UNIVERSITY OF TASMANIA

THIRD YEAR LABORATORY WORK

KYA 312/322

The production and characterisation of X-rays

September 29, 2021

Safety

This experiment involves the use of ionising radiation, specifically X-ray radiation. X-rays are a from of high-energy electromagnetic radiation and have high penetrating power, and therefore the impacts of X-ray radiation can occur throughout the body. Prior to beginning the experiment, you will be provided with a radiation safety induction, but as a general set of guidelines:

- Only operate the X-ray apparatus when the chamber in closed, and do not attempt to open the chamber whilst X-ray emission is occurring

This experiment also makes use of a variety of powdered-chemical samples for scattering, some of which are heavy-metal compounds and care must be taken to avoid exposure. Substances include:

- Bismuth oxide
- Germanium dioxide
- Lead dioxide
- Mercury oxide
- Potassium bromide
- Selenium
- Silver nitrate
- Strontium sulfate
- Tungsten oxide
- Zinc

As with any hazardous chemical, appropriate care must be taken when samples are handled in order to mitigate any potential hazard. For specific information pertaining to each chemical, including what action should be taken in the event of exposure, one should consult the relevant material safety data sheet. As a general reminder

- Do not handle chemicals with which you are unfamiliar
- Wash your hands and face thoroughly prior to eating or drinking after handling potentially hazardous material

The production and characterisation of X-rays

Summary

This experiment investigates the energy spectrum of X-rays produced from copper, and aims to measure the Rydberg constant by studying the scattering of X-rays as a function of atomic number.

Objectives and learning outcomes

- Understand the means of X-ray production

- Study the intensity of characteristic X-rays from a copper target

- Characterise the short-wave onset of bremsstrahlung radiation

- Calculate the Rydberg constant through measurement of K-edge absorption features for different elements

Introduction

X-rays are commonplace in daily life, with uses ranging from medical imaging to border security, as well as their use in cutting-edge research in fields such as structural biology and astronomy. X-rays and their interactions are characterised by the energy scales over which they exist and occur respectively, with typical X-ray energies in the range of $100\,\mathrm{eV}$ to $100\,\mathrm{keV}$. High-energy or so-called *hard* X-rays provide an excellent probe into the large-scale structure of materials due to their weak interactions with matter, whereas low-energy or *soft* X-rays provide probe for atomic structure as they are strongly interacting.

The uses of X-rays are myriad, but more fundamentally we might be inclined to ask: what is an X-ray? From where do they come? What distinguishes an X-ray from a gamma ray, or from a UV photon? It may be surprising, but there is not a clear consensus for exactly what is the distinction between X-rays and other forms of electromagnetic radiation. The most widely adopted definition of what constitutes an X-ray is the origin the radiation, and it is exactly this origin we seek to probe in this experiment.

Background

In 1895, Wilhelm Röntgen discovered a new form of *ray*, which he denoted X-rays, with the X referencing the designation as an unknown object. It was during the course of his initial experiments that he accidentally produced an absorption image of his own hand, although the first recorded image is the hand of his wife Anna Bertha Ludwig. The discovery earned Röntgen the inaugural Nobel prize in physics; however, it was not until 1912 when Max von Laue observed the diffraction of X-rays from crystals [1] (for which he was awarded the Nobel prize in physics in 1914) that said rays were determined to be a form of electromagnetic radiation. 1912 was also the year that Lawrence Bragg discovered the formalism that relates the scattering of X-rays from crystalline materials to atomic structure of the crystals [2] (for which he was also awarded the Nobel prize in physics in 1915). It was then the work of Henry Moseley in developing X-ray spectroscopy which established the link between X-ray emission/absorption and atomic structure [3], providing the first evidence of Bohr's theory of the atom and cementing the concept of the atomic number (it is speculated that he would have been awarded the Nobel prize in physics were it not for his death whilst serving in World War I). X-rays and their interactions have proven to be robust and versatile tool for probing the structure of matter, and it is in this context that our investigation is to be conducted.

The production of X-rays

As part of this experiment, we are going to investigate the radiation produced by copper under bombardment from energetic electrons. In the apparatus is a Cu X-ray tube, which, in essence is a vacuum tube containing both a tungsten filament and a copper block. The filament is heated which results in electrons being produced and when a potential is applied to the copper block, electrons are accelerated towards the target and, provided that the accelerating voltage is sufficient, X-rays are produced. In our setup, the voltage of the electron source is zero and the copper is held at a high voltage U_A, where A denotes that we are talking about the anode of the system. Figure 1 shows a schematic of the setup.
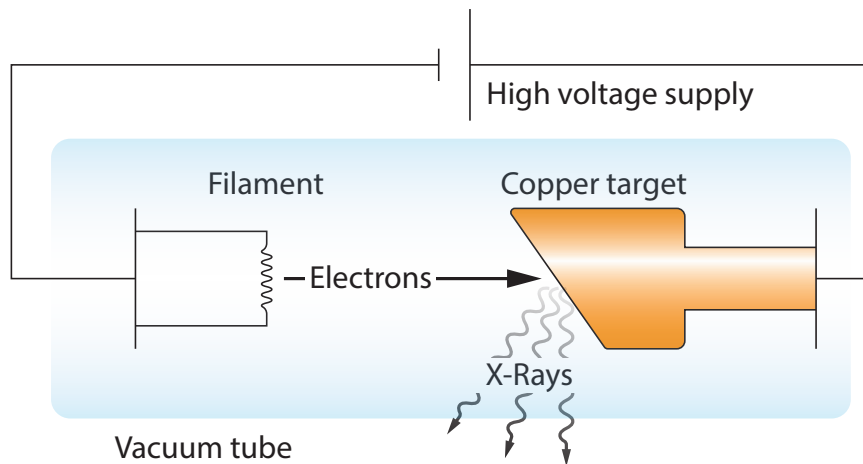

Figure 1: Schematic of the X-ray source to be used in this experiment

> **Exercise 1**
>
> Describe the physical processes by which the above arrangement produces X-rays. What kind of energy spectra would one expect for X-rays produced by the above process?

In simple terms, we are interested in probing that nature of the X-rays produced from the copper target, and in particular we are interested the distribution of the X-ray energies. Moreover, we want to probe the effects of varying the potential U_A along with the associated anode current I_A on the energy spectrum. The first task is to determine how we detect the X-rays, and then determine how we collect an energy spectrum?

The detection of X-rays

With the understanding of the physical processes that might lead to the production of X-rays, it is natural to ask how we might go about verifying if they have been produced.

> **Exercise 2**
>
> How might one detect the X-rays? And how might the energy spectrum of said X-rays be measured? To help answer these questions, consider how ionising radiation is normally detected, and how energy spectra are normally collected.

The apparatus we shall be using makes use of a Geiger–Müller tube to detect the X-rays, and the combination of a goniometer and a lithium fluoride crystal to measure the energy dispersion.

> **Exercise 3**
>
> Describe in detail how the described setup can be used to collect X-ray spectra; include a diagram in your explanation. What are the limitations of the setup?

The interaction of X-rays and matter

Like all photons, X-rays can interact with matter via a range of mechanisms, which are the photoelectric effect, Compton scattering and pair production. The likelihood of a given interaction occurring is strongly dictated by the X-ray energy and the properties of the scattering material. In this particular experiment, the available X-ray energy range is such that we need only consider the photoelectric effect. In this case, a photon can interact with a bound electron, with an energy transfer resulting in the emission of the electron from the parent atom. Scattering via this process can only occur when the energy of the photon is greater than the binding energy of the electron, meaning that the transmission of X-rays through a material will depend on the energy. Indeed, the phenomenon of absorption *edges* is key to utility of X-ray spectroscopy. Edges are a sudden increase in X-ray absorption which occur when the X-ray energy is just above the binding energy of a given state, and thus are element specific.

Absorption edges are seen when the photon energy matches that of an atomic transition, and in the case of X-rays we are talking about the inner-shell electronic transitions. From empirical work conducted by Johannes Rydberg and shored up later by the theoretical work of Niels Bohr, a basic atomic energy spectrum for hydrogenic atoms can be predicted through the Rydberg formula:

$$\frac{1}{\lambda} = R_\infty Z^2 \left(\frac{1}{n_2^2} - \frac{1}{n_1^2} \right) \tag{1}$$

where Z is the atomic number, n_1 and n_2 are the principle quantum numbers for the initial and final state and R_∞ is the Rydberg constant. In essence, the Rydberg constant can be thought of as the wavenumber of the lowest-energy photon capable of ionising a hydrogen atom in the ground state, which unsurprisingly can be expressed as the combination of fundamental constants:

$$R_\infty = \frac{m_e e^4}{8\epsilon_0^2 h^3 c}. \tag{2}$$

A note on notation: despite X-ray physics, especially as it pertains to spectroscopy, being effectively an arm of atomic physics, the language spoken by those in the atomic physics and X-ray physics communities is somewhat different. Clearly, given one is discussing the same phenomena, translations are unique and well-formed but are not always obvious. In general, physics tends to use the atomic physics notation to identify electronic states (term symbols) whereas the X-ray spectroscopy community uses IUPAC notation, which is a modernisation of the historic Siegbahn notation. As scientists, we should strive towards a universal, standardised systems; however, it is also important that one be exposed to, and fluent in, other systems which are commonplace. Therefore, given this experiment is in the realm of X-ray spectroscopy, the primary notation used to describe transitions and states will be primarily from the X-ray community.

For the most prominent transition edges, we are usually considering a transition involving an excited state and the ground ($n = 1$) state. In Seigbahn notation, the ground state is labeled as K, and radiation emitted from a decay to this state is denoted by K with a subscript, indicating the state from which the decay occurred ($\alpha, \beta, \dots$). An additional numerical subscript is used to denote the fine-structure of the exited state, this convention is not always followed, especially in the case where the energy splitting of the excited state is not readily resolvable. Shown in figure 2 is an energy level diagram depicting the states, their different labels and the labels of some transmissions between states. Table 2 collates the different labeling conventions and will hopefully serve as a reference for transliteration.

> **Exercise 4**
>
> Figure 2 and table 2 do not indicate the naming conventions for the $L_1 \to K_1$, $M_1 \to K_1$ or $M_1 \to L_1$ transitions. Speculate why this might be the case.

It was during the study of characteristic K_α emission as a function of atomic number by Henry Moseley that he empirically determined that the frequency of the emission followed [3]
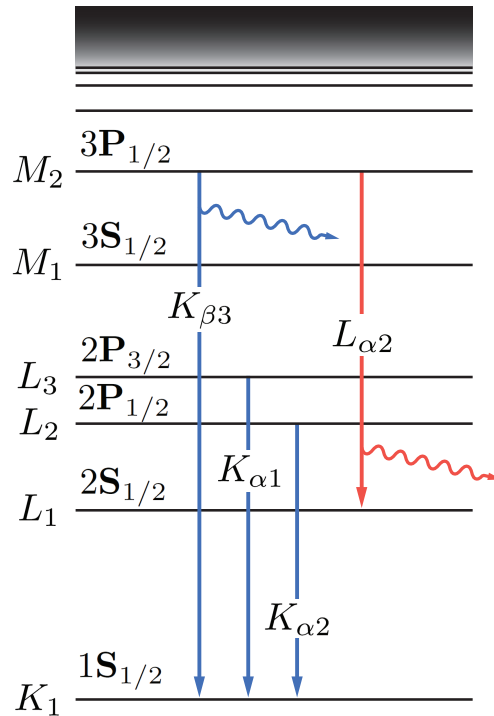
$$\nu = \frac{3}{4} c R_\infty (Z - \sigma)^2 \tag{3}$$

Figure 2: Energy-level diagram for K_α and K_β radiation

X-ray transition nomenclature			
Lower state	Upper state	Siegbahn notation	IUPAC notation
	L_3 ($2\mathbf{P}_{3/2}$)	$K_{\alpha 1}$	$K - L_3$
K_1 ($1\mathbf{S}_{1/2}$)	L_2 ($2\mathbf{P}_{1/2}$)	$K_{\alpha 2}$	$K - L_2$
	M_2 ($3\mathbf{P}_{1/2}$)	$K_{\beta 3}$	$K - M_2$
L_1 ($2\mathbf{S}_{1/2}$)	M_2 ($3\mathbf{P}_{1/2}$)	$L_{\alpha 2}$	$M_2 - L_1$

Table 1: Transliteration of atomic states and X-ray transitions

where σ is the so-called screening constant. Upon careful consideration of the above equation, it is perhaps not all that surprising a result: equation 1 does not account for the fact that the nuclear charge of the atom is partially screened by inner-shell electrons, thus reducing the effective charge. For K_α, the electron in the K state *screens* the electron from the nucleus, meaning the effective charge is $Z - 1$.

Exercise 5

Verify that for K_α emission that equation 1 and equation 3 are consistent.

The importance of the observations which led to equation 3 cannot be overstated: a connection between Bohr's theory to explain the optical spectrum of hydrogen could accurately explain X-ray spectra, despite the wildly different energy scales. It was also the work that led to the electronic charge of an atom becoming linked to the position of the element within the periodic table, and firmly established the order in which elements should be arranged in the periodic table. A testament the success of theory was the accurate prediction of multiple elements yet to be discovered, along with the correction of the existing *mass order* which resolved inconsistencies with chemical periodicity.

Apparatus

To produce and detect X-rays, we are going to use a commercial system, the PHYWE *XR 4.0 expert X-ray unit*. The system is a simple X-ray beamline, with an X-ray tube source, a small (2 mm) diaphragm used to collimate the X-rays and a sample stage mounted on a goniometer, which allows the angle of both the sample and detector relative to the X-rays to be independently controlled. The system is capable of producing a source potential of 35 kV and uses a target of copper. To interface with the system, ensure the X-ray unit and associated computer are powered on, and then open the `measure` program.

The experimental interface is comprised of two sections; the control and the analysis sections. Of primary interest is the control section which appears in the `Device` tab of the `Navigator` window, which shows an image of the apparatus (see figure 3). Here it is possible to control experimental parameters: by clicking on the left of the image, one can access the source parameters, and by clicking on the right of the image, one can access the goniometer parameters.

Figure 3: Screenshot of the PHYWE `measure` interface

Prior to running any experiments, the apparatus must be internally calibrated. For the apparatus to function, the sliding lead-glass door must be closed and locked in place: this is done by sliding the door into the closed position until the locking mechanism is engaged, and then press the lock symbol on the front panel. The mechanism functions as an interlock, meaning that unless the door is locked shut, the device will not function. Thus, if you are having trouble with the system, verify that the door is correctly shut and the lock engaged. Next, to perform the calibration, open the `Goniometer adjustments` panel and click on `calibrate`. Presuming that calibration was achieved, we are now ready to collect our first X-ray spectrum.

X-ray emission from copper

The first investigation which is to be undertaken is to characterise the emission of X-rays from copper. This requires not only an understanding of X-ray production but also X-ray detection. In brief, we want to collect energy spectra of X-rays produced by copper over a range of different parameters, and verify that the energy spectra behave in a manner commensurate with our understanding.

First light

To produce the first X-rays, begin by setting the anode voltage and current to $U_A = 35\,\text{keV}$ and $I_A = 1.0\,\text{mA}$ respectively. These are the maximum values for both current and voltage, and thus maximises the likelihood that that X-ray emission is occurring. It is under these conditions that parameters to scan the goniometer can be set which allows us to collect X-ray spectra; the scan parameters are set and then the experiment is started by selecting `Start measurement` from the toolbar and upon completion you should see the results in the `Multigraph` window. You should take some time to familiarise yourself with the system and the software by collecting spectra: try to understand the different parameters and see if your intuitions align with what you observe.

Detector gadget

Before we delve into the characterisation of X-rays, it is important to understand how we are detecting the X-rays, and any limitations the detection system might impose on our experiments.

As stated earlier, the apparatus makes use of a Geiger–Müller tube. The detector operates on the principle of facilitating Townsend discharge events in order to detect ionising radiation. In our case, a small cylinder containing a low-pressure gas has a potential applied between a central rod and the chamber wall. Radiation with sufficient energy entering the chamber will ionise gas molecules, and the ionisation products will be strongly accelerated due to the electric field between the rod and wall, and these charges can subsequently ionise other gas molecules, and the process continues in an avalanching fashion, allowing a large electrical signal to be measured from a single ionisation event. A schematic of the detector is shown in figure 4.
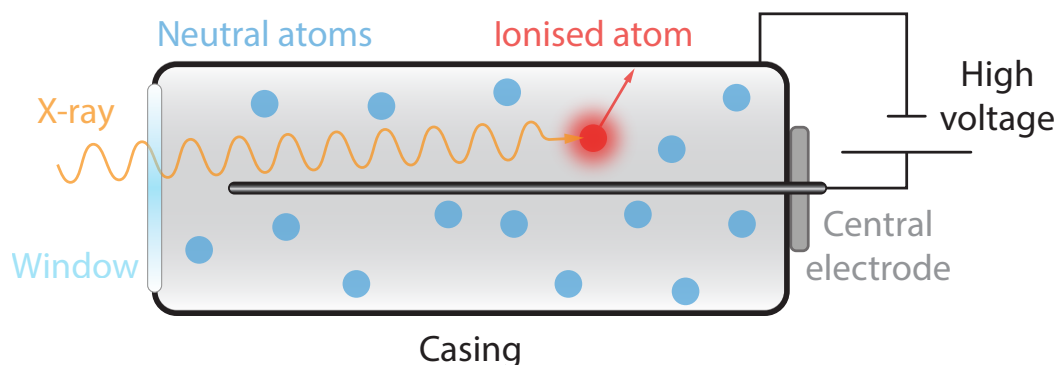

Figure 4: Schematic of a Geiger–Müller tube

Geiger–Müller tubes are highly effective radiation detectors; however, they provide no spectral information and are not suitable to measure high intensity sources of radiation.

> **Exercise 6**
>
> What is *dead time* in the context of a detector? Why is it important to consider the dead time? How does one compensate for a known dead time of duration τ? What is the physical origin of the dead time in a Geiger–Müller detector?

In order to better understand the functioning of the detection system, we want to measure the output of the Geiger–Müller tube. The signal is extracted by monitoring the tube supply voltage, which is done in by systems within the apparatus. However, it is easy enough to separate the signal from the supply voltage using a simple circuit, which has been prepared and is labelled *X-ray HV decoupler*.

Do not connect or disconnect the cable connecting the Geiger–Müller tube to the decoupler whilst the detector is live: the cable will also be energised. Adjust the detector voltage in the `X-ray adjustment` menu → `GM voltage`.

Under safe conditions, with the detector supply connected to the input of the decoupler circuit, monitor the output signal on a digital oscilloscope. Should you have difficulty observing a signal, ensure that the conditions for the detection of X-rays are satisfied.

> **Exercise 7**
>
> Vary the supply voltage of the Geiger–Müller tube and note the effect on the signal. Choose an appropriate voltage and then measure the detector dead time at his voltage. Note that this will be the detector voltage used for the rest of the experiment.

Seeing is believing

We now wish to investigate the X-rays emitted from copper. Ultimately, the goal is to collect and identify any features on a high-resolution energy spectrum; however, prior to making any measurements, we can perform a few quick calculations to test our predictive powers.

Characteristic emission

The primary characteristic X-ray emission lines we expect to observe and the K_α and K_β emissions.

> **Exercise 8**
>
> Estimate the energy of K_α and K_β emissions from copper and hence speculate over what range of angles would you expect to see a signal? As a reminder, the lattice spacing of LiF is 201.4 pm

Bremsstrahlung

Besides characteristic emission, we also expect Bremsstrahlung emission.

> **Exercise 9**
>
> What is the expected emission spectrum of the Bremsstrahlung radiation? What identifiable features would be present at a source potential of $U_A = 35\,\mathrm{kV}$?

Characteristic emission

Now we have a prediction of how the copper spectrum should look, let's put the background theory to the test.

> **Exercise 10**
>
> Collect a high-resolution scan of X-ray emissions from copper. Identify all prominent features and compare these to the predicted values.

The intensity of radiation

It can be shown that the intensity of characteristic K-shell X-rays, I_K, is related to the anode current I_A, anode voltage U_A and the ionisation potential $U_K = 8.979\,\mathrm{keV}$ through [?]

$$I_K \propto I_A(U_A - U_K)^{3/2} \tag{4}$$

The Duane-Hunt displacement law

You may have noticed by varying the anode voltage U_A in the previous section that the Bremsstrahlung background was altered. Indeed, a relationship pertinent to the phenomenon is the Duane-Hunt displacement law:

$$U_A \lambda_{\min} = 1.2398 \times 10^{-6}\,\text{Vm} \tag{5}$$

Edge absorption and Moseley's law

With a firm understanding of the X-ray emissions of copper, we now seek to probe the interaction of X-rays with matter. In particular, we seek to investigate K-edge absorption for a range of elements and relate the findings to Moseley's law. To execute this experiment, it is required that materials of differing atomic number be placed as absorbers in the X-ray beamline and an energy spectrum collected for each absorber. You have access to a range of scattering materials; however, it is critical that you handle the materials in a safe manner.

Scattering samples for X-ray absorption		
Chemical	Atom	Atomic number
Zinc powder	Zinc	30
Germanium dioxide	Germanium	32
Selenium powder	Selenium	34
Potassium bromide	Bromine	35
Strontium sulfate	Strontium	38
Silver nitrate	Silver	47
Tungsten oxide	Tungsten	74
Mercury oxide	Mercury	80
Lead dioxide	Lead	82
Bismuth oxide	Bismuth	83

Table 2: Scattering targets

When absorption samples are being used, ensure to replace the small (2 mm) diaphragm in the beamline with the larger (5 mm) diaphragm to permit a greater X-ray flux. You may also need to adjust the acquisition time to ensure sufficient counts are detected.

Extension projects

L edge absorption Relativistic corrections [4]

Appendix

Scan parameters

Provided your scan parameters are in the correct ballpark, there is often no "correct" set of scan parameters; however, some combinations of parameters can produce cleaner signals. The following parameters should yield an acceptable scan; however, it is advised that you modify and set the parameters to the values you fell appropriate.

High-resolution emission spectra

Goniometer parameters	
Parameter	Value
Mode	1:2 coupling mode
Detector angle	8°
Crystal	LiF
Absorber	None
Crystal start angle	4°
Crystal stop angle	55°
Crystal increment	0.1°
Integration time	2.0 s

Edge-absorption spectra

Goniometer parameters	
Parameter	Value
Mode	1:2 coupling mode
Detector angle	8°
Anode voltage	35 kV
Anode current	1 mA
Crystal	LiF
Absorber	None
Crystal start angle	4°
Crystal stop angle	21°
Crystal increment	0.1°
Integration time (no scattering material)	2.0 s
Integration time (with scattering material)	3.0 s

X-ray transition energies

A database of X-ray transitions is maintained by the National Institute of Standards and Technology (NIST), and can be accessed here.

References

[1] Walter Friedrich, Paul Knipping, and Max Laue. Interferenzerscheinungen bei roentgenstrahlen. *Annalen der Physik*, 346(10):971–988, 1913.

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[3] Henry Gwyn Jeffreys Moseley. Lxxx. the high-frequency spectra of the elements. part ii. *The London, Edinburgh, and Dublin Philosophical Magazine and Journal of Science*, 27(160):703–713, 1914.

[4] Tomas Soltis, Lorcan M Folan, and Waleed Eltareb. One hundred years of moseley's law: An undergraduate experiment with relativistic effects. *American Journal of Physics*, 85(5):352–358, 2017.