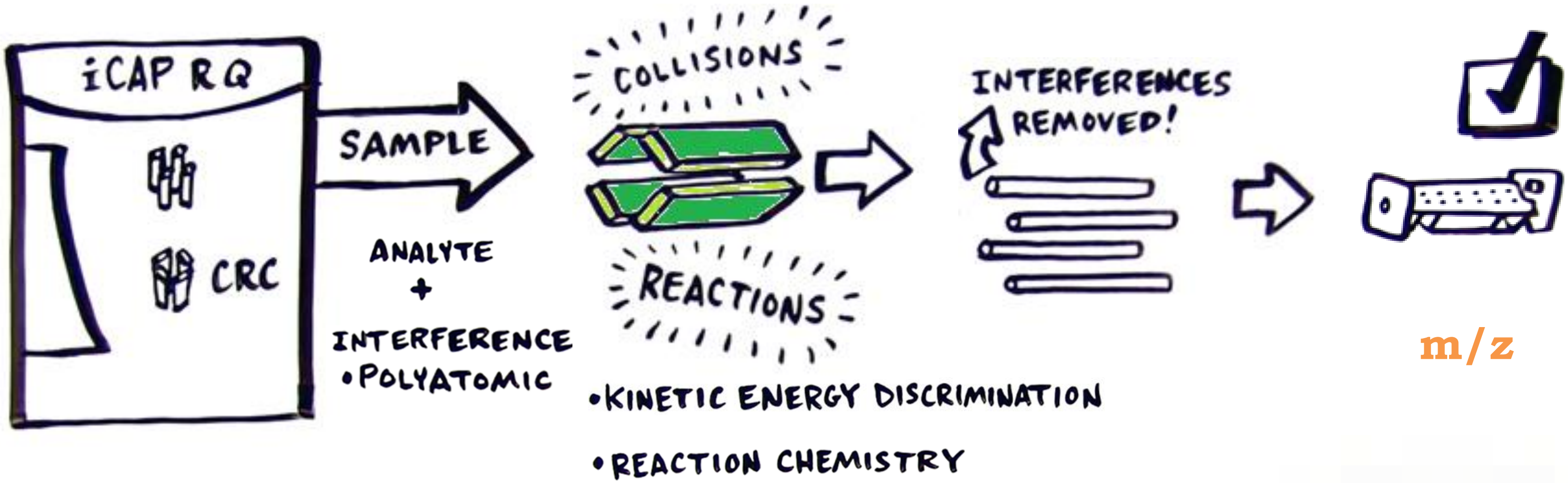


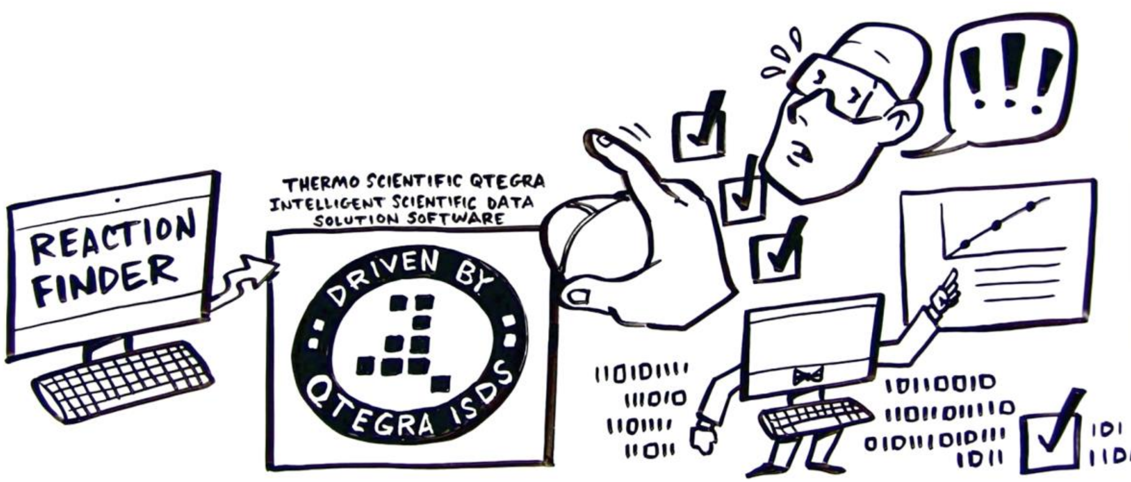


Process occurring during Atomization and Ionization



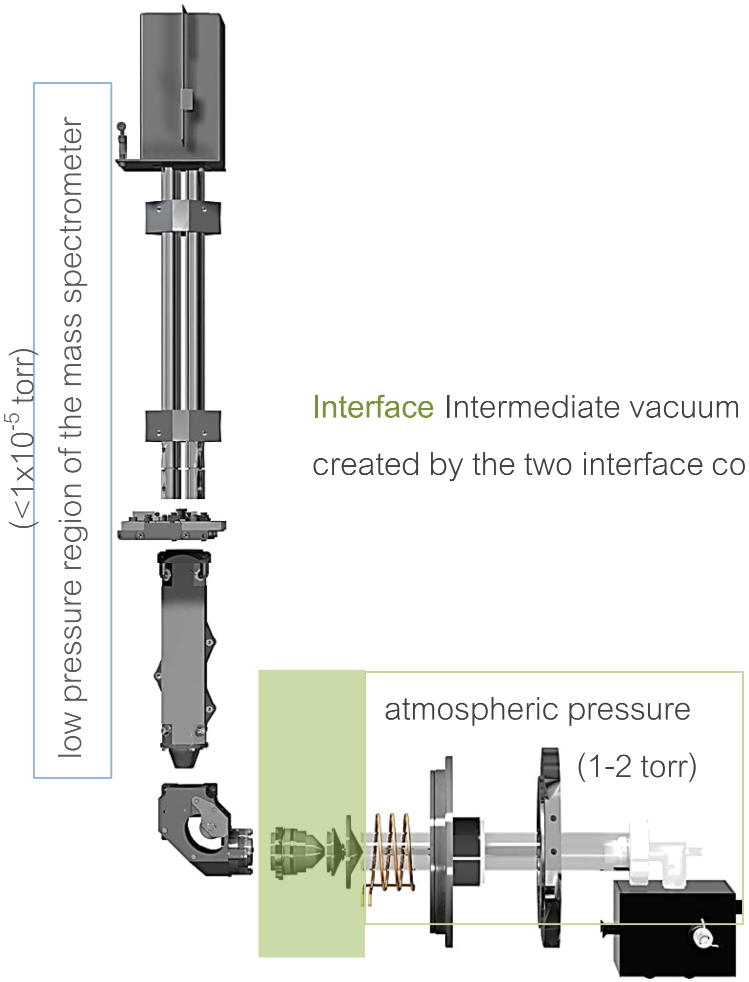
Inductively Coupled Plasma Mass Spectrometry (ICP-MS)



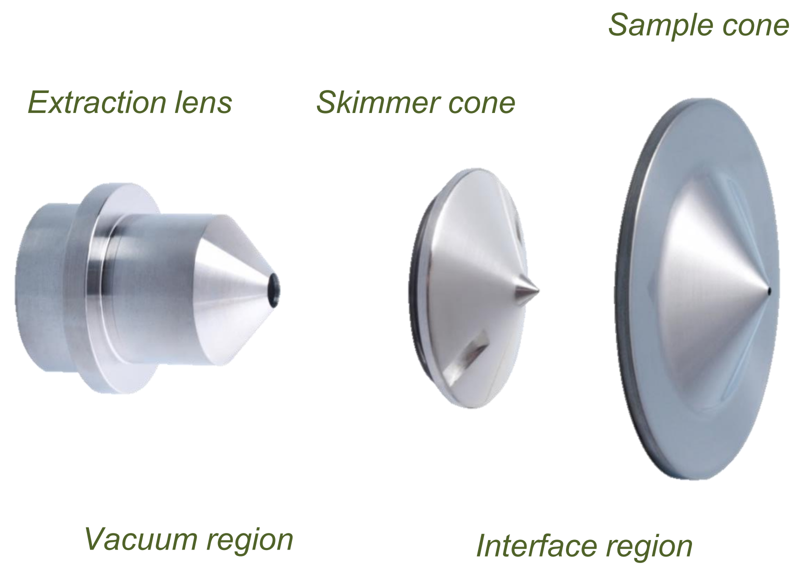


How to extract the ions into MS ?

The positively charged ions that are produced in the plasma are extracted into the vacuum system, via a pair of interface “cones” and the “extraction lens”



Interface Intermediate vacuum region created by the two interface cones.



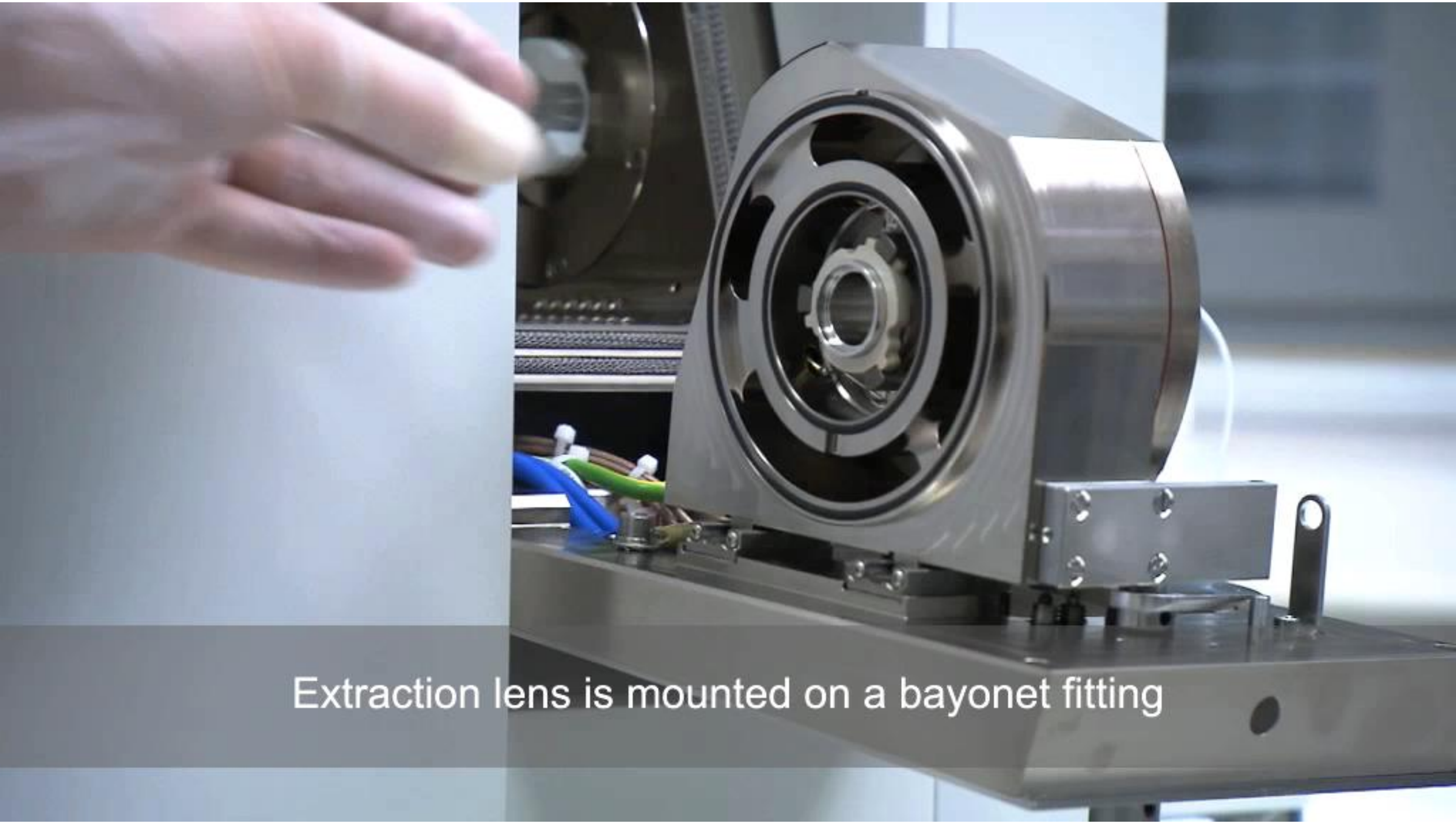
https://www.youtube.com/watch?v=mPDxFpWkceA

Simplified torch and injector maintenance

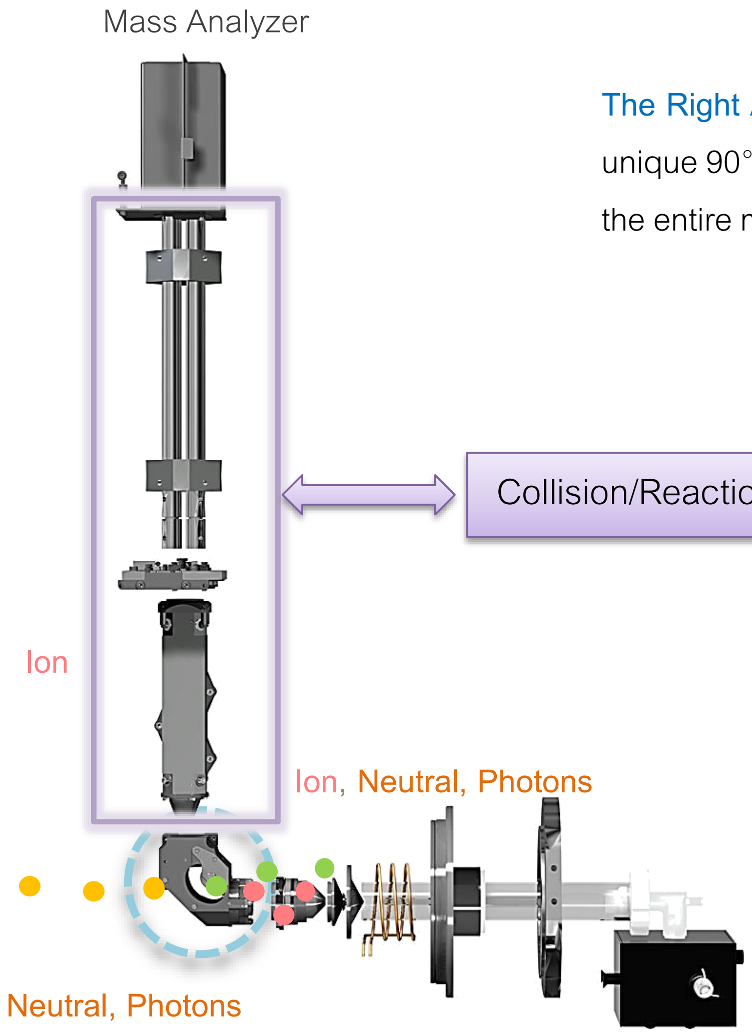
https://www.youtube.com/watch?v=4xM_hNrOZOU

Rapid replacement of sample and skimmer cones

https://www.youtube.com/watch?v=r_6SZCb8f9A



How to maintain low background and drift ?



Collision/Reaction cell and Mass Filter (Quadrupole)

The Right Angular Positive Ion Deflection (RAPID) lens

unique 90° cylindrical ion lens providing high ion transmission across the entire mass range.

Elimination of neutral species

Polyatomic interference

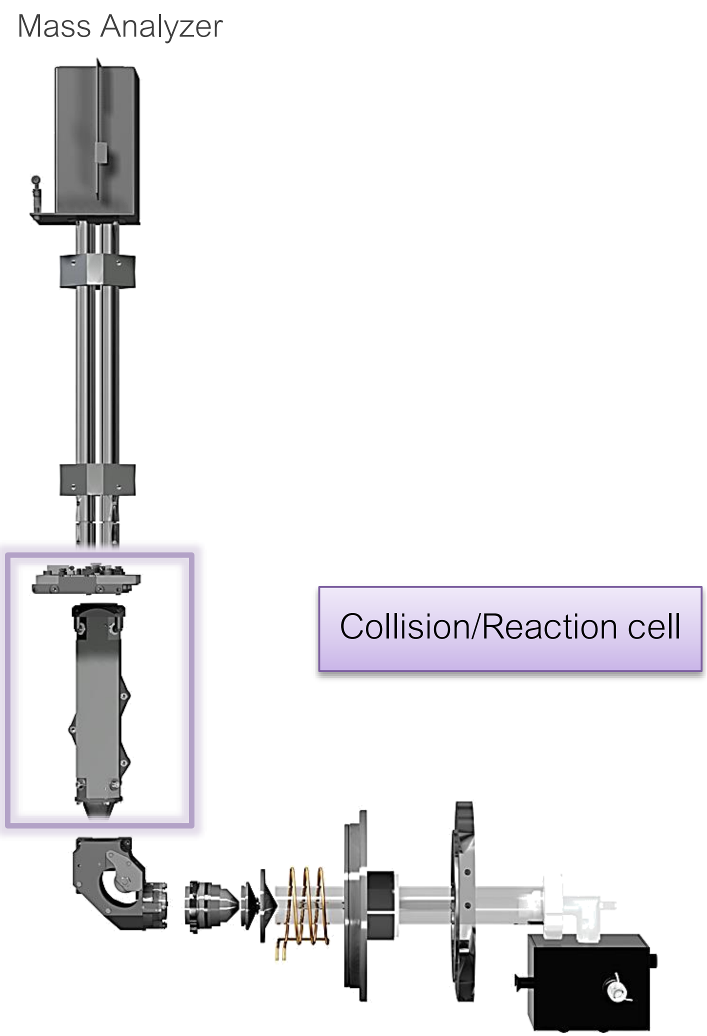
$^{40}Ar^{16}O$

$^{40}Ca^{16}O$

$^{40}Ar^{35}Cl$

ANALYTE	POTENTIAL INTERFERENT	PRECURSORS
^{45}Sc	$^{13}C^{16}O_2$, $^{12}C^{16}O_2H$, ^{44}CaH, $^{32}S^{12}CH$, $^{32}S^{13}C$, $^{33}S^{12}C$	H, C, O,S, Ca
^{47}Ti	$^{31}P^{16}O$, ^{46}CaH, $^{35}Cl^{12}C$, $^{32}S^{14}NH$, $^{33}S^{14}N$	H, C, N, O, P, S, Cl, Ca
^{49}Ti	$^{31}P^{18}O$, ^{48}CaH, $^{35}Cl^{14}N$, $^{37}Cl^{12}C$, $^{32}S^{16}OH$, $^{33}S^{16}O$	H, C, N, O, P, S, Cl, Ca
^{50}Ti	$^{34}S^{16}O$, $^{32}S^{18}O$, $^{35}Cl^{14}NH$, $^{37}Cl^{12}CH$	H, C, N, O, S, Cl
^{51}V	$^{35}Cl^{16}O$, $^{37}Cl^{14}N$, $^{34}S^{16}OH$	H, O, N, S, Cl
^{52}Cr	$^{36}Ar^{16}O$, $^{40}Ar^{12}C$, $^{35}Cl^{16}OH$, $^{37}Cl^{14}NH$, $^{34}S^{18}O$	H, C, O, N, S, Cl, Ar
^{55}Mn	$^{37}Cl^{18}O$, $^{23}Na^{32}S$, $^{23}Na^{31}PH$	H, O, Na, P, S, Cl, Ar
^{56}Fe	$^{40}Ar^{16}O$, $^{40}Ca^{16}O$	O, Ar, Ca
^{57}Fe	$^{40}Ar^{16}OH$, $^{40}Ca^{16}OH$	H, O, Ar, Ca
^{58}Ni	$^{40}Ar^{18}O$, $^{40}Ca^{18}O$, $^{23}Na^{35}Cl$	O, Na, Cl, Ar, Ca
^{59}Co	$^{40}Ar^{18}OH$, $^{43}Ca^{16}O$, $^{23}Na^{35}ClH$	H, O, Na, Cl, Ar, Ca
^{60}Ni	$^{44}Ca^{16}O$, $^{23}Na^{37}Cl$	O, Na, Cl, Ca
^{61}Ni	$^{44}Ca^{16}OH$, $^{38}Ar^{23}Na$, $^{23}Na^{37}ClH$	H, O, Na, Cl, Ca
^{63}Cu	$^{40}Ar^{23}Na$, $^{12}C^{16}O^{35}Cl$, $^{12}C^{14}N^{37}Cl$, $^{31}P^{32}S$, $^{31}P^{16}O_2$	C, N, O, Na, P, S, Cl
^{64}Zn	$^{32}S^{16}O2$, $^{32}S_2$, $^{36}Ar^{12}C^{16}O$, $^{38}Ar^{12}C^{14}N$, $^{48}Ca^{16}O$	C, N, O, S, Ar, Ca
^{65}Cu	$^{32}S^{16}O2H$, $^{32}S_2H$, $^{14}N^{16}O^{35}Cl$, $^{48}Ca^{16}OH$	H, N, O, S, Cl, Ca
^{66}Zn	$^{34}S^{16}O$, $^{32}S^{34}S$, ^{33}S, ^{48}C, ^{18}O	O, C, S
^{69}Ga	$^{32}S^{18}O_2H$, $^{34}S_2H$, $^{37}Cl^{16}O_2$	H, O, S, Cl
^{70}Zn	$^{34}S^{18}O_2$, $^{35}Cl_2$	O, S, Cl
^{75}As	$^{40}Ar^{34}SH$, $^{40}Ar^{35}Cl$, ^{40}Ca ^{35}Cl, $^{37}Cl_2H$	H, S, Cl, Ca, Ae
77Se	^{40}Ar ^{37}Cl, ^{40}Ca ^{37}Cl	Cl, Ca, Ar
78Se	$^{40}Ar^{38}Ar$	Ar
80Se	$^{40}Ar_2$, $^{40}Ca_2$, $^{40}Ar^{40}Ca$, $^{32}S_2{}^{16}O$, $^{32}S^{16}O_3$	O, S, Ar, Ca

How to remove Polyatomic Interference?

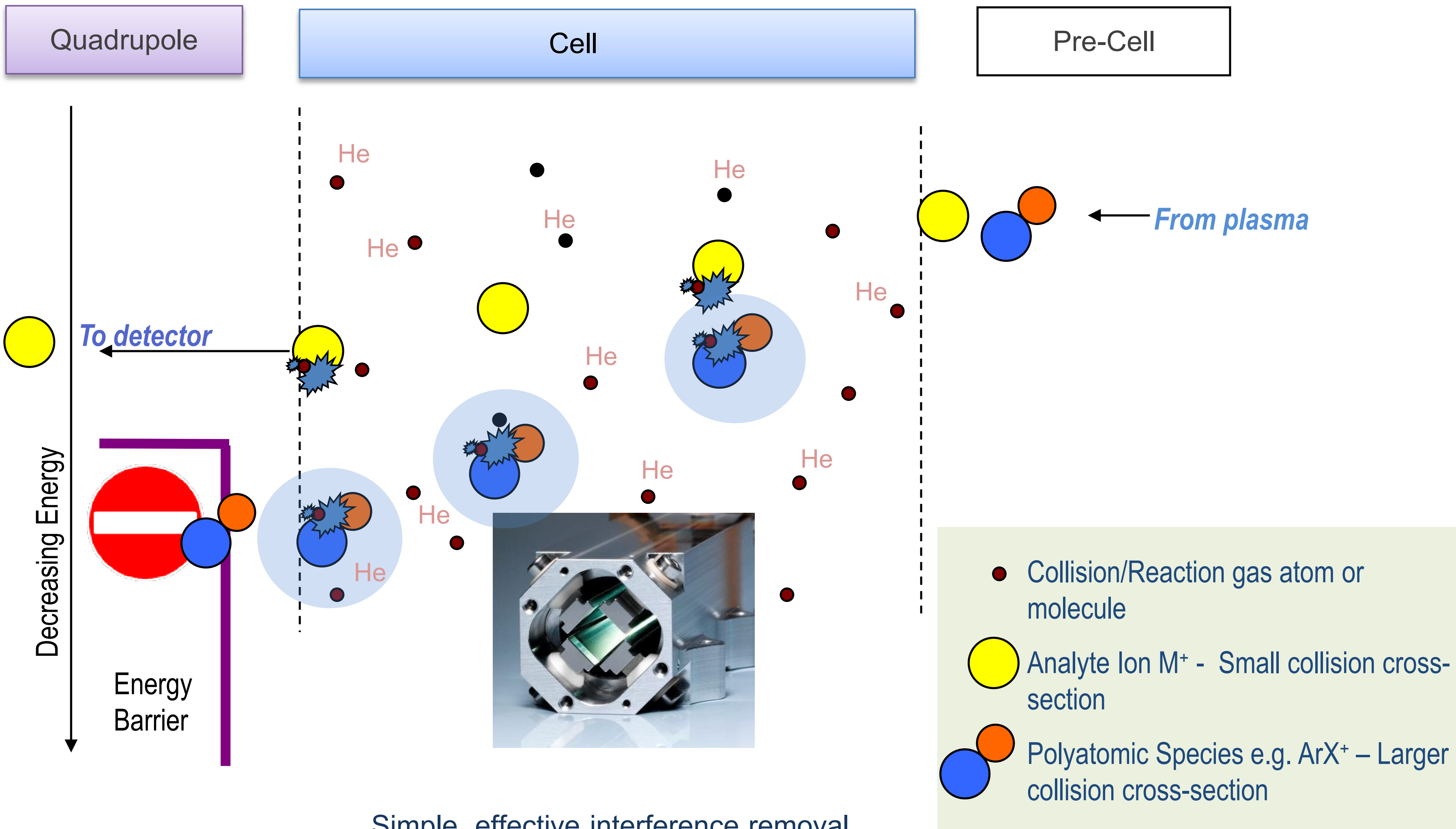


Qcell Collision/Reaction Cell (CRC)

- Flatapole technology for improved transmission
- Low mass cut off filters out unwanted precursor ions
- Single mode interference removal with He
- He KED filters out unwanted polyatomic interferences
- Small CRC volume for fast gas exchange, <10s
- Flexibility to work with reactive gases, such as mixtures of O_2 7% dilute H_2 or 1% NH_3
- Non-consumable, zero-maintenance

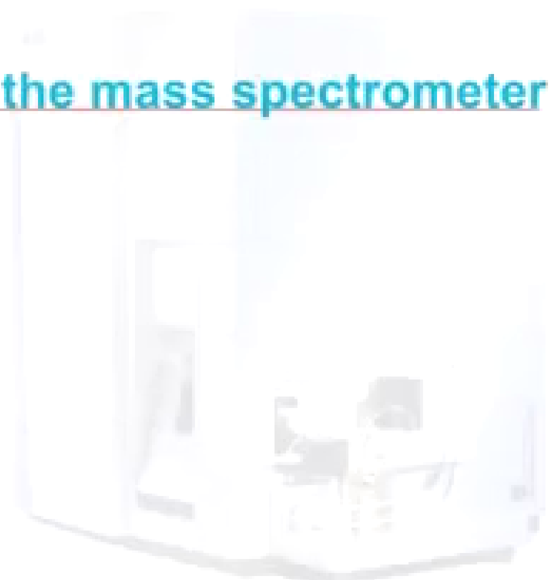
How to remove Polyatomic Interference? He KED mode

Collisional retardation / energy filtering



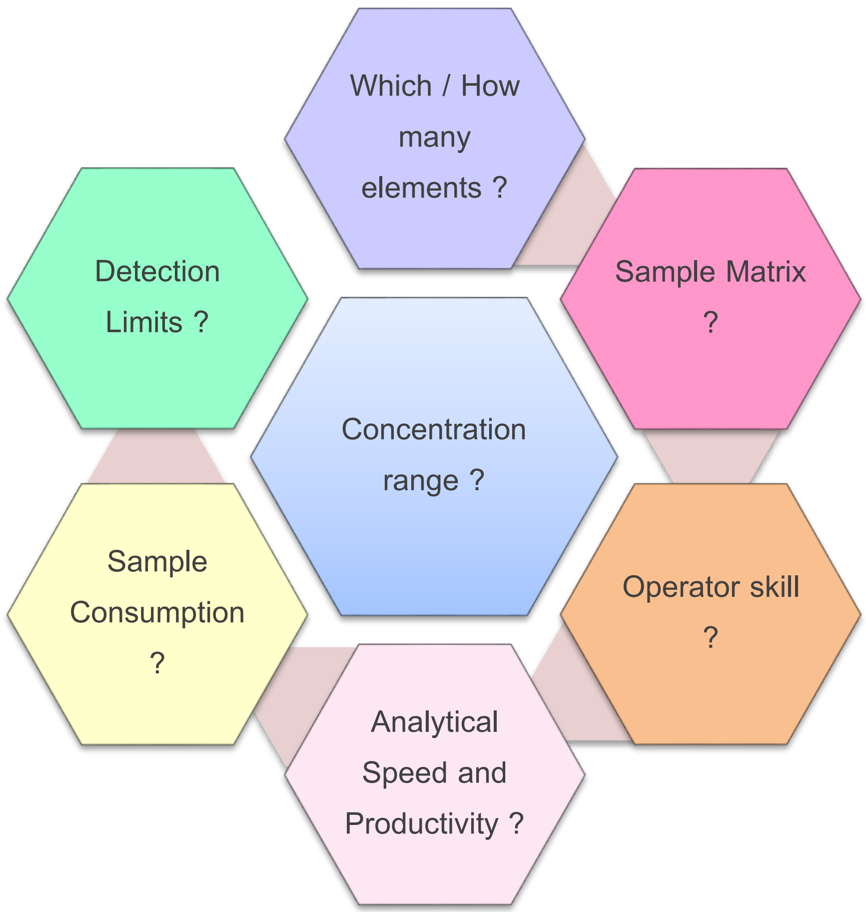
Simple, effective interference removal

Flight of ions through the mass spectrometer



Selection Criteria

- Typical selection criteria includes:



A cross-technique comparison

Flame AA	GFAAS	ICP-OES	ICP-MS
• Easy to use	• Very good detection limits	• Easy to use	• Excellent detection limits
• Very fast	• Small sample size	• Multi-element	• Multi-element
Lowest capital cost	• Moderate price	• High productivity	• High productivity
• Very compact instrument	• Very compact instrument	• Very economical for many samples and/or elements	• Very economical for many samples and/or elements
• Good performance		• Robust interface	• Wide dynamic range
• Robust interface		• Excellent screening abilities	• Fast semi-quantitative screening
• Very compact instrument		• High total dissolved solids	• Hybrid techniques LA-ICP-MS (solids)*, IC or LC-ICP-MS (speciation)*
			• Excellent detection limits

Sample Requirements Criteria

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

Precision

"Precision" is a measure of the confidence you can have in your measured results

Technique	Precision
Flame AAS	Short term : 0.1-1.0% Long term : 1-2% (2beam optic)
GFAAS	Short term 0.5-5% Long term : highly dependent on the tube type and condition
ICP-OES	Short term : 0.1-2% Long term : <1-5%
ICP-MS	Short term : 0.5-2% Long term : <4%

- Long-term precision in any of the techniques can be improved by more frequent instrument calibration or drift correction techniques.
- The use of internal standardization can significantly improve precision in ICP and ICPMS

Speed of Measurement

- How many samples can a particular technique analyze in a given time?
- How many elements can be determined?

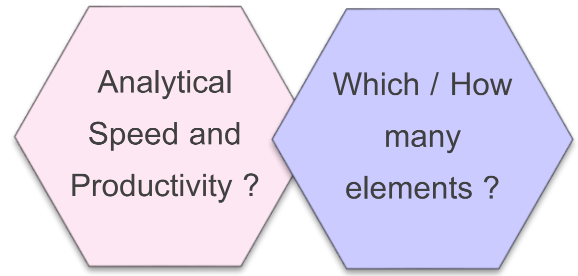
Sequential

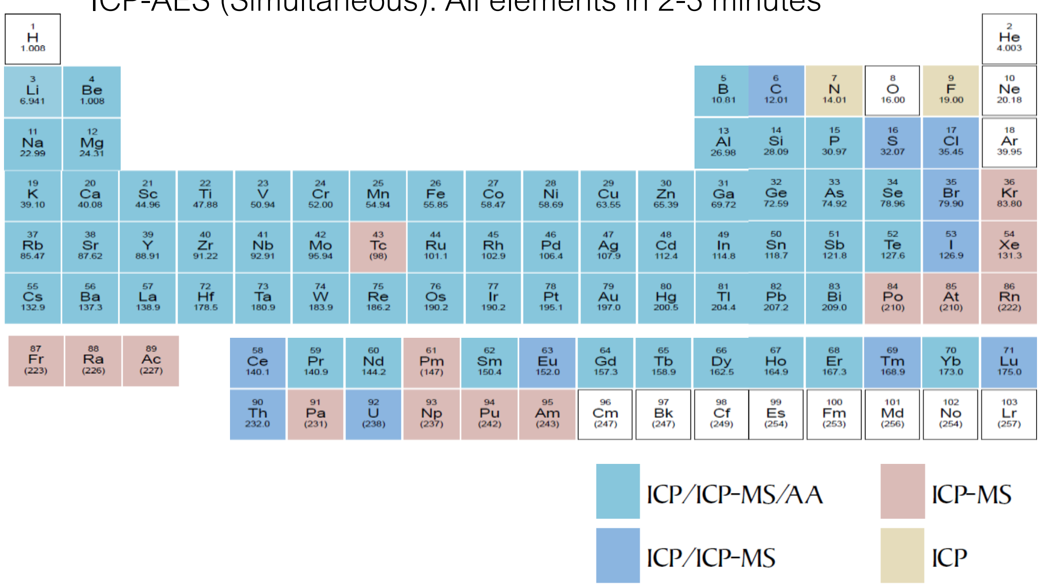
- ICP-AES (Sequential): 5-6 elements per minute for each sample
- FAAS: 4 seconds per element for each sample
- GFAAS: 2-3 minutes per element for each sample

Simultaneous

ICP-MS: All elements in 2-3 minutes

ICP-AES (Simultaneous): All elements in 2-3 minutes

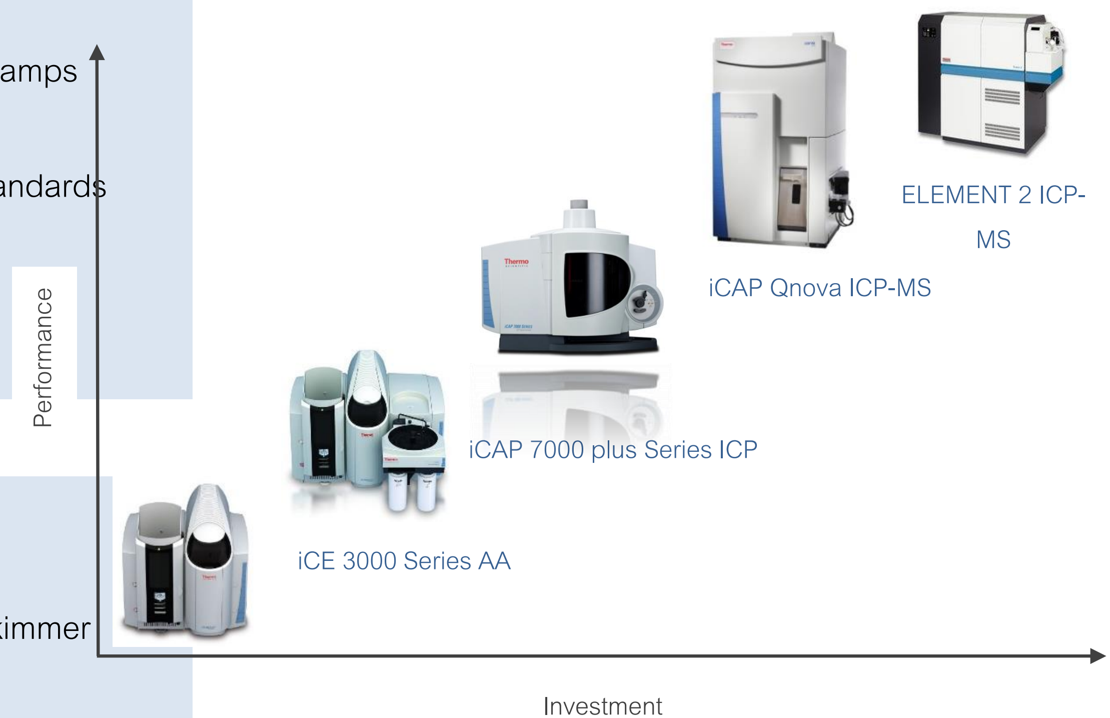




- For less than 5 elements per sample, FAAS is often the quickest technique, depending on the total number of samples.
- For 5-15 elements, sequential ICP-AES is the optimum choice.
- Above 15 elements, either ICP-MS or simultaneous ICP-OES is the best choice.
- GFAAS will always be the slowest of the techniques

Operating cost

FAAS	GFAAS
• acetylene/nitrous oxide gases • compressed air source • hollow cathode lamps • reagents and standards • power	• argon gas • hollow cathode lamps • graphite tubes • reagents and standards • power • cooling water
ICP-OES	**ICP-MS**
• argon gas • quartz torches • reagents and standards • pump tubing • power • cooling water	• argon gas • quartz torches • sampling and skimmer cones • reagents and standards • pump tubing • power • cooling water



Overall Technique Selection Summary

- For ultimate throughput, choose ICP-OES and ICP-MS
- If ultimate DLs, choose ICP-MS
- For strictly regulated industries/applications such as USEPA, please check that data is accepted

Application Fields

Which Instrument would you recommend for analysis of Cadmium in Chocolate ?

Cadmium is a heavy metal used in a variety of applications, such as steel plating, as a pigment in plastics and glasses, and in the production of batteries. These industrial activities are the main route through which cadmium is released into the environment where it accumulates in water and soil, and subsequently plants, animals and fish through uptake and ingestion. One of the main routes of human exposure to cadmium is therefore through the ingestion of foodstuffs.

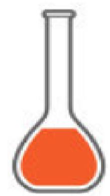
- Typical maximum levels of cadmium in foodstuffs are currently between *0.05 – 0.2 mg/kg* wet weight.
- The main ingredients in chocolate consist of cocoa, milk and fats, each of which is a potential source of cadmium.

Sample Preparation

0.3 g

+ 7 mL HNO_3 + 1 mL H_2O_2

Left to stand for 5 mins



diluted to 100 ml with DI water

1 mg/l cadmium sub-standard was prepared in deionised water for spiking of samples prior to digestion

Analysis of Cadmium in Chocolate by GFAAS

- 10 µg/l sub-standard was made up in 7% nitric acid and 1% hydrogen peroxide to matrix match to the digested samples.
- Blank and diluent were also prepared at 7% nitric acid and 1% hydrogen peroxide.
- A matrix modifier : 2 g/l of ammonium nitrate
- Cadmium was analyzed at 228.8 nm and Zeeman background correction

Furnace Method

Phase	Temperature / °C	Time / s	Ramp / °C/s
Dry	110	30	10
Ash	400	20	150
Atomize	1300	3	0
Clean	2500	3	0

Thermo Scientific iCE3500 AAS

Results for the analysis of cadmium in chocolate following analysis by GFAAS

Sample	Measured Concentration µg/l	Concentration in original sample mg/kg	Calculated Recovery Spiked / %
USA Origin, Milk	0.030	0.010	
USA Origin, Milk, Spiked	5.095		101
UK Origin, Milk	0.038	0.012	
UK Origin, Milk, Spiked	5.182		103
USA Origin, Dark	0.124	0.042	
USA Origin, Dark, Spiked	4.761		93

Analysis of toxic elements in drinking and bottled waters

China and India have seen a huge increase in the consumption of bottled water in the last decades

Chinese regulations:

GB 8537–2008 - Drinking natural mineral water

GB 17324–2003 - Hygienic standard of bottled purified water for drinking

GB 5749–2006 - Standards for drinking water quality

GB 3838–2002 - Environmental quality standard for surface water

Indian regulations:

IS 10500:2012 - Drinking Water

IS 13428:2005 - Packaged natural mineral water

IS 14543:2004 - Packaged drinking water (other than packaged natural mineral

Table 1. Maximum permissible levels in $mg \cdot kg^{-1}$.

Element	GB 8537-2008	GB 17324-2003	GB 5749-2006	GB 3838-2002 (I)[1]	IS 10500:2012	IS 13428:2005	IS 14543:2004
Arsenic	0.01	0.01	0.01	0.05	0.01	0.05	0.05
Cadmium	0.003	-	0.005	0.001	0.003	0.003	0.01
Chromium*	0.05	-	0.05	0.01	0.05	0.05	0.05
Copper	1	0.01	1	0.01	0.05	1	0.05
Iron	-	-	0.3	0.3	0.3	-	0.1
Lead	0.01	0.01	0.01	0.01	0.01	0.01	0.01
Mercury	0.001	-	0.001	0.00005	0.001	0.001	0.001
Nickel	0.02	-	0.02	0.02	0.02	0.02	0.02
Zinc	0.2	-	1	0.05	5	5	5

มาตรฐานคุณภาพน้ำเพื่อบริโภค

มาตรฐานผลิตภัณฑ์อุตสาหกรรมน้ำบริโภค				
คุณลักษณะ	ดัชนีคุณภาพน้ำ	หน่วย	มาตรฐาน	
			เกณฑ์กำหนดสูงสุด (Maximum Acceptable Concentration)	เกณฑ์อนุโลมสูงสุด[a] (Maximum Allowable Concentration)
ทางกายภาพ	1.สี (Colour)	ปลาตินัม-โคบอลต์ (Platinum-Cobalt)	5	15
	2.รส (Taste)	-	ไม่เป็นที่รังเกียจ	ไม่เป็นที่รังเกียจ
	3.กลิ่น (Odour)	-	ไม่เป็นที่รังเกียจ	ไม่เป็นที่รังเกียจ
	4.ความขุ่น (Turbidity)	ซิลิกา สเกล ยูนิต (Silica scale unit)	5	20
	5.ความเป็นกรด-ด่าง(pH)	-	6.5-8.5	9.2
ทางเคมี	6.ปริมาณสารทั้งหมด (Total Solids)	มก./ล.	500	1,500
	7.เหล็ก (Fe)	มก./ล.	0.5	1.0
	8.มังกานีส (Mn)	มก./ล.	0.3	0.5
	9.เหล็กและมังกานีส (Fe& Mn)	มก./ล.	0.5	1.0
	10.ทองแดง (cu)	มก./ล.	1.0	1.5
	11.สังกะสี (Zn)	มก./ล.	5.0	15.0
	12.คัลเซียม (Ca)	มก./ล.	75[b]	200
	13.แมกนีเซียม (Mg)	มก./ล.	50	150
	14.ซัลเฟต (SO_4)	มก./ล.	200	250[c]
	15.คลอไรด์ (Cl)	มก./ล.	250	600
	16.ฟลูออไรด์ (F)	มก./ล.	0.7	1.0
	17.ไนเตรต (NO_3)	มก./ล.	45	45
	18.อัลคิลเบนซิลซัลโฟเนต (Alkylbenzyl Sulfonate,ABS)	มก./ล.	0.5	1.0
	19.ฟีโนลิกซับสแตนซ์(Phenolic substances as phenol)	มก./ล.	0.001	0.002
สารเป็นพิษ	20.ปรอท (Hg)	มก./ล.	0.001	-
	21.ตะกั่ว (Pb)	มก./ล.	0.05	-
	22.อาร์เซนิก (As)	มก./ล.	0.05	-
	23.ซีลีเนียม (Se)	มก./ล.	0.01	-
	24.โครเมียม (Cr hexavalent)	มก./ล.	0.05	-
	25.ไซยาไนด์ (CN)	มก./ล.	0.2	-
	26.แคดเมียม (Cd)	มก./ล.	0.01	-
	27.แบเรียม (Ba)	มก./ล.	1.0	-

มาตรฐานคุณภาพน้ำดื่มในภาชนะบรรจุที่ปิดสนิท			
คุณลักษณะ	ดัชนีคุณภาพน้ำ	หน่วย	ค่ามาตรฐาน (เกณฑ์อนุโลมสูงสุด)
ทางกายภาพ	1.สี (Colour)	ฮาเซนยูนิต(Hazen)	20
	2.กลิ่น(Odour)	-	ไม่มีกลิ่น (ไม่รวมกลิ่นคลอรีน)
	3.ความขุ่น(Turbidity)	ซิลิกาสเกลยูนิต (silica scale unit)	5
	4.ค่าความเป็นกรด-ด่าง (pH)	-	6.5-8.5
ทางเคมี	5.ปริมาณสารทั้งหมด(Total Soilds)	มก./ล.	500
	6.ความกระด้างทั้งหมด(Total Hardness) (คำนวณเป็นแคลเซียมคาร์บอเนต)	มก./ล.	100
	7.สารหนู (As)	มก./ล.	0.05
	8.แบเรียม (Ba)	มก./ล.	1.0
	9.แคดเมียม (Cd)	มก./ล.	0.005
	10.คลอไรด์ (Cl, คำนวณเป็นคลอรีน)	มก./ล.	250
	11.โครเมียม (Cr)	มก./ล.	0.05
	12.ทองแดง (cu)	มก./ล.	1.0
	13.เหล็ก (Fe)	มก./ล.	0.3
	14.ตะกั่ว (Pb)	มก./ล.	0.05
	15.แมงกานีส (Mn)	มก./ล.	0.05
	16.ปรอท (Hg)	มก./ล.	0.002
	17.ไนเตรต (NO_3-N, คำนวณเป็นไนโตรเจน)	มก./ล.	4.0
	18.ฟีนอล (Phenols)	มก./ล.	0.001
	19.ซีลีเนียม (Se)	มก./ล.	0.01
	20.เงิน (Ag)	มก./ล.	0.05
	21.ซัลเฟต (SO_4)	มก./ล.	250
	22.สังกะสี (Zn)	มก./ล.	5.0
	23.ฟลูออไรด์ (F) (คำนวณเป็นฟลูออรีน)	มก./ล.	1.5
	24.อะลูมิเนียม	มก./ล.	0.2
	25.เอบีเอส (Alkylbenzene Sulfonate)	มก./ล.	0.2
	26.ไซยาไนด์	มก./ล.	0.1

http://www.pcd.go.th/info_serv/reg_std_water01.html#s2

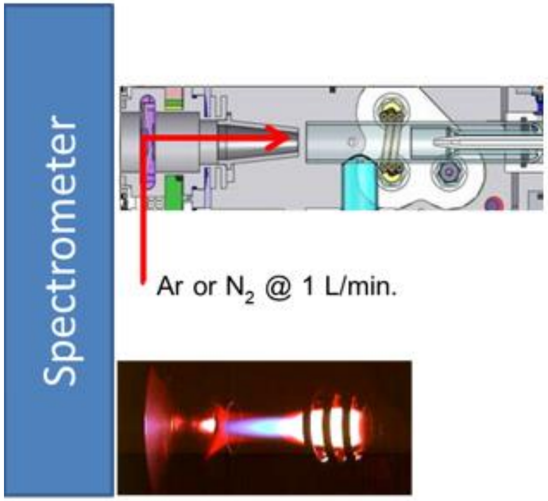
Analysis of toxic elements in drinking and bottled waters by ICP-OES

Thermo Scientific™ iCAP™ 7200 ICP-OES Duo with Qtegra™ Intelligent Scientific Data Solution™ (ISDS) Software

- Tap water sample from Dingpu river area, Shanghai
- Tap water sample from Jinqiao lake area, Shanghai
- Waterman (packaged drinking water)
- Nestle (natural mineral water)
- Evian (natural mineral water)

- Samples did not require any pre-treatment
- Samples were analyzed directly after preservation in 0.5% AR grade nitric acid (HNO_3)

Parameter	Setting
Pump Tubing	Sample Tygon® orange/white Drain Tygon® white/white
Pump Speed	45 rpm
Nebulizer	Glass concentric
Nebulizer Gas Flow	0.19 MPa
Spray Chamber	Glass cyclonic
Auxiliary Gas Flow	0.5 L·min^{-1}
Coolant Gas Flow	12 L·min^{-1}
Center Tube	2 mm
RF Power	1150 W
Plasma View	Axial
Exposure Time	5 s

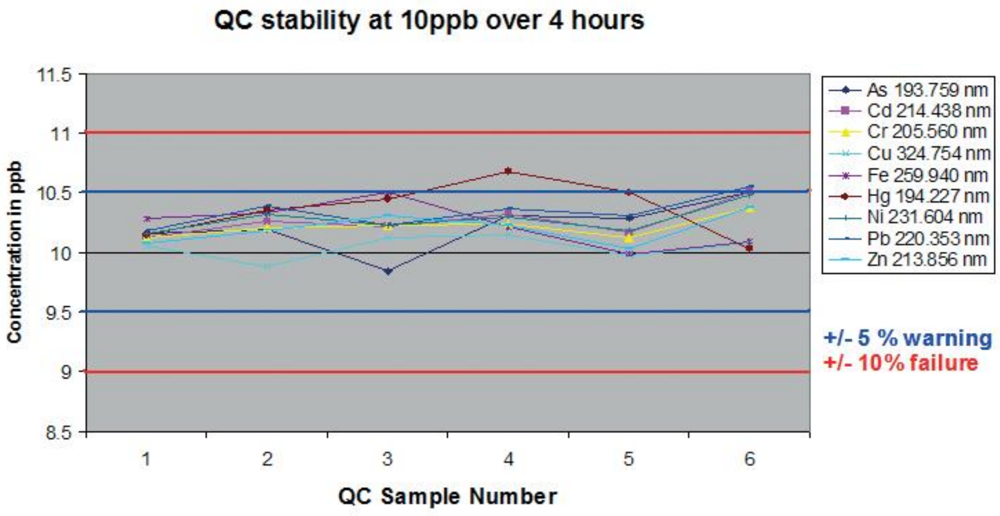


Averaged results and method detection limits in µg·kg-1.

Element and wavelength (nm)	MDL	Dingpu River	Jinquiao Lake	Waterman	Nestle	Evian
As 193.759	2.14	<DL	1.27	<DL	<DL	<DL
Cd 214.438	0.07	<DL	<DL	<DL	<DL	<DL
Cr 205.560	0.21	<DL	<DL	<DL	<DL	<DL
Cu 324.754	0.39	<DL	1.52	<DL	<DL	<DL
Fe 259.940	0.25	1.14	1.53	0.41	0.78	0.74
Hg 194.227	0.66	<DL	<DL	<DL	<DL	<DL
Ni 231.604	0.36	1.05	0.57	<DL	<DL	<DL
Pb 220.353	1.06	<DL	<DL	<DL	<DL	<DL
Zn 213.856	0.19	<DL	<DL	<DL	<DL	<DL

✓ All QC recoveries were within 10%

Stability of the 10 µg·kg-1 QC check over 4 hours



Which technique?

Which technique would you use for the analysis of Lead in blood??

They do not have detection limits but would like to detect as low as possible

A fully quantitative research method for the analysis of Lead in whole blood

Dust, Paint, Soil, Industrial, Water, Toy, Food

- The United States Centers for Disease Control and Prevention (CDC) states that Blood Lead Levels (BLL) >70 µg/dL (700 ng/mL) can cause serious health effects.
- BLL as low as 10 µg/dL (100 ng/mL) are associated with cognitive development, growth, and behavioral issues in children between the ages of 1-5 years.

✓ As, Cd, Cr, Pb, Hg and Se in whole blood and Certified reference materials (Seronorm Trace Elements Whole Blood)

A fully quantitative research method for the analysis of Lead in whole blood using ICP-MS

Add Ultrapure water

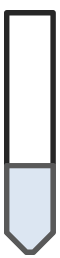
Vortex mixing for 15 minutes before use

Sample is 50 fold diluted with diluent

Plastic tube

- Add 100 mL of blank or standard or sample or QC
- Add 4900 mL of Diluent*

Vortex mixer



Analysis by ICP-MS

* Tetramethylammonium hydroxide (TMAH, 1.5%), Hydrochloric acid (HCl, 1.5%), Ammonium Pyrrolidine dithiocarbamate (APDC), Triton-X and 0.1 µg/L of ^{103}Rh (Internal standard)

A fully quantitative research method for the analysis of Lead in whole blood using ICP-MS

Possible interferences for 82Se

Symbol	Mass	Abundance
82Kr	81.9135	11.600
1H + 81Br	81.9241	49.303
16O + 1H + 65Cu	81.9305	30.752
16O + 66Zn	81.9209	27.834
12C + 70Ge	81.9242	20.275
14N + 68Zn	81.9279	18.731
13C + 69Ga	81.9289	0.661
40Ar + 42Ca	81.9210	0.644
12C + 70Zn	81.9253	0.593
16O + 3H + 63Cu	81.9405	0.000
17O + 2H + 63Cu	81.9428	0.000
18O + 1H + 63Cu	81.9366	0.138
164Dy++	81.9646	28.200
163Dy++	81.4644	24.900

Close

^{82}Se is chosen based on less possible argon based interferences compare to ^{80}Se ($^{40}Ar_2^+$).

Selected analyte isotopes		
^{75}As	^{114}Cd	^{82}Se
^{52}Cr	^{202}Hg	
^{63}Cu	^{208}Pb	

Internal standard isotope
^{103}Rh

^{114}Cd is chosen based on it abundance.

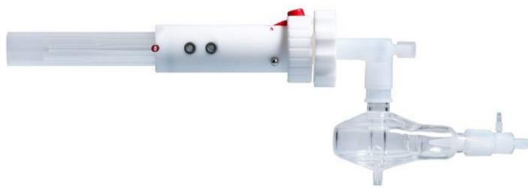
	Symbol	Mass	Abundance	Interferences
☐	106Cd	105.9065	1.25	106Pd(27.330%); 16..
☐	108Cd	107.9042	0.89	108Pd(26.460%); 1H .
☐	110Cd	109.9030	12.49	110Pd(11.720%); 16..
☐	111Cd	110.9042	12.80	12C + 99Tc(0.000%);
☐	112Cd	111.9028	24.13	112Sn(0.970%); 40Ar
☐	113Cd	112.9044	12.22	113In(4.300%); 14N +
☑	114Cd	113.9034	28.73	114Sn(0.650%); 40Ar
☐	116Cd	115.9048	7.49	116Sn(14.530%); 16..

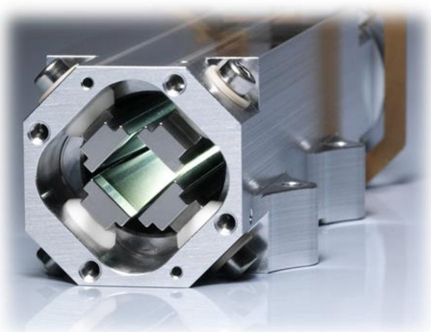
Possible interferences for 114Cd

Symbol	Mass	Abundance
114Sn	113.9028	0.650
40Ar + 74Ge	113.8836	36.354
12C + 102Ru	113.9043	31.252
16O + 98Mo	113.9003	24.073
14N + 100Ru	113.9073	12.554
1H + 113Cd	113.9122	12.218
14N + 100Mo	113.9105	9.595
16O + 1H + 97Mo	113.9088	9.526
1H + 113In	113.9119	4.299
16O + 98Ru	113.9002	1.876
40Ar + 74Se	113.8849	0.896
12C + 102Pd	113.9056	1.009
15N + 99Tc	113.9064	0.000
13C + 101Ru	113.9089	0.187

Close

How to remove Polyatomic Interference? Collision Cell Technology (CCT)



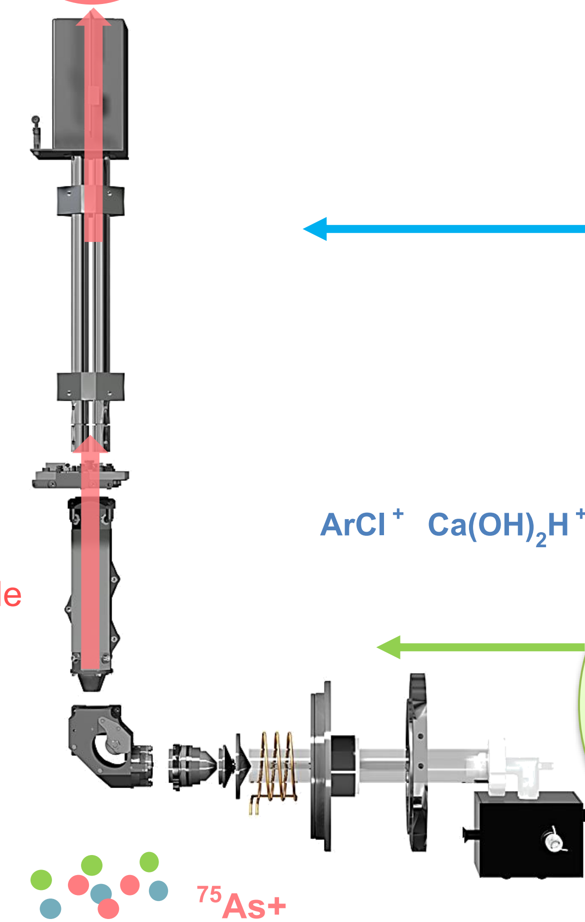


Unique Flatapole Design

$^{75}As^{+}$

Comprehensive Interference Removal

Quadrupole isolates ions wanted for measurement



He KED mode

$ArCl^{+}$ $Ca(OH)_2H^{+}$

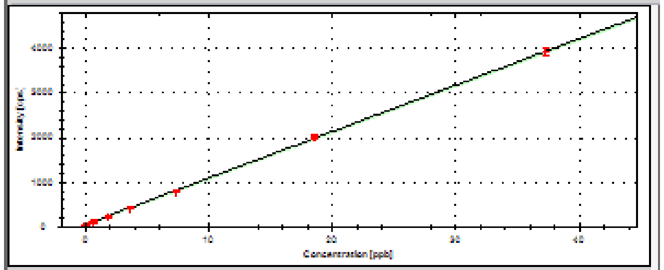
He KED filters out unwanted polyatomic interferences, based on difference in cross-sectional size of the analyte and polyatomic

$^{75}As+$

$40Ar35Cl^{+}$ $Ca(OH)_2H^{+}$

A fully quantitative research method for the analysis of Lead in whole blood using ICPMS

75As (KEDS) - IS:103Rh (KEDS)

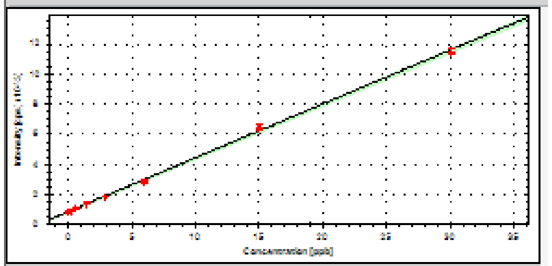


f(x) = b*x + a

Parameter	Value	Confidence Del	StdErr	Rel. StdEr
b	104.221	+/-0.391	0.276	0.265
a	30.330	+/-0.000	0.000	0.000
R^2	1.000			
BEC	0.291 ppb			
IDL(LOD)	0.081 ppb			

MDL include 50 times dilution

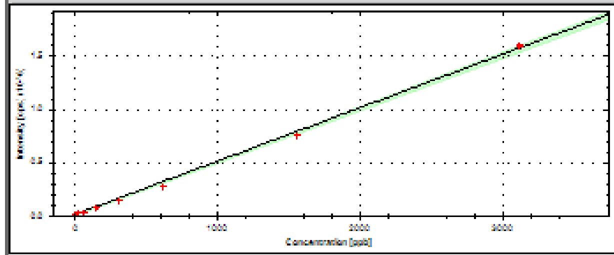
52Cr (KEDS) - IS:103Rh (KEDS)



f(x) = b*x + a

Paramet	Value	Confidence	StdErr	Rel. Std
b	357.573	+/-4.636	3.276	0.916
a	787.121	+/-0.000	0.000	0.000
R^2	0.999			
BEC	2.201 ppb			
IDL(LOD)	0.181 ppb			

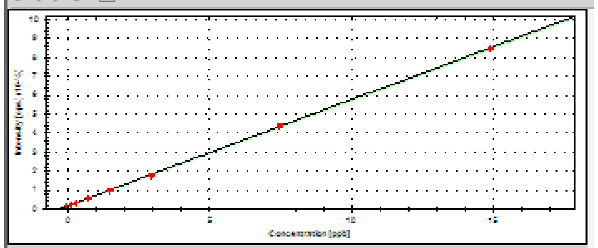
63Cu (KEDS) - IS:103Rh (KEDS)



f(x) = b*x + a

Parameter	Value	Confidence Del	StdErr	Rel. StdEr
b	501.486	+/-10.242	7.239	1.443
a	4,354.447	+/-0.000	0.000	0.000
R^2	0.998			
BEC	8.683 ppb			
IDL(LOD)	0.945 ppb			

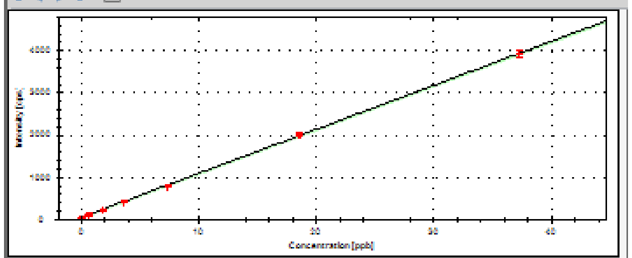
114Cd (KEDS) - IS:103Rh (KEDS)



f(x) = b*x + a

Parameter	Value	Confidence De	StdErr	Rel. StdEr
b	560.533	+/-2.758	1.949	0.348
a	116.691	+/-0.000	0.000	0.000
R^2	1.000			
BEC	0.208 ppb			
IDL(LOD)	0.031 ppb			

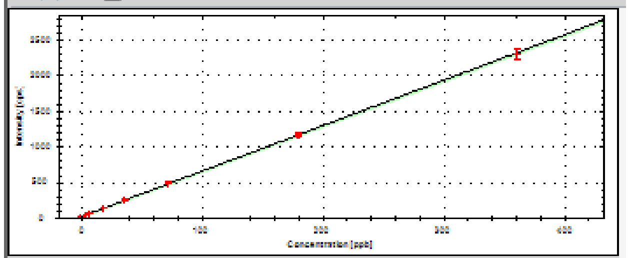
75As (KEDS) - IS:103Rh (KEDS)



f(x) = b*x + a

Parameter	Value	Confidence Del	StdErr	Rel. StdEr
b	104.221	+/-0.391	0.276	0.265
a	30.330	+/-0.000	0.000	0.000
R^2	1.000			
BEC	0.291 ppb			
IDL(LOD)	0.081 ppb			

82Se (KEDS) - IS:103Rh (KEDS)

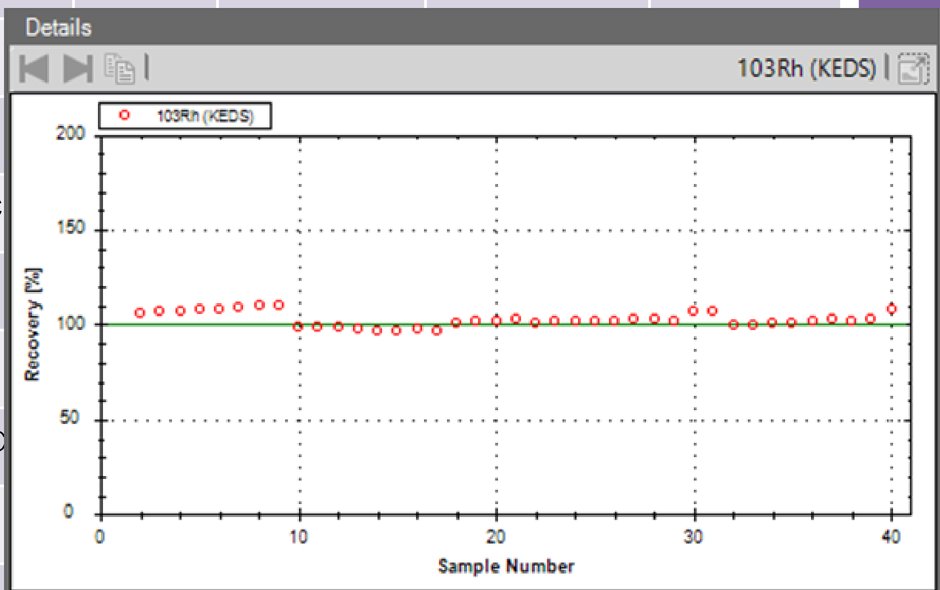


f(x) = b*x + a

Parameter	Value	Confidence De	StdErr	Rel. StdEr
b	6.390	+/-0.037	0.026	0.404
a	7.327	+/-0.000	0.000	0.000
R^2	1.000			
BEC	1.147 ppb			
IDL(LOD)	0.693 ppb			

A fully quantitative research method for the analysis of Lead in whole blood using ICPMS

Element	CRM	Certified Value (ng/mL)	Acceptable range (ng/mL)	Found (± SD, n = 3) (ng/mL)
As	L1	2.4	1.4-3.4	2.77±0.09
C				
C				
	L3	23.2	18.5 – 27.9	24.27 ± 0.64



Element	CRM	Certified Value (ng/mL)	Acceptable range (ng/mL)	Found (± SD, n = 3) (ng/mL)
	L1	1.5	0.90 – 2.10	2.24 ± 0.24
	L2	16	9.60 – 22.40	20.47 ± 0.39
	L3	37.1	29.6 – 44.6	40.41 ± 1.55
	L1	10.2	6.00 – 14.40	10.68 ± 0.36
	L2	310	186 - 434	394 ± 13
	L3	447	401 - 493	536 ± 22
	L1	59	35 – 69	69.03 ± 0.59
	L2	112	66 – 158	131 ± 5
	L3	272	217 – 327	258 ± 8

Thank you for your attention!!!

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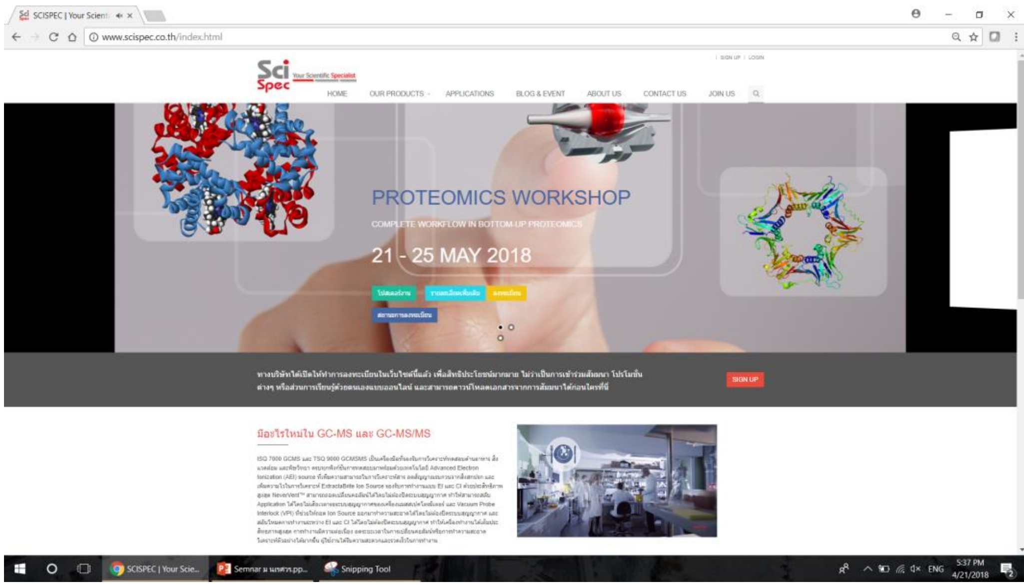

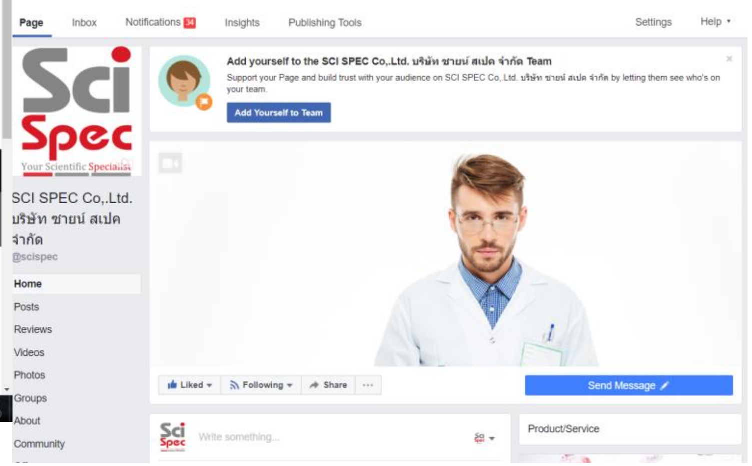

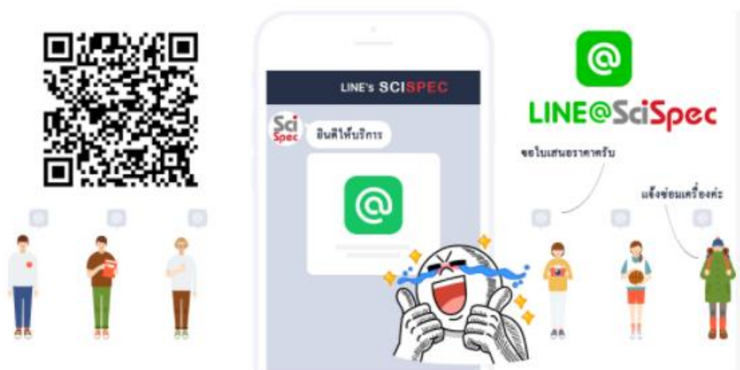

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