

X-ray Spectroscopy: Characterization of X-ray Emission of Different Metals

Related Topics: X-ray notation, Siegbahn notation, bremsstrahlung radiation, characteristic X-ray spectrum, X-ray tube, X-ray fluorescence.

Before reading the experiment guide, look up the definitions of all the related topics and make sure you understand their meaning.

Purpose

In this experiment, you will measure the characteristic X-ray lines emitted from different metals. The energies of these lines are unique for each metal and a careful analysis of the lines can reveal the inner constellation of the atomic orbitals of the element.

In order to construct an energy levels diagram for a specific metal, one has to solve two different problems. The first is a practical one – how can you measure high energy radiation that has a wavelength of a few angstroms? What method is best used for such purpose?

The second problem is how to decipher the data you measured – what energy level transitions caused each line? Can there be more than one transition? What is the selection rule that can be applied for these transitions?

By solving these two problems, you can construct a model of the atomic levels for the element you are researching. This experiment demonstrates only one of the many spectroscopy methods that can be applied for modeling atomic orbitals and its results can be compared to those that are found in different articles and databases.

Theory

X-ray Tube Emission

When high energy electrons interact with an atom, a few phenomena can occur. The Electric field of the atom's nucleus can decelerate the electron and cause the emission of a continuous spectrum called **bremsstrahlung** ("braking radiation" in German). This type of radiation is emitted from every X-ray tube, regardless of the tube's type and operation conditions.

If the incident electron has sufficient energy, it can also ionize the atom, freeing an electron from the K-shell of the atom. Then, another electron from a higher energy level in the atom will make the transition to occupy the vacant quantum state of the freed electron, causing the emission of a photon of a specific energy correlating to the energy gap between the energy levels:

$$h\nu_{\text{photon}} = E_{\text{initial}} - E_{\text{final}} \quad (1)$$

where h is Planck's constant, ν_{photon} is the photon's frequency and $E_{\text{initial}}, E_{\text{final}}$ are the initial and final energies of the electron.

Since the energies of the photons emitted through this process are dependent only on the type of the atom and its inner atomic structure, this radiation is called the **characteristic X-ray spectrum**. It can be used to identify the content of alloys and other materials. Note that the characteristic X-ray spectrum is **quantized** (X-ray "lines"), as opposed to the continuous bremsstrahlung radiation. If an x-ray tube is operated with a voltage that is high enough to cause ionization, its emission spectrum will be the sum of those two radiations and will have a continuous part overlapping a quantized one.

X-ray Fluorescence

A different method of generating the characteristic X-ray spectrum of an element is to use **X-ray fluorescence**, where the element is bombarded by high energy photons (instead of electrons). When the photons ionize the element's atoms it emits its characteristic lines, in the same mechanism that occurred after being ionized by electrons. The difference between the two methods is that in X-ray fluorescence there is no bremsstrahlung being emitted, since photons cannot "decelerate", and the emitted spectrum contains only the characteristic lines.

In this experiment you will generate high energy photons using an X-ray tube and set the emitted X-ray flux to hit a metal or an alloy. Then you will measure the characteristic spectrum emitted from the material with an energy detector and identify its contents from the spectrum.

The System and Equipment



Fig. 1: Experiment System – An X-ray Unit, an energy detector and a multi-channel analyzer (MCA).

This experiment is performed using an X-ray unit (see Fig. 1) which includes an X-ray tube on the left side and a measurement chamber on the right. The energy detector is installed on the goniometer in the measurement chamber, and connected to the multi-channel analyzer (MCA, on the right). The output of the MCA is transmitted to the program.

In the X-ray tube, a beam of accelerated electrons is incident on the tube's anode, causing the emission of bremsstrahlung radiation and the characteristic X-ray lines of the anode's metal. The X-ray radiation then passes through a small hole into the measurement chamber. The size of the hole can be adjusted by inserting a diaphragm into it. This controls the focus of the X-ray beam and the X-ray flux. After passing through the diaphragm, the X-ray beam is incident on a material and ionizes it. The emitted spectrum from the alloy is measured with the energy detector.

There are four different X-ray tubes: Cu, Fe, Mo and W. In each tube a beam of electrons is accelerated using a high voltage (ranging from 10keV to 35keV). The voltage of the tube determines the energy of the radiation that can be emitted and the tube's current controls the intensity of the radiation.

The X-ray unit is controlled through a control panel (Fig. 4) on the front lower part of the unit. The MCA and energy detector is operated with a computer program (the Icon "measure" on the Desktop).



Fig. 4: Control Panel on the X-ray Unit.

Preparation Questions

1. Read "Nomenclature system for X-ray spectroscopy" and explain the terms: "normal X-ray lines", "Diagram levels", "orbital notation".
2. What do the letters V, X, C^* denote in IUPAC notation? What are the rules for denoting an X-ray emission in IUPAC?
3. Find the atomic X-ray levels (electron binding energies) of Fe, Cu, Mo, W. For each metal, between which levels the transitions have an energy of $5 - 30 \text{ keV}$? What are their notations in Siegbahn notation? (you can use sources from the course website)
4. According to the energy detector manual, how does it measure the energy of the X-ray photon? What is its output?
5. What is the purpose of the intrinsic region in the X-ray energy detector's diode?
6. How does the Multichannel Analyzer (MCA) determine the energy of an incident radiation on the detector (see manual)?
7. What is the MCA calibration process? What is its purpose and how is it performed?
8. What do the GAIN (of the main amplifier) and OFFSET parameters of the MCA mean?

Experiment Procedure

Do not perform experiment without receiving safety and operational instructions beforehand!

Operating the Goniometer

When the system is turned on, the Goniometer is in the position in which it was left after previous use. After the system door is first opened, closed and **locked**, the system resets itself, so both the sample holder and the goniometer arm are at angle 0. In this stage you can toggle between 3 modes of the goniometer and sample rotation: coupled, only sample moves, and only goniometer arm moves, see Fig. 5. This is done by successive pressing of the **Enter** button.

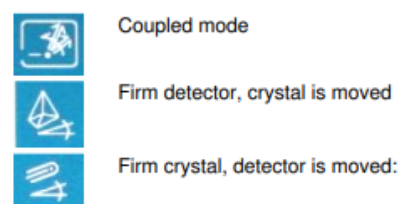


Figure 5 : Goniometer modes and symbols

There is a different way to control goniometer, not recommended for use in this experiment, outlined in [Operating the Goniometer II](#) at the end of the tutorial.

Preparing the Energy Detector for Calibration

- 1) Make sure the Mo- or the W-tube is in the X-ray unit and note it in your log (for later analysis).

- 2) Insert a 2mm diaphragm tube in the X-ray beam hole. This attenuates the beam so it wouldn't saturate the detector.
- 3) Set the goniometer on the farthest end from the beam outlet hole, by releasing the screw and moving it horizontally.



- 4) Close and lock the door by pressing the lock button:
- 5) Set the goniometer about 3.5° (to reduce the total rate of radiation reaching the detector).
- 6) Press "Menu", "X-ray Parameters" and set the voltage to 35keV and the current to 0.01mA (the minimal current possible).
- 7) In the program click "Gauge", "Multi channel analyzer", "spectra recording". This interface displays the histogram of the energy detector's measurements as they are acquired. Set the x-axis to display the channels.
- 8) Set offset to zero, what is the origin of these "measurements"? Note that the X-ray tube is off. What is the counting rate of this signal? Does changing the gain changes the rate? set the offset to the minimal value that cancels this signal.
- 9) Turn on the X-ray tube by pressing the "on-off" button (the "wave" icon). What is the counting rate? Set the tube's current and the goniometer angle so that the rate would be approximately $200 \frac{\text{counts}}{\text{sec}}$ (or lower). This rate is low enough so that the detector will give correct readings.
Whenever you don't actively measure the spectrum, turn the tube off!
- 10) Set the Gain so that the X-ray tube's spectrum will "spread" over most of the channels. Write down the settings you used. You will not change the Offset and Gain from this point on.

Energy Detector Calibration

- 1) Clear the histogram ("reset") and measure the spectrum emitted from the tube. Let the histogram accumulate until the peaks in the spectrum are clear and the SNR (the ratio of each peak amplitude to the "noise" level surrounding it) is approximately 3. This can take a few minutes. **Turn off the tube when you're done.**
- 2) Identify the spectral lines you see. The spectrum should contain the characteristic lines of the tube's anode (Molybdenum or Tungsten). It can also contain the characteristic lines of the Copper (Cu) which surrounds the anode metal. Use NIST database to find the energy of each line.
- 3) Save the calibration spectrum measurement by pressing "accept data" and then "Measurement", "Export Data", "save to file".
- 4) Use MATLAB to plot the spectrum you measured. Find what channel is associated with each line and calculate the linear relation between channel and energy ($E = a \cdot (\#channel) + b$) for the settings you used (GAIN and OFFSET). **Later, you will add more datapoints to the calibration set in order to get a precise calibration, see item (8) in "Spectrum Measurement and Additional Calibration"**

Spectrum Measurement and Additional Calibration

- 1) Move the goniometer horizontally to the nearest position to the beam outlet hole and remove the diaphragm.
- 2) Put a pure metal sample (not an alloy) into the crystal holder and set it on the goniometer. Make sure the metal isn't loose and can rotate without hitting anything.

- 3) Set the goniometer at about 90° and the metal sample at 45°.
- 4) In the program, click "Gauge", "Multi channel analyzer", "spectra recording". Set the offset and gain to the values you used in the calibration.
- 5) Clear the histogram ("reset") and turn on the X-ray tube. Change the tube's current so that the counting rate will be around $200 \frac{\text{counts}}{\text{sec}}$.
- 6) Let the histogram accumulate until the SNR of the peaks is approximately 3, then turn off the tube. Press "accept data", save the measurement and export the data you measured. If the peaks are overlapping let the histogram accumulate for a longer time, until you are certain that analysis with cftool can separate them into two Gaussians.
- 7) Plot the spectrum you measured in MALTAB, identify its lines and add them to the calibration set.
- 8) To improve the calibration's precision, measure the spectrum of more metals and **add their lines to the calibration set**. Do this until you are certain the calibration cannot be improved by additional lines.
- 9) Measure the spectrum of the alloys available and determine their composites. Compare the elements you identified with literature.

Bonuses

1. Measure the spectrum of a crystal and determine its composites.
2. Measure the tube's emission spectrum as a function of its voltage and current. Find an explanation for the results you've got.
3. Plan an experiment to determine the effect of the counting rate on the measurement and execute it. Why do you need to keep the counting rate below ~250 counts/sec to get accurate measurements?

Operating the Goniometer II

You can change goniometer settings by pressing "**Menu**", "**Goniometer**", "**Parameters**", "**Modify**".

The first parameter to change is the crystal type. This is for the Bragg experiment, you don't need it. Press **Enter**: this will take you to the **Scan Type**. Here you can switch between the three scan types using **up** and **down** arrows. Press **Esc** to finish.



Coupled mode



Firm detector, crystal is moved



Firm crystal, detector is moved: