MEDICAL GEOLOGY/GEOCHEMISTRY

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Session 3A - January 18, 2006

Session 3 January 18, 2006

Objective

Session 3A

Overview of Analytical Techniques:

- Atomic Absorption and Emission
- Inductively Coupled Plasma Mass Spectrometry
- Instrumental Neutron Activation Analysis
- Electron Microprobe - Wavelength and Energy
- Dispersive X-ray Spectroscopy

Session 3B

11AM-12PM:

- (EAPS - Neutron Activation Analysis Laboratory)
- Concepts of Sample Preparation
- Hands on Experience with instruments for
- Trace Element Determination by Neutron Activation Analysis –
- Hand out of review quiz

Introduction

Analytical technique is a tool to determine

- abundances of elements
- information about minerals
- information about organics

May be categorized as

- inorganic and organic
- qualitative and quantitative
- spectroscopic and classical

Introduction …

- Qualitative means – identification.
- Quantitative means - determining the abundance.

  The basic concept of quantitative analysis:

  Take a material, with known abundances, called the standard.

  Using the known amount of abundance(s) in the standard, estimate the abundance(s) in the unknown called the sample, maintaining all the conditions and parameters same for the sample and the standard.

Spectroscopic vs. Classical Techniques

- Spectroscopic analytical techniques utilize electromagnetic radiation interaction with the materials for analysis.
- Classical techniques utilize physical properties:

  color, conductivity, density,

  electric charge, mass,

  refraction, volume

Electromagnetic Radiation – Spectroscopic Techniques

Electromagnetic radiation consists of two sinusoidal waveforms , namely electric and magnetic, propagated along the same axis in planes perpendicular to each other.

The electromagnetic wave has two properties:

Energy E

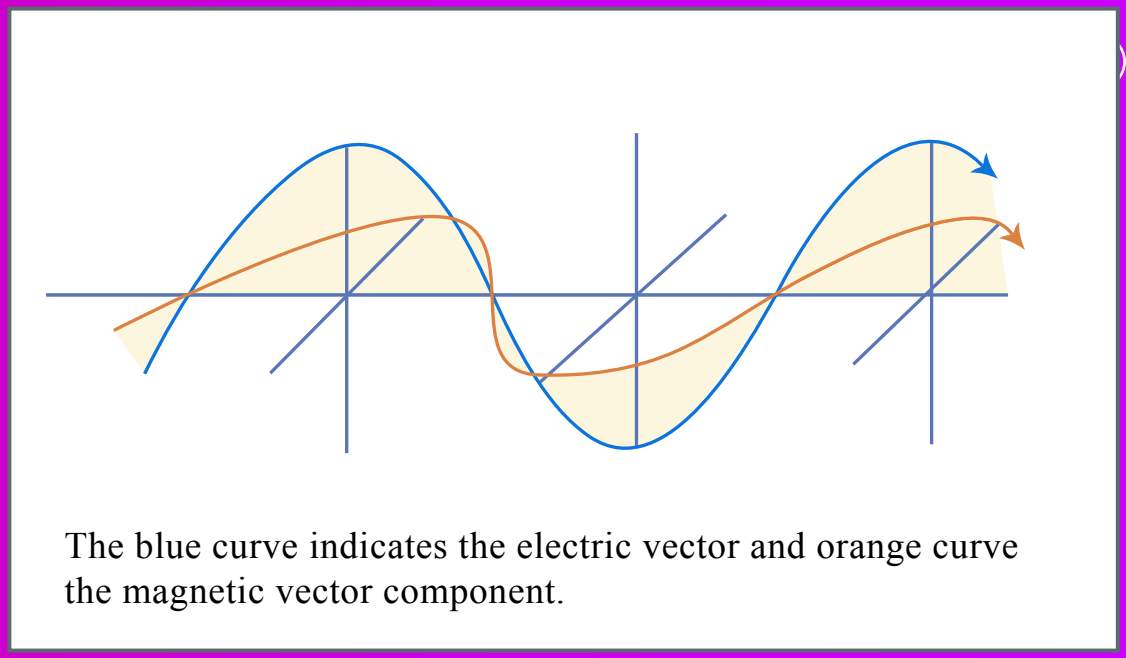
Wavelength λ (or frequency υ)

$E = hc / \lambda = h\upsilon$

h is Planck's constant,

c is velocity of light

Light is a well known example of electromagnetic radiation.



The blue curve indicates the electric vector and orange curve the magnetic vector component.

Figure by MIT OCW.

Figure 1. Components of electromagnetic radiation

Figure 2. Calibration Curve

Quantitative analysis involves determination of a calibration curve by measuring the analytical signal as a function of known concentrations of the standard(s), conducted in a range of values.

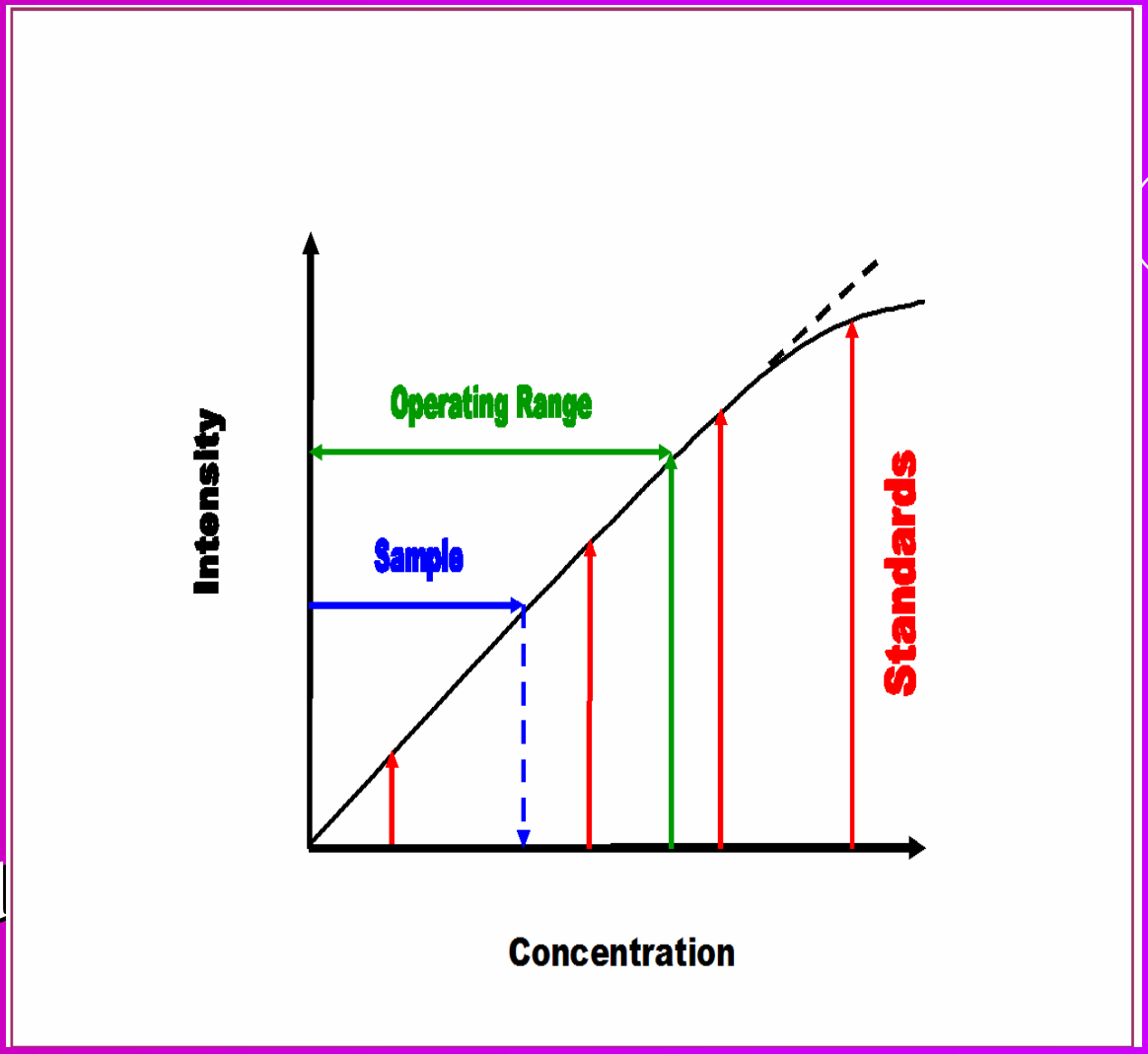
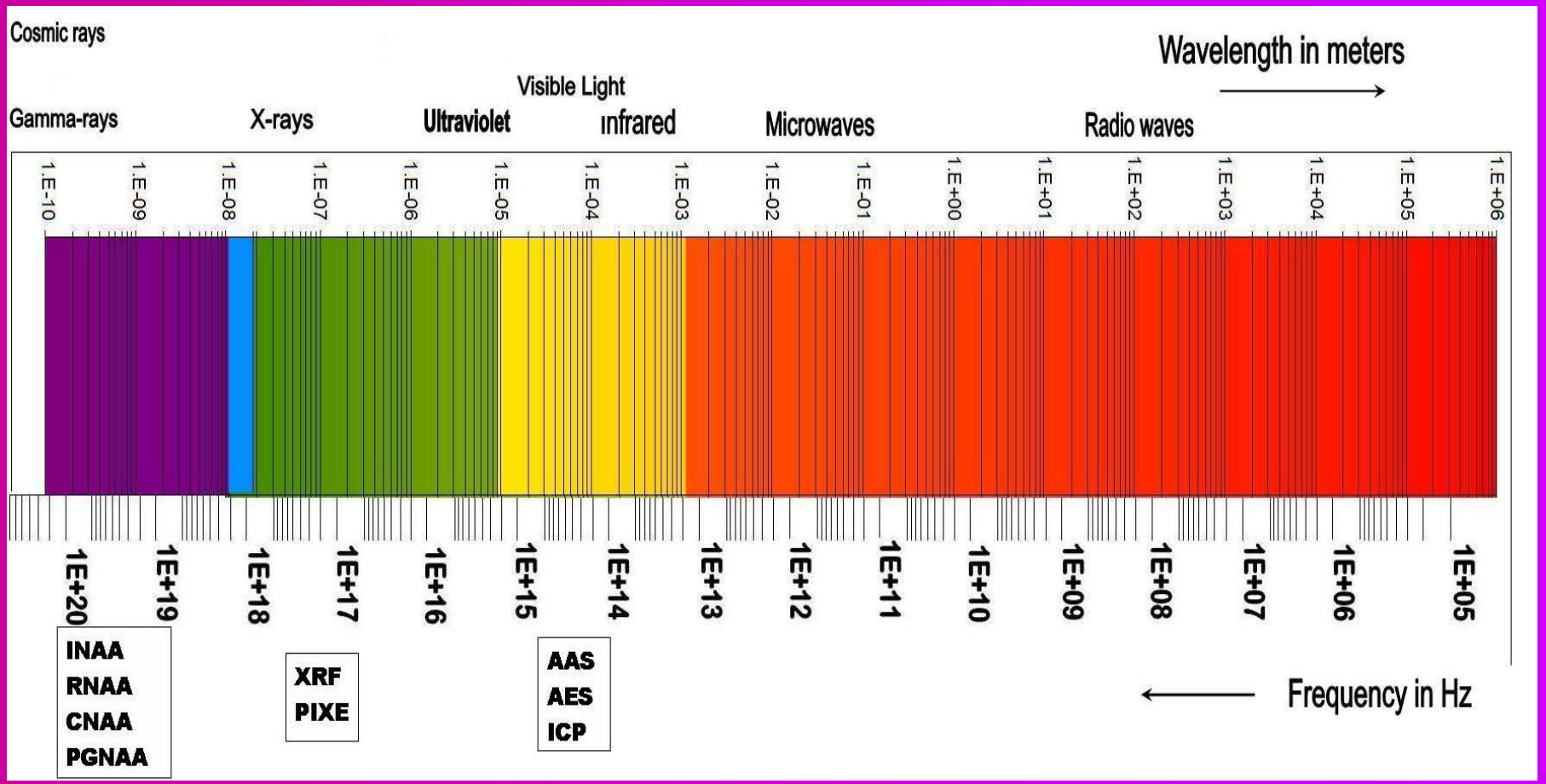


Figure 2. Calibration curve for quantitative analysis

Figure 3.
Electromagnetic Spectrum and Spectroscopic Techniques



Based on Figure 3.1 , pp 78, A Handbook of Silicate Rock Analysis, P. J. Potts.

Spectroscopic Techniques …

- The different energies of the photons in the electromagnetic spectrum are representative of different types of interactions in the atoms and molecules; and are detected and measured by different types of spectroscopic techniques.
- Microwave and infrared spectroscopy use the properties of molecular **rotations and vibrations**.
- Ultra violet and visible light spectroscopy utilize **absorption** and **emission** of energies of outer electron transitions.
- X-ray **fluorescence** – inner electrons
- Gamma rays – **nuclear transitions**.

Figure 4A.
Pictorial depiction of Atomic Nucleus – Electron Orbitals

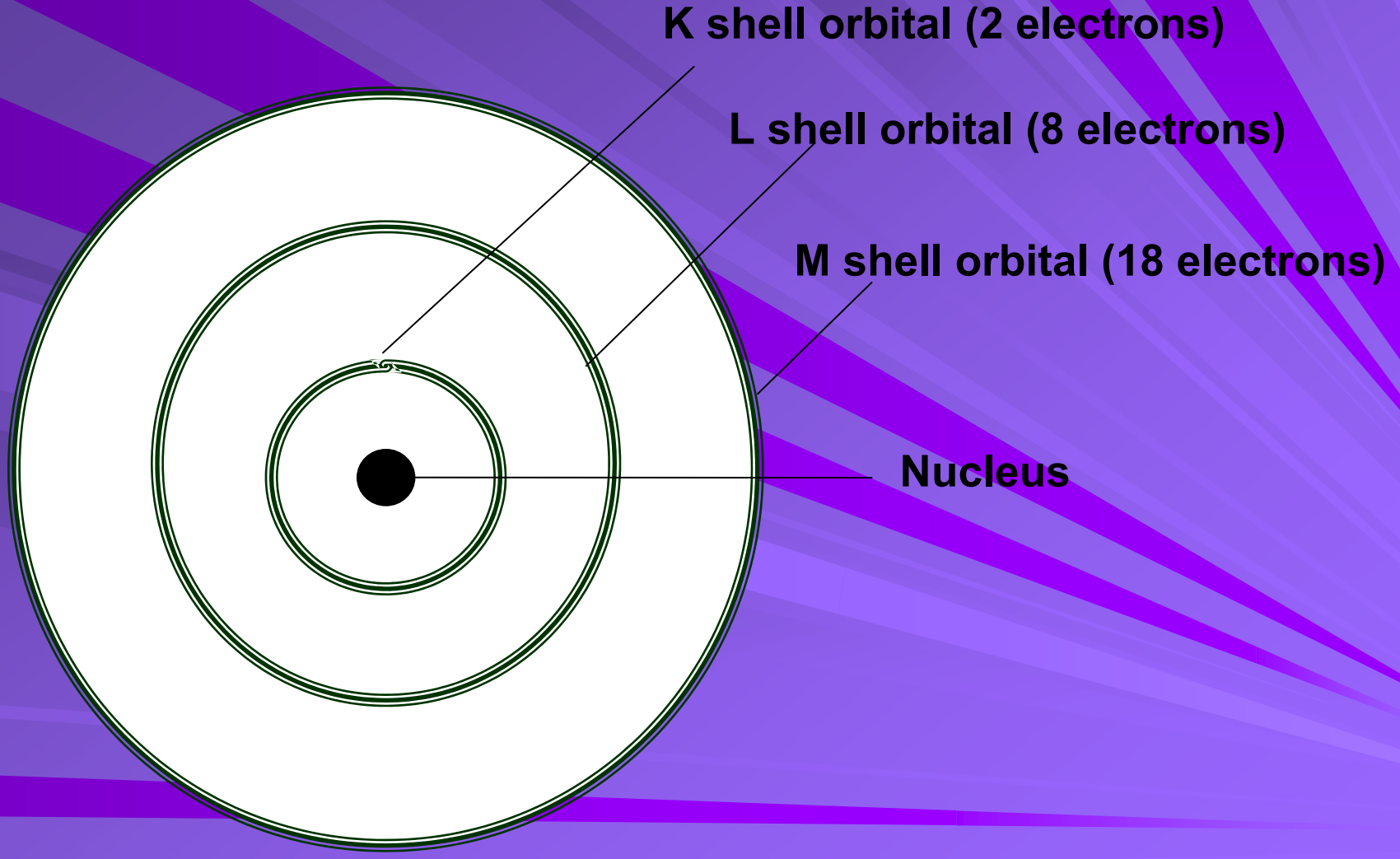


Figure 4B.
Atomic Absorption and Emission

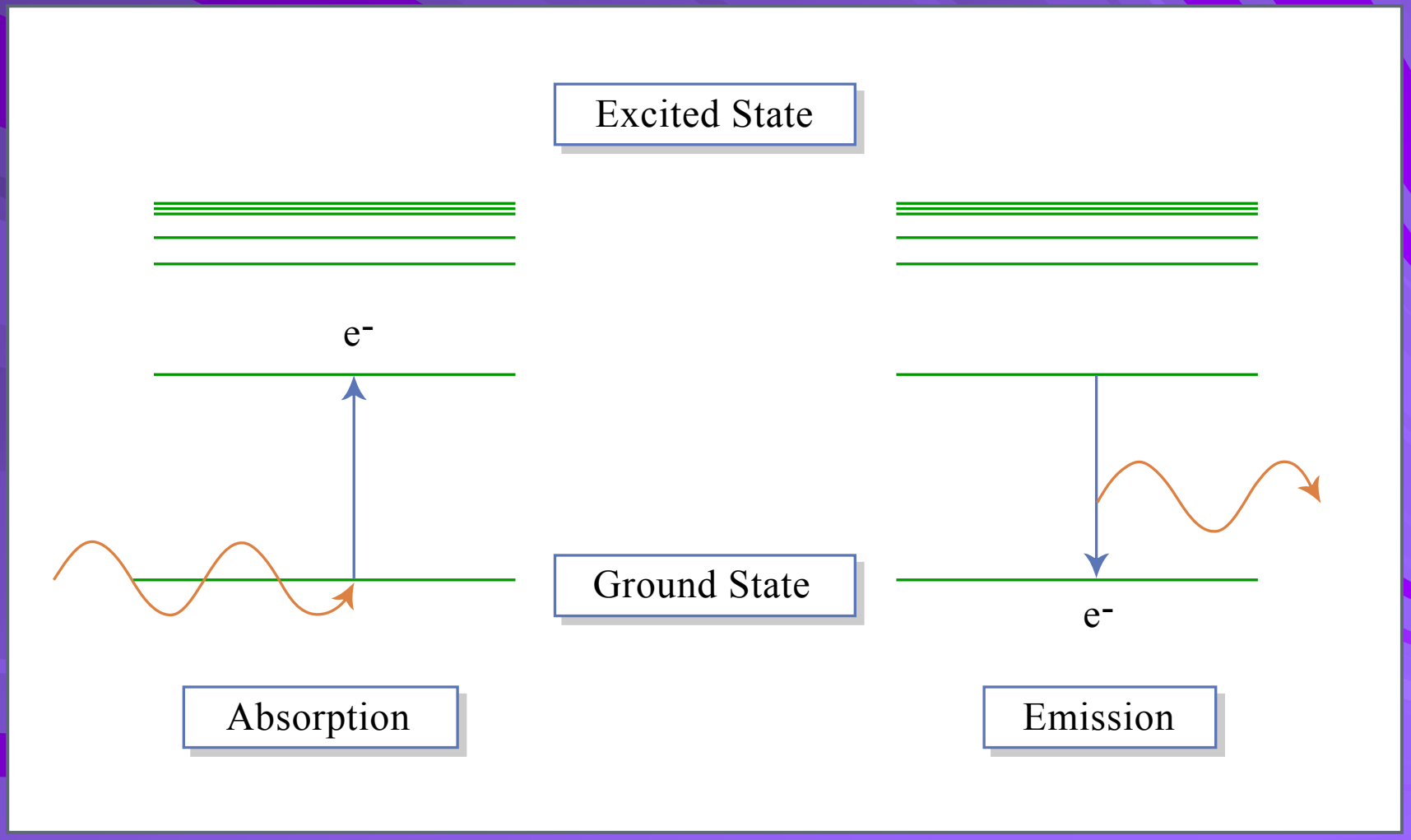
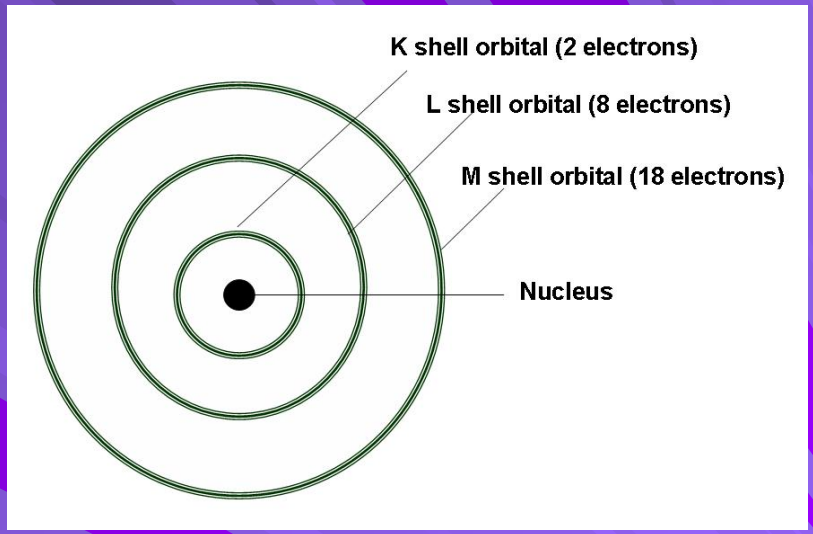


Figure by MIT OCW.

Atomic Spectroscopy
Atomic Absorption and Atomic Emission



Principles:

Atomic spectra are generated by transitions of electrons from one discrete orbital to another in an atom .

The difference in energy between respective orbitals corresponds to the energy of the electromagnetic radiation in the UV-Visible region.

Two processes, namely, absorption and emission provide analytical capability.

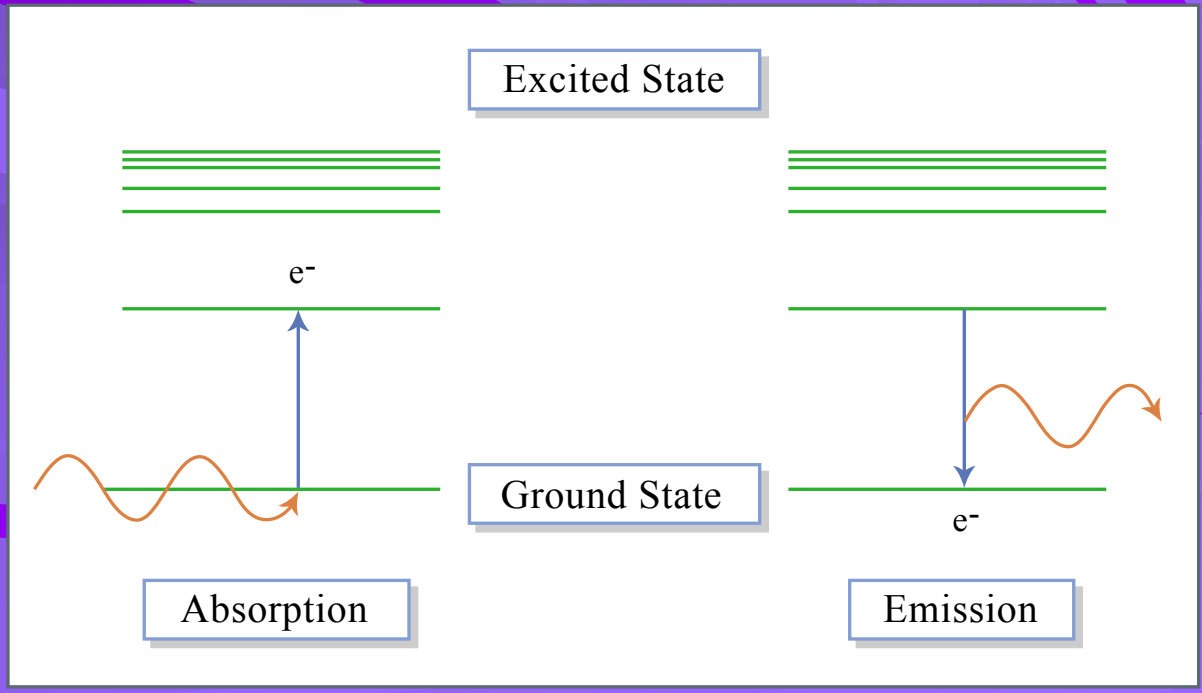


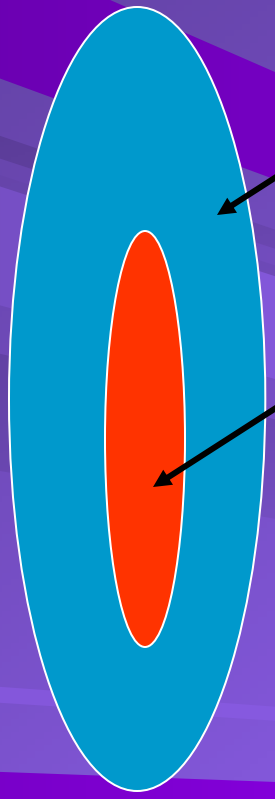
Figure by MIT OCW.

Atomic Absorption Technique

- This technique was developed out of the phenomenon – observation of the spectral lines of solar radiation.
- The understanding of this observation is that (the spectral lines) the observed spectrum is due to the **absorption of light** in the **atomic vapor** in the Sun's atmosphere. - Discovery in the 1925s.
- Strong **absorption of optical radiation** by atoms of an element could be **induced** if the sample were **excited** by the **atomic radiation** of that element.

Atomic Absorption …

- Simple explanation



Induced radiation of the element, excites the sample material, causing excitement of the electrons of the specific element from lower to higher orbitals.

Absorbs radiation from the sample

Atomic Absorption Spectroscopy

Principle:

The sample material is excited by electromagnetic radiation causing the excitation of the electrons from lower orbital state to higher. The intensity of absorbed light is proportional to the concentration of the element in the sample material.

Hence the intensity of the inducing incident light radiation must be exactly the same as the energy difference of the orbitals.

Hence the requirement for a hollow cathode lamp – that enables the atomized sample material to be excited with an atomic line spectrum of precise wavelength.

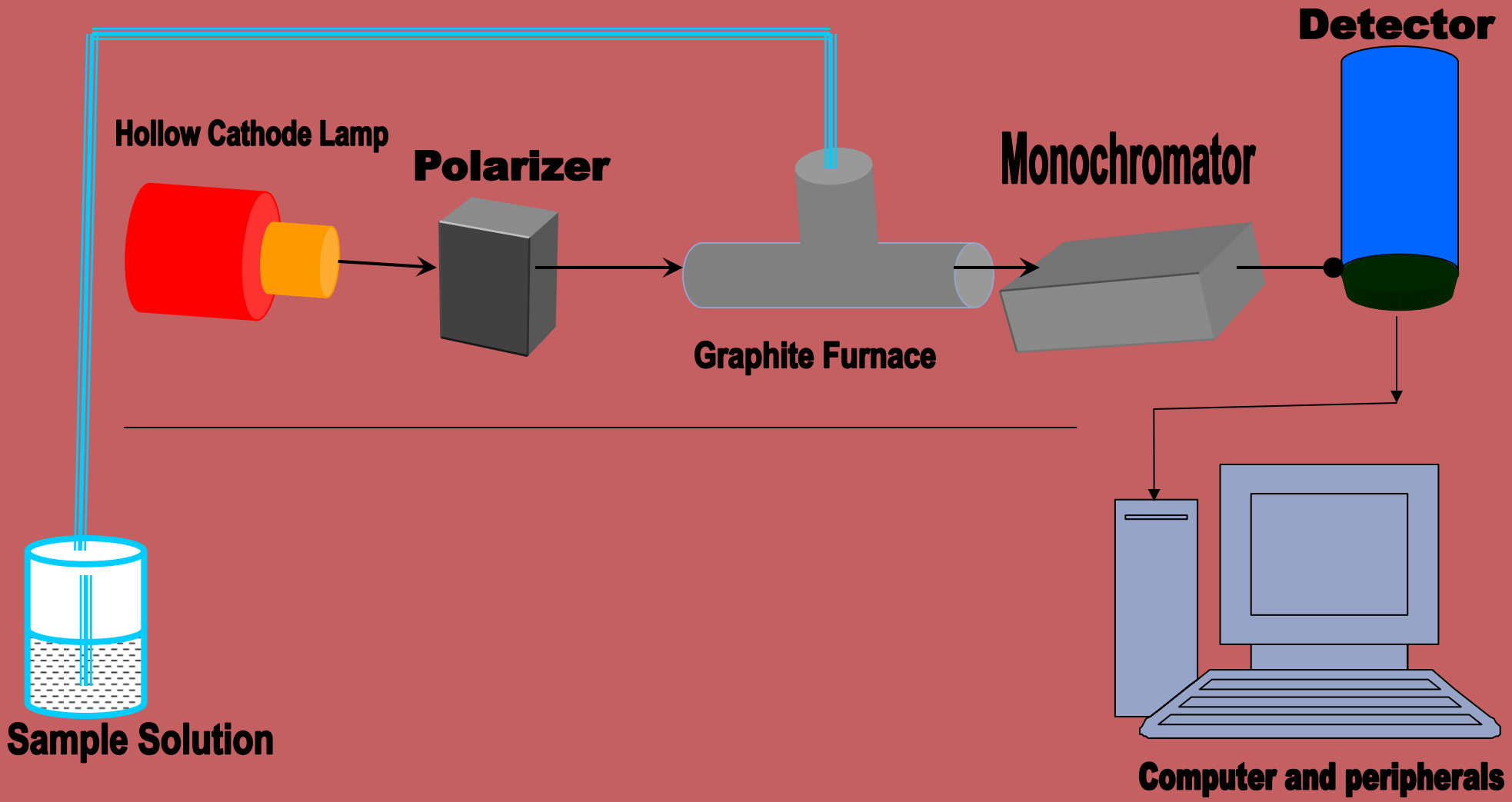
Flame Atomic Absorption Spectroscopy (FAAS) and **Graphite Furnace Atomic Absorption Spectroscopy (GFAAS)** have similar measurement technique, but differ in sample injection and atomization.

Atomic Absorption Spectrometer

An atomic absorption spectrometer consists of:

- Atomic Light Source:Hollow cathode tube or electrodeless discharge lamp
- Nebulizer for making the solution into aersols
- Atomizer for atomizing the aerosols
- Monochromator: To disperse incident polychromatic radiation into constituent wavelengths.
- Photomultiplier detector
- Read out system: Computer and peripherals

Figure 5. Schematic of Graphite Furnace Atomic Absorption Spectrometer



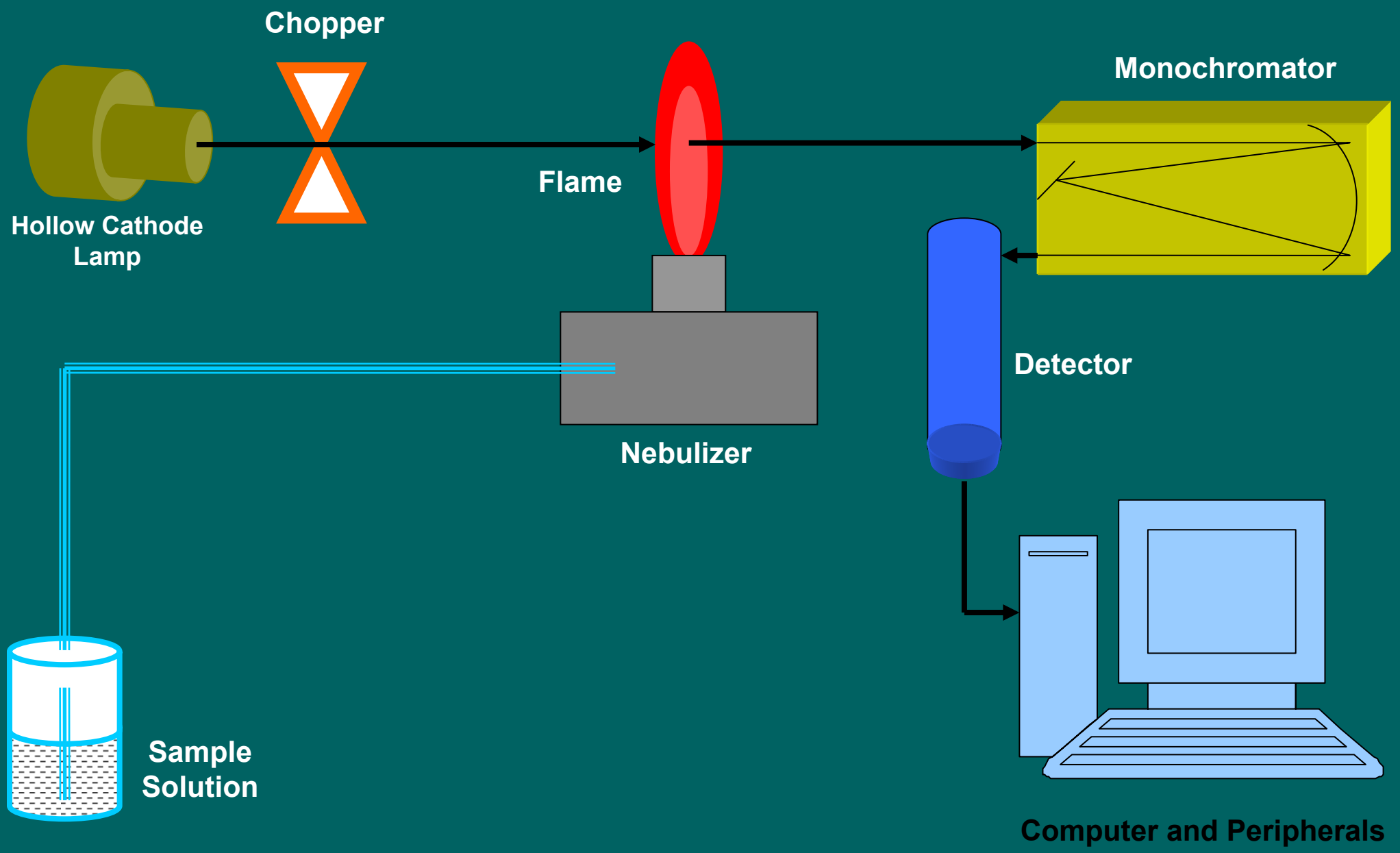
Flame Atomic Absorption Spectroscopy

The sample solution is sprayed into the flame by the nebulizer. The flame is made from the Air-Acetylene or Nitrous Oxide-Acetylene gas torch. The hollow cathode lamp consists of the filament of the element to be analyzed and is filled with argon or neon gas.

High voltage is applied to the lamp to generate the characteristic radiation which is isolated from the radiation from the flame by a chopper.

The detector consists of a photomultiplier tube which converts the incident EM radiation energy into an electrical signal.

Figure 6. Schematic of Flame Atomic Absorption Spectrometer



Atomic Absorption Spectrometry

E.g.: Absorption Lines

Element	Wavelength nm
As	228.812
Cu	324.754
Iron	271.903
Iron	279.470
Iron	352.414

Atomic Emission Spectroscopy

Principles:

Atomic emission is induced when some external source of energy such as an argon plasma is utilized to provoke the electron excitement transitions. When the excited electrons de-excite to the ground or lower state orbitals; the released energy is the intensity of the emission radiation.

Other sources: Arc-Spark

Inductively Coupled Plasma Atomic Emission Spectroscopy

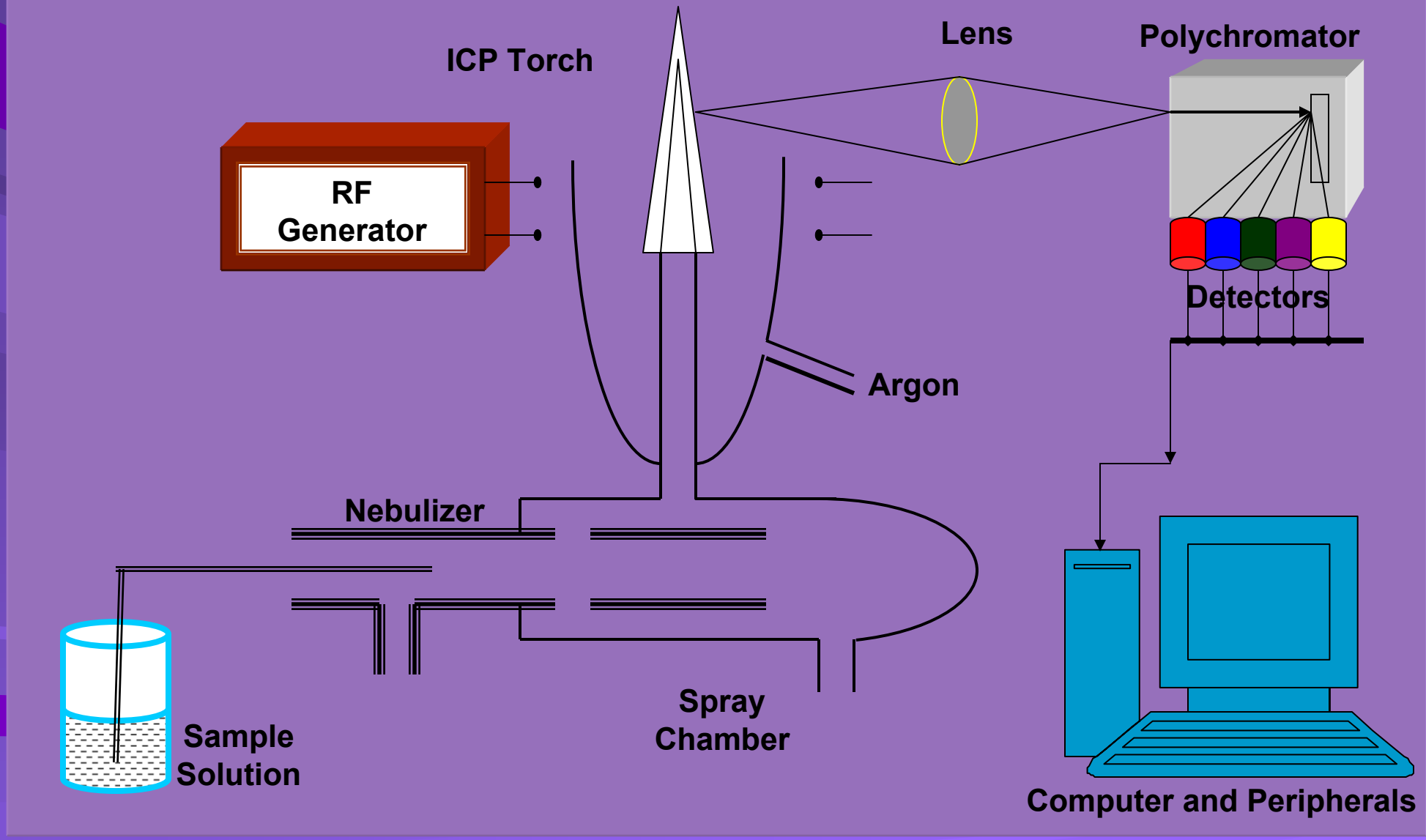
Principle:

The sample aerosol is ‘heated’ in a plasma. The plasma is an ionized argon gas at high temperatures (6000K -10,000K).

The plasma, at these high temperatures , excites the atoms of the sample aerosol and there by emitting EM radiation of characteristic wavelengths of different elements.

This is thus a multi-element analytical technique.

Figure 7. Schematic of Inductively Coupled Plasma Atomic Emission Spectrometer



ICPAES

E.g.: Emission lines

Element	wavelength nm
As	193.696
Cu	324.724
Iron	259.940

Analysis of liquids by Inductively Coupled Plasma Mass (ICPMS) Spectroscopy

ICPMS technique is useful for multi-element analysis of geological, environmental and medical sample materials.

ICPMS provides information about the abundances as well as isotopic ratios of the nuclides.

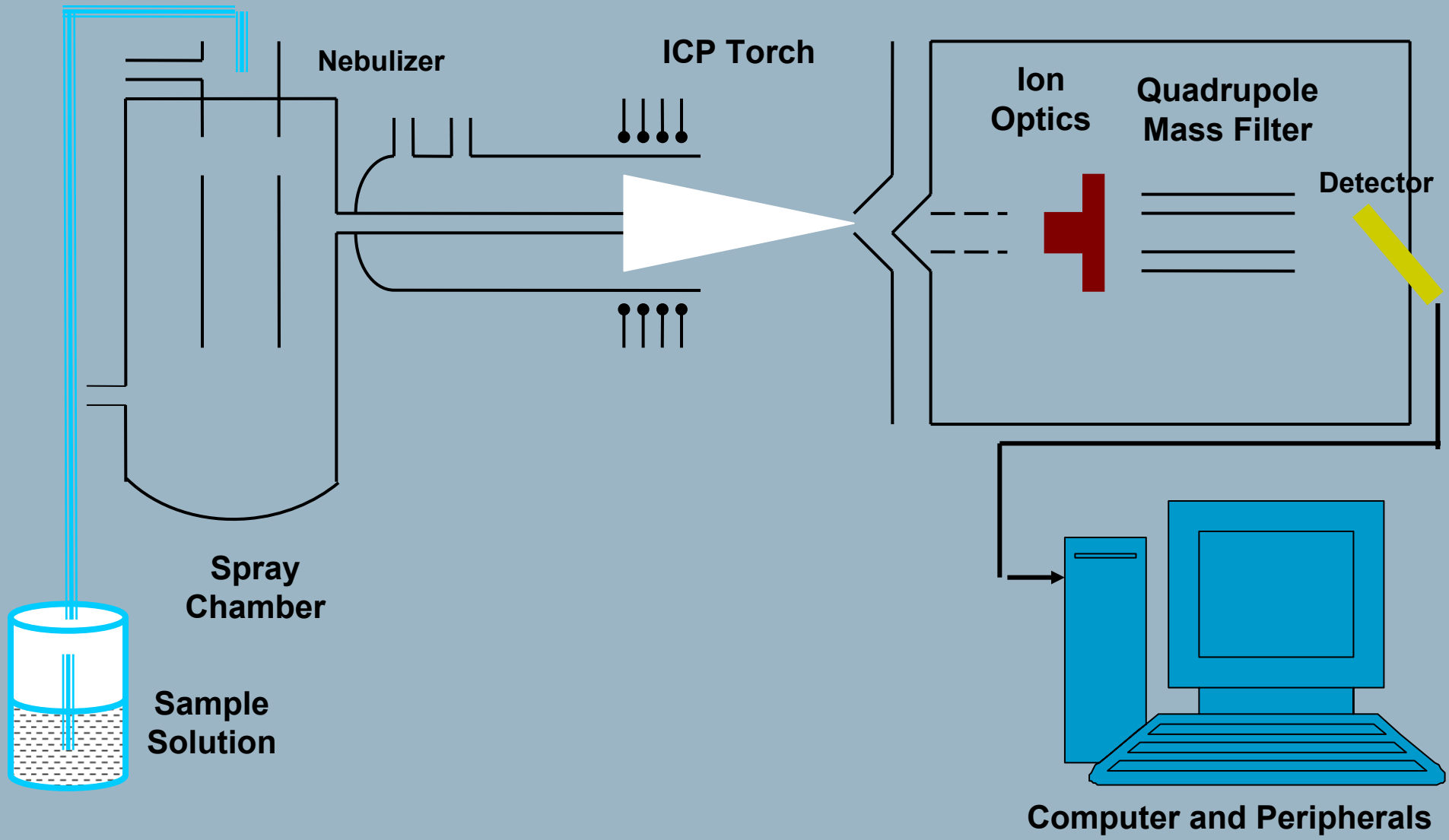
Inductively Coupled Plasma Mass Spectrometer

Principle:

- The ICPMS technique consists of a high temperature plasma, into which the sample aerosol is injected and positively charged ions are generated by the interaction.

- A mass spectrometer quantifies the ionization based on the mass to charge ratio.

Figure 8.
Schematic of Inductively Coupled Plasma Mass Spectrometer



Analysis of Solids by Neutron Activation Analysis (NAA) and Gamma Spectroscopy

Principle:

Neutron Activation Analysis is a nuclear analytical technique that involves irradiating a sample with neutrons. The stable isotopes of different elements in the sample become radioactive. The radioactivity of different radionuclides can be detected and quantified by gamma spectroscopy.

Neutron Activation Analysis …

- A stable isotope when bombarded with neutrons, absorbs a neutron; and by the most common type of nuclear reaction, namely, (n, gamma) reaction, gets transformed into higher mass unstable nucleus.

$$ {}^{A}_{Z}X \; (n,\gamma) \; {}^{A}_{N}X^{*} \longrightarrow {}^{A+1}_{Z}X \quad ({}^{A}_{N}X^{*}: \text{unstable}) $$

- When the unstable nucleus de-excites by prompt gamma rays, and gets transformed into a radioactive nucleus (with next higher neutron number). This radioactive nucleus decays mainly by beta rays and (or) characteristic gamma-rays.

Neutron Activation Analysis …

Nuclear Reaction

Nuclear reaction occurs when target nuclei are bombarded with nuclear particles, depicted pictorially

$$X + a \longrightarrow Y + b + Q$$

Or

X(a,b)Y

Target X is bombarded by particle "a",

Y is the product nuclei with resulting particle "b" .

Q is the energy of the nuclear reaction, which is the difference between the masses of the reactants and the products.

Ex:

$$^{59}_{27}Co + {}^{32}_{0}n \longrightarrow {}^{1}_{0}\gamma + {}^{60}_{27}Co$$

or

$$^{59}_{27}Co\ (n, \gamma)\ ^{60}_{27}Co$$

Neutron Activation Analysis…

- **1)Neutron capture:**

  The target nucleus absorbs (captures) a neutron resulting in a product isotope, the mass number of which is incremented by one. If the product nucleus is unstable, it usually de-excites by emission of gamma rays and/or β.

- **Ex:**

$$^{58}_{26}\mathrm{Fe} \quad (n, \gamma) \quad ^{59}_{26}\mathrm{Fe} \quad .$$

Gamma Spectrometer

- An irradiated material is radioactive emitting radiations – α, β, γ, ……
- For Neutron Activation Analysis – usually gamma radiation is selected.
- **Gamma spectrometer** is the detection system that measures gamma ray intensity.

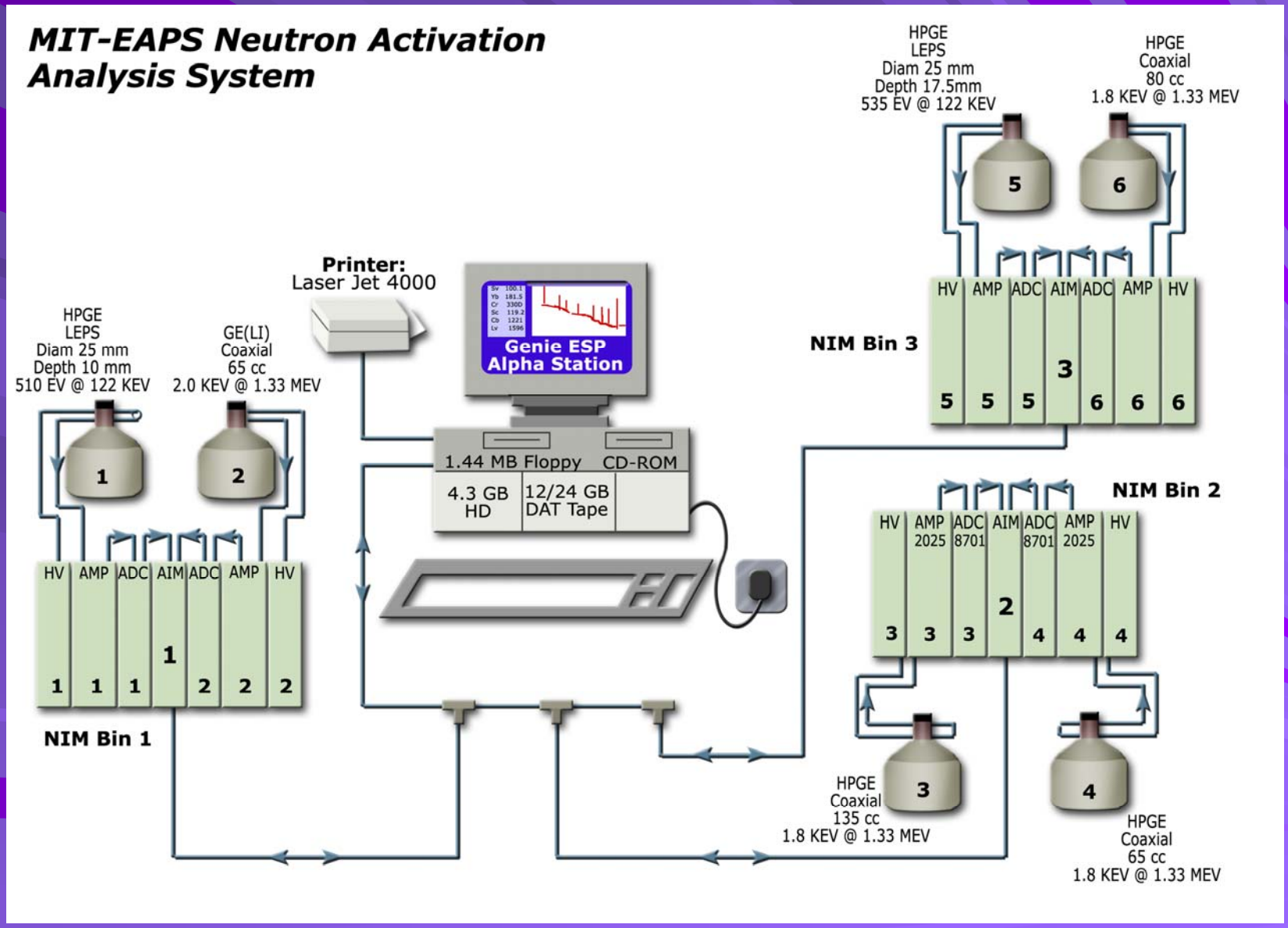
Gamma Spectrometer

Gamma spectrometer system for measuring the gamma-ray activity of an irradiated material consists, typically, of

1) Detector
2) Amplifier
3) Multi Channel Analyzer
4) Computer & peripherals

This is shown pictorially:

Figure 9. Schematic of Gamma Spectrometer



Gamma detector…

The energy of nuclear radiation is converted into an electrical signal by a device that is the nuclear radiation detector.

The three major categories of gamma detectors used in Neutron Activation Analysis are:

1)Scintillators : **NaI(Tl), CsF, ZnS(Ag)**

2)Semiconductors : Si, Ge, CdTe, GaAs

3)Gas Filled : He, Air, H_2, N_2

Gamma detector…

- The nuclear radiations emanating from the irradiated material will cause ionization in the detector medium by means of charged particle products of their interactions.

- The scintillators and the semiconductors have energy discrimination capacity better than the gas filled detectors.

Gamma detectors…

The nuclear radiations incident on the detector crystal initiate ionizations by creation of electrons (negative charge) and holes (positive charge).

An electric field is created by applying high voltage to the electrodes mounted on opposite sides of the detector crystal. The charge carriers get attracted to the electrodes of opposite polarity because of the electric field. The charge collected at the electrodes is proportional to the energy lost by the incident radiation.

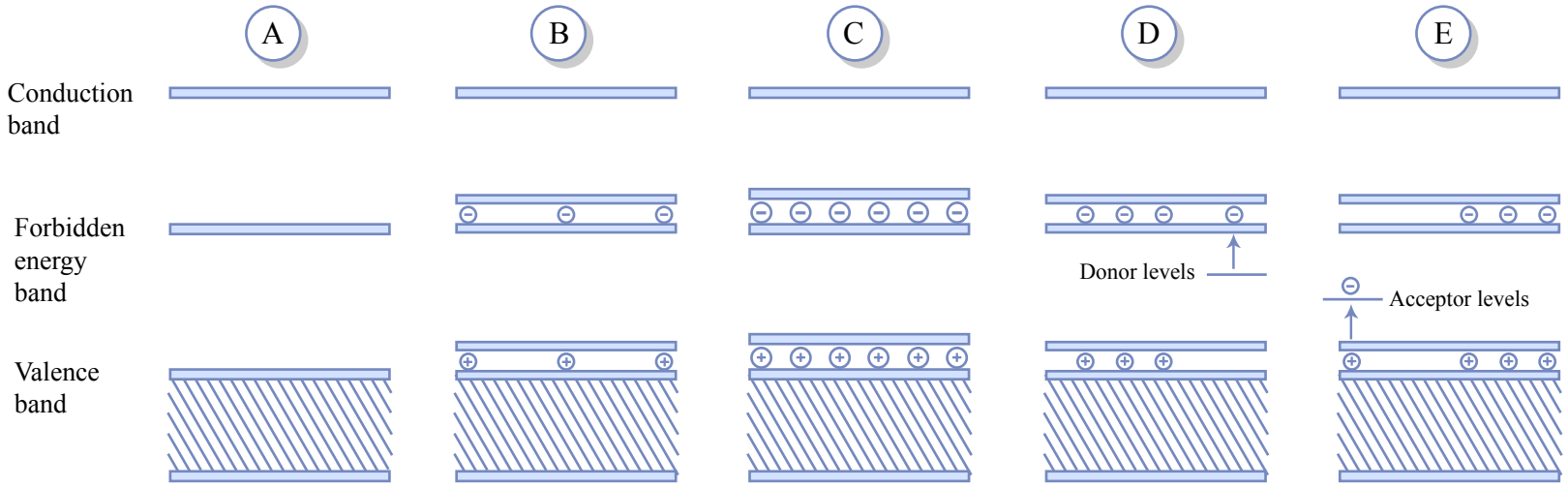
Chapter IV : Instrumentation in neutron activation analysis by P. Jagam and G. K. Muecke p 77, Figure 4.3

Mineralogical Association of Canada. Short Course in Neutron Activation Analysis in the Geosciences, Halifax May 1980, Ed: G. K. Muecke.

Figure 10.
Schematic diagram of conduction and forbidden bands of a semiconductor detector crystal

Neutron Activation Analysis

Properties of semiconductor materials



KEY:- Shading indicates valence band fully occupied by electrons. Arrows indicate direction of ionization of electrons to or from impurity atoms.

Schematic behaviour of a semiconductor crystal

A: Perfect (intrinsic) semi-conductor at 0 K, the valence band is fully occupied by electrons, and the conduction band is empty, in this state the semiconductor cannot conduct.

B: Semiconductor at 77 K; vast reduction in thermal ionization to conduction band.

C: Semiconductor at room temperature; significant thermal excitation of electrons from valence to conduction band; in this state the semiconductor will conduct.

D: Effect of 'donor' atom impurities in *n*-type semiconductor material.

E: Effect of 'acceptor' atom impurities in *p*-type semiconductor material.

Reference: A Handbook of Silicate Rock Analysis by P. J. Potts, Blackie Chapman and Hall New York page 406 Figure 12.7

Interaction of gamma radiation with matter

- **Photoelectric effect** is the interaction between the incident gamma-ray and orbital electron of the atom of the detector crystal. The energy of the gamma-ray is completely transferred to the electron. The electron overcomes the ionization potential by utilizing part of the transferred energy, the remainder becomes the kinetic energy of the electron. Photoelectric interaction predominantly takes place with orbital shells close to the nucleus (K-shell). The vacancy created by the ionized electron gets filled by an electron falling from the next higher shell simultaneously emitting the characteristic K X rays of Ge. Thus characteristic X rays of the detector material are emitted when photoelectric interaction takes place.

Figure 11.
Schematic depiction of Photo Electric Effect

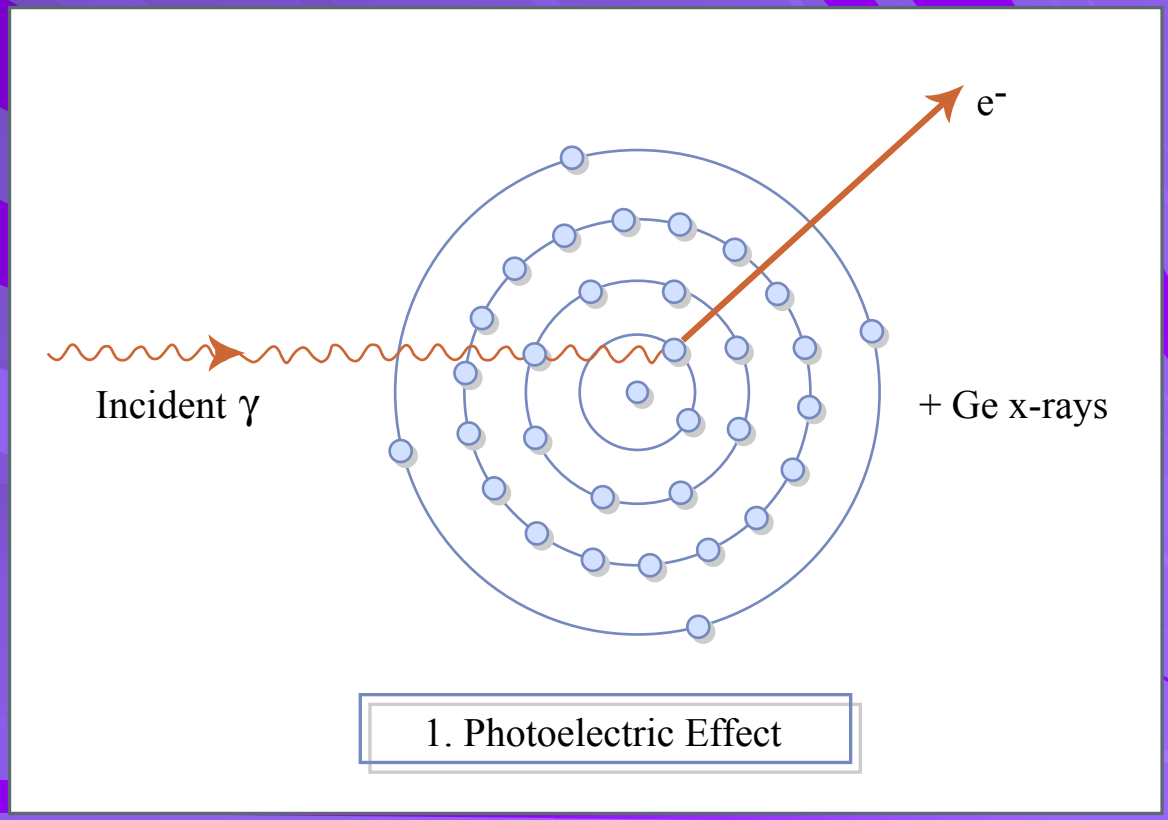


Figure by MIT OCW.

Interaction of gamma radiation with matter…

Compton scattering is the interaction between the incident gamma ray and an outer orbital electron in which only part of the gamma energy is transferred to the electron and the the remainder is reirradiated as a lower energy gamma ray (scattered inelastically) preserving the total energy and momentum. In a head-on collison maximum transfer of energy occurs following which the secondary gamma ray is emitted at 180 to the first. The secondary gamma photon can itself interact by further compton or photoelectric interactions. However, there is a probability that this gamma will itself escape from the detector. Compton scattering in the detector is the main cause of the high background contnuum below the energies of the principal gamma photo peaks recorded on Ge detectors.

Figure 12.
Schematic depiction of Compton Scattering

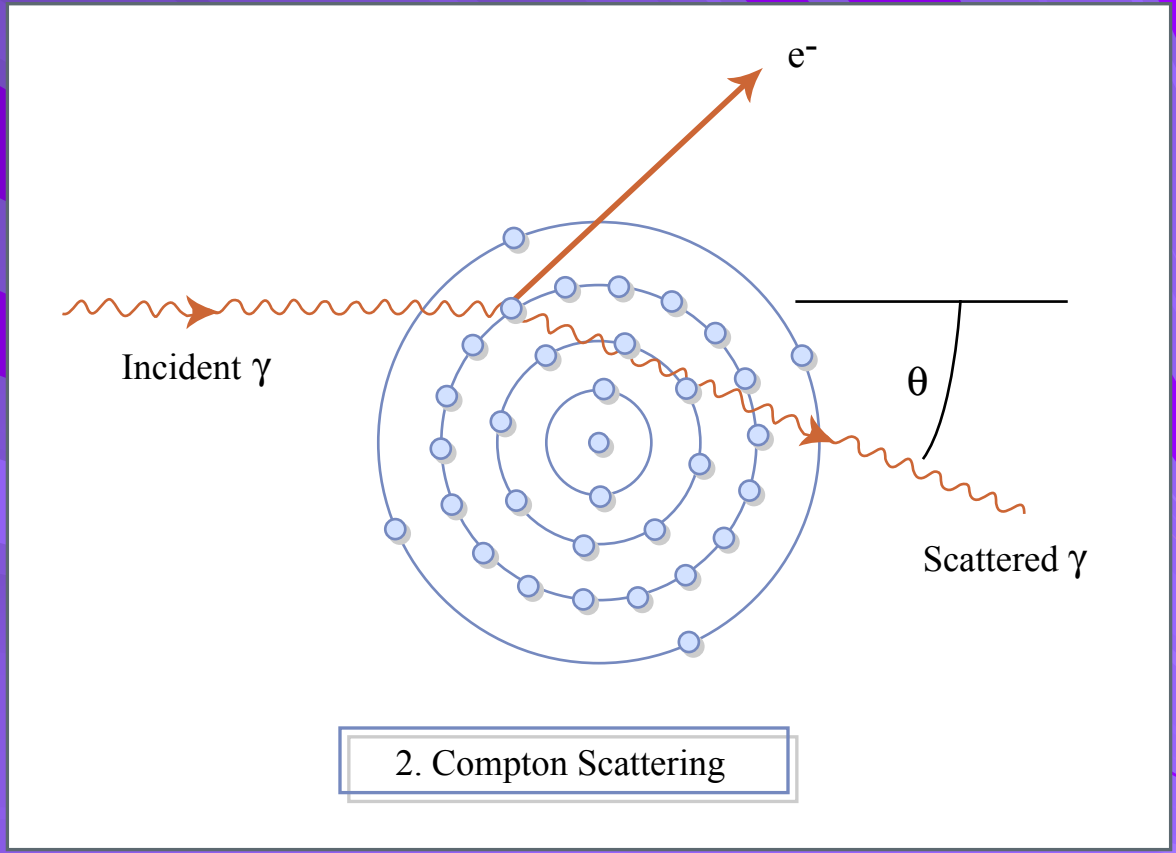


Figure by MIT OCW.

Interaction of gamma radiation with matter…

Pair production interaction becomes significant when incident gamma ray energies exceed 1.022 MeV. The interaction of the incident gamma-ray in the strong electromagnetic field surrounding the nucleus results in complete transmutation of gamma photon energy into an electron – positron pair. The particles, which are very short lived, lose their kinetic energy very quickly, by further collison with the atoms of the detector and then spontaneously annihilate to generate two 511 keV gamma –rays emitted at 180 degrees to one another.

Figure 13.
Schematic diagram of Pair Production

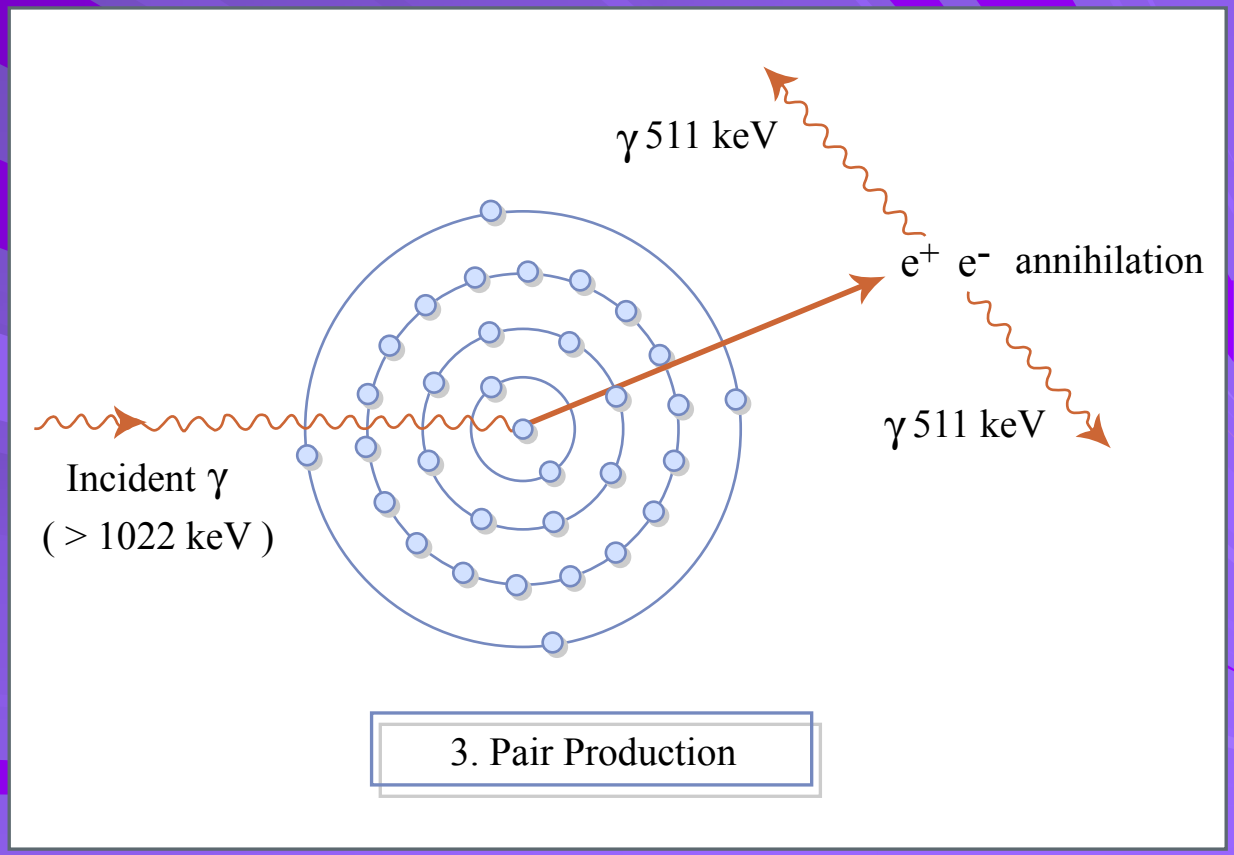


Figure by MIT OCW.

Interaction of gamma radiation with matter…

Bremsstrahlung

- **Bremsstrahlung continuum** radiation is also created in the detector by the deceleration of energetic electrons within the electrostatic fields surrounding the nucleus.
- Bremsstrahlung radiation can randomly contribute to the continuum spectrum.

Figure 14.
Schematic diagram of Bremsstrahlung interaction

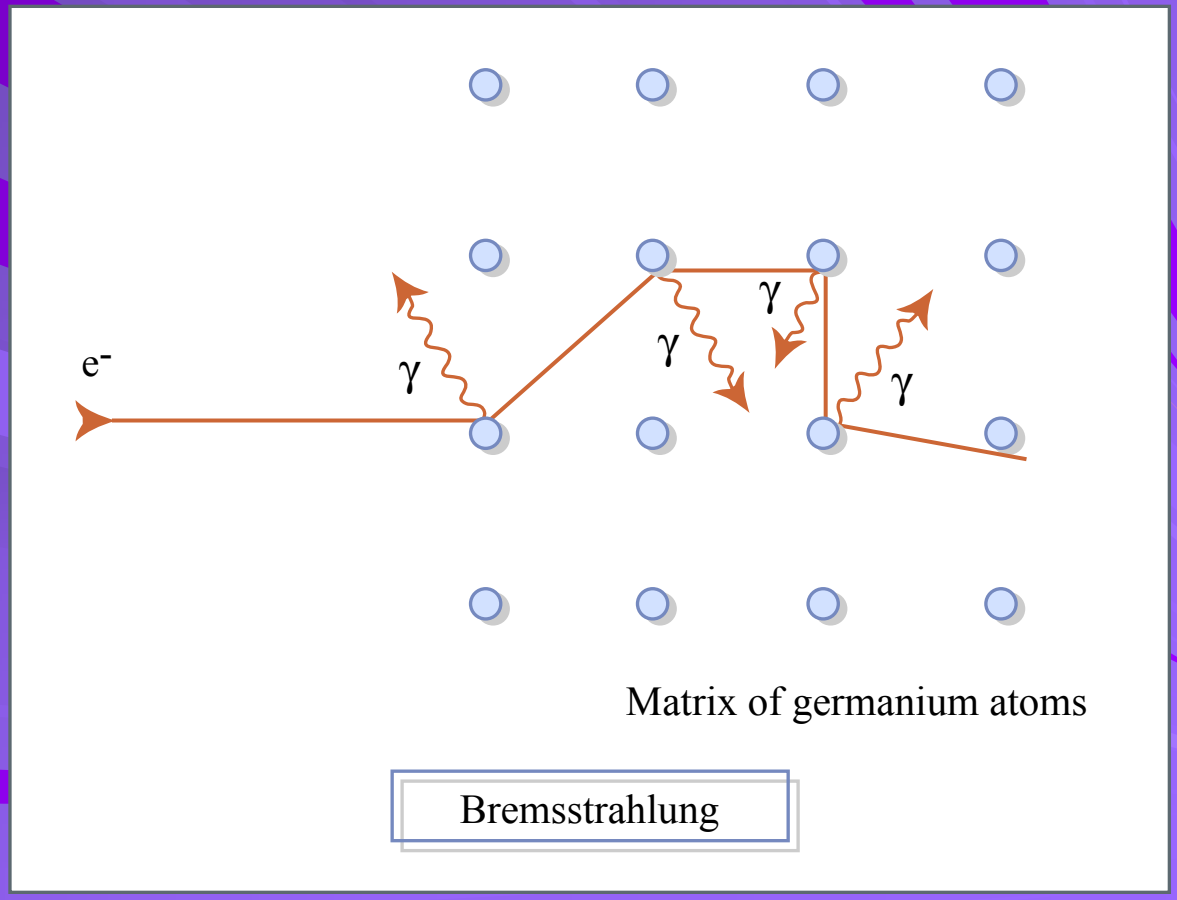
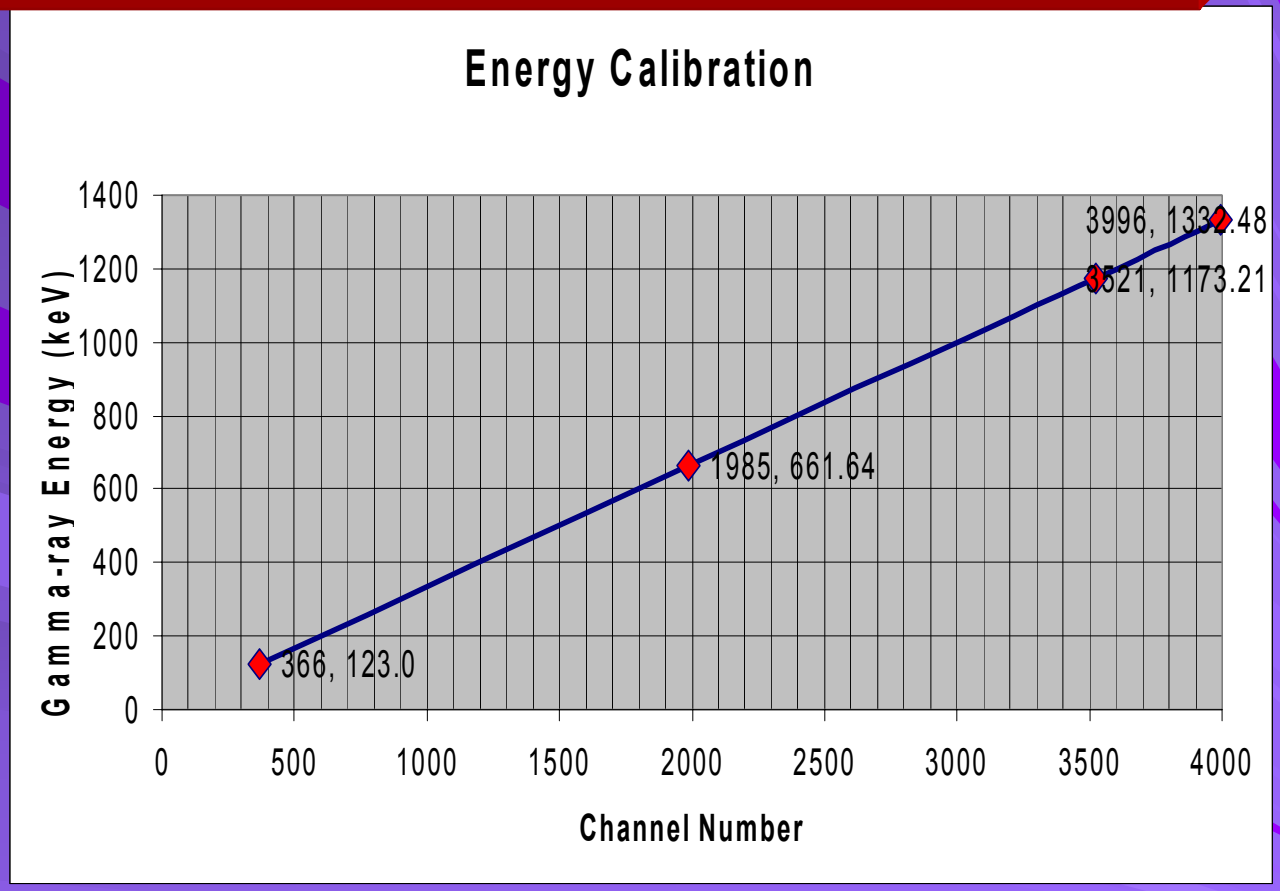


Figure by MIT OCW.

Figure 15.
Energy Calibration of a Gamma Spectrometer using Standard Calibration Sources

Source	Gamma-ray Energy keV	Channel Number
^{57}Co	123.0	366
^{137}Cs	661.64	1985
^{60}Co	1173.21	3521
	1332.48	3996



Gamma Spectrum - Multielement

Reference:
Multielement analysis of food spices by instrumental neutron activation analysis,
P. Ila and P. Jagam,
Journal of Radioanalytical and Nuclear Chemistry, 57 (1980) 205-210.

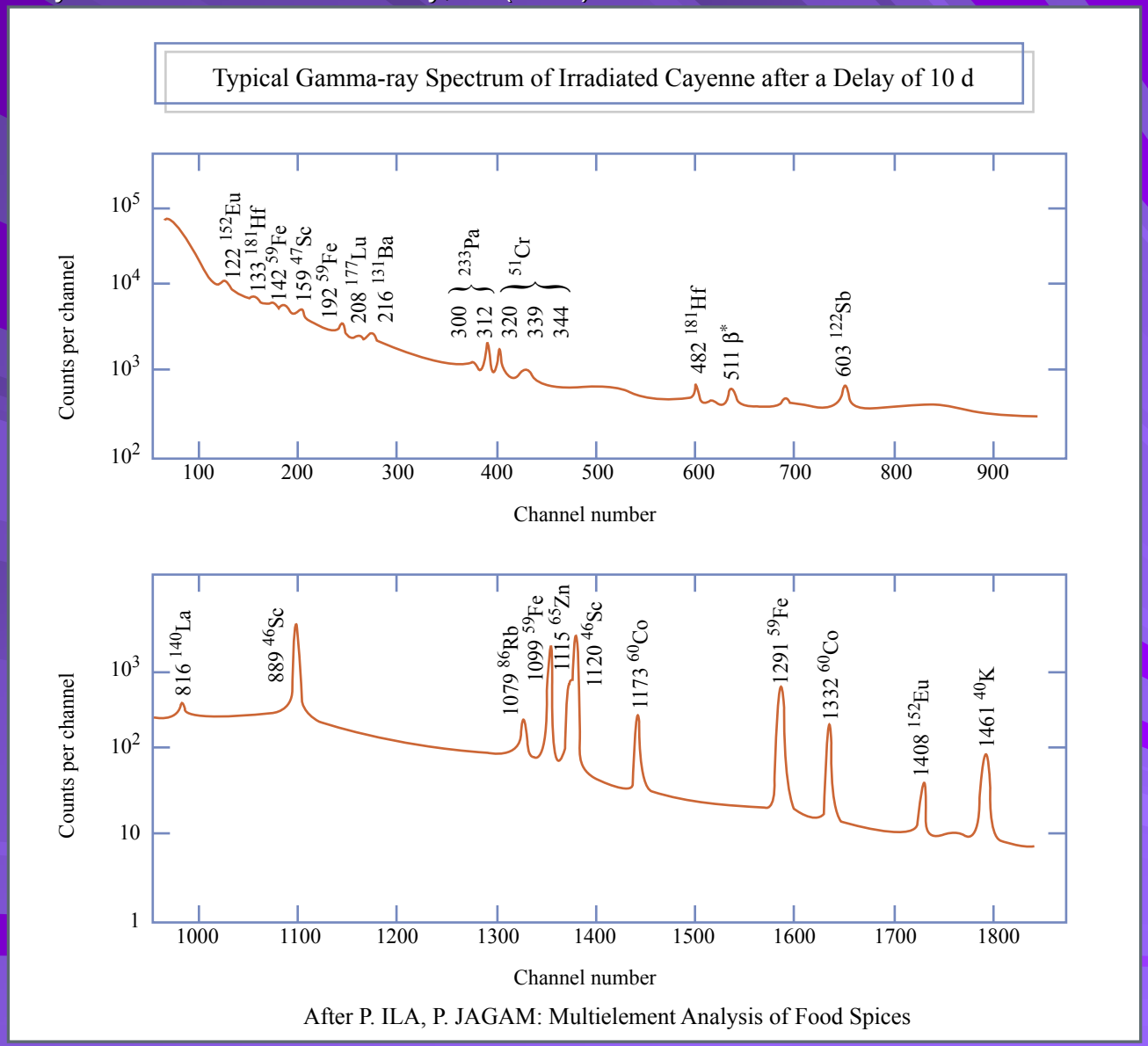


Figure 16. Multi-element gamma-ray spectrum of a food material

Interaction of gamma radiation with matter

Gamma radiation interacts with matter causing ionization in matter by three principal processes:

1)Photoelectric effect
2)Compton scattering
3)Pair production

Reference: Chapter 12.6 Interaction of gamma-radiation with Ge detectors, A Handbook of Silicate Rock Analysis by P. J. Potts, Blackie Chapman and Hall New York page 412, Figure 12.17

Activity Equation

A = number of decays per second (Activity) dps

N = number of atoms of the target isotope

$$= \frac{m}{W} \times q \times 6.023 \times 10^{23}$$

m = mass of the element in the irradiated sample g

θ = isotopic abundance

w = Atomic weight of the element

λ = decay constant = $0.693/t_{1/2}$

$t_{1/2}$ = Half-life of the isotope

ϕ = neutron flux $n.cm^{-2}.sec^{-1}$

σ = activation cross-section 10^{-24} cm^2

t_{irr} = irradiation time sec

Activity Equation …

$A = N \sigma \phi [1 - \exp(-\lambda t_{irr})]$

After a delay of time t_d

$A = N \sigma \phi [1 - \exp(-\lambda t_{irr})] \exp(-\lambda t_d)$

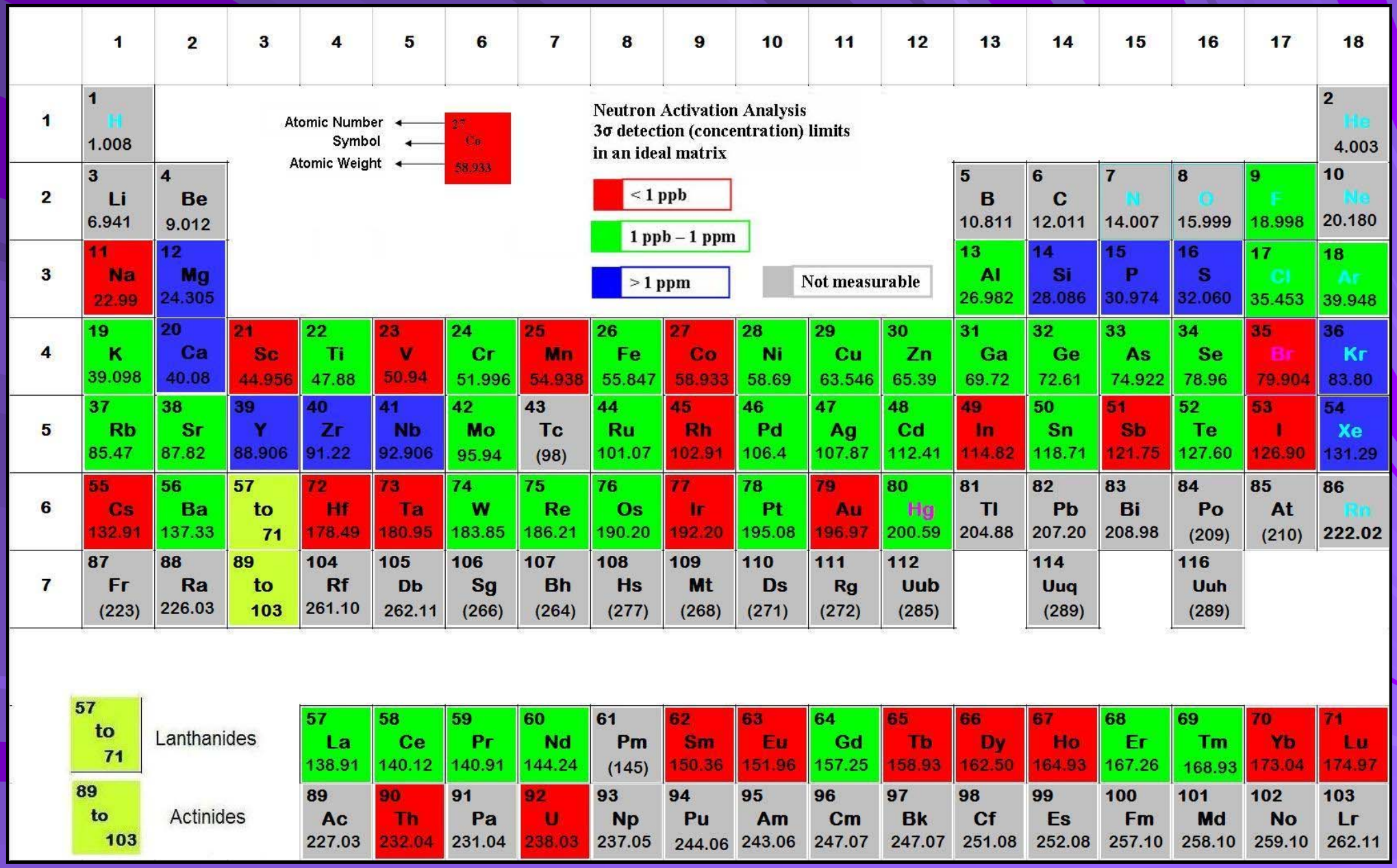
For a counting time of t_c

$A = N \sigma \phi [1 - \exp(-\lambda t_{irr})] \exp(-\lambda t_d) \ [1 - \exp(-\lambda t_c)]$

Neutron Activation Analysis by comparator method

- AStandard = Activity of an isotope of an element in the known (Standard) is proportional to the amount present.
- ASample = Activity of the isotope of the same element in the unknown (Sample)
- AmountStandard/ AmountSample = AStandard / ASample

Figure 17. Trace element abundance determination by Neutron Activation Analysis of different elements



Based on: Neutron Activation Analysis, Modern Analytical Geochemistry, pp 116-135.

Conclusion

Neutron Activation Analysis:

1. **Nuclear technique that measures the intensity of gamma rays of " characteristic" energy using gamma spectroscopy.**
2. **Multielement Analysis.**
3. **Rapid analyses of multiple samples.**
4. **Sample size can be variable (typically 1 mg to 1 gm).**

Conclusion …

5. Nondestructive - that is valuable and safe, samples are not destroyed.

6. No Chemical processing; therefore samples are not contaminated during sample preparation, no uncertainty about total dissolution of sample, no need for dilutions of solutions, making the technique valuable and safe. Samples are not destroyed.

7. No need for repeated blank measurements because no memory effects.

8. Gamma ray spectroscopy is largely free from matrix interferences

9. Depending on the sample matrix, elemental concentrations can be determined at parts per million (ppm), parts per billion (ppb) and parts per trillion (ppt) level.

10. Versatile (in use for more than half a century), well established and reliable.

Table 1. Summary of features of Atomic and Nuclear analytical Techniques

Features	FAAS	GFAAS	ICP-AES	ICPMS	INAA
Elements analyzed	60+	50+	70+	75+	70+
Multi-element	No	No	Yes	Yes	Yes
Sample throughput	Fast (<5 elements/sample)	Slow (3-5 minute/element)	Fast multi-element analysis	Fast multi-element analysis	Fast multi-element analysis
Semi-quantitative analysis	No	No	Yes	Yes	Yes
Isotopic analysis	No	No	No	Yes	Yes (some limitations)
Detection limit	Good	Excellent	Very good	Excellent	Excellent
Dynamic range	10^3	10^2	10^5	10^5 - 10^8	Not applicable
Precision	< 1%	< 5%	< 2%	< 3%	< 1 % (based on counting statistics)
Sample volumes	Large	Small	Small	Small	Small - Large
Dissolve solids	< 5%	< 20%	< 20%	< 0.5%	Not necessary
Interferences					
Spectral	Very few	Very few	Many	Few	Few
Chemical	Many	Many	Few	Some	None
Physical	Some	Very few	Very few	Some	None
Memory effect	Yes	Yes	Yes	Yes	No
Blank measurement	Necessary	Necessary	Necessary	Necessary	Not necessary
Method development	Easy	Difficult	Moderately easy	Difficult	Easy
Ease of us	Very Easy	Moderate	Easy	Moderate	Very easy
Capital and running costs	Low	Medium	High	Very high	Very high

Based on: Table VII, pp 716, Essentials of Medical Geology.

Electron Probe Microanalysis

- Electron probe microanalysis technique is useful to analyze the composition of a selected surface area of diameter size of few microns (micron = 0.001 meter = 0.1 cm) of the sample.
- For example in geological materials – can determine
  - composition of individual minerals
  - variation of concentration within a single grain
- For this type of analysis – the samples are to be polished thin sections mounted
  - in a resin block, or
  - glass slide backing.

Based on: pp 326, Handbook of Silicate Rock Analysis, P. J. Potts.

Figure 18. Schematic of Electron Microprobe

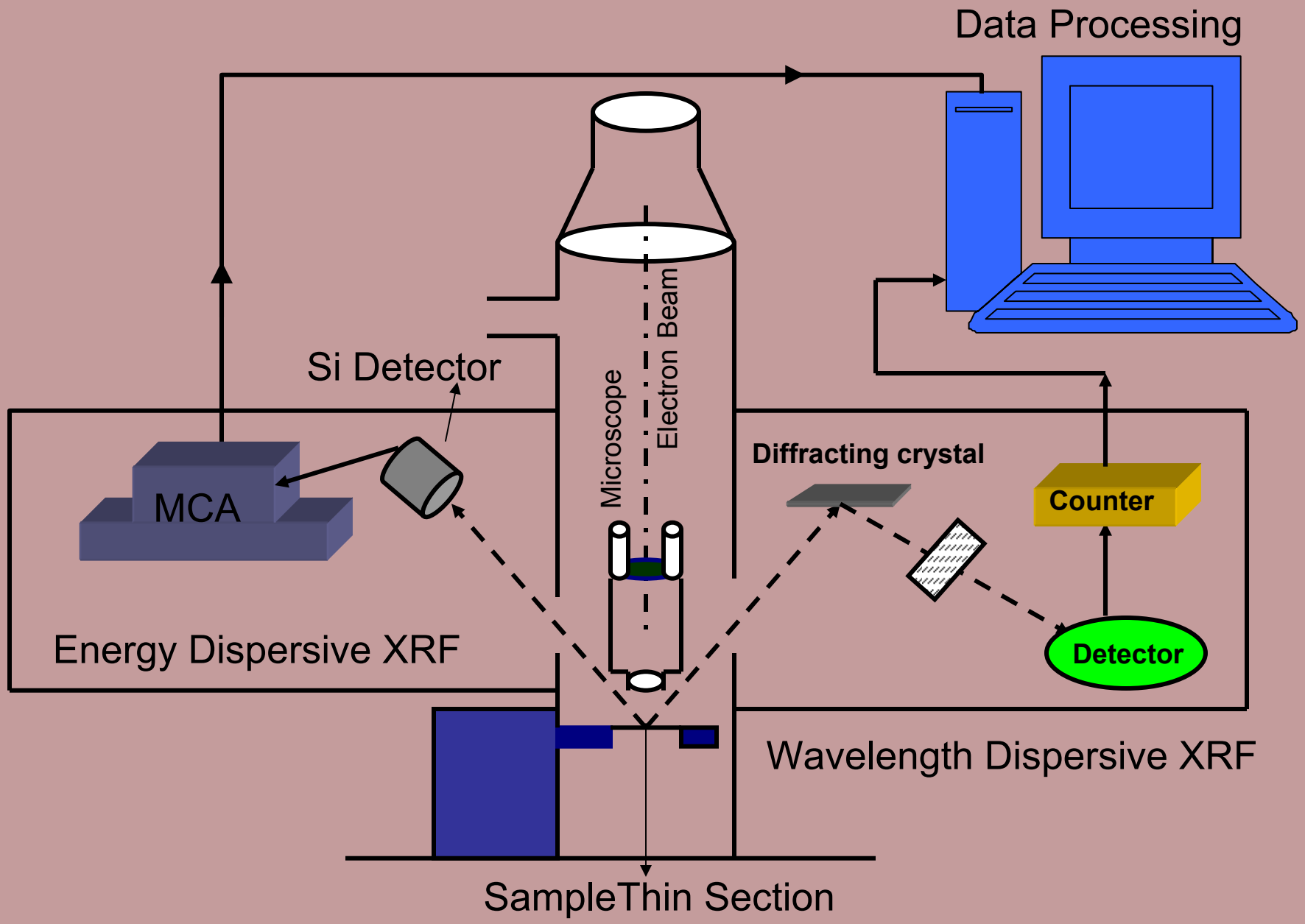


Figure 19.
Wavelength Dispersive XRF (WDXRF)
Energy Dispersive XRF (EDXRF) …

Principles:

- In a stable atom, electrons occupy in discrete energy orbitals; the notation of these orbitals in decreasing binding energy level is K, L, M, … .
- The sample is excited by means electromagnetic radiation generated by radioisotopes, X-ray tubes, charged particles (electrons, protons and alpha particles).
- WDXRF use X-ray tubes
- EDXRF uses both X-ray tube and radio-isotopes.

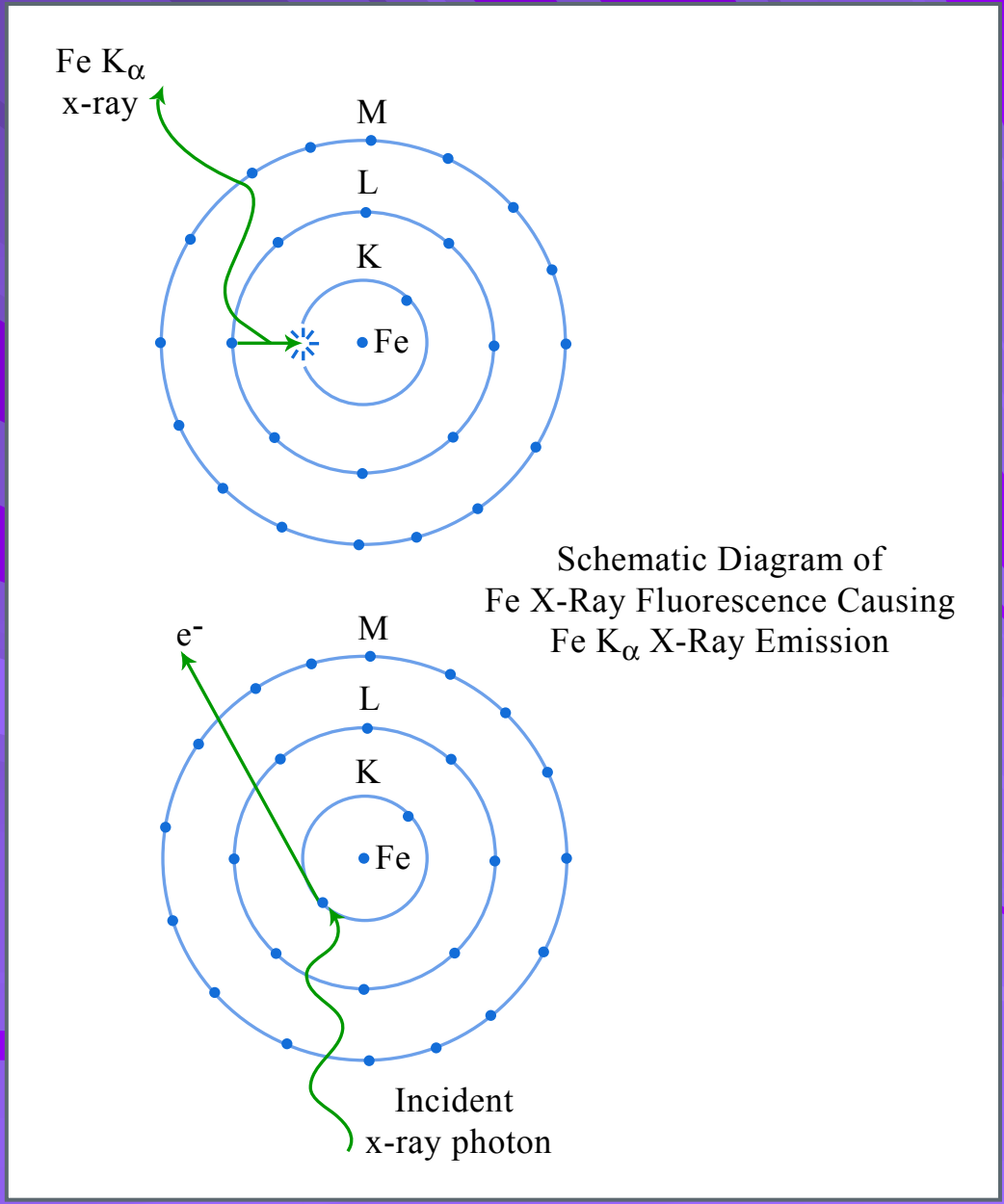
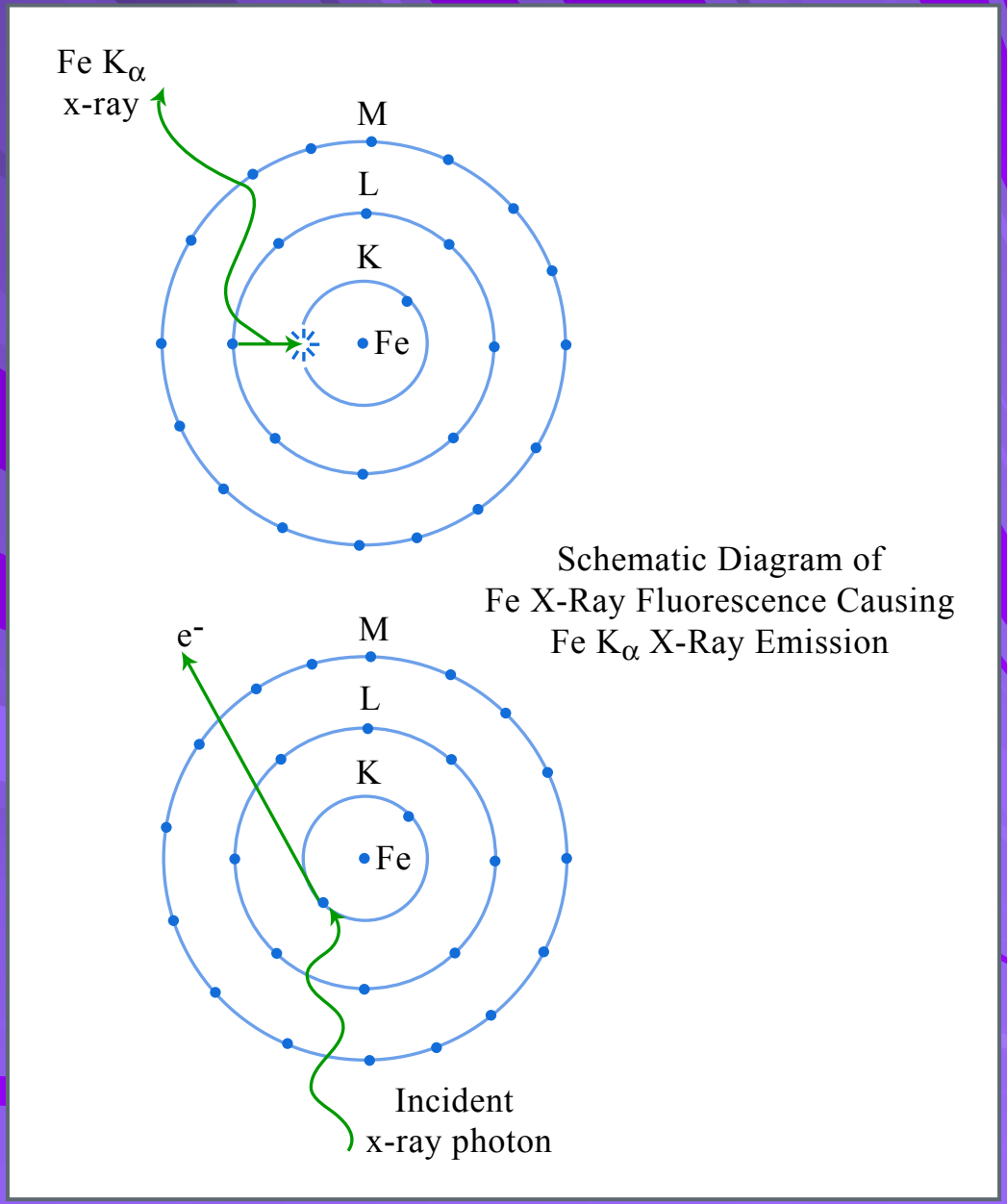


Figure by MIT OCW.

Wavelength Dispersive XRF (WDXRF) Energy Dispersive XRF (EDXRF) …

- When the energy of the exciting source radiation is higher than the binding energy of an electron in the inner orbital, the electron gets ejected and the atom becomes ionized. But the vacancy created by the ejected electrons filled by a higher energy electron in the outer orbital. As a result of this event, a photoelectron will be emitted with characteristic **wavelength or energy** (difference between the energies of the two levels). This emitted photon sometimes may be reabsorbed immediately (causing no emission).
- Fluorescence yield is the probability of emission of characteristic K, L, M, … X-ray lines. It increases with increasing atomic number and decreases with K>L>M ….



Schematic Diagram of
Fe X-Ray Fluorescence Causing
Fe K_α X-Ray Emission

Figure by MIT OCW.

Wavelength Dispersive XRF (WDXRF) Energy Dispersive XRF (EDXRF)

- Dispersive means separation and measurement.
- WDXRF – Separation is done by collimators and diffraction crystals. Measurement is done by detecting the characteristic wavelengths by scintillation detectors and proportional counters providing a pulse height distributed spectrum.
- EDXRF – the wavelength dispersive crystal and detector system is replaced by solid state energy dispersive system consisting of Si(Li) detector coupled to a Multichannel analyzer system.

Review Quiz

1. Explain the dose response curve with reference to essentiality and non-essentiality and health effects.
2. List 5 essential elements and briefly describe their health effects due to deficiency and toxicity.
3. List 5 toxic elements and their effects on health.
4. List the components and brief description of any one analytical technique.
5. In a fictional town called Cleanland, the town people are concerned about a piece of land they want to designate for vegetable gardening. They come to you for consultation – what will you advise?? Explain.

Summary

I gave the overview of analytical techniques:

- Atomic Absorption and Emission
- Inductively Coupled Plasma Mass Spectrometry
- Instrumental Neutron Activation Analysis
- Electron Microprobe - Wavelength and Energy Dispersive X-ray Spectroscopy

Internet Keywords

- Atomic absorption, atomic emission, wavelength dispersive X-ray spectroscopy, energy dispersive X-ray spectroscopy,
- Neutron activation analysis
- Gamma spectrometer
- Interaction of gamma rays with matter
- Electron probe

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