X-Ray Source

Spectrometer

X-Ray Fluorescence

Amptek Mini-X X-Ray Tube
Amptek X-123 X-Ray Spectrometer

Bennet Schwab

Phys 3340, Spring 2014
Markus Raschke
5/6/14

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Basics of X-Ray Fluorescence

K-alpha Emission

X-Ray Fluorescence (XRF) may occur when a high-energy x-ray becomes incident on an element. This may lead to the ejection of one or more electrons from an inner orbital. If this occurs, the atomic structure becomes unstable, leading to electrons from higher orbitals to transition into the lower orbital to fill the hole left by the ejected electron. In the process, a photon is emitted with energy equal to the energy difference between the two orbitals. The wavelength of emitted wave can be calculated using Planck's Law:

$$\lambda = (h * c)/(\Delta E)$$

Emissions due to transitions to the lowest orbital, the K orbital, from the second orbital, the L orbital, are called K-alpha emissions. Emissions due to transitions to the K orbital from the third orbital, the M orbital, are called K-beta emissions. The subscript alpha or beta refers to a transition from one or two orbitals above the named orbital respectively. There are also observable L-alpha, L-beta, M-alpha, etc. emissions which occur for elements with higher atomic masses. The energy of a photon from a K-beta transition is always greater than from a K-alpha transition from the same element, which in turn is always greater than an L-beta transition, etc. This is due to the fact that higher orbitals have higher energies and due to the change in energy between orbitals decreasing as they get further from the nucleus.

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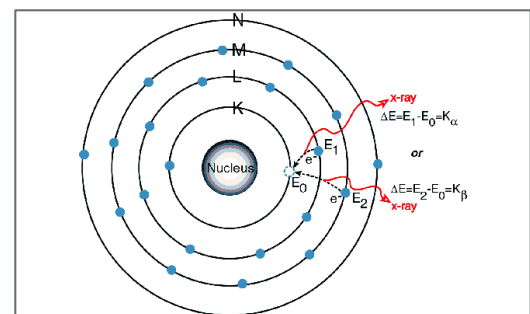
Objective

The goal of this experiment is to measure the energy spectra from various known elements using an x-ray spectrometer. This is done by firing an x-ray from an x-ray tube directed towards the sample for an extended period of time. Fluorescence will occur that can then be picked up by the spectrometer and the data fed to a computer program that plots channel vs. counts. With the known emission spectrum of the sample and measured peak values, the x-axis can be calibrated to fit the linear relationship of channel to units of keV. Once calibrated, the program can identify spectra of unidentified elements and compositions of elements. The final goal was to perform x-ray fluorescence spectroscopy on a rare earth rock to discover its composition.

Experimental Design

The device producing the x-ray is the Amptek Mini-X X-Ray Tube shown as (1) in the Figure. This device has a silver transmission target and a beryllium end window. It is operated at 15 keV when the interlock, shown as (3), is in the closed position and automatically shuts down if the interlock is disengaged. The Mini-X is positioned 15.93 mm from the sample at 22.5° below normal to the sample plane, and equidistant at 22.5° above is the X-123 Spectrometer shown as (2). With a Si-PIN detector, the X-123 is operated with a gain of 50. It has a resolution of 145 eV to 260 eV. The spectrometer data is sent to the computer program DppMCA.exe that plots the energy values against number of counts.

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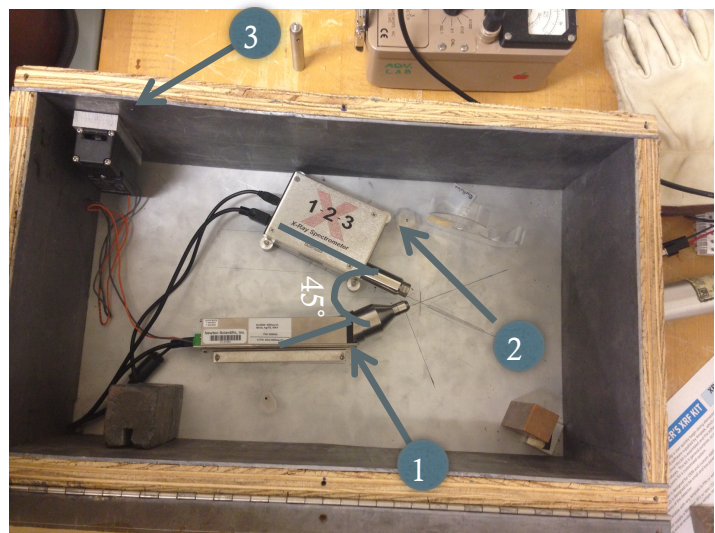
Samples

The samples used were copper, lead, argon gas, aluminum, brass, molybdenum, tungsten, silver and lastly a rare earth rock. All were used for calibration except brass and the rare earth rock. Copper was used to calibrate middle-energy spectra, lead for high-energy spectra, and aluminum for low-energy spectra.
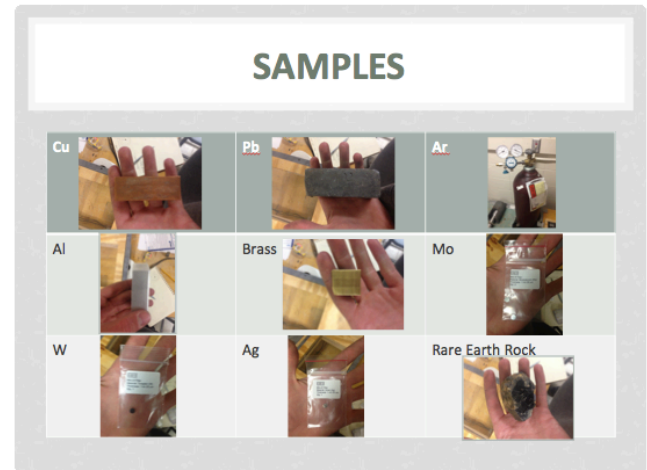
Data & Analysis

Copper   Expectation   Measured
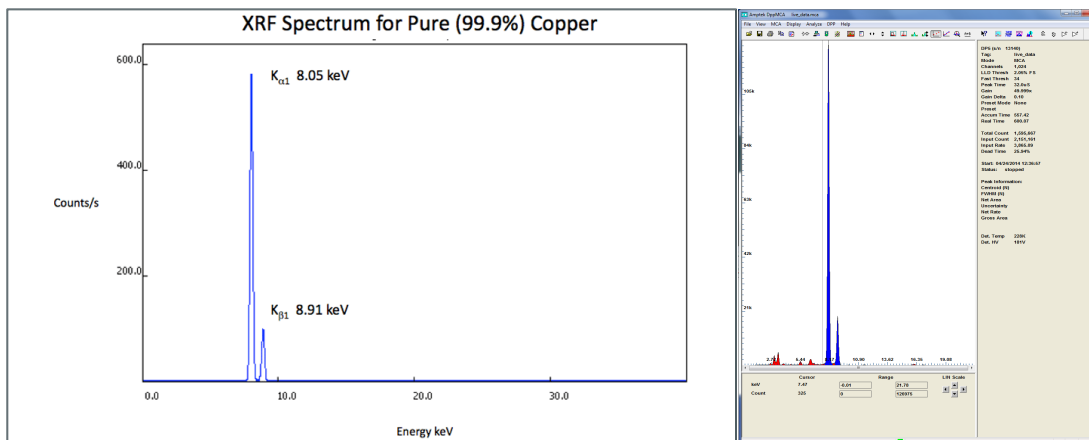
Two well defined peaks that agree with expectation. More frequent of the two peaks is the K-alpha emission at 8.05 keV and the higher energy peak is the K-beta emission at 8.91 keV. These two peaks were used for initial calibration from channel value to keV on the software. The smaller bumps were identified as Argon, Chromium, and Iron from lowest to highest.

Lead   Expectation   Measured
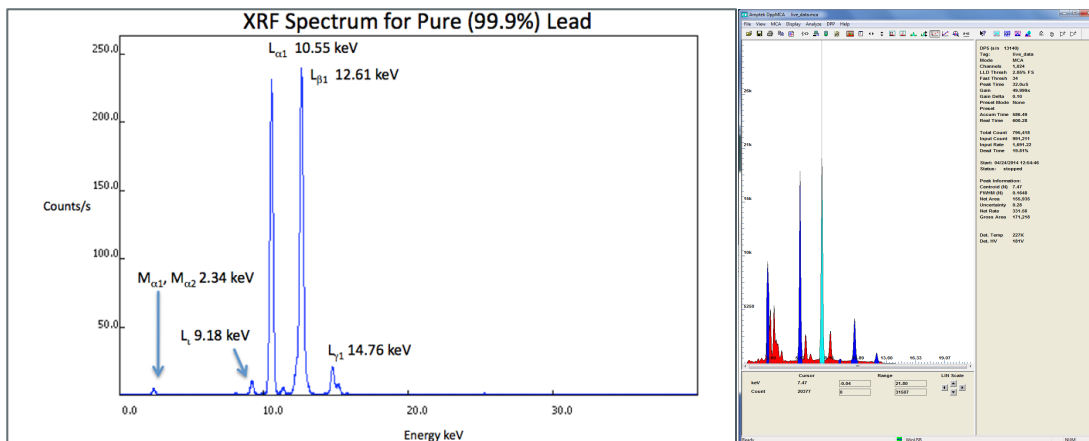
Three observable energies on the L order, shown as the three highest energy highlighted peaks, with energies of 9.18 keV, 10.55 keV, and 12.61 keV. Additionally, a low energy observable M-alpha emission is shown as the lowest highlighted peak at 2.34 keV. The two large peaks are Chromium and Nickel K-alpha energies, each with their respective K-beta.

Aluminum   Expectation   Measured
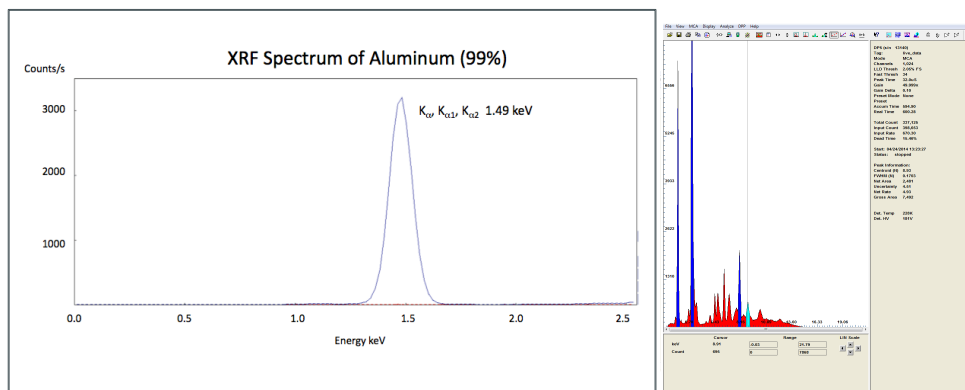
There is only one distinguishable peak for aluminum, shown as the lowest highlighted peak at 1.49 keV. The peak to the right of it is Argon and the other two highlighted peaks are the Copper spectrum; the K-alpha peak being the taller of the two and the K-beta the other.

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Argon Gas   Expectation   Measured
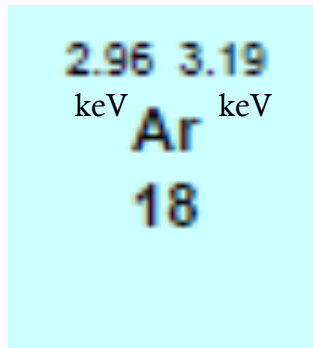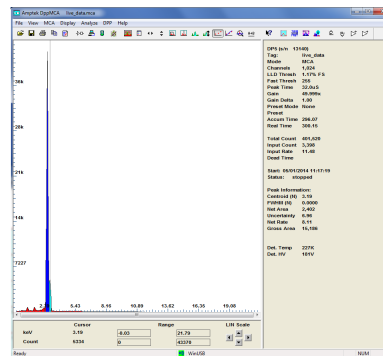
The large spike is clearly the Argon K-alpha emission and the smaller peak on shoulder is measured to be the K-beta emission. These are the only relevant observable peaks. In most of the spectra from other samples there are other peaks including Argon since Argon makes up some of the composition of the air in the box (which is not vacuum).

Molybdenum   **Tungsten**   **Silver**
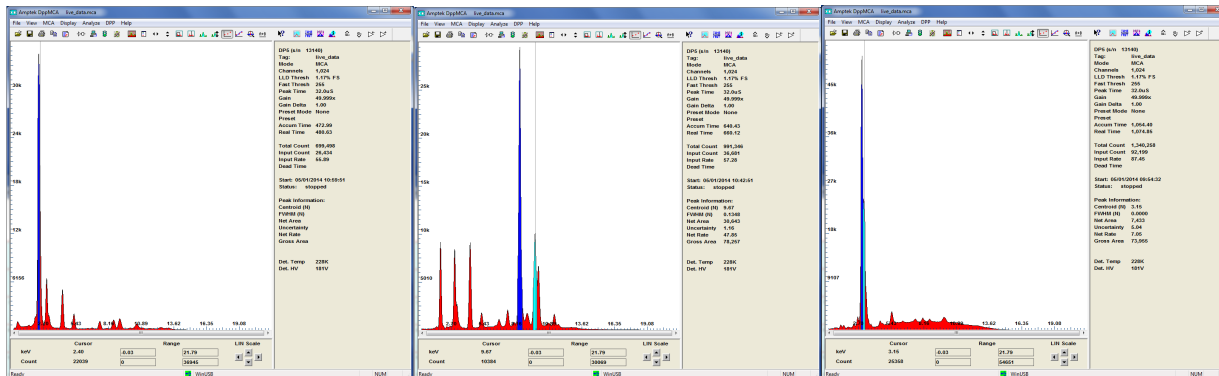
The spectra of Molybdenum, Tungsten and Silver were measured to double check calibration and to add more data points to the calibration. This improved the accuracy of the linear fit of channel to keV. The K-alpha peaks (highlighted in dark blue) for all and the K-beta peak (highlighted in light blue) for Tungsten were input to the calibration.

Calibration

Seven different elements were used in the process of calibration with a total of 14 known energy relationships (two high energy lead relationships not visible in picture). This allows for a more accurate linear fit for channel to keV. As shown below, the fit is very linear with little deviation from the line from any plotted point besides small random error.
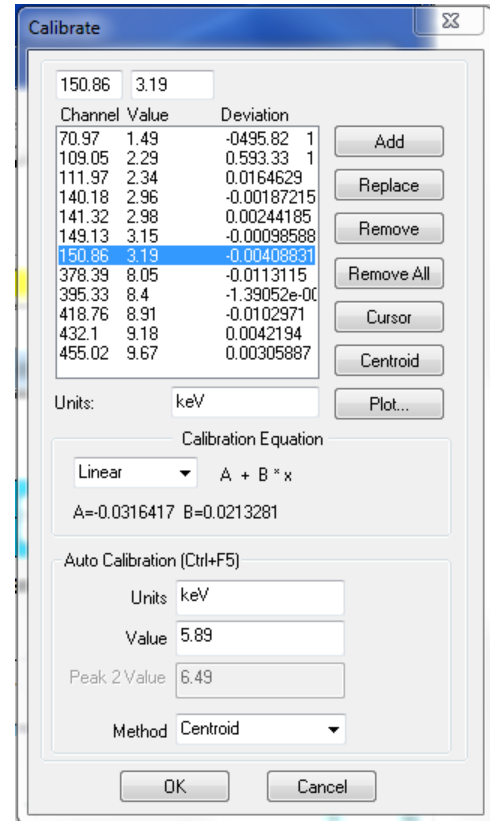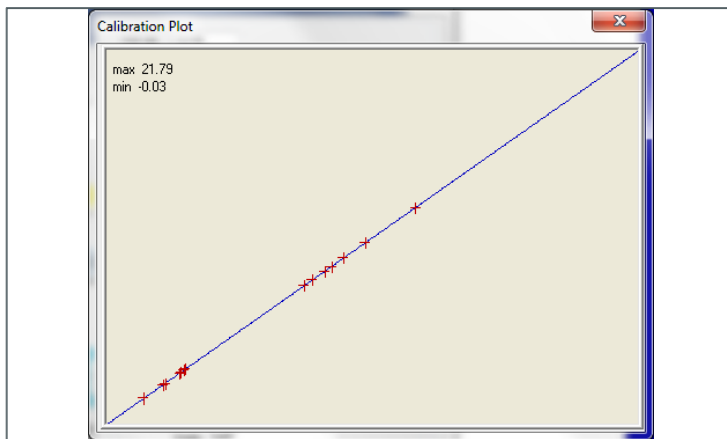
Now the spectrometer measures accurately in keV. This allows for the easy identification of elements from a table of spectra. The observable peaks are matched with their respective element to conclude the composition of unknown metals, compounds, rocks, etc.
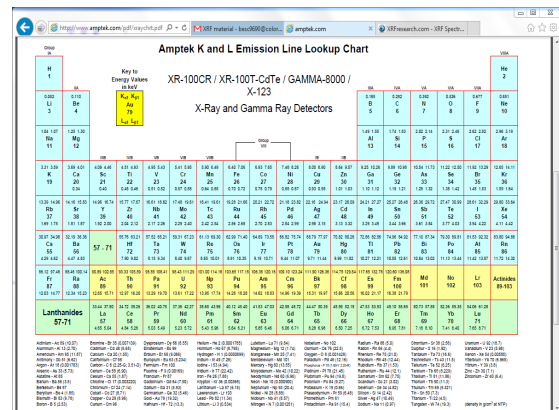
Data & Analysis Pt. 2

Brass   Expectation   Measured
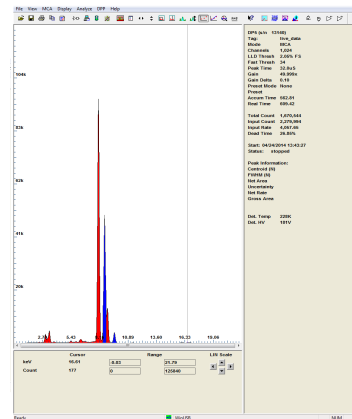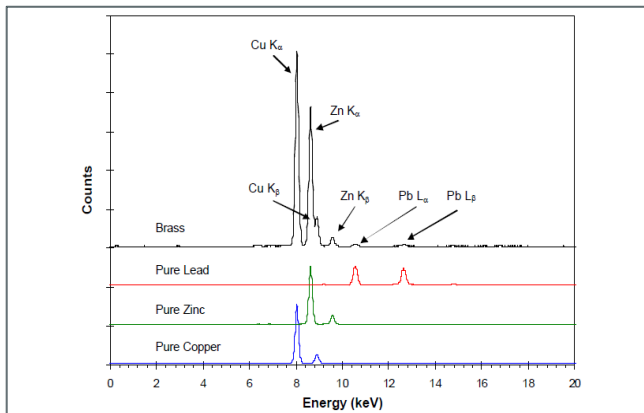
Brass has a composition of mainly three elements: Copper, Zinc, and Lead. The largest peak is from the K-alpha emission of Copper, and the accompanying K-beta peak shown as red in the middle of the two blue peaks. The two blue peaks are from the Zinc spectrum; K-alpha and K-beta energies. Lead is difficult to see unless zoomed in on.

Rare Earth Rock
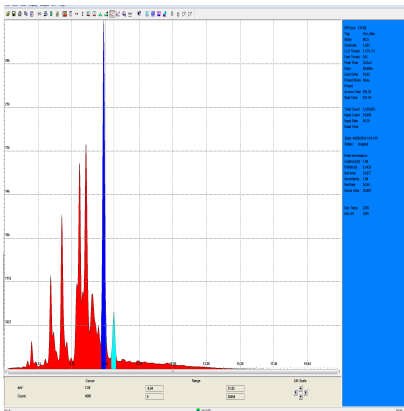
The image to the left shows the many peaks found in the XRF spectrum of the rare earth rock. The highlighted K-alpha (dark blue) and K-beta (light blue) peaks are from the spectrum of Iron, which is 6.40, keV and 7.04 keV respectively. The Cerium L-alpha and L-beta emissions sre the next two tallest peaks at 4.84 keV and 5.26 keV. The other main peaks are Calcium and Argon. There are also spectra from Aluminum, Silicon, Phosphorus, Sulfur, Chlorine, Potassium, Thorium, Lanthanum, Samarium, Europium and Gadolinium.

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Discussion

From the analysis of the data we see peaks at a number of different energies. These peaks form the overall spectrum of the rare earth rock. As we look at each energy peak directly, we determine the identity of each element in the rock by finding its known emission values on an emission look up chart. This rock has a peak at 3.15 keV, which is unique to a Thorium M-beta emission. Additionally there are peaks around 3.00 keV, the value of a Thorium M-alpha emission, but the Argon peak is overlying and hides the peak. If the experiment were to be performed in vacuum the Argon peak would presumably disappear and we would expect to see a clean peak at the Thorium M-alpha emission energy value, 3.00 keV. I conclude from the large spikes at the K-alpha and K-beta emission energies characteristic to Iron, 6.40 keV and 7.06 keV respectively, that Iron makes up a high percentage of the rock's composition. There is a noticeable decay in the amount of counts per second read by the spectrometer for higher energy emissions. I predict that this is in part because the ejection of electrons for lower energy elements, such as Aluminum, is easier than for higher energy elements, such as Zinc, due to the higher difference in the energy of the orbital and the energy of the x-ray. This decay would have to be factored into the data counts to get a good calculation of the composition percentage of each element in the rock. Yet the fact that that Iron has peaks at higher energies than other measured elements and is still has the highest number of counts per second leads to the conclusion that Iron makes up a major component of the rock's composition.

Outlook/Perspective

This experiment could be improved by reducing the amount of air in the chamber, ideally have the chamber in vacuum. This would negate the Argon spectrum from the air and allow for easy identification of elements with emission spectra around 3.00 keV. Additionally, a formula for the decay of counts per second for higher energies could be equated. This would allow for the composition percentage of elements in a sample to be calculated.

Appendix