Inherent fluorescence radiation of the X-ray energy detector (Item No.: P2544201)

Curricular Relevance

| Area of Expertise: ILIAS | Education Level: Physik | Topic: Hochschule | Subtopic: Moderne Physik | Experiment: Röntgenphysik |

Difficulty	**Preparation Time**	**Execution Time**	**Recommended Group Size**
Difficult	1 Hour	2 Hours	2 Students

Additional Requirements:	**Experiment Variations:**
• PC	

Keywords:

Bremsstrahlung, characteristic X-radiation, fluorescence radiation, fluorescent yield, interference of X-rays, crystal structures, Bragg's law, Compton scattering, escape peaks, semiconductor energy detectors, multi-channel analysers

Overview

Short description

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.

Fig. 1: P2544201

This experiment is included in the upgrade package: XRM 4.0 X-ray material analysis.

Equipment

Position No.	Material	Order No.	Quantity
1	XR 4.0 expert unit, X-ray unit, 35 kV	09057-99	1
2	XR 4.0 X-ray goniometer	09057-10	1
3	XR 4.0 X-ray plug-in unit W tube	09057-81	1
4	Multichannel analyser	13727-99	1
5	XR 4.0 X-ray energy detector (XRED)	09058-30	1
6	XR 4.0 X-ray specimen set of 7 metals for X-ray fluorescence	09058-31	1
7	XR 4.0 X-ray Universal crystal holder for X-ray unit	09058-02	1
8	XR 4.0 X-ray LiF crystal, mounted	09056-05	1
9	measure Software multi channel analyser	14452-61	1
10	XR 4.0 XRED cable, 2 m	09058-35	1
11	XR 4.0 X-ray Diaphragm tube d = 1 mm	09057-01	1
12	XR 4.0 X-ray Diaphragm tube d = 2 mm	09057-02	1
13	Screened cable, BNC, l = 750 mm	07542-11	1

Tasks

1. Calibrate the semiconductor energy detector with the aid of the characteristic fluorescence radiation of the calibration sample.

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 samples.

Setup and Procedure

Setup

- Screw the adapter ring onto the inlet tube of the energy detector.

- 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 from the MCA to the appropriate connections in the experiment chamber of the X-ray unit (signal cable: red, supply cable: green (see Fig. 2)).

- Connect the external ports for the X RED of the x-ray unit (signal cable red, supply cable green, see Fig. 3) to the multi-channel analyse (MCA). Connect the signal cable via a screened BNC-cable to the "Input" port of the MCA 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 swivelling range.

- 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. 4).

Fig. 2: Connections in the experimentation area

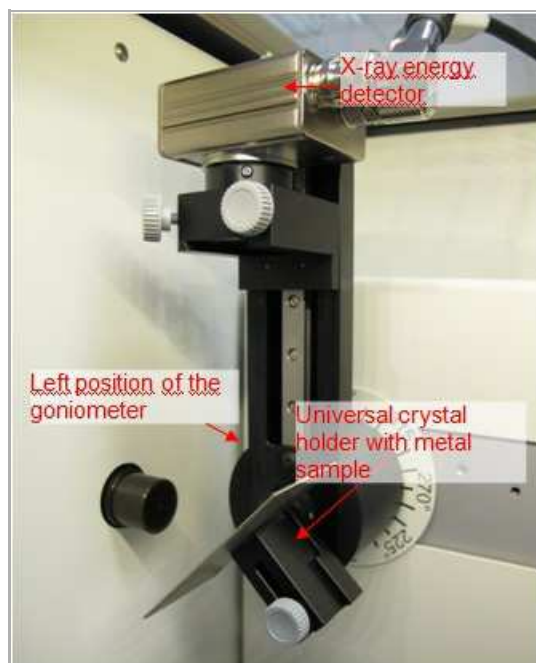Fig. 3: Connection at the external panel of the XR 4.0 X-ray expert unit to the MCA

Fig. 4: Set-up at the goniometer

Calibration of the multi-channel analyser
(if there is no other already existing calibration that can be used)

PHYWE excellence in science

Robert-Bosch-Breite 10
D - 37079 Göttingen

Tel: +49 551 604 - 0
Fax: +49 551 604 - 107

info@phywe.de
www.phywe.com

- 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

- 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 5.

- 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. P2544701) $E(L_3M_5/L_3M_4)$ = 8,41keV and $E(L_2N_4)$ = 9,69 keV are entered into the corresponding fields, depending on the colour. (Note: Since a separation of the lines L_3M_5 and L_3M_4 Lines is not possible, the mean value of both lines is entered as the energy of the line).

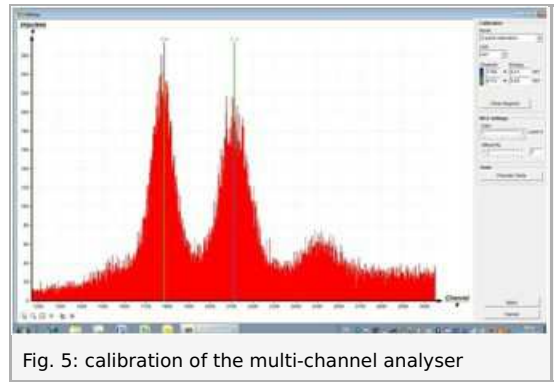- Name and save the calibration.

Fig. 5: calibration of the multi-channel analyser

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: Adjust an anode voltage 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.

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 ≤200 c/s.
- Measuring time: ≤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"
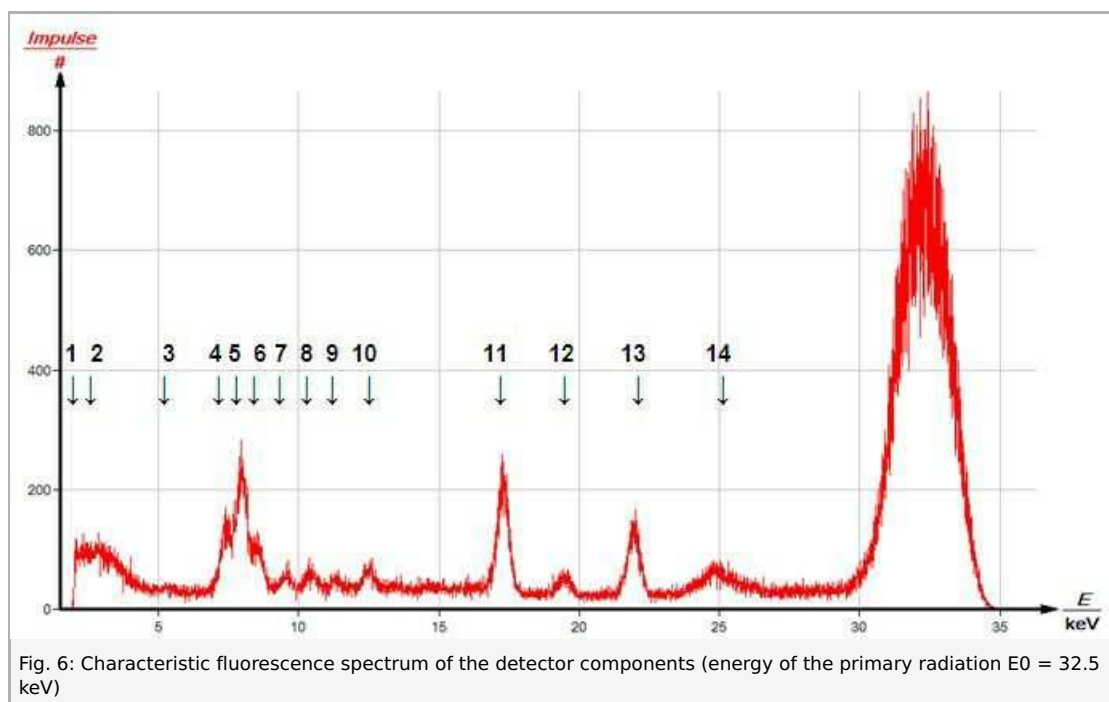
Theory and Evaluation

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.

Figure 6 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 ϑ = 5.4°).

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

$$2d \cdot sin5,4° = \lambda = 37,9pm \rightarrow \approx 32,5keV \qquad (1)$$
(d (LiF) = 201.4 pm)

Fig. 6: Characteristic fluorescence spectrum of the detector components (energy of the primary radiation E0 = 32.5 keV)

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 $\Delta j = 0, \pm1 and \Delta l = \pm1$ (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 K_β-line for every high-intensity K_α-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. 6.

Table 1: Assignment of the lines shown in Fig. 6

Line number	E / keV	Element	Name / transition	E_{Lit} / keV
1	2,46	Mo	see below	2,54
2	3,13	Ag	$L_{\beta1}/L_2M_4$	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_α/KL_3	8,04
6	8,58	Au und, od. Zn	L_l/L_3M_1 und, od. K_α/KL_3	8,49 / 8,61
7	9,60	Au	L_α/L_3M_4	9,63
8	10,50	As ?		10,51
9	11,42	Au	s.u.	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		

Line assignment:

- Line 1: unresolved, 4 molybdenum lines $L_{\beta2,3,4,6}$ ($L_1M_{2,3}$ 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_β-transitions
- Lines 2, 5, 13, and 14: assembly materials inside the housing
- Lines 8 and 10: no clear assignment

Figures 7-9 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 6 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. 7), no fluorescence is caused inside the detector.

Fig. 7: Fluorescence spectrum of a pure molybdenum sample

Fig. 8: Fluorescence spectrum of a pure zircon sample

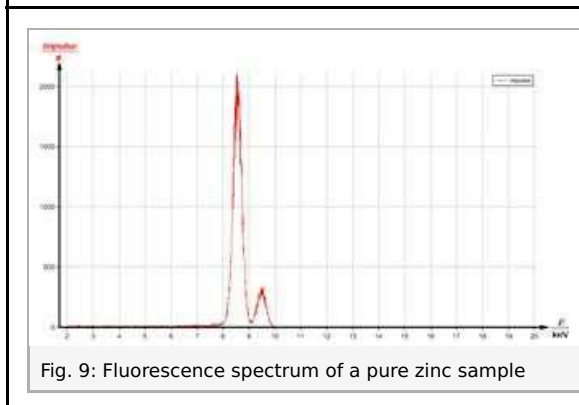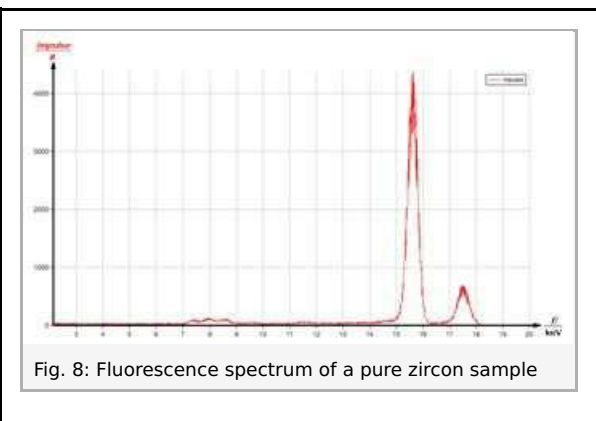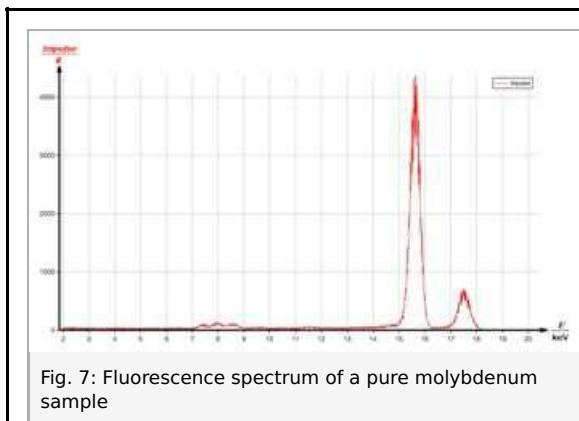Fig. 9: Fluorescence spectrum of a pure zinc sample

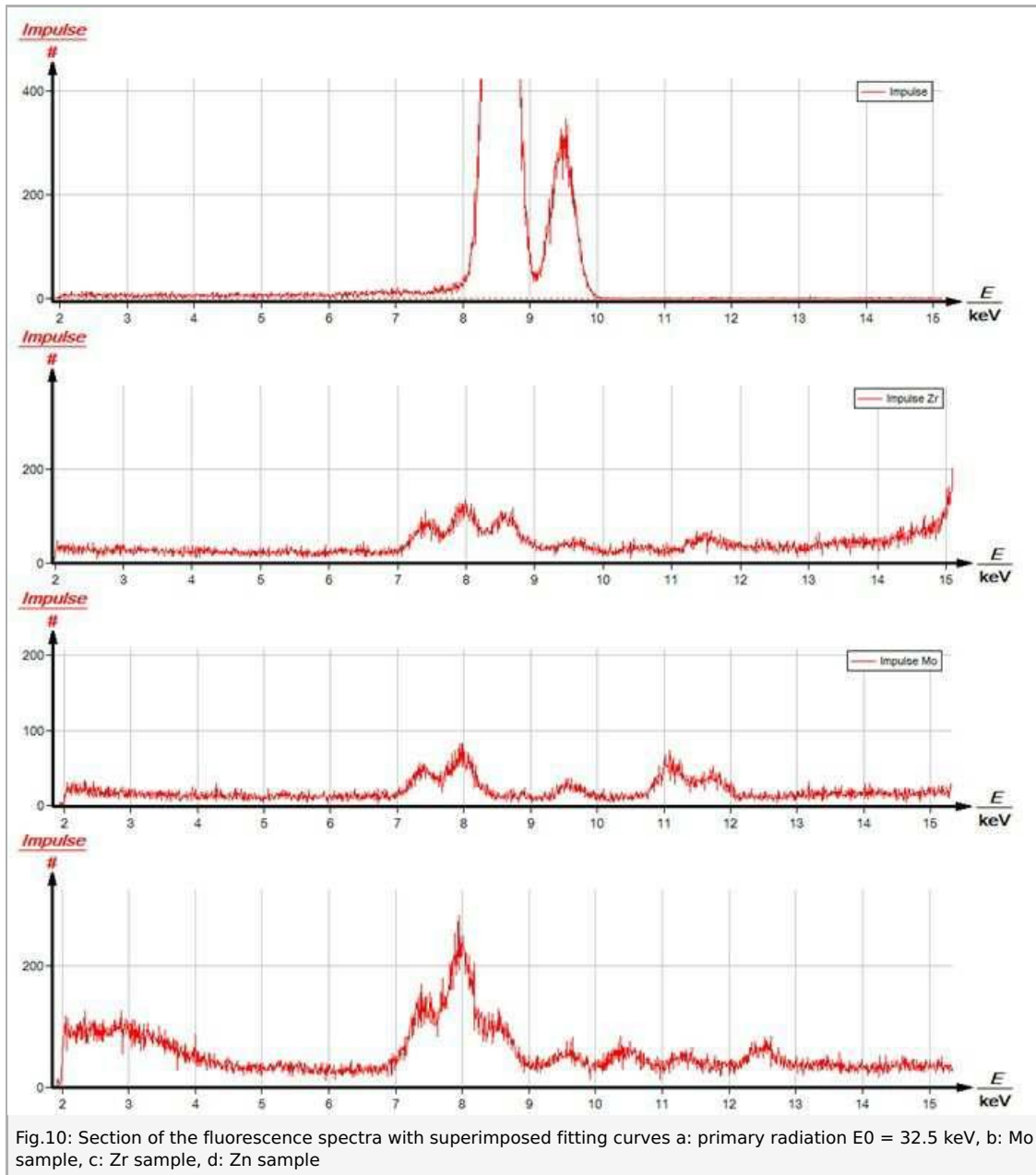Figure 10 shows the fluorescence spectra with an identical intensity scale up to an energy level of 15 keV.

Fig.10: Section of the fluorescence spectra with superimposed fitting curves a: primary radiation E0 = 32.5 keV, b: Mo sample, c: Zr sample, d: Zn sample

Appendix

Artefacts that may affect the analysis of fluorescence spectra:

- 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_α-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.