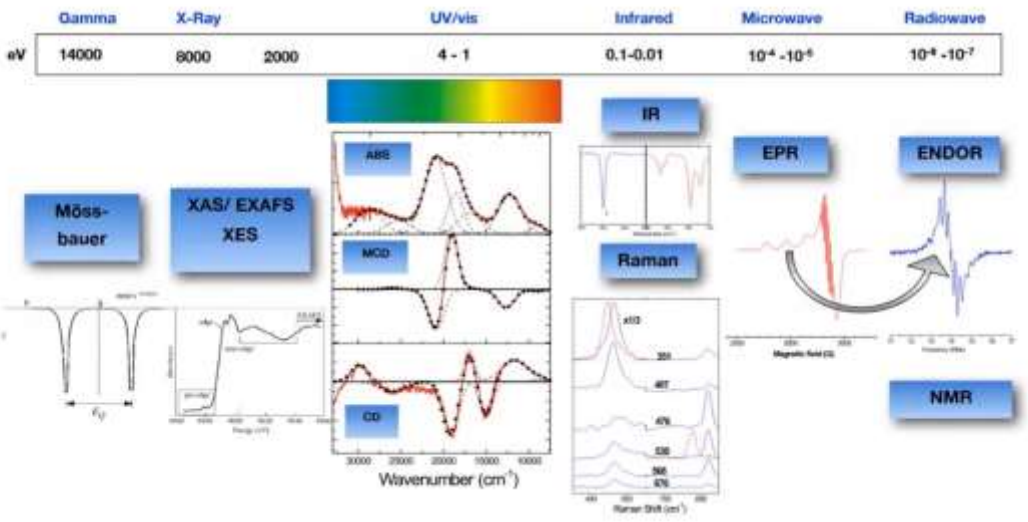
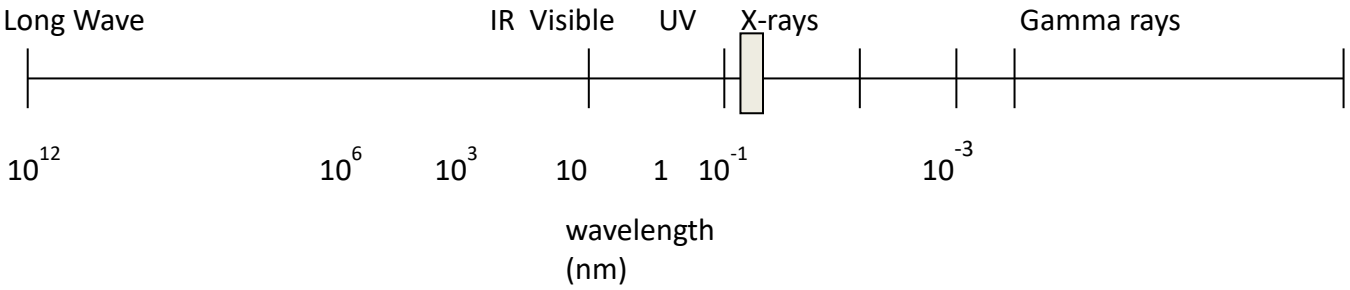
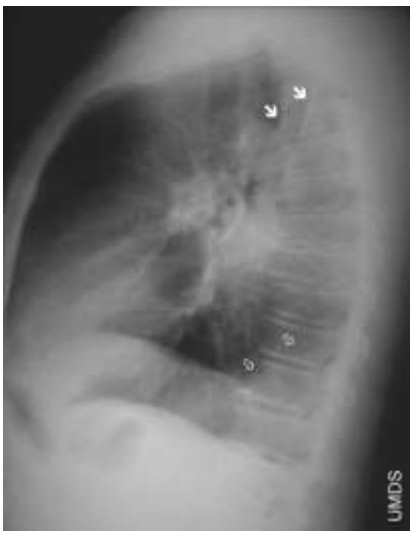
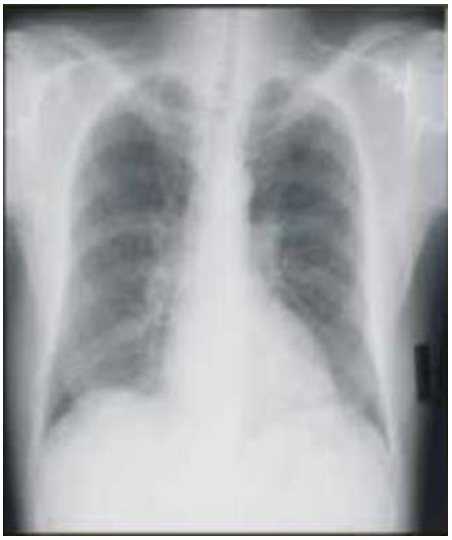
What is X-ray Spectroscopy and How Can it Help Me?

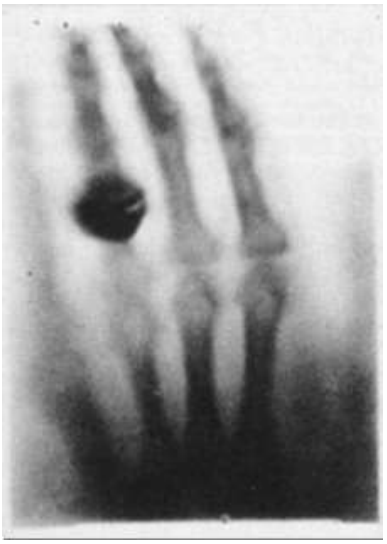
X-ray spectroscopy uses x-rays to excite core electrons in a molecule or material











1/ X-Ray Spectroscopy:

Radiation is energy in the form of waves or particles. Radiation which is high enough in energy to cause ionization is called ionizing radiation**.** It includes particles and rays given off by radioactive material and high-voltage equipment. Ionizing radiation includes x-rays, gamma-rays, beta particles, alpha particles, and neutrons.
X-rays were discovered in 1895 when Wilhelm Conrad Roentgen observed that a screen coated with a barium salt fluoresced when placed near a cathode ray tube. Roentgen concluded that a form of penetrating radiation was being emitted by the cathode ray tube and called the unknown rays, X-rays.

2/ Production of X-rays:

The most common X-ray photon sources are X-ray tubes in which radiation is produced by the bombardment of a target with electrons.
An x-ray tube requires a source of electrons, a means to accelerate the electrons, and a target to stop the high-speed electrons. (As shown in figure 01)

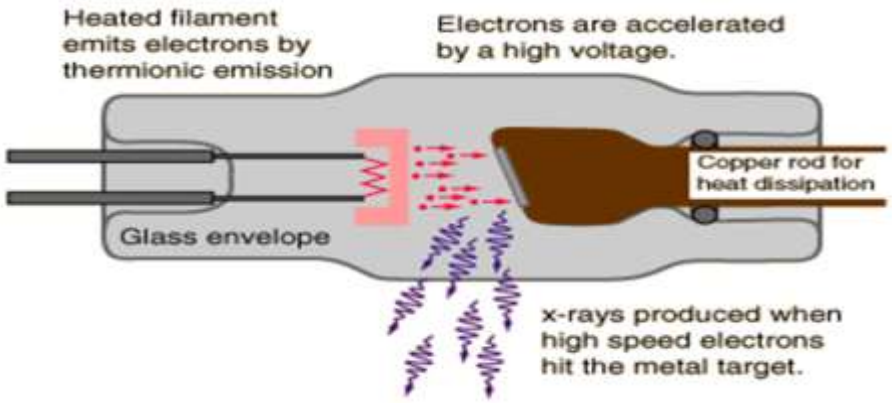
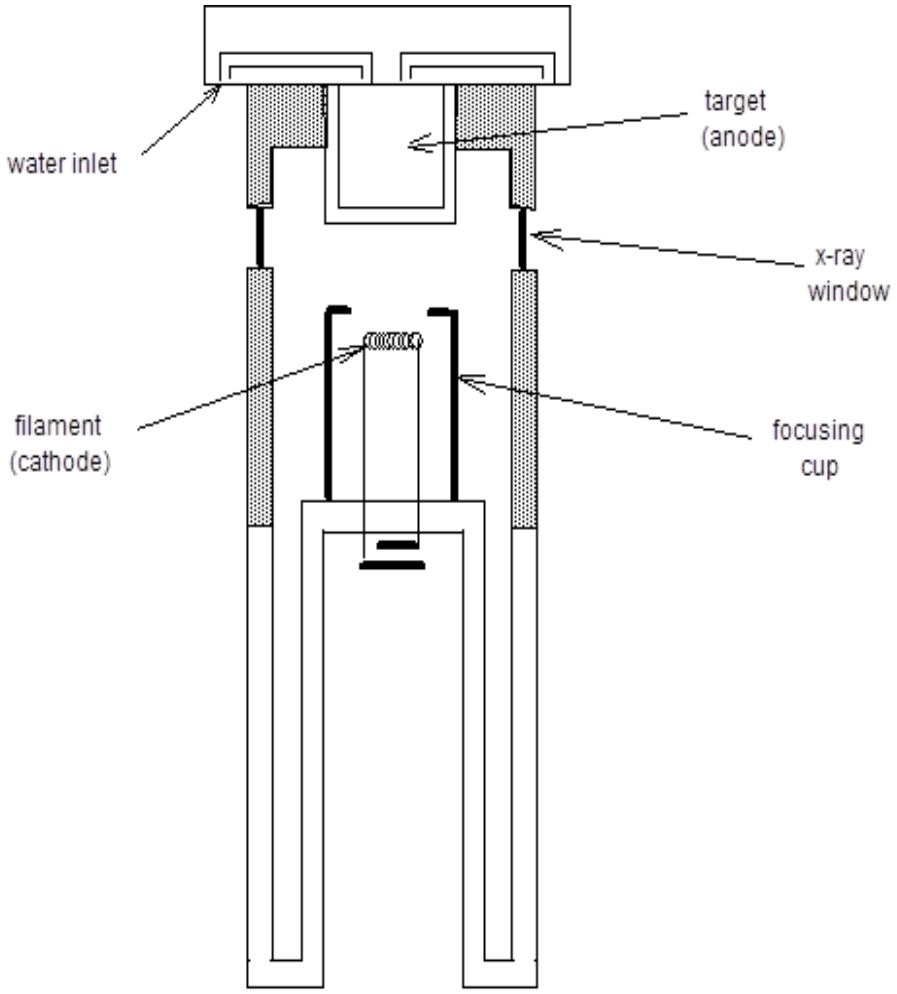


Figure 01

The cathode, formed by a tungsten filament, is heated by Joule effect. The electrons emitted by the filament are accelerated by the potential difference applied between the cathode and the anticathode. Under the impact of these electrons, the anticathode (the target), made of pure metal (Mo, Cu, Fe, Ag…), emits a spectrum of X-rays and heats up, which requires its cooling.


water inlet
target
(anode)
x-ray
window
filament
(cathode)
focusing
cup

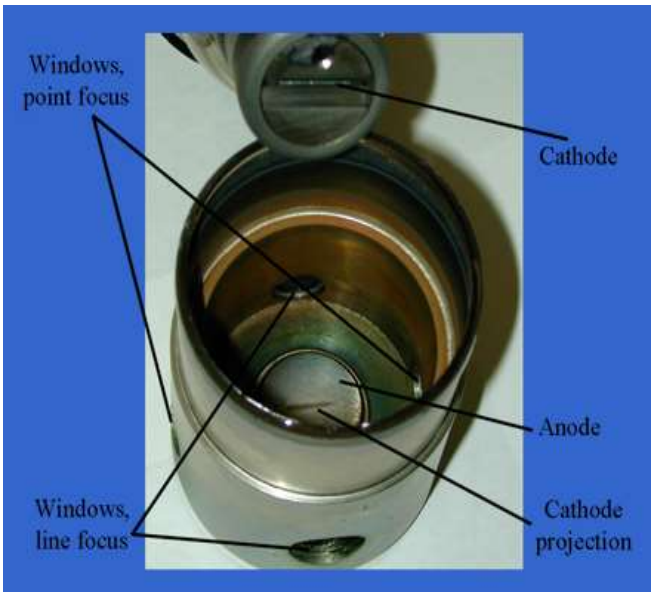

Windows,
point focus
Cathode
Anode
Windows,
line focus
Cathode
projection

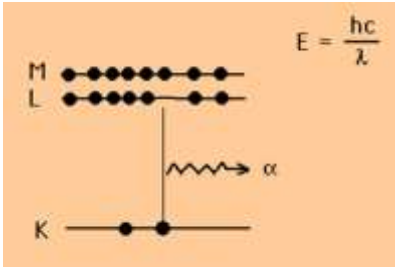
Figure 02 (X-ray device)

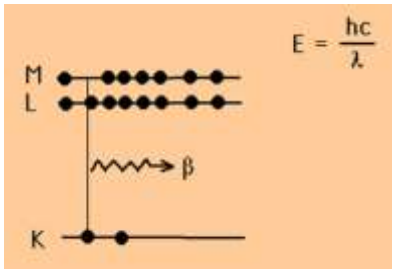
The tubes are protected by a shield and are equipped with low thickness beryllium exit windows transparent to X-rays.

The X-ray tube, detector and sample are contained in a housing that provides shielding to the user and others in lab. The access doors are interlocked with safety switches and will shut off X-rays when opened. The large viewing area is made possible by effective internal shielding and use of special glass or plastic windows.

3/ Properties and characteristics of X-rays:

- Frequency: 3.10^{16} Hz – 3.10^{19} Hz.
- Wavelength: 0,01 nm – 10 nm (shorter than UV and longer than γ) that is why it is very dangerous.
- Energy: 100 eV – 100 KeV.
- Principles of Non-Destructive Inspection :
  Most of X-ray images are monochromatic. The shading of the color is mainly related to the amount of X-ray penetration.





- X-rays penetrate matter:

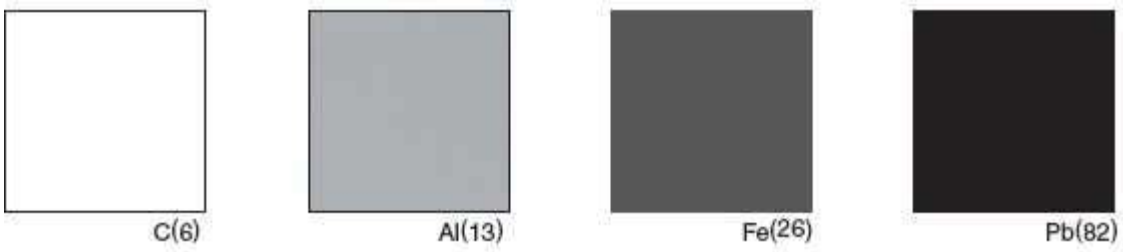
  X-rays are electromagnetic waves with very short wavelengths. Therefore, they can pass between the atoms that make up the substance. However, not all X-rays penetrate an object without being changed. Some X-rays are attenuated as they pass through. A variety of interactions (photoelectric effect, characteristic X-ray radiation, inelastic scattering, etc.) occur when X-rays hit the electrons circling around a nucleus. X-rays for which such phenomena do not occur and pass straight through are transmission X-rays. The areas on an image where these X-rays are present appear bright and whiter as the dose increases. On the other hand, attenuated areas appear darker and black.

- Strength of transmission X-rays:

  Generally, the strength of transmission X-rays is determined by the following factors.

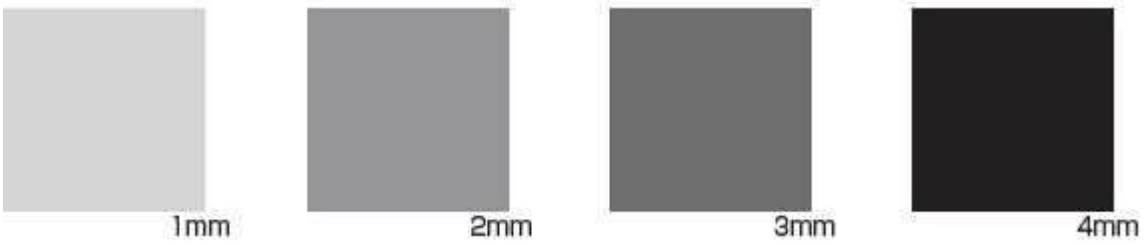
  a- More X-rays are blocked as atomic number and density increase:

  Changes in X-ray image with different materials of the same thickness
  * Atomic numbers are shown in parentheses.



  b-More X-rays are blocked as thickness increases:

  Changes in X-ray image with the same material in different thicknesses



  c- X-ray intensity is determined by tube voltage (V) and tube current (A):

  Increasing the tube voltage shortens the wavelength of X-rays and makes transmission easier. The value of the tube voltage required for transmission will differ depending on the material of the sample, for example, Al (atomic number 13) or Fe (atomic number 26). Increasing the tube current increases the dose and intensity of X-rays generated, but since the wavelength does not change, objects that are not penetrated are

not visible, no matter how much the tube current is increased. Determining the optimal settings for these two conditions (tube voltage and tube current) according to the object you want to see is the key to taking clear X-ray images.

4/ X-ray interactions:

In passing through matter, energy is transferred from the incident x-ray photon to electrons and nuclei in the target material. An electron is ejected from the atom with the subsequent creation of an ion. There are three basic methods in which x-rays interact with matter: photoelectric effect, Compton scattering, and pair production. (NB:All these interactions are bad for living cells).

5/Applications of X-Ray Spectroscopy:

a- Radiography:

Diagnostic radiology is the branch of medicine that involves taking and reading X-rays. The physicians that prescribe the shots and the technologists that operate the machines are specifically trained and licensed to perform these tasks. They also stay current through continuing education. Institutions are always striving to get quality images with the minimum patient exposure.

**difference in x-ray intensity transmitted through various parts of subject Depends on:

- Thickness difference.
- Density difference.
- Atomic number difference.
- Radiation quality (kVp, HVL).

**Subject Contrast and Radiation Quality:

- high kVp = lower subject contrast: long scale contrast (less difference between areas receiving varying amounts of radiation)
- low kVp = high subject contrast: short scale contrast (more black and white; more difference between areas receiving varying amounts of radiation), but increases patient dose.

b- X-ray Fluorescence:

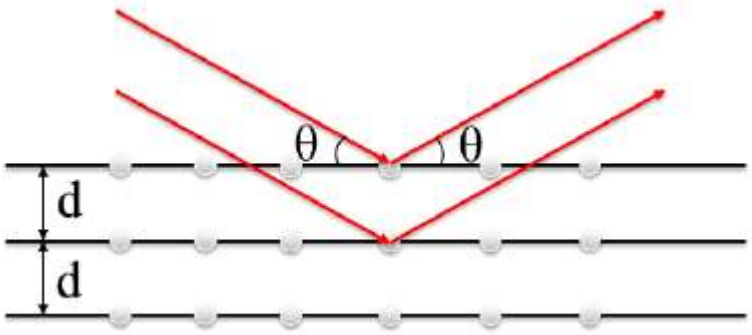
XRF uses X-ray fluorescence spectrum analysis technology and the method of energy dispersion to analyze the element content in the material. Materials are exposed to rays with suitable energy so that atoms in pulps are ionized and in excited state. Inner-shell electrons are excited and therefore vacancies appear. Such vacancies are filled by outer-shell electrons at high levels rapidly (within 10-15s) so that energy level transition occurs. Energy difference between energy levels is released in the form of characteristic X-rays. When pulps contain several elements, each element will emit several types of characteristic X-rays. Such X-rays received by detector and small electronic pulses will be generated. The peak amplitude of these pulses is proportional to the energy of incident X-rays. The intense of X-rays is proportional to the content of elements in slurry. Scattered X-rays can be used for slurry concentration correction.

c- Crystallography:

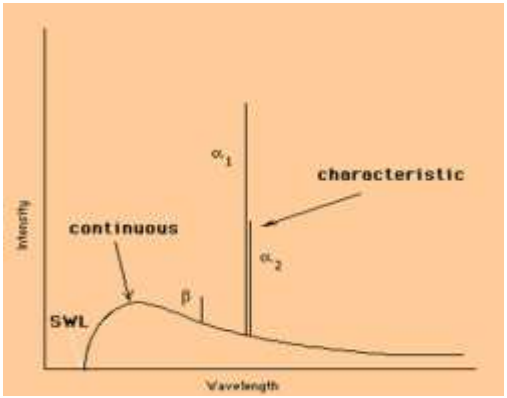
X-ray diffraction (XRD) is one of the most important characterization tools used in solid state chemistry and materials science, which could provide most definitive structural information (e.g. interatomic distances, bond angles, crystallinity, and etc…). The extensive use of X-rays for the analysis of atomic structural arrangements is based on the fact that the wavelength of the X-ray is in the 1 x 10-10m range, which is the same order magnitude of the atomic spacings in crystalline solids.

When x-rays interact with crystal lattice, a simple model called Bragg's law can be used to understand the required conditions for diffraction. The Bragg's law can be expressed as: $2d\sin\theta = n\lambda$

Where λ is the wavelength of X-ray, d is the spacing between layers of atom, θ is angle between incident X-ray beam and scattering plane, and n is integer.



Thus, the diffracted waves will consist of sharp interference maxima (peaks) with the same symmetry as in the distribution of atoms if the atoms are arranged in a periodic fashion in crystals. And the structural information of the crystals can be revealed based on the diffraction peaks.



AIF X-ray Diffraction Laboratory provides access to 3 state-of-the-art X-ray diffractometers for characterizing microstructural and crystallographic properties of powders, thin films, fibers and other solid materials. Below is a list of the applications that AIF

c-1/ Crystal Phase Identification and Quantitatively Phase Analysis:

XRD users may access to the International Centre for Diffraction Data (ICDD) database, which updates every year at AIF.

c-2/ Crystal Structure and Unit Cell Lattice Parameter

The crystal structural information of the sample may obtain by indexing peak positions or Rietveld refinement. AIF also offers Rietveld refinement workshop annually. For identification purposes, the diagram of the sample can be compared to the diagrams in the A.S.T.M (American Society for Testing Materials) file.

The ASTM data sheet for a given compound contains:

- The complete list of indexed dhkl.
- The ratio of their intensities.

And can contain the crystallographic characteristics (lattice parameters, space group, etc.)

c-3/ Crystallite Size: The information of crystallite size of the sample may acquire by analyzing peak broadening.

c-4/ Epitaxial,Texture and Orientation in the Samples: Bruker AXS General Area Detector Diffraction System (GADDS) at AIF equipped with a High-Star area detector and a Four-circle Eulerian cradle that allow for rapid analysis of polycrystalline and single crystalline samples, texture in the samples, and coarse-grained materials.

c-5/ Crystallinity of polymeric materials or fibers can be determined by comparing the intergrated peak area between amorphous phase and crystalline phase.

c-6/ Thickness, Roughness, and Density of the thin films: Some X-ray diffractometers are capable of performing X-ray reflectivity technique on thin films, which provide the information of film thickness, roughness, and density.

d- XPS (ESCA) :

XPS (also referred to as Electron Spectroscopy for Chemical Analysis or ESCA) is an analytical technique where x-rays are used to bombard a specimen and the energies of emitted electrons are analyzed. Typical x-ray sources are Mg K_α at 1253.6 eV and Al K_α at 1486.6 eV. Analysis with an ultraviolet source at 21.2 eV can also be made to provide high count rates for the low energy region which contains valence band information.

X-rays penetrate the specimen surface to a depth of a few micrometers but only the electrons near the surface can be emitted without losing energy due to collisions with other atoms. The kinetic energy (KE) of the electrons is measured and the binding energy (BE) of the electrons can be determined with a simple relationship:

$$h\nu = KE + BE + \varphi$$

where $h\nu$ is the x-ray energy and φ is the spectrometer work function (usually only a few eV). An energy spectrum is obtained with a scan over the kinetic energy range from 0 eV to the incident x-ray energy. The energy spectrum is different for each element and permits elemental identification of the species present in the top 1-2 nm. The detection limit is approximately 0.1% atomic. XPS is more sensitive for higher atomic number elements.