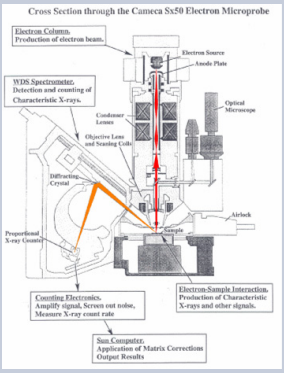
Lunar and Planetary Laboratory
1960 – 2010

The University of Arizona Electron Microprobe Laboratory

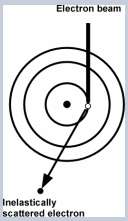
How Does a Microprobe Work?



In an electron microprobe, a solid sample is bombarded with a focused beam of high energy electrons (5 - 30keV).

This produces a variety of different types of **interactions between the beam electrons and the atoms in the sample**.

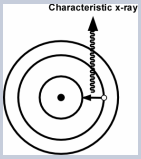
1) Ejection of an Inner Shell Electron



For electron microprobe analysis, the most important of these interactions is when a beam electron collides with an atom in the sample and causes an **inner shell electron to be ejected from the atom**.

2) Production of a Characteristic X-ray

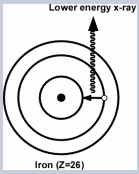
The resulting vacancy in the inner shell can be filled by an electron moving in from an outer shell.



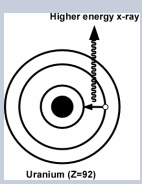
When this happens a **characteristic x-ray photon is released** with an energy equal to the difference in energy between the two shells.

3) The Energy of the Characteristic X-ray depends on the Element

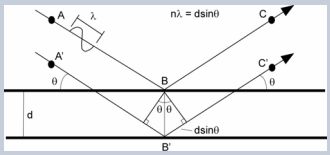
For a given transition between two shells, the energy of the emitted x-ray photon will uniformly increase with increasing atomic number (Z).



As a result, for a given transition between two shells, **the atoms of a each element will emit a characteristic x-rays having a specific and well known energies**.



4) Wavelength Dispersive Spectrometers



To separate the characteristic x-rays of a given element from those of other elements in the sample, the electron microprobe uses a crystal to **diffract x-rays having the energy of the element of interest into an x-ray counter**. The device that does this is called a Wavelength Dispersive Spectrometer (WDS).

Wavelength Dispersive Spectrometers are far superior at resolving x-ray energies than are other methods. These spectrometers are the defining feature of an electron microprobe.

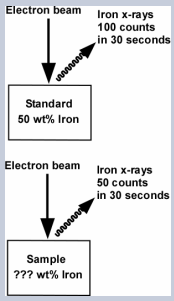
5) Standards Based Technique

Quantitative electron probe microanalysis is a **standards based technique**. In order to turn the x-ray count rates obtained for an element in an unknown sample into a concentration (in wt%) it is necessary to compare the unknown count rate to the count rate obtained on a standard with a known composition under the same analytical conditions.

6) Quantification

For example, as a first approximation, (known as **Castaing's First Approximation**), if under the same conditions you get 50 counts in 30 sec. for iron in an unknown and 100 counts in 30 sec. on a standard containing 50 wt% iron, to a first approximation the unknown is calculated to contain:

(50/100) * 50 wt% Fe = 25 wt% Fe.



In practice, this first approximation must be adjusted to account for the **effects of other elements present in the sample and standard** that may differentially affect x-ray production for the element of interest (i.e. **matrix effects**) in order to arrive a final estimate of the concentration of elements present in the sample.

7) Result – Quantitative Chemical Analysis

After analyzing for all the elements in the sample in the manner described above, a **full chemical analysis of the sample** is obtained. A routine analysis typically has a 1σ accuracy of 0.5 - 1.5% relative for major elements and minimum detection limit or 100 – 300 ppm. If desired, both accuracy and detection limits can be greatly increased by adjusting measurement conditions.

In almost all cases, electron microprobe analyses are much more accurate than can be attained using other non-destructive analysis techniques.