

Updated: 21 September 2014

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CEE 772: Instrumental Methods in Environmental Analysis

Lecture #7

Atomic Spectroscopy: Sample Treatment and
Methodology

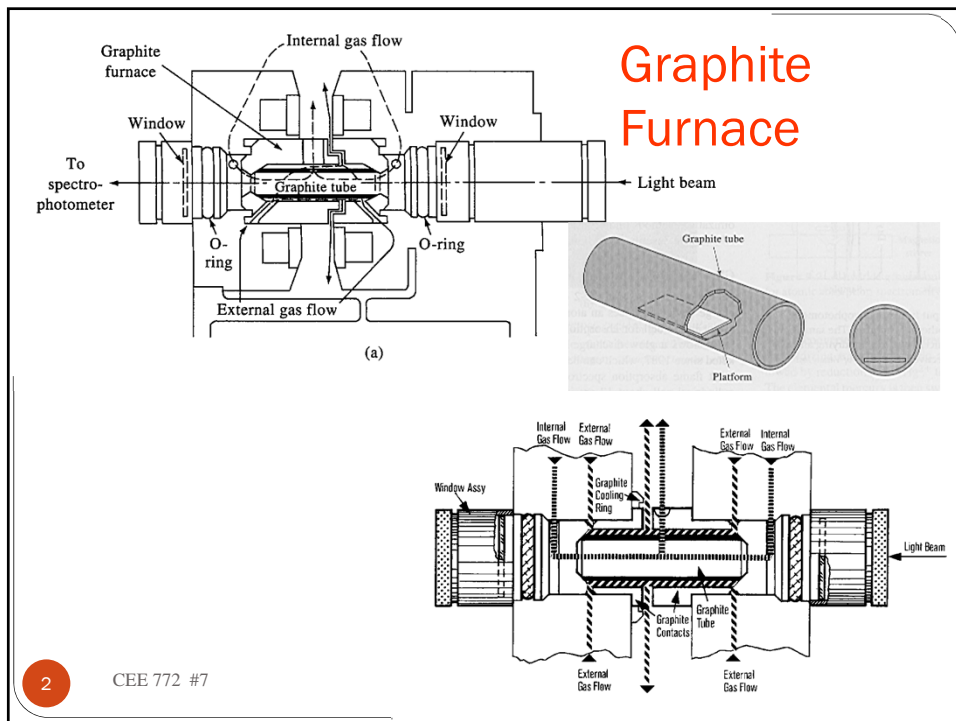
(Skoog, Chapt. 8 & 9; pp.192-203, 206-227)

(Harris, Chapt. 22)
(pp.615-635)

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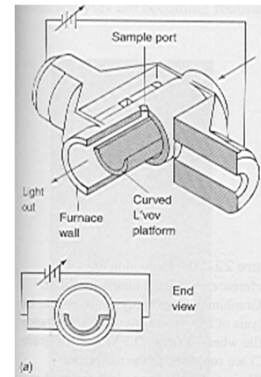
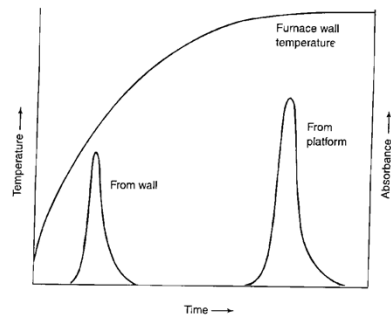
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Platform position

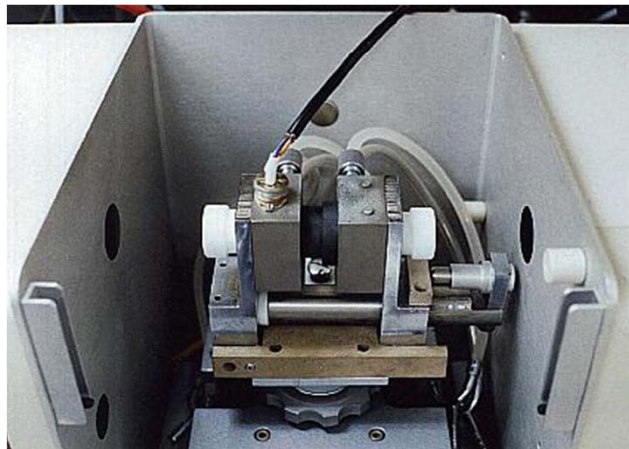


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Graphite Furnace



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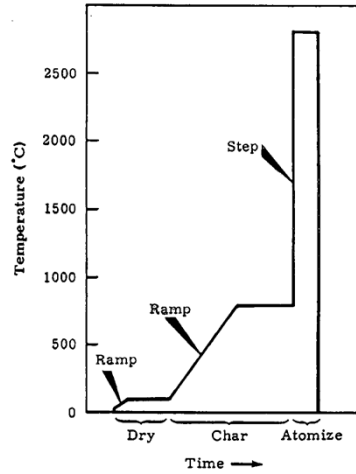
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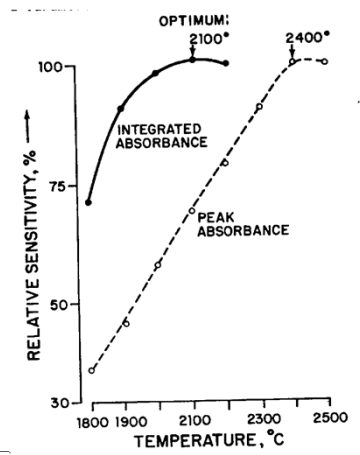
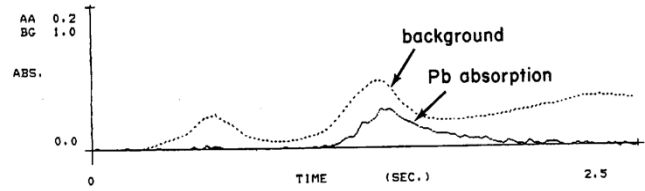
Temperature Programming

- Dry
 - Remove solvent
- Char
 - Remove organic matter and other volatiles
- Atomize
 - Convert analyte to an atom vapor

GRAPHITE FURNACE PROGRAM



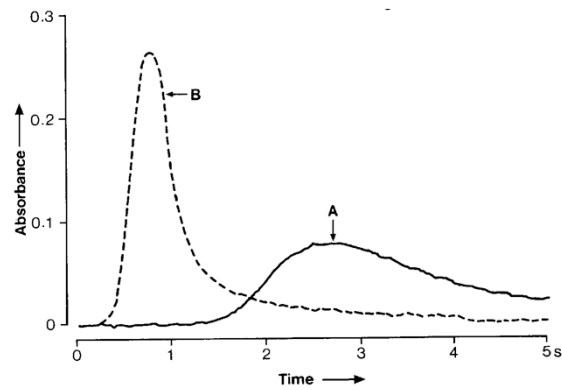
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Signals

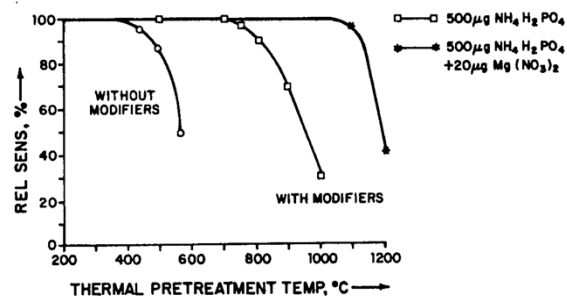
- Analysis of Molybdenum
 - A: uncoated tube
 - B: pyro-coated tube



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Use of Matrix Modifiers

- Thermal pretreatment for Cd using different matrix modifiers

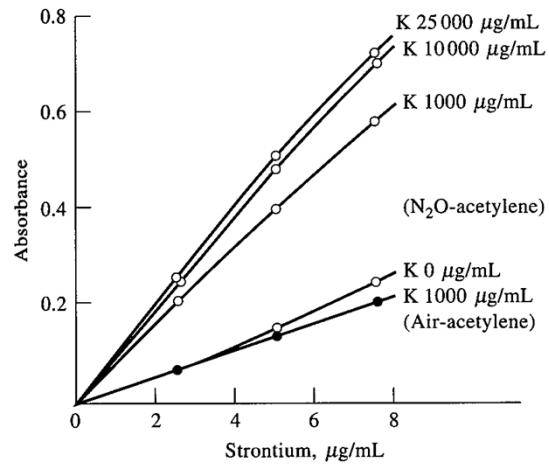


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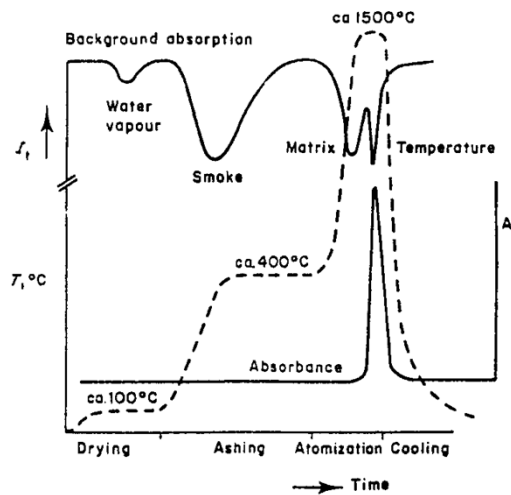
Matrix Modifiers, cont.

- Impact of K on calibration curve for Sr



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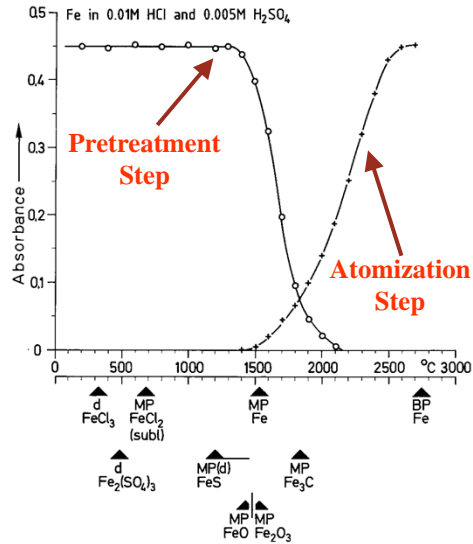
Thermal Progression



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Pretreatment/Atomization 1

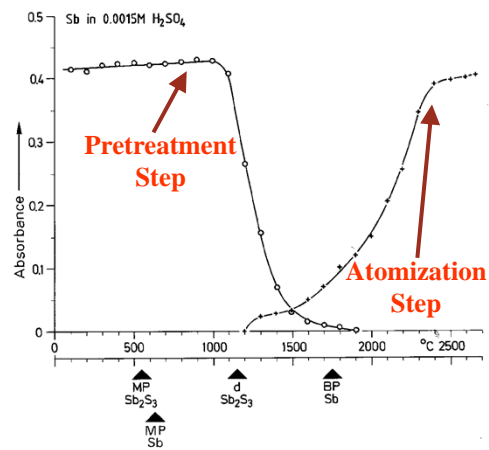
- Case for Iron
 - Pretreatment
 - First losses just below melting point
 - Atomization
 - Probably directly from metal as many salts decompose at lower temps



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Pretreatment/Atomization 2

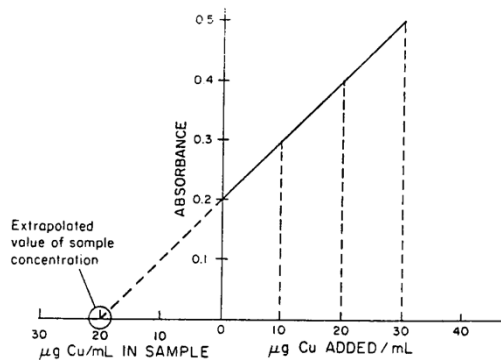
- Case for Antimony
 - Pretreatment
 - First losses just below melting point
 - Atomization
 - Probably delayed due to high temp of salt decomposition



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Standard Addition

- Use of standard addition to compensate for matrix effects

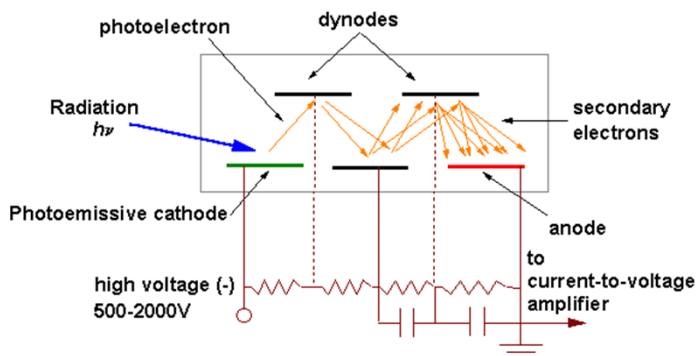


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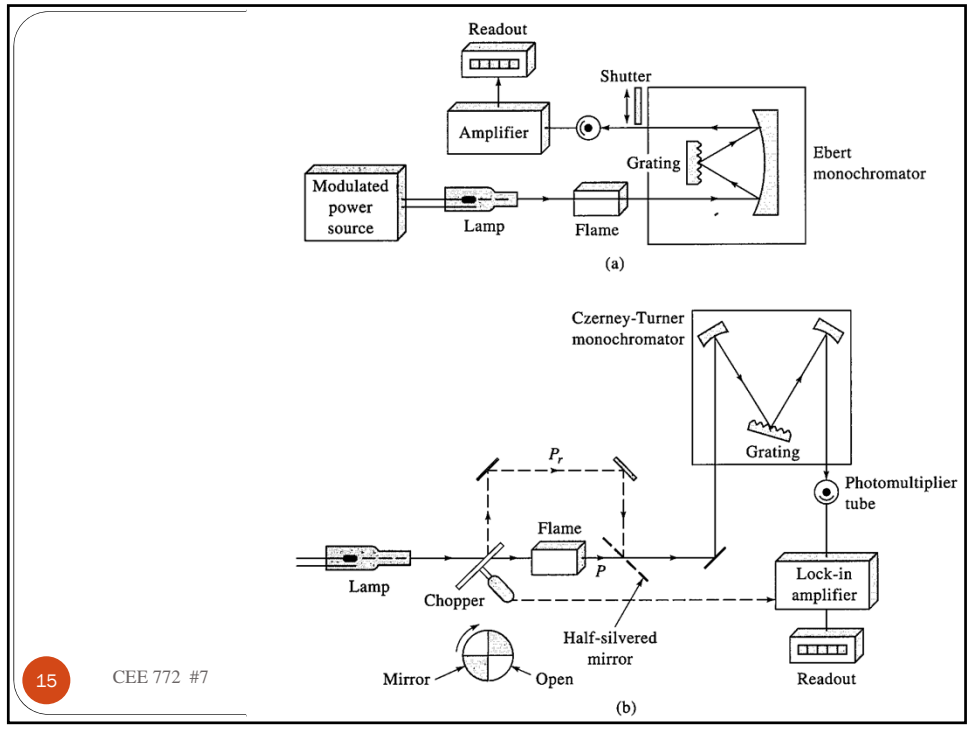
Photomultiplier



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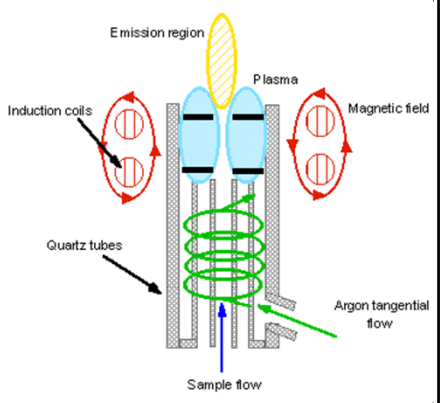
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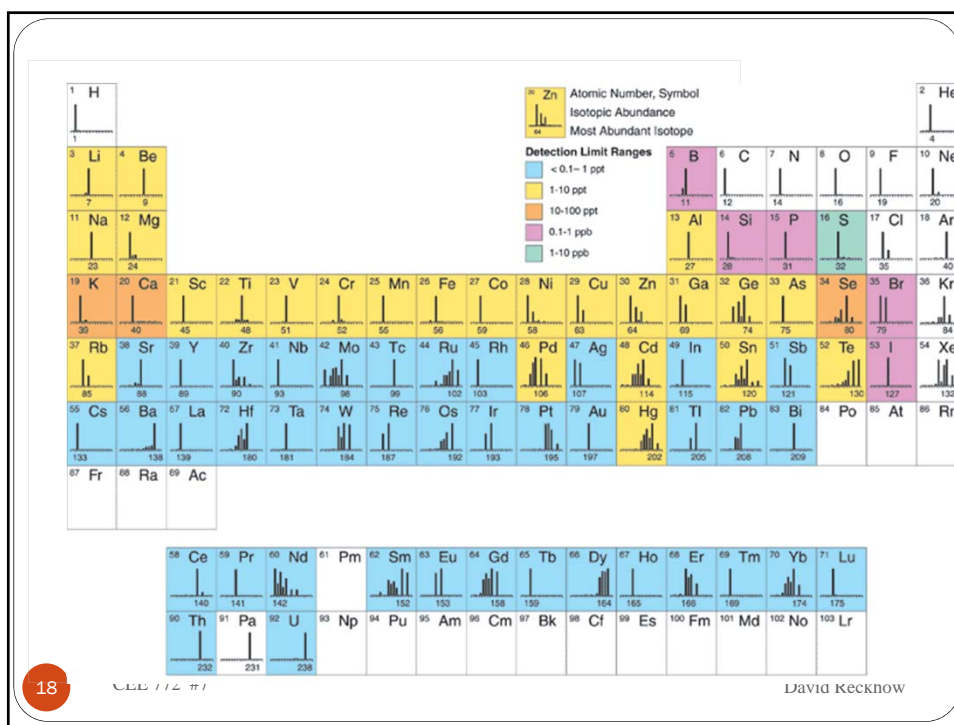
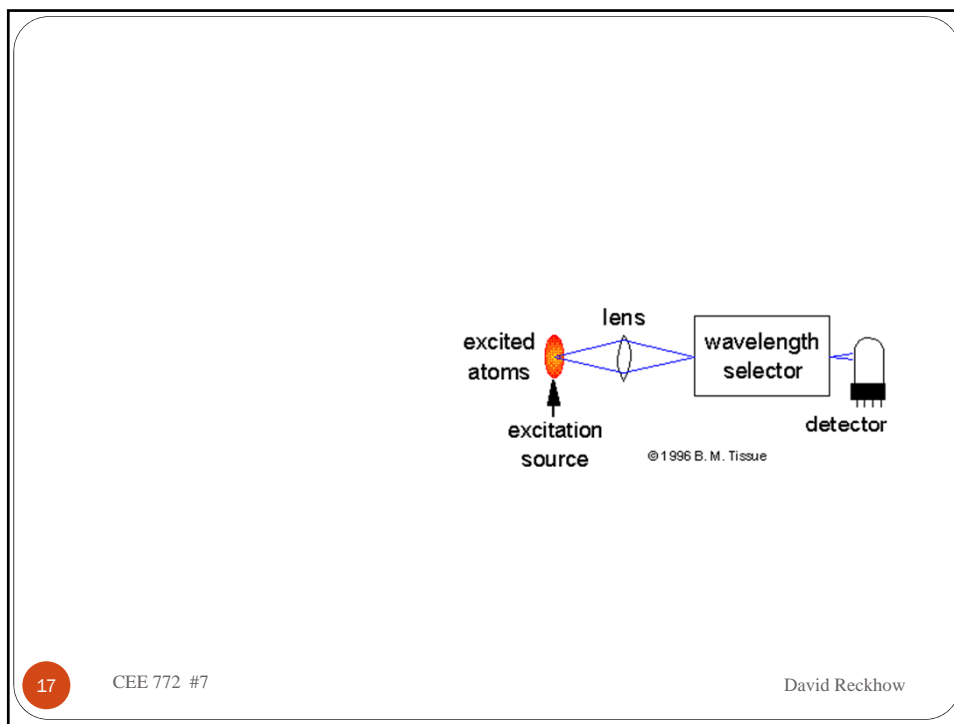
Plasma source

- The sample is nebulized and entrained in the flow of plasma support gas, which is typically Ar. The plasma torch consists of concentric quartz tubes, with the inner tube containing the sample aerosol and Ar support gas and the outer tube containing an Ar gas flow to cool the tubes (see schematic). A radiofrequency (RF) generator (typically 1-5 kW @ 27 MHz or 41 MHz) produces an oscillating current in an induction coil that wraps around the tubes. The induction coil creates an oscillating magnetic field, which produces an oscillating magnetic field. The magnetic field in turn sets up an oscillating current in the ions and electrons of the support gas. These ions and electrons transfer energy to other atoms in the support gas by collisions to create a very high temperature plasma.



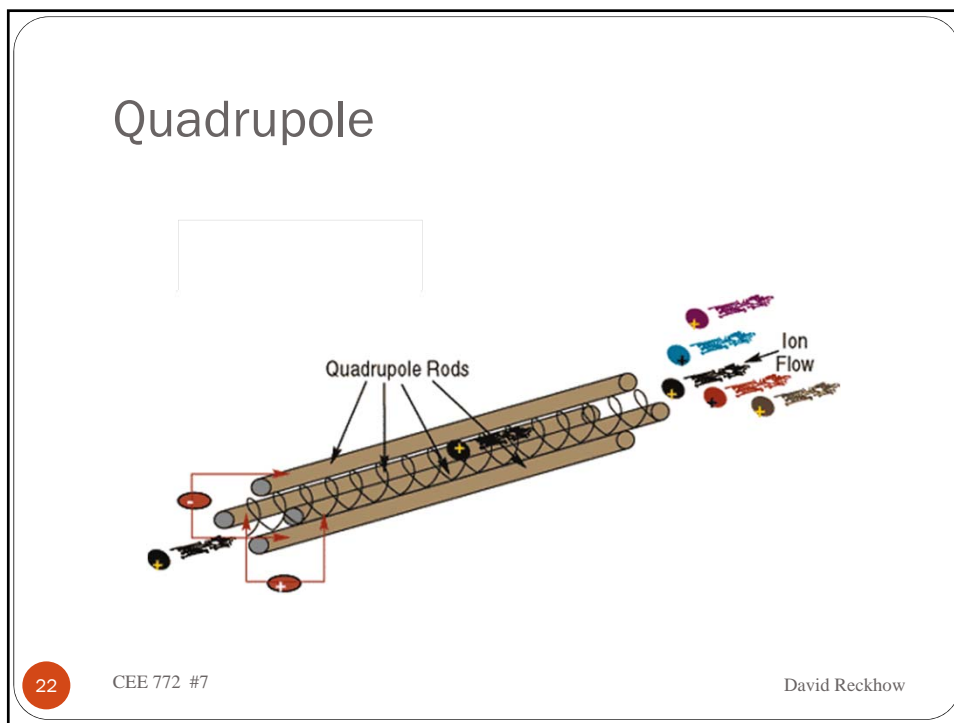
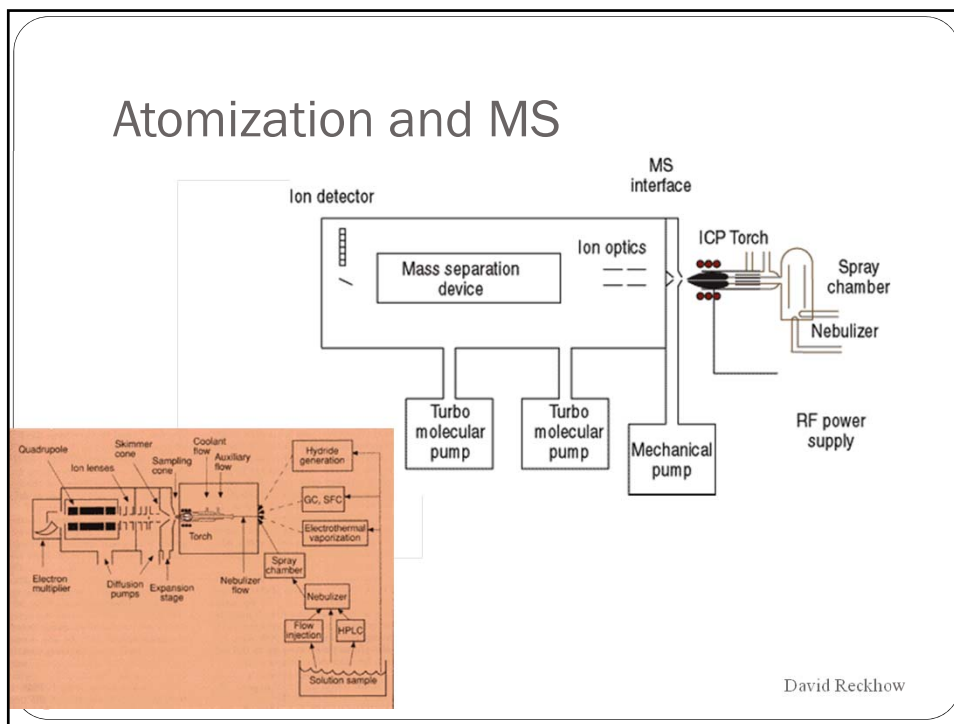
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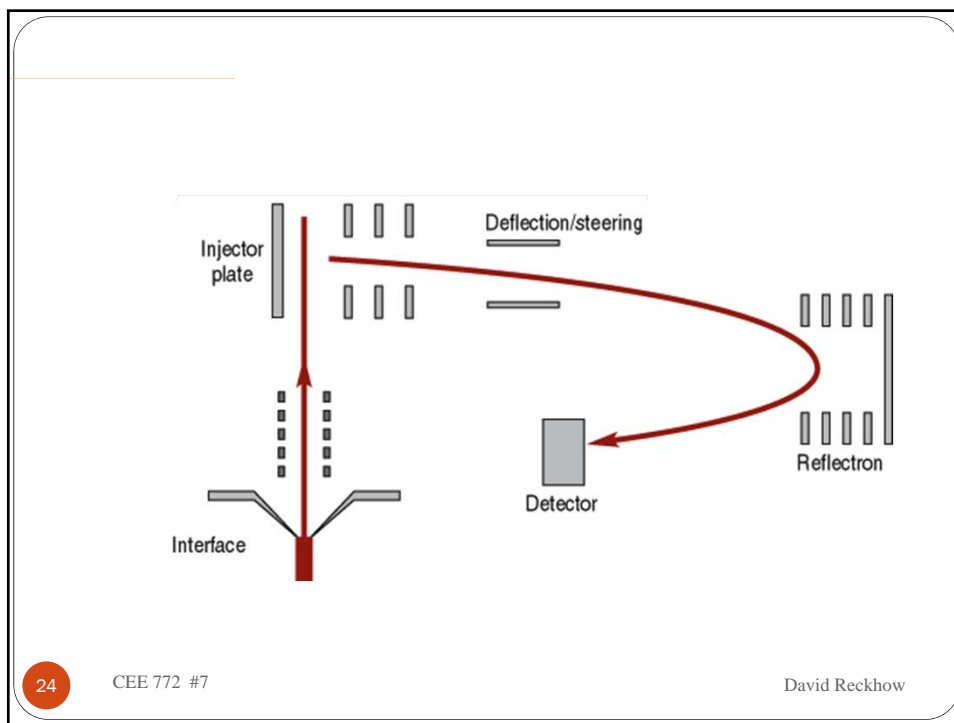
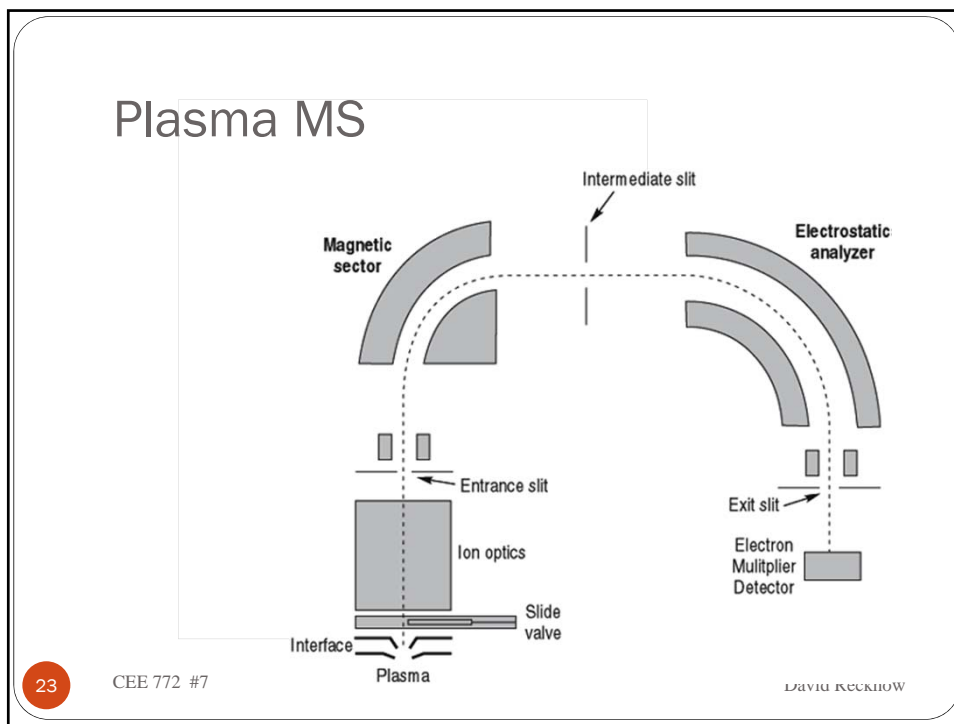
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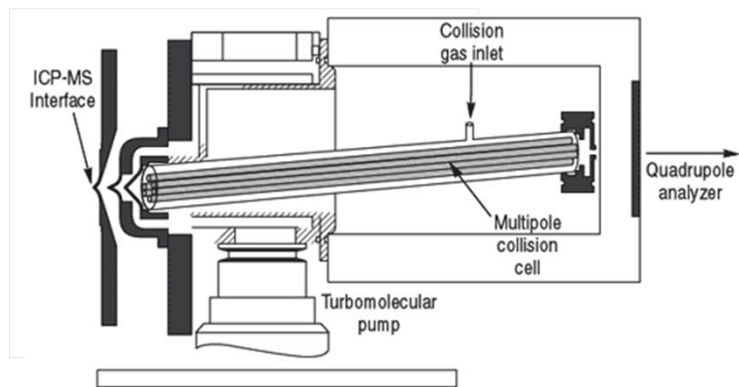
		Detection limits (ng/mL)																																		
		— Inductively coupled plasma emission		— Flame atomic absorption		— Graphite furnace atomic absorption																														
Li 0.7 0.1	Be 0.07 1 0.02	Fe 0.7 5 0.02	B 1 500 15	C 10 —	N	O	F	Ne	Al 2 30 0.01	Si 5 100 0.1	P 7 40,000 30	S 3 —	Cl	Ar	K 20 3 0.1	Ca 0.07 0.5 0.01	Sc 0.3 40 —	Ti 0.4 70 0.5	V 0.7 50 0.2	Cr 2 3 0.01	Mn 0.2 2 0.01	Fe 0.7 5 0.02	Co 1 4 0.02	Ni 3 90 0.1	Cu 0.9 1 0.02	Zn 0.6 60 0.001	Ga 10 60 0.5	Ge 20 200 —	As 7 200 0.2	Se 10 250 0.5	Br	Kr				
Rb 1 7 0.05	Sr 0.2 2 0.1	Y 0.6 200 —	Zr 2 1000 —	Nb 5 2000 —	Mo 3 20 0.02	Tc	Ru 10 60 1	Rh 20 4 —	Pd 4 10 0.3	Ag 0.8 2 0.005	Cd 0.5 10 0.003	In 20 40 1	Sn 9 30 0.2	Sb 9 40 0.15	Te 4 30 0.1	I	Xe	Cs 40,000 4 0.2	Ba 0.6 10 0.04	La 1 2000 —	Hf 4 2000 —	Ta 10 1000 —	W 8 1000 —	Re 3 600 —	Os 0.2 100 —	Ir 7 400 0.2	Pt 7 100 0.1	Au 2 7 10	Hg 7 150 2	Tl 10 20 0.1	Pb 10 10 0.10	Bi 7 40 0.1	Po	At	Rn	
Ce 2 — —	Pr 9 6000 —	Nd 10 1000 —	Pm	Sm 10 1000 —	Eu 0.9 20 0.5	Gd 5 2000 —	Tb 6 500 0.1	Dy 2 30 1	Ho 2 40 —	Er 0.7 30 2	Tm 2 900 —	Yb 0.3 4 —	Lu 0.3 300 —	Th 7 — —	Pa	U 80 40,000 —	Np	Pu	Am	Cm	Bk	Cf	Es	Fm	Md	No	Lr									

Element	Wavelength (nm)	Estimated Detection Limit (µg/L)
Aluminum	308.215	45
Antimony	206.833	32
Arsenic	193.696	53
Barium	455.403	2
Beryllium	313.042	0.3
Boron	249.773	5
Cadmium	226.502	4
Calcium	317.933	10
Chromium	267.716	7
Cobalt	228.616	7
Copper	324.754	6
Iron	259.940	7
Lead	220.353	42
Magnesium	279.079	30
Manganese	257.610	2
Molybdenum	202.030	8
Nickel	231.604	15
Potassium	766.491	See note c
Selenium	196.026	75
Silicon	288.158	58
Silver	328.068	7
Sodium	588.995	29
Thallium	190.864	40
Vanadium	292.402	8
Zinc	213.856	2





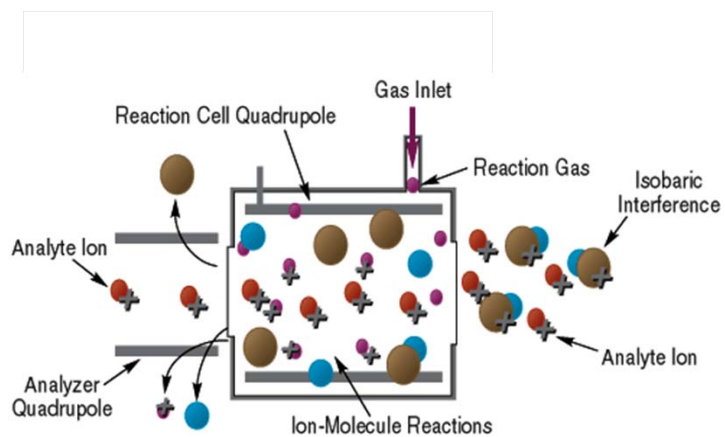
ICP-MS



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- To next lecture