

SS16: X-ray diffraction and X-ray spectroscopy (mini-project)

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Abstract

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1 Introduction

X-rays are an important experimental tool and have been for more than a 100 years now — from the first Nobel Prize for their discovery, and still now. In this experiment, we will first verify Moseley's Law, and then use X-ray spectroscopy to analyse several samples of metal as well as coins from various places around the world.*finish this intro at the end.*

First, we introduce some of the background theory behind X-rays and their production.

1.1 X-ray atomic spectra

The emission spectra of atoms is due to energy transition of shell electrons from a higher energy level to a lower energy level. We are interested in X-rays specifically, which are due to transitions in the inner electron levels of high- Z atoms. These are shielded from the outer electron layers (the higher Z , the better the shielding **CHECK**) and are mostly unaffected by the chemical structure of the sample i.e. by the surrounding atoms. Therefore, we can take an energy level of an electron to be $E_n = -R(Z)/n^2$ where $R(Z) = R_\infty(Z - b)^2$ is the modified Rydberg constant for a given Z , b is some parameter depending on atom, and R_∞ is the hydrogen Rydberg constant. Then, the energy of an emitted photon by a transition from level n_i to n_f is given by:

$$\varepsilon = R(Z) \left(\frac{1}{n_f^2} - \frac{1}{n_i^2} \right). \quad (1)$$

We can rewrite this at fixed n_i , n_f (i.e. for a given line) as:

$$\sqrt{\varepsilon} = m \times Z + C, \quad (2)$$

where m , C are some constants. This relationship is known as 'Moseley's Law'.

The name convention of the energy levels of low- n in X-ray notation is described in Table 1. Usually, the lines which are due to a transition from a higher energy level to a K1 level are referred to as K-series; similarly for L-series and so on.

| Quantum numbers | | | | Atomic notation | X-ray notation |
|-----------------|---|-----|-----|-----------------|----------------|
| n | l | s | j | | |
| 1 | 0 | 1/2 | 1/2 | 1S1/2 | K1 |
| 2 | 0 | 1/2 | 1/2 | 2S1/2 | L1 |
| 2 | 1 | 1/2 | 1/2 | 2P1/2 | L2 |
| 2 | 1 | 1/2 | 3/2 | 2P3/2 | L3 |
| 3 | 0 | 1/2 | 1/2 | 3S1/2 | M1 |
| 3 | 1 | 1/2 | 1/2 | 3P1/2 | M2 |
| 3 | 1 | 1/2 | 3/2 | 3P3/2 | M3 |
| 3 | 2 | 1/2 | 3/2 | 3D3/2 | M4 |
| 3 | 2 | 1/2 | 5/2 | 3D5/2 | M5 |

Table 1: Table describing the connection between X-ray notation and Atomic notation. cite source, fix notation upper/lower index. Maybe add the old Siegbahn notation - used later in text?

1.2 X-ray production

In practice, we produce X-rays by bombarding a sample of some material with high energy electrons or X-rays which removes one of the inner shell electrons from the sample atom. This produces a vacancy in the shell. The vacancy is filled by some electron of a higher n orbit (see Figure 1), and the fall of potential energy of the electron is compensated by an emitted X-ray (ignoring higher order effects like the *Auger effect*).

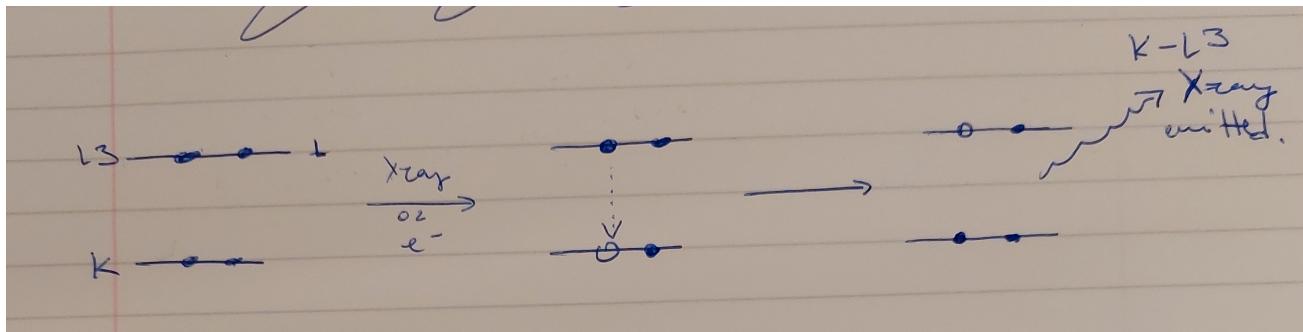


Figure 1: Schematic of the emission process for a K-L3 transition.

Usually, small laboratory X-ray tubes use an electron source. Because of that, we observe a continuum of X-ray radiation imposed on top of the emission lines. This radiation is called *bremsstrahlung* (“braking”) radiation. Its source is the interaction between decelerating electrons and stationary charges in the sample lattice. The maximal energy of a bremsstrahlung continuum is limited by the energy of the decelerating particle. If electrons accelerated by a potential difference V , then the maximal energy is given by:

$$\varepsilon_{\max} = eV. \quad (3)$$

Suppose we use one such continuous X-ray source for the production of X-rays from a sample. We will take the K-series as an example. To produce a vacancy, need an X-ray of energy $\varepsilon_{\text{in}} = -E_1 = R(Z)$, which will be provided by a collision with an electron with maximum energy ε_{\max} in the case of the X-ray source. The resultant energy of the emitted X-ray will be given by (1). Take for example the K-L transition. Its energy will therefore be (ignoring fine-structure):

$$\varepsilon_{\text{out}} = R(Z) \left(1 - \frac{1}{2^2} \right). \quad (4)$$

A process like this one, in which $\varepsilon_{\text{in}} \neq \varepsilon_{\text{out}}$, is called *fluorescence*.

1.3 Composition analysis

As said above, the X-ray emission is relatively independent of the composition of the sample. We can use this fact to perform a composition analysis on a sample by exposing it to the continuous bremsstrahlung of a source; this will allow the different components to fluoresce. The signal will give us information not only *which* elements are present (given that the source is high enough to allow fluorescence), but their relative contribution — the intensity of the signal will depend only on the number density n . Suppose we measure the intensity of a two-metal alloy with the peaks in the K-L3 line as I_a and I_b . This tells us that:

$$\frac{I_a}{I_b} = \frac{n_a}{n_b}, \quad (5)$$

where n_a and n_b are the number densities of materials a and b. From that, we could easily calculate percentage compositions for number densities or mass densities ($\rho = n \times (\text{molar mass})$).

2 Experiment

Our experiment consists of the setup presented in Figure 2. First, we have an X-ray source with a molybdenum anode, which produces some characteristic K_α X-rays that can be used for diffraction experiments (see the first experiment part of SS16 [put reference](#)) and some continuous bremsstrahlung (see Section 1.2 for more details). These X-rays are focused through a circular aperture towards a target sample. The incident X-rays excite inner shell electrons, and the targets emit mostly in the characteristic X-rays of K,L, and M series. We detect these via an energy spectrometer that is sensitive in the region of our experiment. The setup parameters are: tube voltage $V = 35\text{ kV}$, emission current 1 A, exposure time $t = \text{ask Darryl}$. This, according to (3) and (4), allows us to produce, for example, K_α X-rays with energy up to 26.25 keV from samples with atomic numbers up to at least 50, ignoring contribution of b .

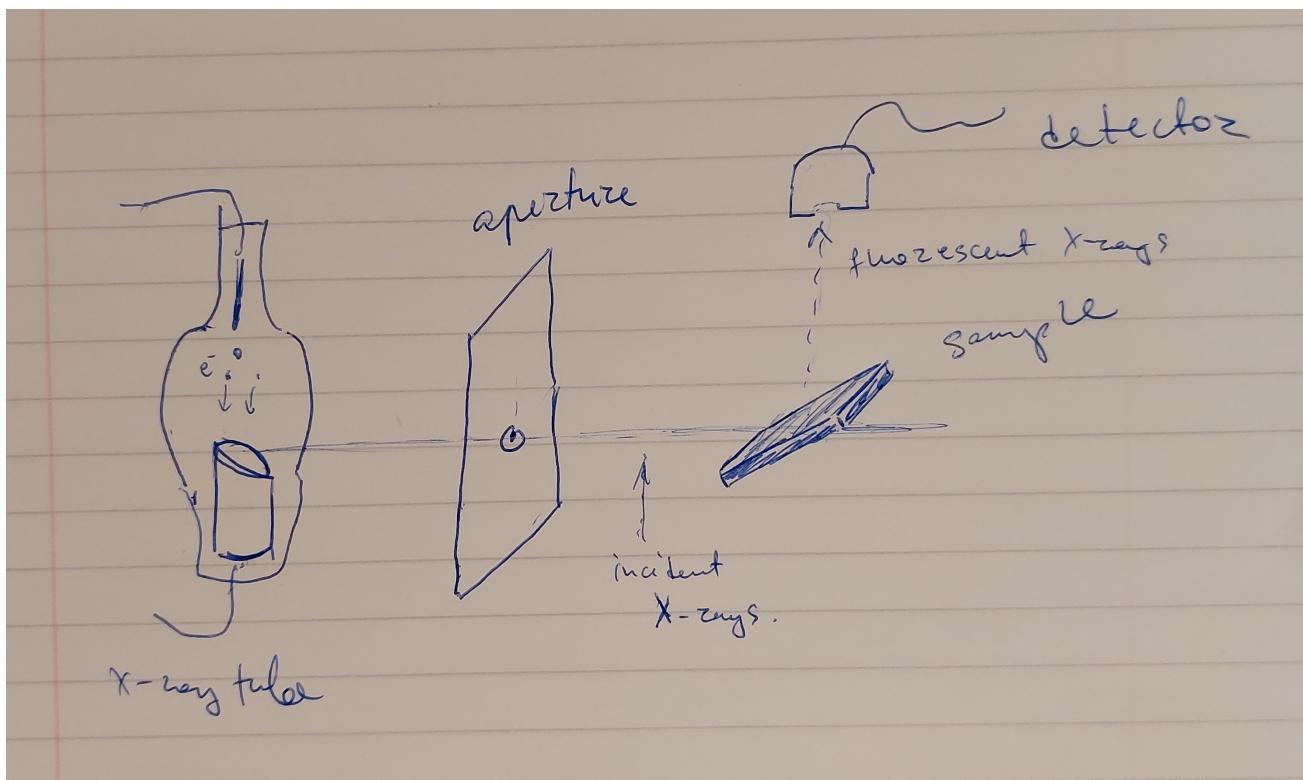


Figure 2: Diagram of the experimental setup. More info in the text of Section 2.

To operate the apparatus and extract data, we use the software provided by the labs *CASSY Lab 2*. The program has a built in database for the X-ray emission lines of most elements. It also allows for calculating a peak centre and fitting gaussian profiles with specified energy to find the intensity of emission lines.

Before we can do any work that has to do with determining energies or referring to energy values from a database, need to set the scaling of the measurement apparatus. We will do so with two of the labelled provided samples - for Fe and Ag. These cover a wide range of values in the bremsstrahlung spectrum, so they are a good way to set the scaling. The particular lines which we use to calibrate are the Fe K_{α} with $E_{Fe} = 6.40 \text{ keV}$ and the Ag K_{α} with $E_{Ag} = 22.17 \text{ keV}$. We also have to note the observational lower and upper limits, respectively 2 keV to 4 keV and $\approx 35 \text{ keV}$.

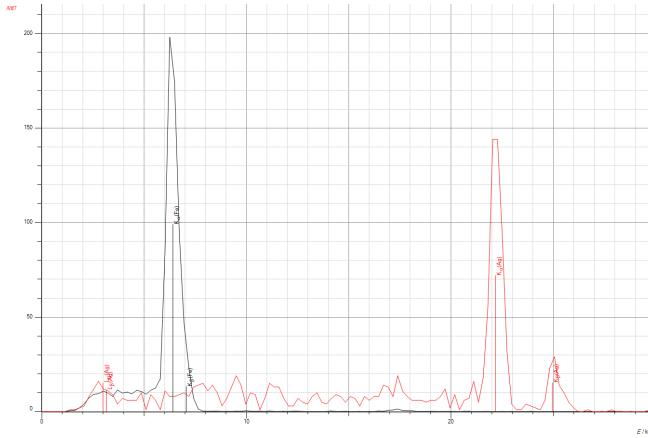


Figure 3: These are the Fe (black) and Ag (red) spectra used for calibration.

The first part of the experiment will test the validity of Moseley's Law with the help of several samples of known substances.

The second part and third part of the experiment will utilise our knowledge about X-rays to learn more about the composition of some samples provided for us, and about some metal coins from around the world.

3 Result and Analysis

3.1 Moseley's Law

We measure the line energy values from the labelled samples. These are mainly in two groups:

- those, whose K_{α} lines lie in the observable range (excluding our two calibration points);
- those, whose L_{α} lines lie in the observable range.

We expect to see a straight line fit for the two groups of lines. The scaling constant m depends purely on the line we are observing, i.e. on the n_i, n_f for the given line. We can predict the ratio of the constants for the two series. Following (1) and Moseley's law, we get:

$$m_K \propto \left(1 - \frac{1}{2^2}\right)^{1/2} = \sqrt{\frac{3}{4}}, \quad m_L \propto \left(\frac{1}{2^2} - \frac{1}{3^2}\right)^{1/2} = \sqrt{\frac{5}{36}} \Rightarrow \frac{m_K}{m_L} = \sqrt{\frac{27}{5}}. \quad (6)$$

Results presented in Figure 4.

We see that these both follow our expectations - have good fits to the model. We can deduce that our description of X-ray emission works sufficiently well for our purposes (given experimental accuracy etc), so we can proceed to use X-ray K and L series lines as a signature of a given element in spectral analysis.

Also, can calculate ratio from given values:

$$\text{predicted: } \frac{m_K}{m_L} = \sqrt{\frac{27}{5}} = 2.324, \quad \text{observed: } \frac{m_K}{m_L} = \frac{0.1026}{0.0411} = 2.496. \quad (7)$$

Agreement is not so good, but we have not taken into account many other effects like change of energy due to being in a state $l \neq 0$ etc. So, not too bad. **can expand a bit maybe.**

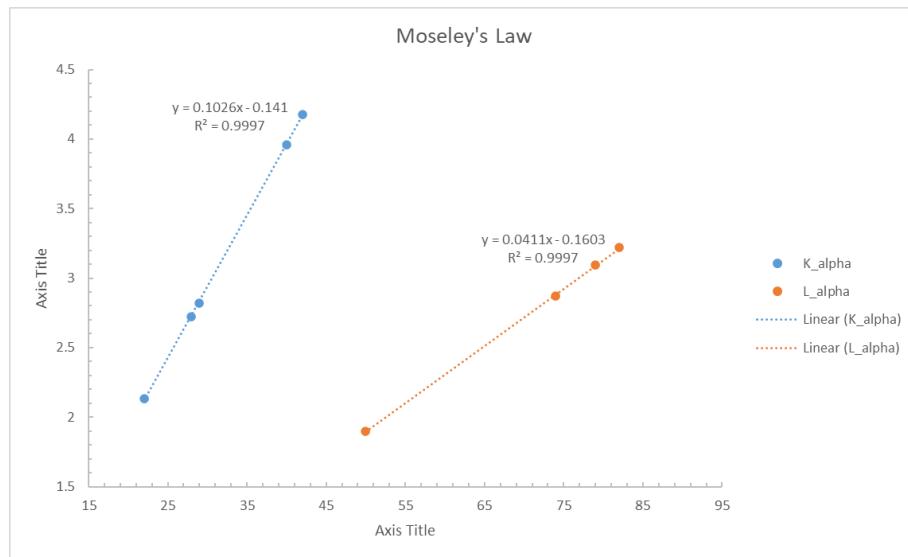


Figure 4: $\text{sqrt}(\text{energy})$ vs Z number for given samples: [list samples](#). produce better final figures.

3.2 Provided alloys and semiconductors

Now, can move to the analysis of samples. We will use fluorescence to determine the composition of the semiconductors from the non-MP part and of some unlabelled samples that are provided in the physics lab.

3.2.1 Semiconductors

The semiconductors are part of the non-MP experiment, where we determined their structure by determination of the lattice constant. Here, we provide an independent confirmation of our previous results.

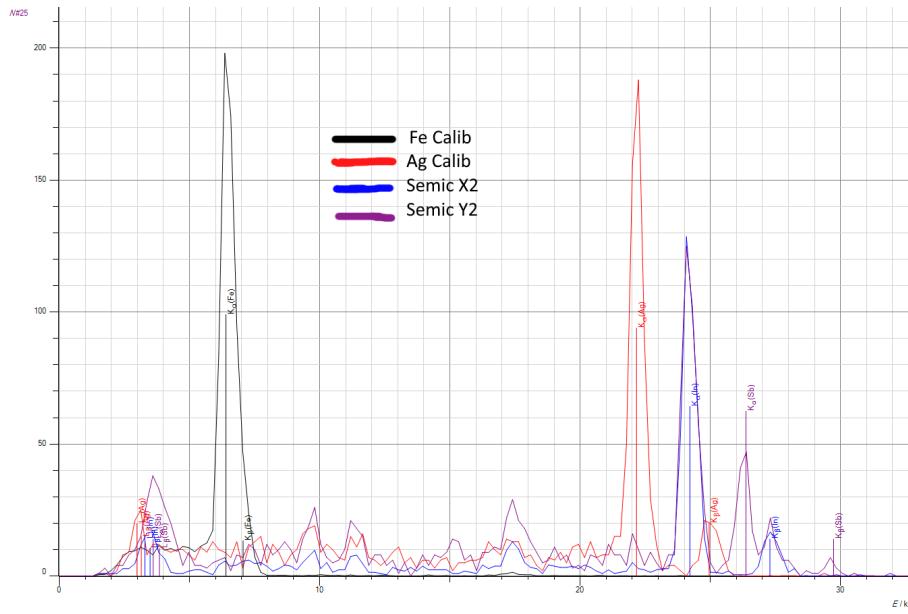


Figure 5: Fluorescence spectra of the Fe, Ag calibration and semiconductors labelled as X2 and Y2 in sample boxes provided. We see that X2 and Y2 show traces of In, and Y2 shows additionally traces of Sb. [prob should go to appendix](#).

We see that semiconductor Y2 shows traces of In and Sb, a group III and V elements respectively. This is a very common structure for semiconductors, so this is reassuring ([expand?](#)). In addition, this confirms our result from the first part of the practical, where we arrived at the conclusion that the semiconductor is an InSb via lattice diffraction. We can measure intensities of the K_α lines for both,

and calculate the ratios:

$$\frac{n_{\text{In}}}{n_{\text{Sb}}} = \frac{I_{\text{In}}}{I_{\text{Sb}}} = \frac{122.5}{42.8} \approx 2.86 \Rightarrow p_{\text{In}} = \frac{n_{\text{In}}}{n_{\text{In}} + n_{\text{Sb}}} = 74\% \quad p_{\text{Sb}} = 26\% \quad (8)$$

For sample X2, we see a trace of In only. Checking with our result from the diffraction analysis, we see that the dopant is P (phosphorus). The characteristic X-rays of P in K-series (most energetic ones) have energies around 2 keV, which is around our detection limit of about 2 keV to 4 keV. This highlights one of the shortcomings of our method: it is limited in detecting only those elements, which have X-ray emission lines in the range of the bremsstrahlung and the range of operation of the detector.

3.2.2 Unlabelled alloys

The lab has two boxes of unlabelled metallic samples - alloys of different metals. We can determine their composition and ratios by using [sec 1.3](#). Result is presented in Table 2.

| sample | element | line | intensity | Kalpha/Kbeta | k | prop | by mass |
|--------|----------|--------|-----------|--------------|----------|----------|-------------|
| b1-1 | Cr | Kalpha | 381.9 | 5.550872093 | 0.422877 | 0.297198 | 0.277555397 |
| b1-1 | Cr | Kbeta | 68.8 | | | | |
| b1-1 | Fe | Kalpha | 903.1 | 4.138863428 | | 0.702802 | 0.722444603 |
| b1-1 | Fe | Kbeta | 218.2 | | | | |
| b1-2 | Cu | Kalpha | 2110.2 | 6.136086072 | 1.783921 | 0.640794 | 0.691408371 |
| b1-2 | Cu | Kbeta | 343.9 | | | | |
| b1-2 | Zn | Kalpha | 1182.9 | 17.09393064 | | 0.359206 | 0.308591629 |
| b1-2 | Zn | Kbeta | 69.2 | | | | |
| b1-3 | Cu | Kalpha | 3947.9 | 14.32994555 | 1.265515 | 0.558599 | 0.613815022 |
| b1-3 | Cu | Kbeta | 275.5 | | | | |
| b1-3 | Zn | Kalpha | 3119.6 | 16.51455797 | | 0.441401 | 0.386184978 |
| b1-3 | Zn | Kbeta | 188.9 | | | | |
| b2-a | unusable | | | | | | |
| b2-b | unusable | | | | | | |
| b2-c | Fe | Kalpha | 1351.9 | 3.736594804 | 2.062081 | 0.673425 | 0.694639809 |
| b2-c | Fe | Kbeta | 361.8 | | | | |
| b2-c | Zn | Kalpha | 655.6 | 8.569934641 | | 0.326575 | 0.305360191 |
| b2-c | Zn | Kbeta | 76.5 | | | | |
| b2-d | Fe | Kalpha | 1443.6 | 3.921760391 | 3.421664 | 0.773841 | 0.790561437 |
| b2-d | Fe | Kbeta | 368.1 | | | | |
| b2-d | Zn | Kalpha | 421.9 | 9.743648961 | | 0.226159 | 0.209438563 |
| b2-d | Zn | Kbeta | 43.3 | | | | |
| b2-e | unusable | | | | | | |
| b2-f | Fe | Kalpha | 1443.3 | 3.885060565 | 2.559496 | 0.719061 | 0.738463241 |
| b2-f | Fe | Kbeta | 371.5 | | | | |
| b2-f | Zn | Kalpha | 563.9 | 9.367109635 | | 0.280939 | 0.261536759 |
| b2-f | Zn | Kbeta | 60.2 | | | | |

Table 2: Table of the fitted intensities in alloy samples. Some of the measurements made had signal comparable with the background due to a human error during data acquisition; included here for completeness. [add a column with line energy, include siunitx for number formatting, prob delete the unusable ones](#)

We can first check for consistency of the fits by looking at the ratios of intensities for K_{α} and K_{β} , which should be approximately the same for all samples as it depends on the structure of the individual atom. Samples b2-c,d,f have close values for both the Fe and Zn lines. Since these two elements do not

overlap in the spectrum (ref appendix here), these ratios should be close to the true ratio. We would expect to observe similar ratios in the other samples for Fe and Zn.

For sample b1-1, the Fe ratio is a little above 4, so we can say that the fit was pretty good. For samples b1-2,3, the Zn ratio is twice the one measured above. The fit here is pretty poor since Cu and Zn are respectively $Z = 29$ and $Z = 30$. Their spectra overlap by a lot — the K_α of Zn is between K_α and K_β of Cu. This, combined with the low resolution of our detector, makes it pretty difficult to fit here. Nevertheless, the K_α lines are much stronger, so we can still try to extract the composition of the alloys (especially since they are close here).

Alloy composition is usually presented by mass in practical applications. These results are in the last column of Table 2.

3.3 International coins

The lab has a box of various coins from around the world, the content of which we can again interpret using our fluorescence method, similar to the analysis in Section 3.2.2.

| No | Country | Denomination | Ni | Cu | Zn | Other |
|----|----------------|----------------|-----|-----|-----|-------|
| 3 | United Kingdom | 50p old | yes | yes | no | no |
| 4 | United Kingdom | 50p new | yes | yes | no | no |
| 5 | United Kingdom | 20p new | yes | yes | no | no |
| 6 | United Kingdom | 10p old | yes | yes | no | no |
| 7 | United Kingdom | 10p new | yes | no | no | no |
| 8 | United Kingdom | 2p new | no | yes | no | no |
| 9 | Singapore | 1 dollar old | yes | yes | no | no |
| 10 | Singapore | 50c old | yes | yes | no | no |
| 11 | Singapore | 20c new | yes | no | no | no |
| 12 | Bulgaria | 50 stotinki | yes | yes | yes | no |
| 13 | Bulgaria | 5 stotinki | no | yes | no | no |
| 14 | European Union | 50c | no | yes | yes | no |
| 15 | European Union | 2c old | no | yes | no | no |
| 16 | European Union | 2c new | no | yes | no | no |
| 17 | Romania | 10 bani | yes | no | no | no |
| 18 | Norway | 1 krone | yes | yes | no | no |
| 19 | Switzerland | 20 centim | yes | yes | no | no |
| 20 | Sweden | 1 krona | yes | yes | no | no |
| 21 | Japan | 1 yen | no | no | no | no |
| 22 | Hong Kong | 10c | no | yes | yes | no |
| 23 | Uganda | 200 schillings | yes | yes | no | no |
| 24 | USA | 25c | yes | yes | no | no |
| 25 | USA | 1c | no | yes | yes | no |
| 26 | Canada | 25c | yes | yes | no | Fe? |
| 27 | Brazil | 5 centavos | no | yes | no | no |

Table 3: Table of metal composition for a selection of international coins.

When looking at plots of the spectra, we see that almost all coins are made predominantly out of Ni, Cu, and Zn with $Z = 28$ to 30 respectively. The spectra are overlapping, so similarly to the b1-2,3 samples in Section 3.2.2, we cannot really fit the samples well for all α, β lines. Nevertheless, we can still determine if one of the three elements is present. Additionally, we note down any additional features of the spectra that are of interest. The results can be summarised in Table 3. Two exceptions to the general rule are obvious - the 1 Yen and 25c Canadian coins. We can compare our results with the information from the institutions that issue these coins.

The official information lists the 1 Yen coin to be made of 100% aluminium cite. The K_α line for aluminium is at 1.49 keV, out of the range of our setup. This is the reason we do not see any signal in

our spectrum.

The official information informs us that the 25c canadian coin is a 94% steel core with 6% Ni-Cu plating. When we look at the spectrum, however, the strength of the Fe line is much less than the Ni and Cu. This is due to the fact that the X-rays do not penetrate deep into the coin. If a thick enough coating is applied, we could even fully shield the steel core. In fact, several coins on our list follow this pattern, e.g. the new 2p and 10p coins.

3.3.1 Why Ni,Cu,Zn?

One obvious question that comes up from these results is why are alloys of Ni, Cu, and Zn the choice? There are several properties of these alloys that make them preferable for coin minting.

Historically, coins were most often made from gold, silver, and copper, with their value determined by the value of the metal in the coin. With the inflation of prices, however, the metal in the coins gradually became more expensive than their monetary value. There was a need for new materials to be implemented while keeping many of the familiar and useful properties of old coins.

Firstly, there are visual considerations of the coins. Ni-Cu and Cu-Zn alloys can have varying colours depending on the ratio of elements used. Ni-Cu are usually silver in appearance, while Cu-Zn alloys are usually golden in appearance, with small amounts of other additives sometimes present, like in the case of the *nordic gold* alloy used for the make of the 50 euro cent coin.

Secondly, the cost considerations required some inexpensive materials to be used so that mass production is practical and coins are not produced at a loss. Due to inflation, this has led to the change of coin composition throughout the years. One example is the UK 10p coin. While the old coin was made from pure Ni25Cu75, the new coin is made from 94% steel clad in 6% Ni. The method of implementing a cheaper core while using Ni,Cu,Zn for an outer layer also keeps the appearance the same or similar, which is one of the reasons for the approach.

Thirdly, the minting of the coins includes making the alloys and producing the coins with intricate detailing on their surfaces. The alloying of the specific ratios required by the specifications can be a difficult task outside of the specialised large scale industrial complexes. The three metals also allow for the detailing on different design coins — their alloys have been used to produce detailing by artisans for centuries (millennia?). Both of these specifics make the production of good counterfeit coins more difficult, which brings more security to the currency.

Finally, these alloys are very practical for everyday use. Coins, in their daily use, are exposed to the elements and physical wear. These alloys are very resistant to rusting (although Ni-Cu are often subject to formation of *patina*) and have high durability against physical stresses like bending, stretching etc. This is another reason for the cladding method — this gives them resistance against the elements and the physical toughness also helps preserve the detailing on the surface.

3.3.2 Cost-based analysis of coins.

This is a potential direction to investigate. Main idea — calculate the “value” of a coin via its weight+official composition to see if anything interesting appears (coins worth more than their value, similar value for all coins etc.)

maybe additional analysis based on raw material cost of coins + a deeper inquiry?

4 Conclusions

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A Energy spectra plots

Add more energy spectra plots here, which do not fit/not vital for main text.

B Extracted intensity data

Add data here as tables, refer to it when needed.

| No in measur | Country | Denomination | Ni, % | Cu, % | Zn, % | Other, % | Weight, g |
|--------------|----------------|----------------|-------|-------|-------|----------------------|------------|
| 3 | United Kingdom | 50p old | 25 | 75 | 0 | 0 | |
| 4 | United Kingdom | 50p new | 25 | 75 | 0 | 0 | |
| 5 | United Kingdom | 20p new | 16 | 84 | 0 | 0 | |
| 6 | United Kingdom | 10p old | 25 | 75 | 0 | 0 | |
| 7 | United Kingdom | 10p new | 6 | 0 | 0 | mild Steel - 94 | |
| 8 | United Kingdom | 2p new | 0 | 6 | 0 | mild Steel - 94 | |
| 9 | Singapore | 1 dollar old | 2 | 92 | 0 | Al - 6 | 8.05+-0.20 |
| 10 | Singapore | 50c old | 25 | 75 | 0 | 0 | 8.56+-0.20 |
| 11 | Singapore | 20c new | ? | 0 | 0 | steel - ? | 6.56+-0.28 |
| 12 | Bulgaria | 50 stotinki | 10 | | | 0 | 5 |
| 13 | Bulgaria | 5 stotinki | | | | steel - ? | 3.5 |
| 14 | European Union | 50c | 0 | 89 | 5 | Al - 5, Sn -1 | 7.8 |
| 15 | European Union | 2c old | | x | | 0 | 3.06 |
| 16 | European Union | 2c new | | x | | 0 | 3.06 |
| 17 | Romania | 10 bani | ? | 0 | 0 | steel - ? | 4 |
| 18 | Norway | 1 krone | 25 | 75 | 0 | 0 | 4.35 |
| 19 | Switzerland | 20 centim | 25 | 75 | 0 | 0 | 4 |
| 20 | Sweden | 1 krona | 25 | 75 | 0 | 0 | |
| 21 | Japan | 1 yen | 0 | 0 | 0 | al-100 | 1 |
| 22 | Hong Kong | 10c | | | | 0 | 1.85 |
| 23 | Uganda | 200 schillings | ~30 | ~70 | 0 | 0 | 8.5 |
| 24 | USA | 25c | | | | 0 | |
| 25 | USA | 1c | 0 | 2.5 | 97.5 | 0 | 2.5 |
| 26 | Canada | 25c | 2.2 | 3.8 | 0 | Steel AISI 1006 - 94 | 4.4 |
| 27 | Brazil | 5 centavos | 0 | | | 0 | |

Table 4: Table of official info about the international coins. Notable are the coins with predominantly steel composition with a coating of one fo the three main elements. [CITATIONS](#)