Basic Concepts of X-ray Fluorescence

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- X-ray Fluorescence (XRF) is a nondestructive method for the elemental analysis of solids and liquids.
- The sample is irradiated by an intense x-ray beam, which causes the emission of fluorescent x-rays.
- The emitted x-rays can either be detected using energy dispersive or wavelength dispersive detector.
- Either the energies or wavelengths of the emitted x-rays are used to identify the elements present in the sample while the concentrations (how much) of the elements are determined by the intensity of the x-rays.
- XRF is a bulk analysis technique with the depth of sample analyzed varying from less than 1 mm to 1 cm depending on the energy of the emitted x-ray and the sample composition.
- The elements commonly detected range from sodium to uranium.
- Lighter elements from boron to fluorine may also be detected.

How XRF works?

- X-ray Fluorescence is a fast and easy analytical technique (Figure 1).

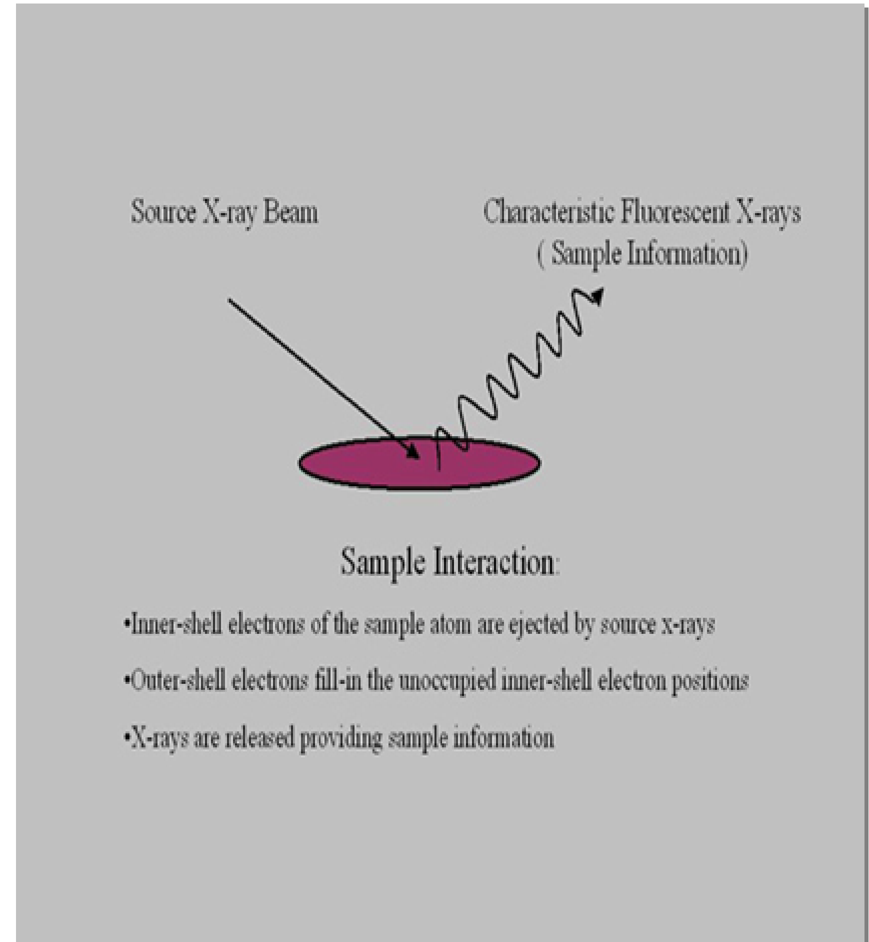

Figure 1- Simple Schematic of XRF

How XRF works?

- First, source x-rays are produced at energies of several thousand electron volts (Figure 2).
- X-rays are generated inside the tube by freeing electrons and accelerating them towards a metal target at energies of several thousand electron volts.
- Once the electrons hit the metal target, x-ray are produced.

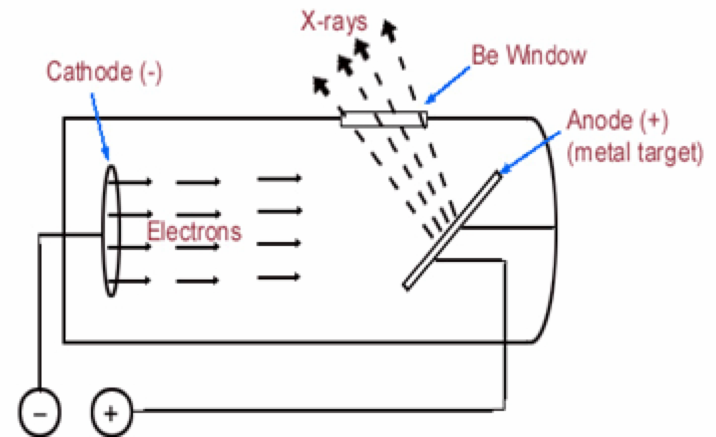

Figure 2 - Schematic of typical X-ray tubes.

How XRF works?

- The x-rays are directed at the "sample" (material being analyzed) from the x-ray tube.
- Second, the beam hits the sample and there is an exchange or "interaction" with the electrons in the atom (Figure 3).
- Finally, is a result of the interaction, x-rays that are characteristic to the elements inside the sample are emitted and analyzed.

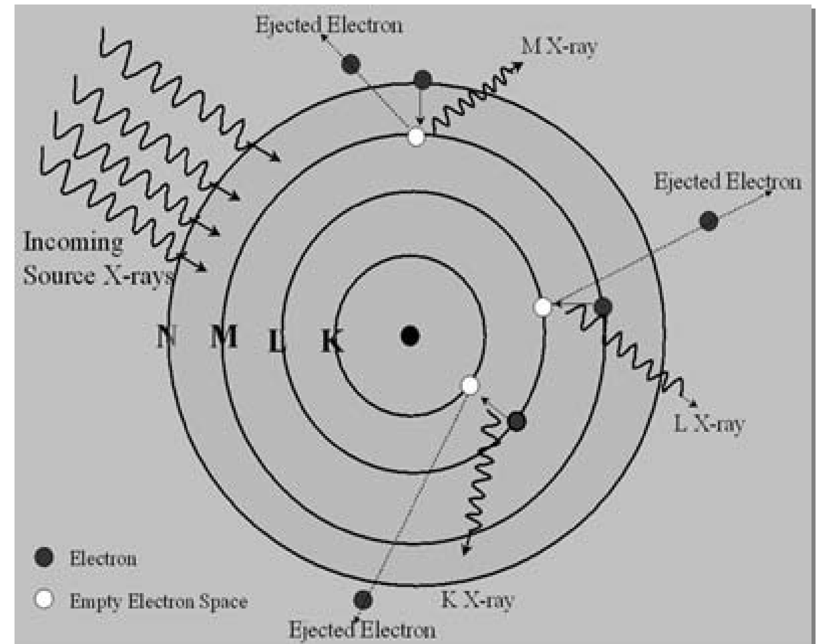

Figure 3 - Simple diagram showing incoming x-rays knocking out inner-shell electrons and having outer-shell electrons fill their space.

How XRF works?

- The outer-shell electrons are bound to the atom with less energy than the inner-shell electrons.
- When the outer-shell electron fills the inner-shell vacancies (empty spaces) the energy difference is accounted for by the energy of the emitted x-ray and therefore energy is conserved.

Principles of X-ray Energy Spectroscopy

- X-ray Energy Spectroscopy (XES) is a technique for rapid, simultaneous multi-element analysis. When excited by an appropriate source, a sample will emit x-rays of energies that are characteristic for the elements composing the sample.
- By measuring the energies of x-rays that are emitted from an excited sample and counting the number of x-rays of each energy, XRF allows us to identify which elements are present in a sample, and also determine the relative concentration of these elements within the sample.

Principles of X-ray Energy Spectroscopy

- The sample can be excited by an x-ray source, by a radioisotope source, or by an electron beam. Specifically, in X-ray Florescence Spectroscopy (XRF), generated x-rays are used as the primacy source. The primary source "excites" the sample by removing / "knocking out“ tightly bound electrons from the inner-shell orbital of the excited atom in the sample.
- Relaxation of the excited atom to the ground state is accompanied by the emission of fluorescent x-rays.

Principles of X-ray Energy Spectroscopy

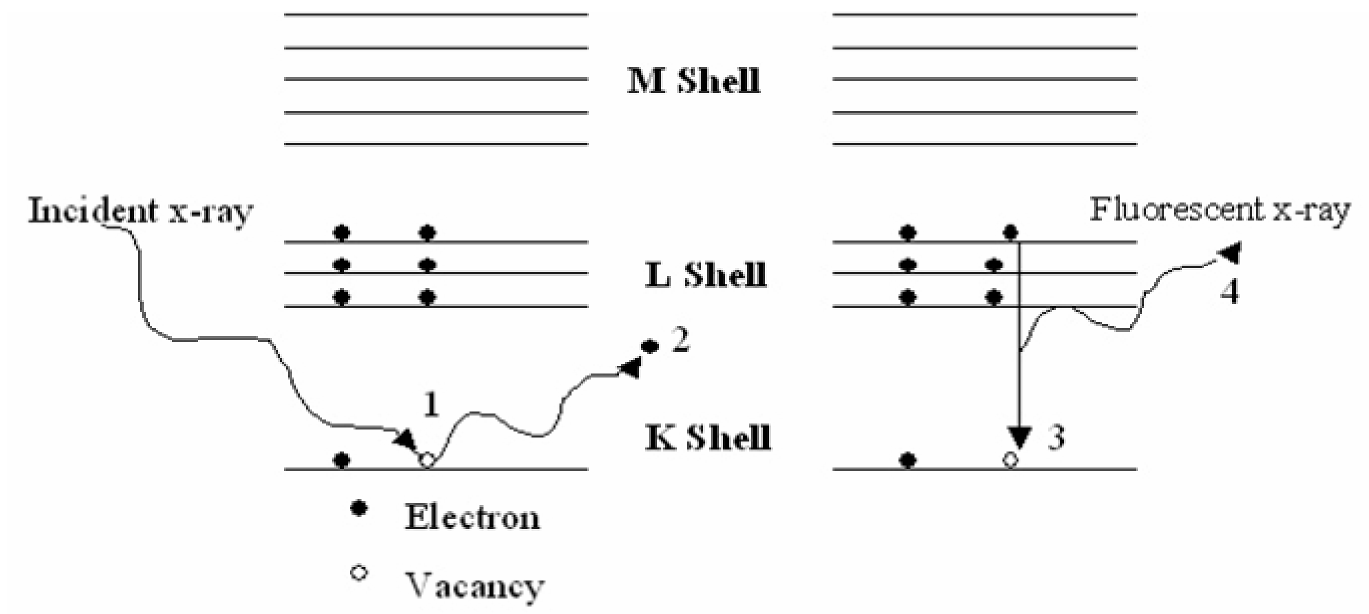

Figure 4: Schematic of the XRF process. Steps 1 & 2, incident x-ray knocks out an inner shell electron, 3, higher shell electron fills the empty vacancy, 4, excess energy given up as an x-ray (photon).

Principles of X-ray Energy Spectroscopy

- In figure-5 shows the X-ray system at ASU that is used for the analysis of the specimens in Images of Nature.
- The X-rays emitted from the exited sample strike a detector, which is typically a Solid- State detector in the case of Energy Dispersive Spectroscopy X-ray Florescence (EDS XRF). The detector used in Kevex 7000, Goldwater Laboratories, is a silicon drifted with lithium, Si (Li) detector (Figure 6a and 6b).

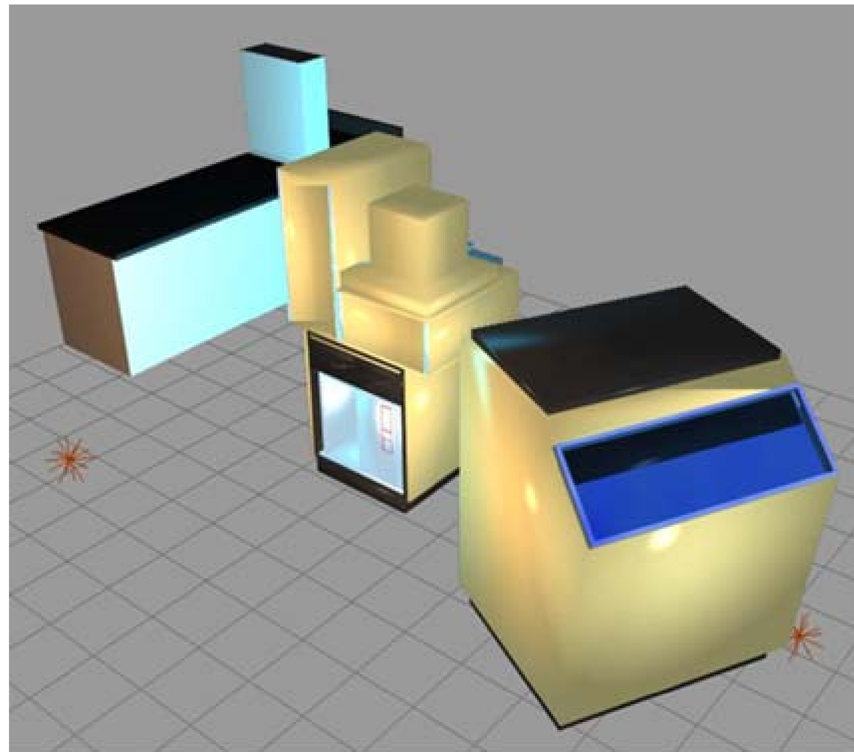

Figure 5: Kevex 7000 X-ray system in the Goldwater Laboratories at ASU.

Principles of X-ray Energy Spectroscopy

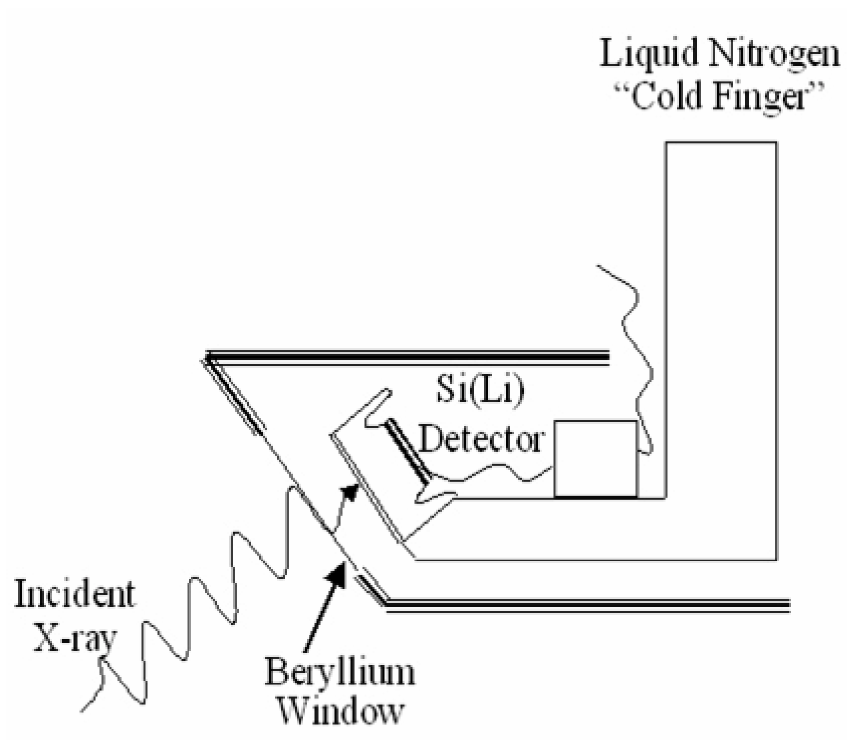

Figure 6(a):The Si (Li) detector schematic

Figure 6(b):The XRF sample chamber with Si (Li) detector

Principles of X-ray Energy Spectroscopy

- The elements that are present in a sample can be identified by the location of their energy peaks along the horizontal axis. Since, in theory, the number of x-rays produced is proportional to the number of atoms present in a sample, quantitative elemental concentrations can be determined from the net intensities of the energy peak.

- Most samples can be analyzed "as is" for most qualitative analysis and quantitative analysis.
- Some samples may require sample preparation such as pelletizing or casting as a fusion disk.
- Vacuum compatibility of the sample depends upon the element to be detected.
- The sample need not be conductive or insulating.
- For quantitative analysis the samples should be homogeneous with a flat smooth surface.
- Quantitative and qualitative elemental information can be obtained from a bulk infinite thickness sample.
- Qualitative elemental concentrations in solids/liquids are of the order of parts per million weight percent (ppm wt%) range and qualitative elemental data from boron to uranium can be acquired.
- The quality of these results depends on the calibration standards.

References

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