# Inherent fluorescence radiation of the X-ray energy detector


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/5f7c40c29d495b0003ea631b

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# General information

## Application

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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**

Fluorescence radiation of the elements of a sample can cause fluorescence radiation inside the detector and its housing if the energy is sufficiently high. As a result, the spectrum may include lines that are not caused by the sample.
For the detection of potential additional lines, the detector is subjected to monochromatic X-radiation with the aid of a monocrystal. For comparison, the fluorescence spectra of pure metal samples are measured.

## Other information (2/2)

**Learning objective**

The goal of this experiment is to investigate the inherent fluoescence radiation of the X-ray energy detector.

**Tasks**

1. Calibrate the semiconductor detector with the aid of the characteristic radiation of the copper X-ray tube.
2. Irradiate the X-ray energy detector with monoenergetic X-rays that are produced by the Bragg reflection on an LiF monocrystal. Measure the resulting fluorescence spectrum.
3. Determine of the energy of the spectrum lines.
4. Assign the lines to elements by comparing the measured values with table values.
5. Comparative measurement and evaluation of the fluorescence spectra of pure metal



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## Theory

For a qualitative analysis of fluorescence spectra, the line peaks are used to determine the associated energy levels. These values are then compared to the corresponding table values (e.g. “Handbook of Chemistry and Physics”, CRCPress, USA). This method provides information concerning the composition of the sample. Due to background noise, peak superimposition, artefacts (see appendix), and the fluorescence radiation of the energy detector it is rather difficult to make a clear statement.
While the first three problems that are mentioned can be corrected by software-assisted evaluation programs, the background radiation can only be identified by preliminary experiments. If the energy level of the fluorescence radiation of the sample is sufficiently high, this radiation may cause additional fluorescence radiation on the detector components.



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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.
- 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.


Fig. 1: Connectors in the experiment chamber


Fig. 2: Connection of the multi-channel analyser



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## Setup (2/2)

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- Connect the multi-channel analyser and computer with the aid of the USB cable.
- 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 2:1 coupling mode (Fig. 3).

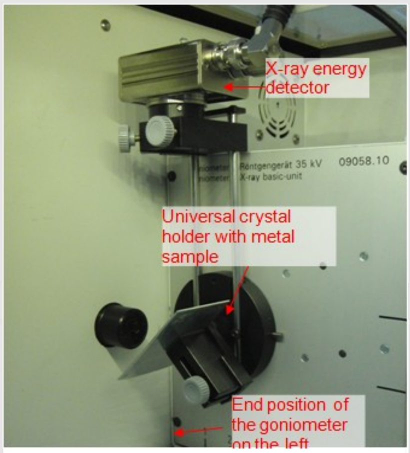


Fig. 3: Goniometer set-up

## 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

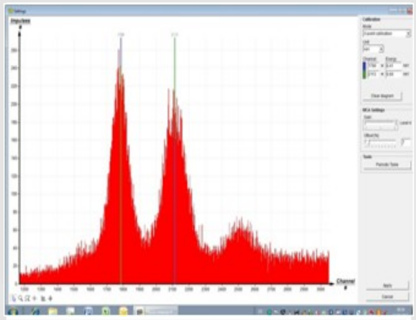

Fig. 4: calibration of the multi-channel analyser



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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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**A: Spectra recording with an LiF monocrystal**

- Use the tube with the 2-mm-aperture.
- Insert the LiF monocrystal and set it to 5.4° (detector at 10.8°).
- Operating data of the molybdenum X-ray tube: anode voltage $\mathrm{U_A}$ = 35 kV and an anode current so that the counting rates are ≤200 c/s.
- In the MEASURE program, select “Multi channel analyser” under “Gauge”. Then, select “Spectra recording”, X-Data = keV, and Interval width [channels] = 1. Use the same offset as before and select Gain = 2.
- Measuring time: ≥15 minutes. Use the timer of the X-ray unit.
- Name and save the measurement.



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## Procedure (4/4)

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**B: Spectra recording with metal samples (changes with regard to A)**

- Insert the universal holder with a metal sample and set it to 45° in the coupling mode (detector at 90°).
- Adjust the anode current so that the counting rates are again $\leq$200 c/s.
- Measuring time: $\leq$10 minutes

**C: Evaluation of the measurement curves**

- In order to determine the line position, switch from the bar display to the curve display. To do so, click “Display options” and then “Interpolation and straight lines”. If necessary, “smooth” the measurement curve slightly with 
- Extend the relevant line section with the aid of the zoom function 
- Find the line centroids with the function “Survey” 

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# Evaluation



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# Task 2

Figure 5 shows the spectrum of the fluorescence radiation of the detector, which was produced by monoenergetic X-radiation of approximately 32 keV via a Bragg reflection on an LiF monocrystal (glancing angle $\theta$ = 5.4°).

In accordance with Bragg's law, the wavelength $\lambda$ of the radiation that hits the detector can be calculated as follows:

$$2\mathrm{d} \cdot \sin(5.4^{\circ}) = \lambda = 57.9\,\mathrm{pm} \rightarrow \mathrm{E} \approx 32.5\,\mathrm{keV} \qquad (1)$$

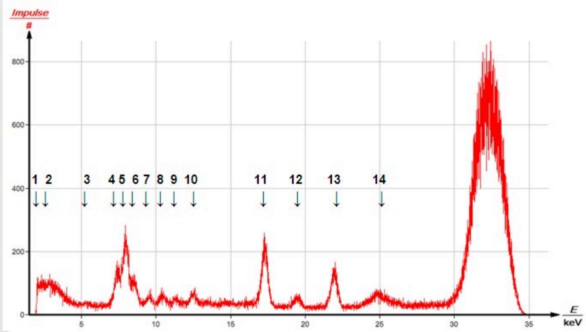


Fig. 5: Characteristic fluorescence spectrum of the detector components (energy of the primary radiation $\mathrm{E}_0$ = 32.5 keV)

# Task 3

For the assignment of the fluorescence lines, the following must be taken into consideration:

- The relaxations that follow the primary ionisation process can only take place if they fulfil the quantum-mechanical selection rules $\Delta \mathrm{j} = 0, \pm 1$ and $\Delta \mathrm{l} = \pm 1$ (j = total angular momentum, l = orbital angular momentum).
- In addition, the energy of the primary photon must be sufficiently high in order to be able to ionise the atom during the transition.
- There must be a low-intensity $\mathrm{K}_\beta$-line for every high-intensity $\mathrm{K}_\alpha$-line.
- In addition, it must be taken into consideration that the fluorescent yield decreases when the principal quantum number (K, L, ...) increases.

Table 1 shows the evaluation of the spectrum of Fig. 5.



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## Task 3 (part 2)

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| A | B | C | D | E |
|---|---|---|---|---|
| Line | E [keV] | Element | Transition | $E_{lit}$ [keV] |
| 1 | 2,46 | Mo | see Task 4 | 2,54 |
| 2 | 3,13 | Ag | $L_{\beta_1}/L_2M_4$ | 3,15 |
| 3 | 5,40 | Cr | $K_\alpha/KL_{2,3}$ | 5,41 |
| 4 | 7,46 | Ni | $K_\alpha/KL_{2,3}$ | 7,47 |
| 5 | 8,01 | Cu | $K_\alpha/KL_3$ | 8,04 |
| 6 | 8,58 | Au/Zn | $L_l/L_3M_1$ or $K_\alpha/KL_3$ | 8,49 / 8,61 |
| 7 | 9,60 | Au | $L_\alpha/L_3M_4$ | 9,63 |
| 8 | 10,50 | As? | | 10,51 |

Table 1: Assignment of the lines shown in Fig. 5

| A | B | C | D | E |
|---|---|---|---|---|
| Line | E [keV] | Element | Transition | $E_{lit}$ [keV] |
| 9 | 11,42 | Au | see Task 4 | 11,44 |
| 10 | 12,57 | Se? | | |
| 11 | 17,36 | Mo | $K_\alpha/KL_{2,3}$ | 17,42 |
| 12 | 19,56 | Mo | $K_\beta/KM_{2,3}$ | 19,61 |
| 13 | 22,02 | Ag | $K_\alpha/KL_{2,3}$ | 22,16 |
| 14 | 24,88 | Ag | $K_\beta/KM_{2,3}$ | 24,94 |
| 15 | 32,5 | E0 | | |

Table 1: Assignment of the lines shown in Fig. 5

## Task 4

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Line assignment:

- Line 1: unresolved, 4 molybdenum lines $L_{\beta_{2,3,4,5}}$ ($L_1M_{2,3}$ and $L_3N_{1,4,5}$) of the primary molybdenum radiation
- Lines 11/12: Compton-scattered, characteristic primary molybdenum radiation
- Lines 3, 4, and perhaps also 6 (zinc): detector housing material
- Lines 6, 7, and 9: semiconductor bonding, line 9 includes several unresolvable $L_\beta$-transitions
- Lines 2, 5, 13, and 14: assembly materials inside the housing
- Lines 8 and 10: no clear assignment



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## Task 5

Figures 6-8 show the fluorescence spectra of pure molybdenum, zircon, and zinc samples for comparison. Apart from the characteristic fluorescence lines of the corresponding primary radiations, the lines of Figure 5 can also be observed in these graphs, although with a reduced intensity (decrease in the fluorescent yield at lower primary energies). Since the energy of the primary fluorescence radiation of the zinc sample is too low (Fig. 6), no fluorescence is caused inside the detector.

Fig. 6: Fluorescence spectrum of a pure molybdenum sample

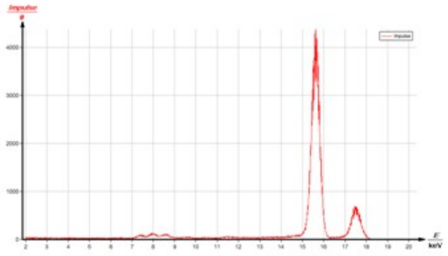
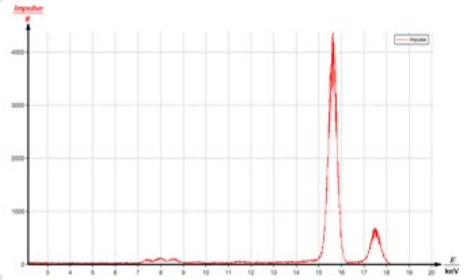


Fig. 7: Fluorescence spectrum of a pure zircon sample



## Task 5 (part 2)

Figure 9 shows the fluorescence spectra with an identical intensity scale up to an energy level of 15 keV.

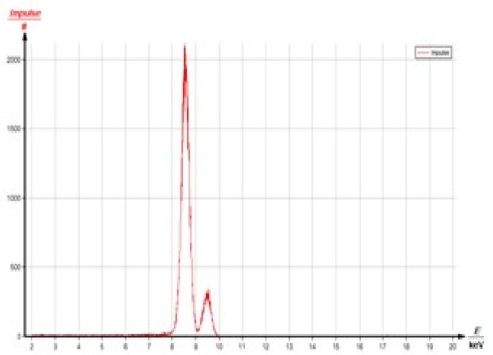
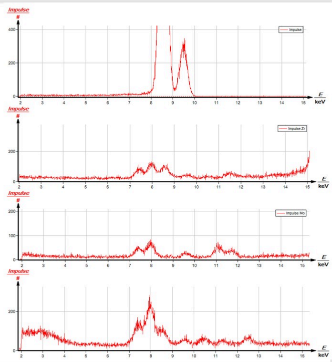


Fig. 8: Fluorescence spectrum of a pure zinc sample

Fig.9: Section of the fluorescence spectra with superimposed fitting curves a: primary radiation $E_0$ = 32.5 keV, b: Mo sample, c: Zr sample, d: Zn sample





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# Appendix

## Artefacts that may affect the analysis

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- Escape peaks: These peaks occur when the fluorescence radiation of energy E of the sample ionises the silicon atoms of the K-shell ($E_K$ = 1.8 keV) in the silicon crystal of the detector before electron-hole pairs are produced. In the spectrum, the fluorescence radiation that is reduced by this amount feigns a line with the energy E - 1.8 keV
- Silicon fluorescence peaks (only in the case of detectors with particularly low-energy sensitivity limits): These peaks occur when the radiation of the sample causes $K_\alpha$-radiation in the Si crystal, which is then detected by the detector.
- Sum peaks (only at high counting rates): These peaks occur when two X-ray quanta hit the detector closely on one another so that they cannot be registered separately by the multi-channel analyser.
- Peak shifts (only at high counting rates): They occur when two X-ray quanta hit the detector closely on one another and if the voltage pulse of the second quantum cannot be amplified sufficiently by the multi-channel analyser.



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