


Atomic spectroscopy **Workshop**

Interferences

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Interferences

Interferences are common with any atomic spectroscopy technique, particularly when trying to measure trace-level concentrations in a sample matrix that contains high concentrations of elements that produce line-rich spectra, such as Fe, Ca, and Si.

Sample matrices that are known to generate these types of interferences include geological, metallurgical, and high-matrix environmental samples, such as soils or wastewaters.

The three common types of interferences are

- physical

- chemical

- spectral.

Physical Interferences

Viscosity

High Dissolved Solids (density)

Acid type or concentration

Surface tension

Organic solvents

How to check matrix interferences

- Spike recovery (spike volume should be negligible)

$$\%R = 100.\frac{(spiked\ sample - sample}{spike}$$

- Dilution (>2) recovery

$$\%R = 100.\frac{(dilutio\quad factor.diluted\ sample)}{sample}$$

- CRM, certified reference material (matrix should be the same than sample)

Solving Physical Interference Problems

Dilution (degrades detection limits)

Matrix matching (must be known)

Internal Standardization

Method of Standard Additions

Internal standard choice

IS should:

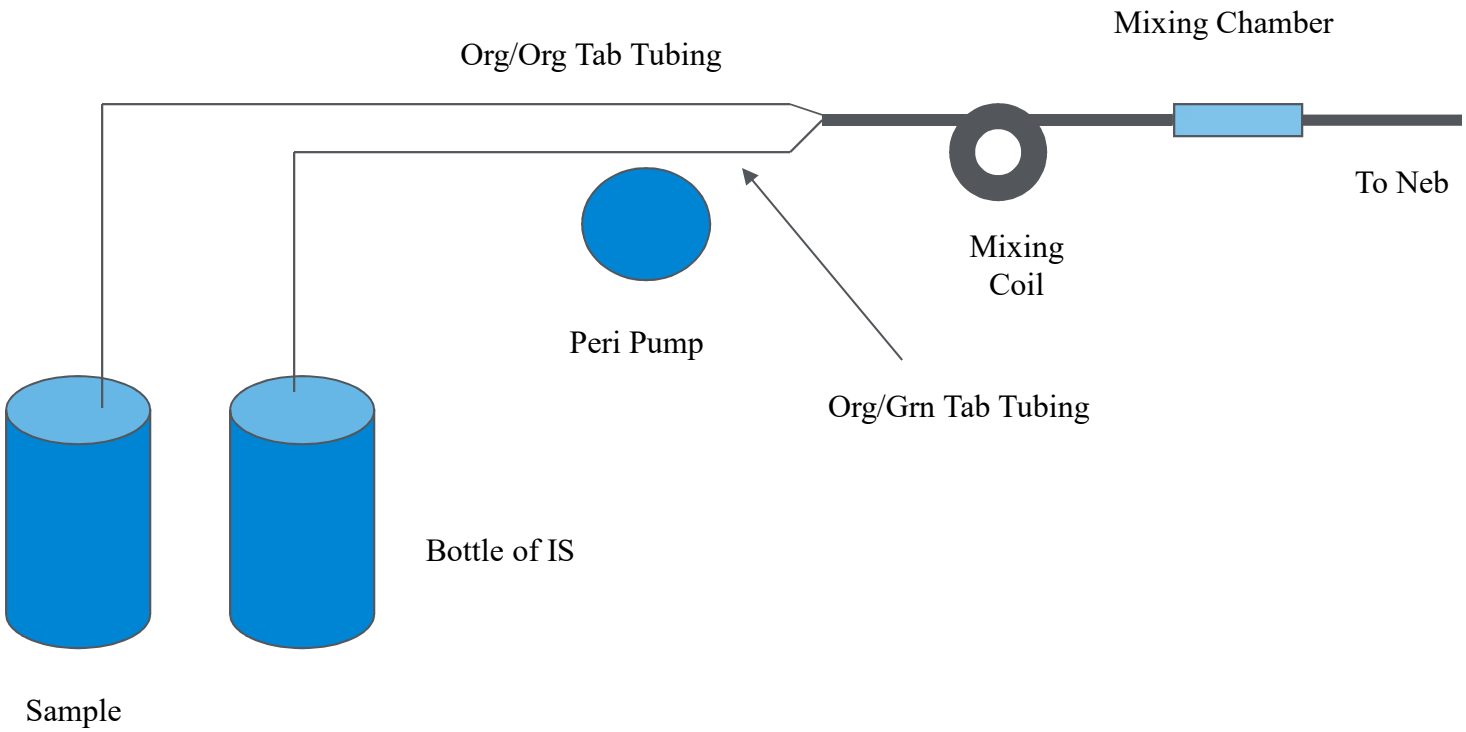
- NOT BE present in sample
- NOT BE polluted
- NOT spectrally interfere analyte lines
- NOT be spectrally interfered by sample

For aqueous samples :

For ionic lines: Y, Sc, Ho 1mg/L or In 10 mg/l (in sample mixture)

For in-line addition : Y, Sc, Ho 5mg/L or In 50 mg/l

Internal Standard Mixing Kit



IS is mixed at a 1 to 5 ratio with Sample

In-line Internal standard addition (in aqueous)

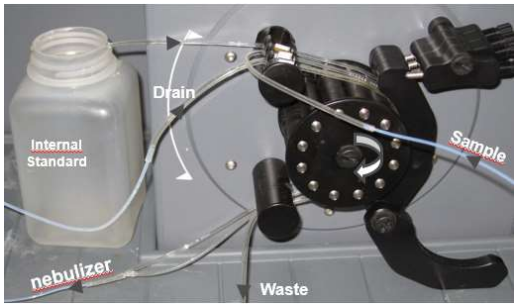
Total nebulizer flow should remain the same with/without IS

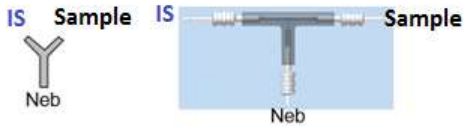
in-line Addition mixing ratio:

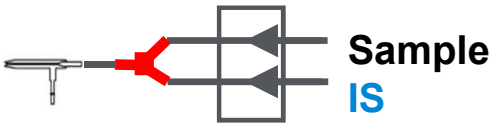
- reduced id ISTD pump tubing to avoid diluting sample
- Ideal relative ratio of ISTD 20-80
- One to one ratio in organics or manual addition

Mixer: Y-piece or T-piece

Clamp pressure: the clamp pressure is even more important when using ISTD, to be sure ISTD is added with same ratio.







Chemical Interferences

A sample matrix characteristic
which causes an analyte to behave
differently in the sample and standard

Types of Chemical Interferences

Ionization (Na, K, Rb, Cs, Li)

Molecular formation (ie oxides)

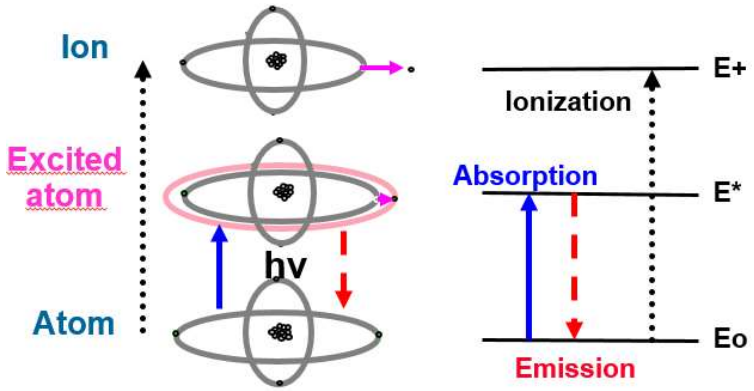
Plasma Loading

Ionization Interferences

As [M] increases, the electron concentration also increases shifting the equilibrium to the left

EIE

- Concerns easily ionizable elements Li, Na, K, Rb, Cs



Ionization Potential eV

Li	5,39
Na	5,14
K	4,34
Rb	4,18
Cs	3,89

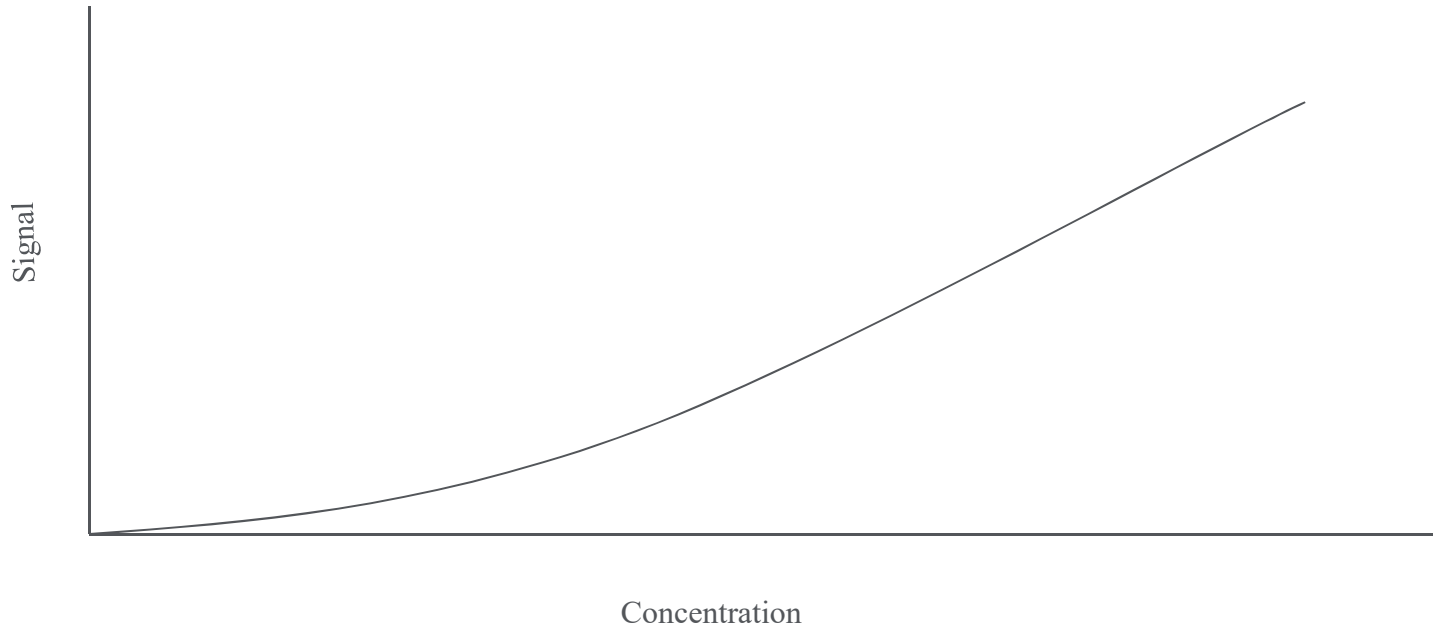
- ionisation buffer addition 2 g/L $Cs(NO_3)_2$

(1% for addition in line)

$$Na \Leftrightarrow Na^+ + e$$

$$Cs \Leftrightarrow Cs^+ + e$$

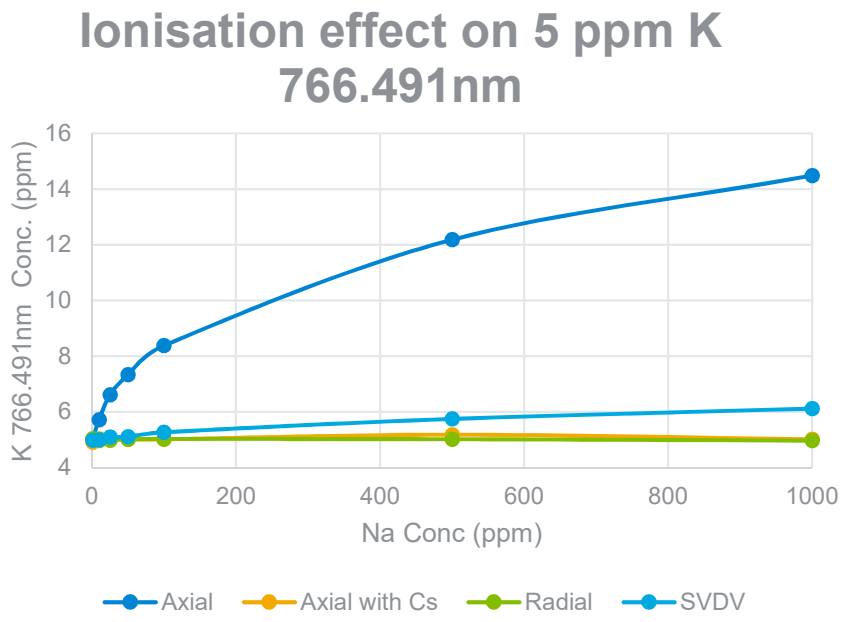
Ionization Interference Problem #1



For a single element solution of an easily ionized element, positive curvature is seen when calibrating

EIE Study – Effect of high Na on 5 ppm K 766.491nm

	Axial	Axial with Cs	Radial
Na (ppm)	ppm	ppm	ppm
1	5.01	4.91	5.04
10	5.72	4.97	5.03
25	6.61	5.03	4.98
50	7.33	5.02	5.02
100	8.38	5.02	5.03
500	12.18	5.03	5.02
1000	14.48	5.02	4.97



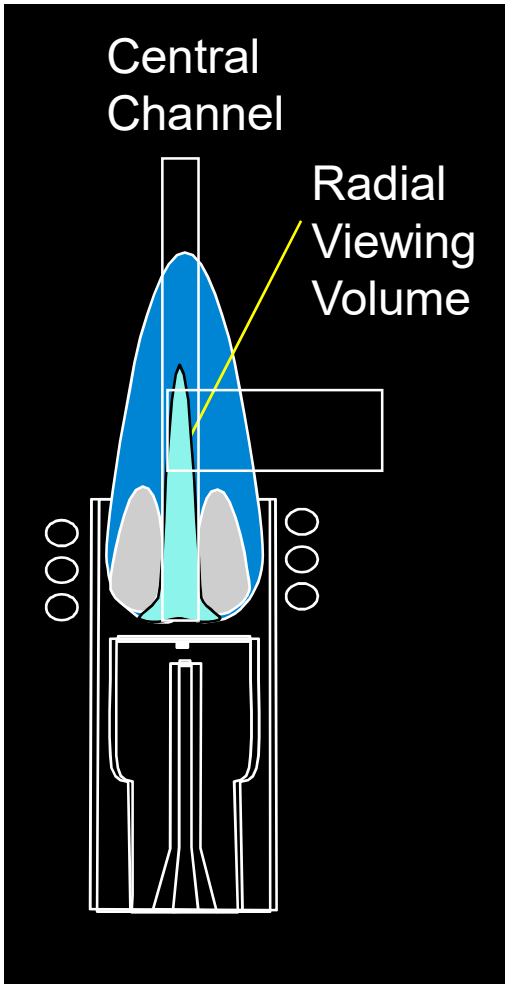
Solving Chemical Interference Problems

Ionisation buffer : CsCl

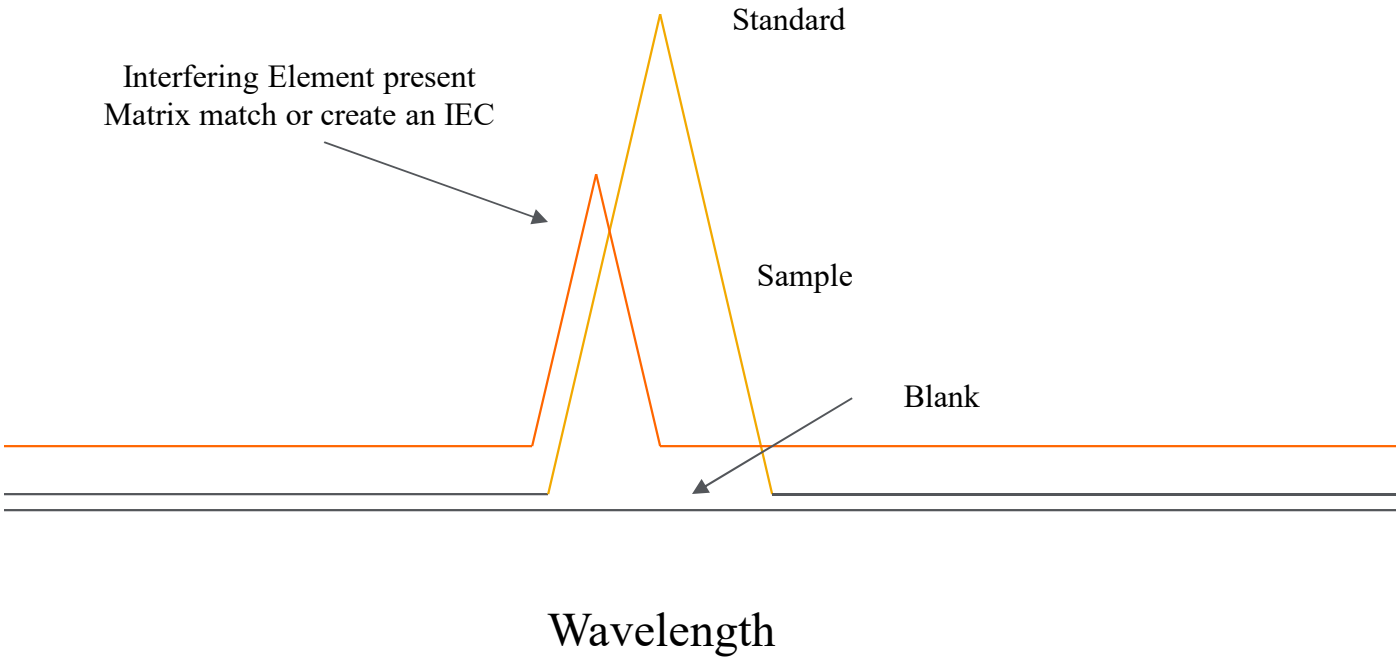
Dilution (degrades detection limits)

Matrix Matching

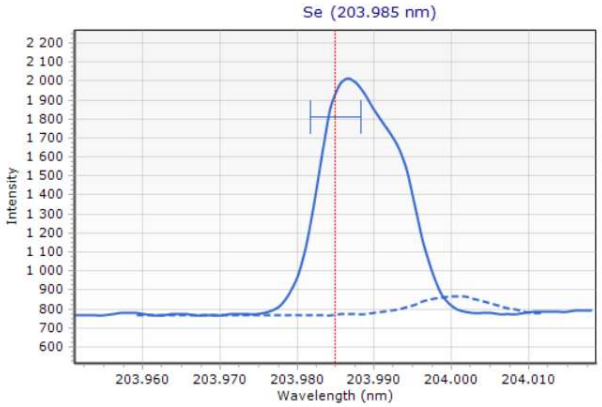
Use radial Viewing



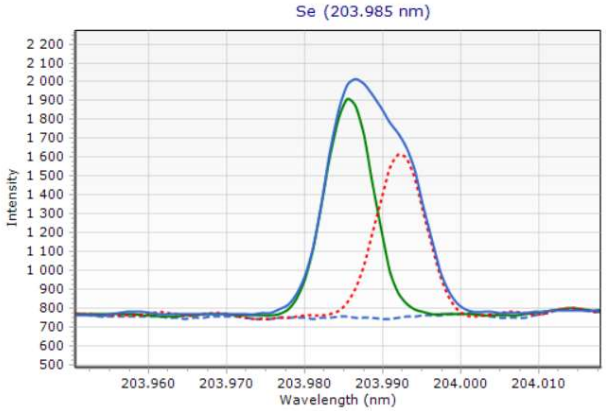
Spectral Interferences



Spectral interference – Cr Interference on Se



Solution Label	Se 196.026 nm ppm	Se 203.985 nm ppm
Blanc	0.00	0.00
Etalon 1	1.00	1.00
Etalon 2	2.00	2.00
Echantillon 1	1.05	1.17



Solution Label	Se 196.026 nm ppm	Se 203.985 nm ppm
Blanc	0.00	0.00
Etalon 1	1.00	1.00
Etalon 2	2.00	2.00
Echantillon 1	1.05	1.07

-- Se Analyte
-- Cr interferent

Possible interferences on Se (203.985 nm)

Symbol	Wavelength (nm)	Ion	Intensity
Hf	203.909	II	45.4
Re	203.920	I	10.8
Cr	203.932	I	7.2
Ir	203.943	I	35.8
Sb	203.977	I	45.2
Se	203.985	I	161.6
Cr	203.991	II	180.2
Mn	203.998	II	18.6
Ta	204.001	II	45.8
Ta	204.061	II	49.2
Sn	204.066	I	18.4
Fe	204.069	II	43.5