# Qualitative X-ray fluorescence spectroscopy of metals - Moseley's law


Physics | Modern Physics | Production & use of X-rays

| Difficulty level | Group size | Preparation time | Execution time |
|---|---|---|---|
| hard | 2 | 45+ minutes | 45+ minutes |

This content can also be found online at:

http://localhost:1337/c/5f7c4b939d495b0003ea63a6


Robert-Bosch-Breite 10
37079 Göttingen

Tel.: 0551 604 - 0
Fax: 0551 604 - 107

info@phywe.de
www.phywe.de

# General information

## Application


Setup

Most applications of X rays are based on their ability to pass through matter. Since this ability is dependent on the density of the matter, imaging of the interior of objects and even peaple becomes possible. This has wide usage in fields such as medicine or security.



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## Other information (1/2)

**Prior knowledge**

The prior knowledge required for this experiment is found in the Theory section.

**Main principle**

Various metal samples are subjected to polychromatic X-rays. The energy of the resulting fluorescence radiation is analysed with the aid of a semiconductor detector and a multi-channel analyser. The energy of the corresponding characteristic X-ray lines is determined, and the resulting Moseley diagram is used to determine the Rydberg frequency and the screening constants.

## Other information (2/2)

**Learning objective**

The goal of this experiment is to get to investigate the spectra of fluorescence radiation.

**Tasks**

1. Calibrate the semiconductor energy detector with the aid of the characteristic radiation of the tungsten X-ray tube.
2. Record the spectra of the fluorescence radiation that are generated by the metal samples.
3. Determine the energy values of the corresponding characteristic $K_\alpha$- and $K_\beta$-lines.
4. Determine the Rydberg frequency and screening constants with the aid of the resulting Moseley diagrams.



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Robert-Bosch-Breite 10
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## Theory (1/3)

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When X-rays interact with matter, they lose energy due to Compton scattering, pair production, and photoelectric effects. In the range of energy that is available during this experiment, the photoelectric effect plays the most important role. This means that in the atom on one of the lower shells an electron is ejected due to the absorbed photon energy.
The now free space is taken by an electron from one of the higher shells. The energy that is produced during this process can be used for the ejection of another electron from one of the higher shells (Auger effect) or for the generation of a photon (fluorescence radiation). In a first approximation, the uninvolved electrons are regarded as fixed during these processes. Their influence is taken into consideration by the introduction of a so-called screening constant $\sigma$, which reduces the effect of the nuclear charge.
If relativistic and spin-orbit coupling effects are neglected, the binding energy $E_n$ of an electron on the nth shell of an atom can be described in an approximate manner by Bohr's atom model:

$$E_n = -\frac{m_e e^4}{8\epsilon_0^2 h^2}(Z-\sigma)^2 \frac{1}{n^2} \qquad (1)$$

## Theory (2/3)

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With the introduction of the Rydberg frequency

$$R = \frac{m_e e^4}{8\epsilon_0^2 h^3}$$

(1) leads to $E_n = -R \cdot h(Z-\sigma)^2 \frac{1}{n^2}$ (2)

The screening constant depends on the location of the excited electron and on the configuration of the other electrons. Due to the migration of an electron from $n_2$ to $n_1$ ( $n_2 > n_1$ ) and in accordance with (2), the energy of the released radiation is:

$$E_n = -R \cdot h \left( \frac{(Z-\sigma_{n_1})^2}{n_1^2} - \frac{(Z-\sigma_{n_2})^2}{n_2^2} \right) \qquad (3)$$

Electron mass $m_e = 9.109 \cdot 10^{-31}$ kg

Elementary charge $e = 1.602 \cdot 10^{-19}$ C

Planck's constant h = 6.6256 $\cdot 10^{-34}$ Js

Permittivity $\epsilon = 8.854 \cdot 10^{-12}\ C^2/Nm^2$

Atomic number Z

Screening constant $\sigma$

Prinicipal quantum number n



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Robert-Bosch-Breite 10
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## Theory (3/3)

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Instead of two screening constants for the migration from $n_2$ to $n_1$, a mean screening constant $\sigma - 1, 2$ is introduced. As a result, (3) is simplified to:

$$E_n = -R \cdot h(Z - \sigma_{1,2})^2 \left(\frac{1}{n_1^2} - \frac{1}{n_2^2}\right) \quad (4)$$

If $\sqrt{E}$ is plotted as a function of Z, the so-called Moseley diagram results. With $n_2$ = 2 and $n_1$ =1 (characteristic $K_\alpha$-line), (4) leads to:

$$\sqrt{E} = \frac{\sqrt{3}}{2}\sqrt{Rh} \cdot Z - \frac{\sqrt{3}}{2}\sqrt{Rh \cdot \sigma_{2,1}} \quad (5)$$

Correspondingly, the following applies from the migration from $n_3$ to $n_1$ with a mean screening constant σ3,1 (characteristic $K_\beta$-line):

$$\sqrt{E} = \frac{\sqrt{8}}{2}\sqrt{Rh} \cdot Z - \frac{\sqrt{8}}{2}\sqrt{Rh \cdot \sigma_{3,1}} \quad (6)$$



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# Equipment

| Position | Material | Item No. | Quantity |
|---|---|---|---|
| 1 | XR 4.0 expert unit, 35 kV | 09057-99 | 1 |
| 2 | XR 4.0 X-ray goniometer | 09057-10 | 1 |
| 3 | XR4 X-ray Plug-in Cu tube | 09057-51 | 1 |
| 4 | XR 4.0 X-ray material upgrade set | 09165-88 | 1 |



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# Setup and Procedure

## Setup (1/2)

- Screw the adapter ring onto the inlet tube of the energy detector and connect the signal and supply cables to the corresponding ports of the detector with the aid of the right-angle plugs.
- Connect the signal and supply cables to the corresponding ports in the experiment chamber of the X-ray unit. In Figure 1, the port for the signal cable is marked in red and the port for the supply cable is marked in green. Connect the external X RED ports of the x-ray unit (see Fig. 2) to the multi-channel analyser (MCA). Connect the signal cable to the “Input” port and the supply cable to the “X-Ray Energy Det.” port of the MCA.


Fig. 1: Connectors in the experiment chamber


Fig. 2: Connection of the multi-channel analyser



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Robert-Bosch-Breite 10
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## Setup (2/2)

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- Secure the energy detector in the holder of the swivel arm of the goniometer. Lay the two cables with sufficient length so that the goniometer can be swivelled freely over the entire range. Connect the multi-channel analyser and computer with the aid of the USB cable.
- Insert the diaphragm tube with the 2-mm-aperture.
- Bring the goniometer block and the detector to their respective end positions on the left. Bring the detector to the 90° position in the 1:2 coupling mode

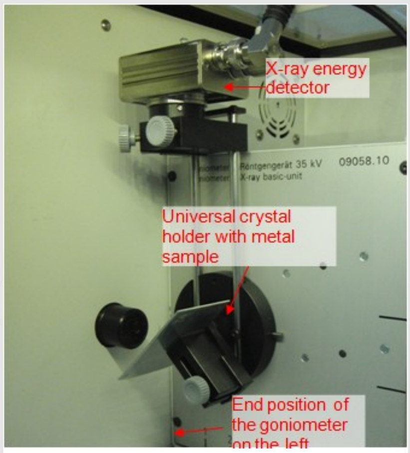


Fig. 3: Goniometer set-up

## Procedure (1/4)

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- Bring the goniometer block and the detector to their respective end positions on the right.
- Insert the tube with the 1mm-aperture into the exit tube of the X-ray tube.
- With the X-ray unit switched on and the door locked, bring the detector to the 0° position. Then, shift the detector by some tenths degree out of the zero position in order to reduce the total rate.
- Operating data of the tungsten X-ray tube: Select an anode voltage $U_A$ = 25 kV and an anode current $I_A$ = 0.02 mA and confirm these values by pressing the “Enter” button.
- Switch on the X-radiation

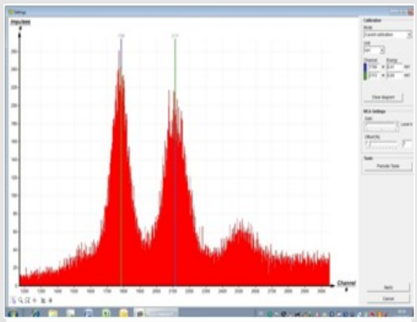

Fig. 4: calibration of the multi-channel analyser



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## Procedure (2/4)

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- In the MEASURE program, select “Multi channel analyser” under “Gauge”. Then, select “Settings and calibration”. After the “Calibrate” button has been clicked, a spectrum can be measured. The counting rate should be < 300 c/s. Energy calibration settings: - 2-point calibration, - Unit = keV, Gain = 2 – Set the offset so that low-energy noise signals will be suppressed (usually a few per cent are sufficient), See Fig 4.
- Measuring time: 5 minutes. Use the timer of the X-ray unit.
- Make the two coloured calibration lines congruent with the line centres of the two characteristic X-ray lines. The corresponding energy values (see e.g. P2544705) $\mathrm{E(L_3M_5/L_3M_4)}$ = 8,41keV and $\mathrm{E(L_2N_4)}$ = 9,69 keV are entered into the corresponding fields, depending on the colour. (Note: Since a separation of the lines $\mathrm{L_3M_4}$ and $\mathrm{L_3M_5}$ Lines is not possible, the mean value of both lines is entered as the energy of the line).
- Name and save the calibration.

## Procedure (3/4)

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**Task 2: Spectrum recording**

- Insert the tube with the 2-mm-aperture.
- Bring the goniometer block and the detector to their respective end positions on the left. Bring the detector to the 90° position in the 1:2 coupling mode.
- Insert the metal sample with the universal crystal holder (sample at 45°).
- Operating data of the molybdenum X-ray tube: Adjust an anode voltage $\mathrm{U_A}$ = 35 kV and an anode current so that the counting rate is $\leq$ 300 c/s.
- Measuring time: 3 minutes (use the timer of the X-ray unit).



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Robert-Bosch-Breite 10
37079 Göttingen

Tel.: 0551 604 - 0
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info@phywe.de
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## Procedure (4/4)

**Task 3: Evaluation of the measurement curves**

- In order to determine the line energy, switch from the bar display to the curve display. To do so, click “Display options” and then “Interpolation and straight lines”.
- Extend the relevant line section with the aid of the zoom function
- Then, select the curve section with . Open the window “Function fitting . Then, select “Scaled normal distribution” and confirm.
- Find the line centroid of the normal distribution with “Peak analysis” or determine it with the function “Survey”

# Evaluation



Robert-Bosch-Breite 10
37079 Göttingen
Tel.: 0551 604 - 0
Fax: 0551 604 - 107
info@phywe.de
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# Task 2

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**Record the spectra of the fluorescence radiation that are generated by the metal samples.**

Fig. 5 shows the fluorescence spectra of different metals.

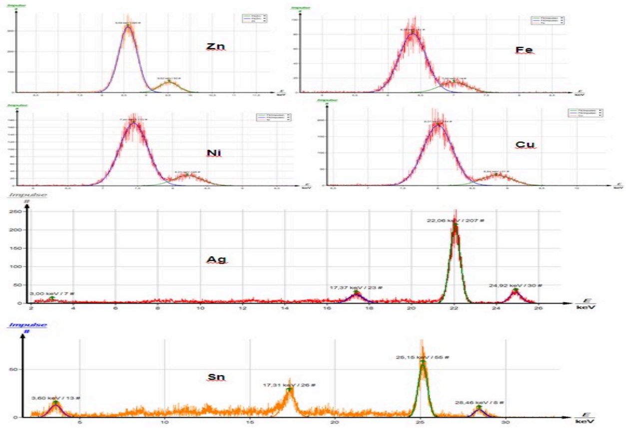


Fig. 5: Fluorescence spectra of various metals

# Task 3

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| A | B | C | D | E | F |
|---|---|---|---|---|---|
| Element | Z | $\mathrm{E}(\mathrm{K}_\alpha)$ exp. [keV] | $\mathrm{E}(\mathrm{K}_\beta)$ exp. [keV] | $\mathrm{E}(\mathrm{K}_\alpha)$ lit. [keV] | $\mathrm{E}(\mathrm{K}_\beta)$ lit. [keV] |
| Fe | 26 | 6.39 | 7,03 | 6,397 | 7,056 |
| Ni | 28 | 7.47 | 8,26 | 7,474 | 8,265 |
| Cu | 29 | 8.04 | 8,90 | 8,039 | 8,905 |
| Zn | 30 | 8.63 | 9,57 | 8,627 | 9,572 |
| Mo* | 42 | 17.38 | 19,56 | 17,427 | 19,608 |
| Ag | 47 | 22.07 | 24,91 | 22,076 | 24,942 |
| Sn | 50 | 25.15 | 28,46 | | |

* The Mo spectrum was obtained through the analysis of the primary radiation of the Mo X-ray tube and is not caused, therefore, by any of the metal samples

**Determine the energy values of the corresponding characteristic $\mathrm{K}_\alpha$- and $\mathrm{K}_\beta$-lines.**

The results of the spectra evaluation are listed in the table. The columns C and D include the energy values of the characteristic $\mathrm{K}_\alpha$- and $\mathrm{K}_\beta$ -lines that were obtained from the spectra that are shown in Figure 6. For comparison, the columns E and F show the corresponding literature, with the mean value of the $\mathrm{K}_{\alpha_1}$- and $\mathrm{K}_{\alpha_2}$-lines as the energy of the $\mathrm{K}_\alpha$-line.



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Robert-Bosch-Breite 10
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## Task 4

**Determine the Rydberg frequency and screening constants with the aid of the resulting Moseley diagrams.**

Figure 6 shows the Moseley lines for the characteristic $\mathrm{K}_\alpha$ and $\mathrm{K}_\beta$ lines. R can be calculated with the gradients of the regression lines that were added.

The gradient of the regression line of the $\mathrm{K}_\alpha$-lines is defined as follows:

$$\mathrm{m} = \frac{\sqrt{3\mathrm{Rh}}}{2}$$

Transformation gives:

$$\mathrm{m}^2 = \frac{4\mathrm{Rh}}{4}$$

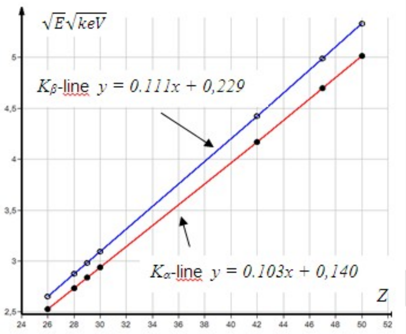


Abb. 6: Moseley lines of the $\mathrm{K}_\alpha$- and $\mathrm{K}_\beta$-lines

## Task 4 (part 2)

Enter the value for the slope of the $\mathrm{K}_\alpha$ Moseley line and transfer keV to J. Thus:

$$\mathrm{R} = 3.41 \cdot 10^{15}\,\mathrm{s}^{-1}$$

With Z = 0 and Rh = 13.6 eV, the intercepts of the corresponding Moseley lines lead to: $\sigma_{2,1} \approx$ 1.5 and $\sigma_{3,1} \approx$ 2.2.

The values of the screening constants that were determined with the aid of Bohr's atom model have only limited validity, since more detailed calculations (Hartree) show that the radial charge density distribution of some electrons, e.g. of the 3p electron, have a secondary maximum near the nucleus. It is nevertheless plausible that $\sigma_{2,1}$ > 1 and $\sigma_{3,1}$ > $\sigma_{2,1}$.since during the $\mathrm{K}_\alpha$ process, the remaining 1s electron and in addition also the uninvolved 2s electrons screen the nuclear charge, whereas during the $\mathrm{K}_\beta$ transition all of the electrons of the L level have an additional screening effect.



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