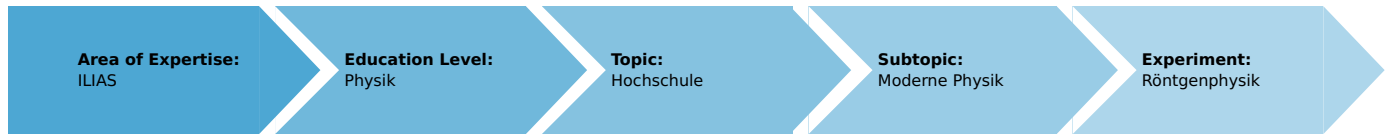


## Characteristic X-rays of molybdenum (Item No.: P2540201)

### Curricular Relevance



#### Difficulty



Difficult

#### Preparation Time



1 Hour

#### Execution Time



2 Hours

#### Recommended Group Size



2 Students

#### Additional Requirements:

- PC

#### Experiment Variations:

#### Keywords:

X-ray tubes, bremsstrahlung, characteristic X-radiation, energy levels, crystal structures, lattice constant, absorption of X-rays, absorption edges, interference, Bragg's law

## Overview

### Short description

#### Principle

An X-ray tube with a molybdenum anode generates X-radiation that is selected with the aid of a monocrystal as a function of the Bragg angle. A Geiger-Müller counter tube measures the intensity of the radiation. The glancing angles of the characteristic X-ray lines are then used to determine the energy.



Fig. 1: P2540201

## 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 Mo tube                | 09057-61  | 1        |
| 4            | Geiger-Mueller counter tube, 15 mm (type B) | 09005-00  | 1        |
| 5            | XR 4.0 X-ray LiF crystal, mounted           | 09056-05  | 1        |
| 6            | XR 4.0 potassium bromide (KBr) crystal      | 09056-01  | 1        |
| 7            | XR 4.0 Software measure X-ray               | 14414-61  | 1        |
| 8            | Data cable USB, plug type A/B, 1.8 m        | 14608-00  | 1        |
| 9            | XR 4.0 X-ray Diaphragm tube d = 1 mm        | 09057-01  | 1        |

## Tasks

1. Analyse the intensity of the molybdenum X-radiation as a function of the Bragg angle and with the aid of a **LiF** monocrystal.
2. Repeat task 1 with a **KBr** monocrystal as the analyser.
3. Determine the energy values of the characteristic X-rays of molybdenum based on the spectra and compare them with the values that were determined based on the corresponding energy-level diagram.

## Set-up and procedure

### Set-up

Connect the goniometer and the Geiger-Müller counter tube to their respective sockets in the experiment chamber (see the red markings in Fig. 2). The goniometer block with the analyser crystal should be located at the end position on the right-hand side. Fasten the Geiger-Müller counter tube with its holder to the back stop of the guide rails. Do not forget to install the diaphragm in front of the counter tube (see Fig. 3). Insert a diaphragm tube with a diameter of  $2\text{ mm}$  into the beam outlet of the tube plug-in unit for the collimation of the X-ray beam.

**For calibration:** make sure, that the correct crystal is entered in the goniometer parameters. Then, select "Menu", "Goniometer", "Autocalibration". The device now determines the optimal positions of the crystal and the goniometer to each other and then the positions of the peaks.

**Note:** Details concerning the operation of the X-ray unit and goniometer as well as information on how to handle the monocrystals can be found in the respective operating instructions.



Fig. 2: Connectors in the experiment chamber



Fig. 3: Set-up of the goniometer

### Procedure

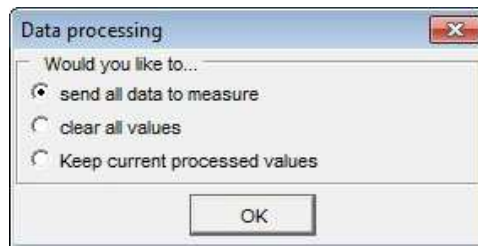
- Connect the X-ray unit via USB cable to the USB port of your computer (the correct port of the X-ray unit is marked in Fig. 4).



- Start the “measure” program. A virtual X-ray unit will be displayed on the screen.
- You can control the X-ray unit by clicking the various features on and under the virtual X-ray unit. Alternatively, you can also change the parameters at the real X-ray unit. The program will automatically adopt the settings.
- Click the experiment chamber to change the parameters for the experiment. Select the parameters as shown in Figure 6 for the LiF crystal. If you use the **KBr** crystal, select a start angle of  $3^\circ$  and a stop angle of  $30^\circ$ .
- If you click the X-ray tube, you can change the voltage and current of the X-ray tube. Select the parameters as shown in Fig. 7.
- Start the measurement by clicking the red circle:



- After the measurement, the following window appears:



- Select the first item and confirm by clicking OK. The measured values will now be transferred directly to the “measure” software.
- At the end of this manual, you will find a brief introduction to the evaluation of the resulting spectra.

**Note:** Never expose the Geiger-Müller counter tube to the primary X-radiation for an extended period of time.



Fig. 5: Part of the user interface of the software

Overview of the settings of the goniometer and X-ray unit:

- 1:2 coupling mode
- Gate time 2 s; angle step width 0.1°
- Scanning range 3° - 65° (LiF monocrystal), and 3° - 30° (**KBr** monocrystal)
- Anode voltage  $U_A = 35 \text{ kV}$ ; anode current  $I_A = 1 \text{ mA}$

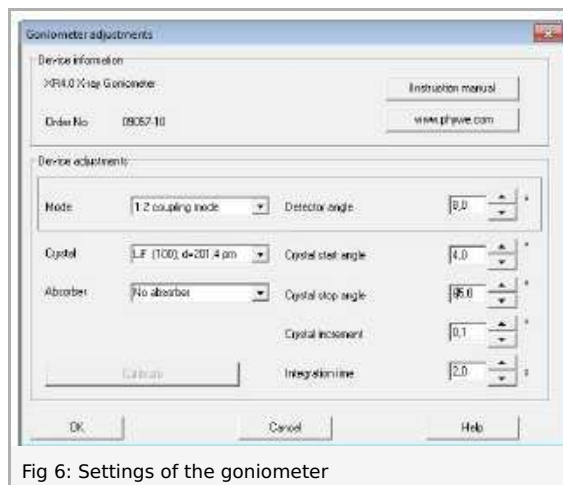


Fig 6: Settings of the goniometer

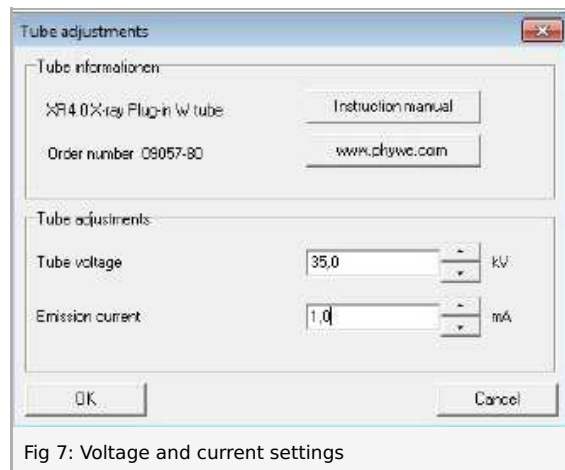


Fig 7: Voltage and current settings

## Theory and evaluation

### Theory

When electrons impinge on the metallic anode of the X-ray tube with a high level of kinetic energy, X-rays with a continuous energy distribution (the so-called bremsstrahlung) are produced. The spectrum of the bremsstrahlung is superimposed by additional discrete lines. If an atom of the anode material is ionised on the K shell following an electron impact, an electron from a higher shell can take up the free place while emitting an X-ray quantum. The energy of this X-ray quantum corresponds to the energy difference of the two shells that are involved in this process. Since this energy difference is atom-specific, the resulting radiation is also called characteristic X-radiation.

Figure 8 shows the energy level diagram of a molybdenum atom. Characteristic X-radiation that is produced following a transition from the L shell to the K shell is called  $K_{\alpha}$  radiation, while the radiation that is produced following a transition from the M shell to the K shell is called  $K_{\beta}$  radiation ( $M_1 \rightarrow K$  and  $L_1 \rightarrow K$  transitions are not allowed due to quantum-mechanical selection rules).

$\Delta l = \pm 1$  and  $\Delta j = 0, \pm 1$  selection rules for the dipole radiation

( $l$  = orbital angular momentum,  $j$  = total angular momentum)

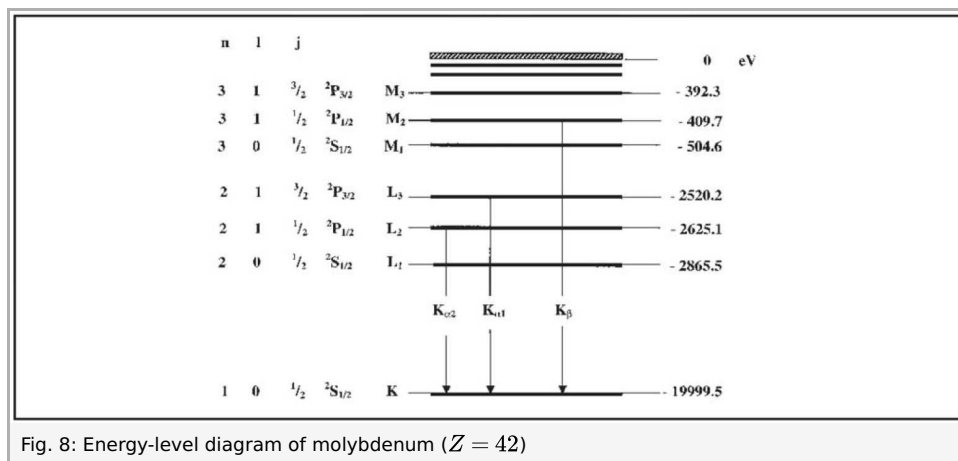


Fig. 8: Energy-level diagram of molybdenum ( $Z = 42$ )

The characteristic X-ray lines of molybdenum have the following energy levels (Fig. 8):

$$E_{K\alpha^*} = E_K - \frac{1}{2}(E_{L_2} + E_{L_3}) = 17.427 \text{ keV} \quad (1)$$

$$E_{K\beta} = E_K - E_{M_{2,3}} = 19.590 \text{ keV}$$

$E_{K\alpha^*}$  is the energetic mean value of the  $K_{\alpha 1}$  and  $K_{\alpha 2}$  lines.

The analysis of polychromatic X-rays is made possible through the use of a monocrystal. When X-rays of the wavelength  $\lambda$  impinge on the lattice planes of a monocrystal under the glancing angle  $\vartheta$ , the rays that are reflected on the lattice planes interfere with each other in a constructive manner provided that their path difference  $\Delta$  corresponds to an integral multiple of the wavelength. In accordance with Figure 9, Bragg's law applies to constructive interference:

$$2d \sin \vartheta = n\lambda \quad (2)$$

( $d$  = interplanar spacing;  $n = 1, 2, 3, \dots$ )

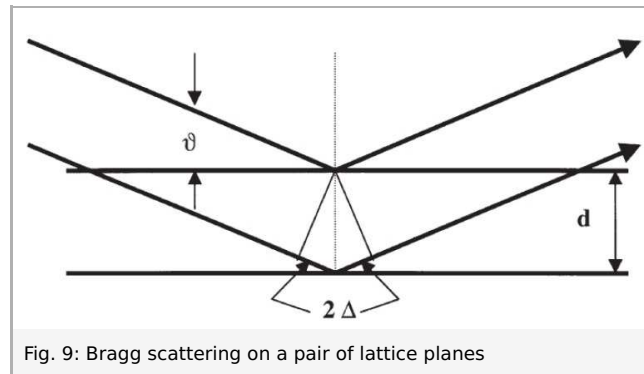


Fig. 9: Bragg scattering on a pair of lattice planes

If the interplanar spacing  $d$  is known, the wavelength  $\lambda$  can be determined with the aid of the glancing angle  $\vartheta$ . The energy of the radiation then results from:

$$E = h \cdot f = \frac{hc}{\lambda} \quad (3)$$

When combining (2) and (3), we obtain:

$$E = \frac{(n \cdot h \cdot c)}{(2d \sin \vartheta)} \quad (4)$$

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

Velocity of light  $c = 2.9979 \cdot 10^8 \text{ m/s}$

Interplanar spacing **LiF** (200)  $d = 2.014 \cdot 10^{-10} \text{ m}$

Interplanar spacing **KBr** (200)  $d = 3.290 \cdot 10^{-10} \text{ m}$

Equivalent  $1\text{eV} = 1.6021 \cdot 10^{-19} \text{ J}$

## Note

The data of the energy-level diagram were taken from the "Handbook of Chemistry and Physics", CRC Press Inc., Florida.

## Evaluation

In the following section, the evaluation of the data is described based on example results. Your results may differ from the results given below.

*Task 1: Analyse the intensity of the molybdenum X-radiation as a function of the Bragg angle and with the aid of a **LiF** monocrystal.*

Figure 10 shows the X-ray spectrum of molybdenum that was analysed with a **LiF** monocrystal. Welldefined lines are superimposed on the continuous bremsstrahlung spectrum. The glancing angles of these lines remain unaltered when the anode voltage is varied. This indicates that these lines are characteristic X-ray lines. The first three pairs of lines can be assigned to first-, second-, and third-order interferences ( $n = 1, 2, 3$ ). Due to the decreasing intensity of the spectrum in the direction of larger glancing angles, fourth- and fifth-order interferences are only visible (very faintly) for the  $K_{\alpha}$  line. The separation of the  $K_{\alpha}$  doublet can already be observed (very faintly) for  $n = 4$  and  $n = 5$  (see also the P2540701 and P2540801 experiments).



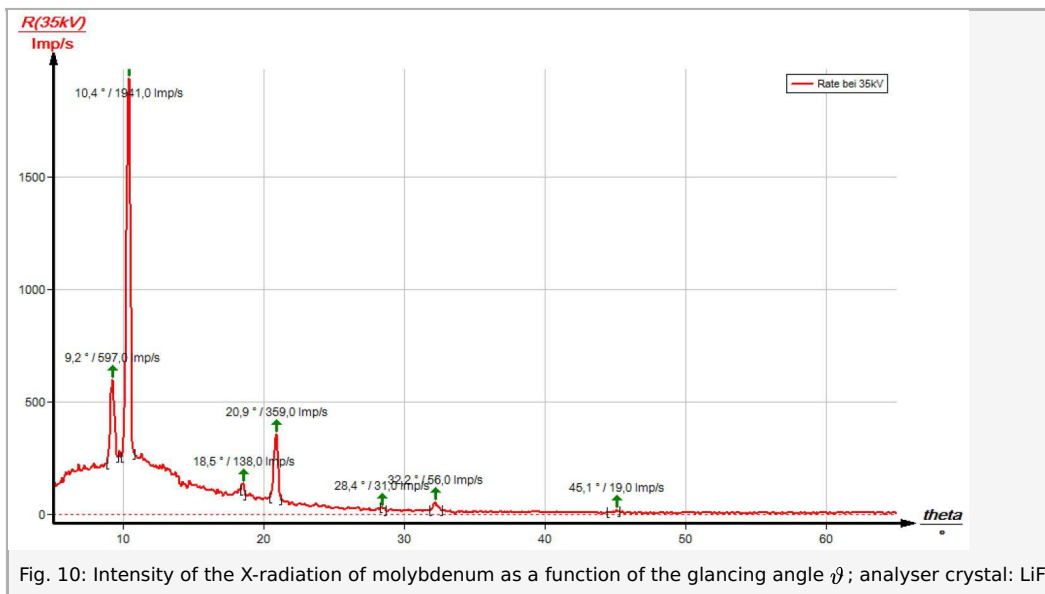


Fig. 10: Intensity of the X-radiation of molybdenum as a function of the glancing angle  $\vartheta$ ; analyser crystal: LiF

**Task 2:** Analyse the intensity of the molybdenum X-radiation as a function of the Bragg angle and with the aid of a **KBr** monocrystal.

If the LiF monocrystal is replaced by the KBr monocrystal (Fig. 11), interferences up to the fourth order can be observed. The spectrum of the bremsstrahlung in Fig. 11 shows clear intensity steps at  $\vartheta = 8.2^\circ$  und  $\vartheta = 16.5^\circ$ . They correspond to the *K*-edge absorption value of bromine ( $E_K = 13.474 \text{ keV}$ ) with  $n = 1$  or  $n = 2$  that can be expected in theory. The *K*-edge absorptions of potassium, lithium, and fluorine cannot be observed in this area of the bremspectrum, since the intensity is too low (for *K*- and *L*-edge absorption experiments, please refer to experiment P2541201).

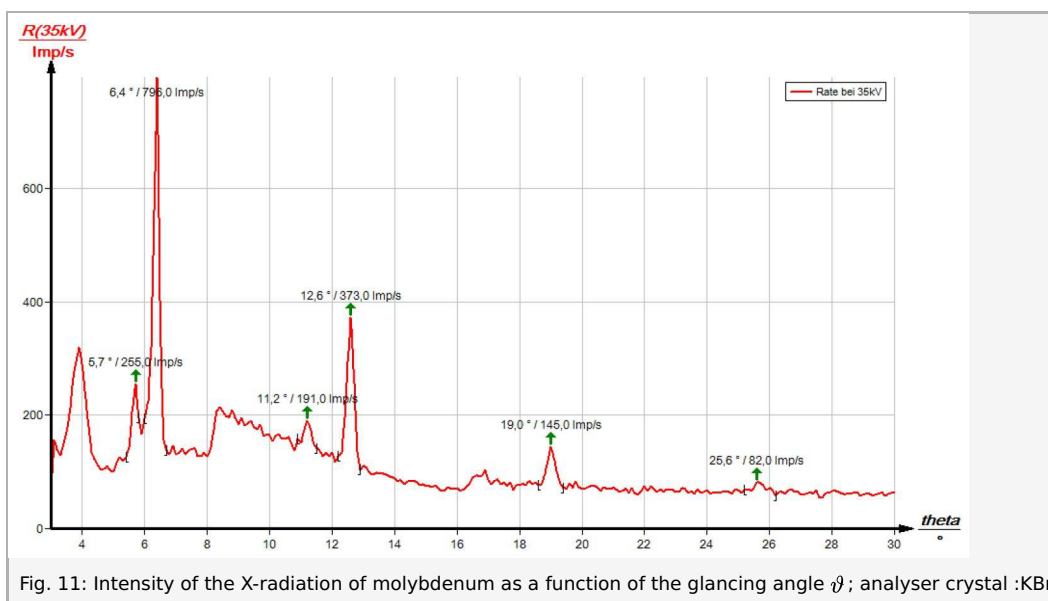


Fig. 11: Intensity of the X-radiation of molybdenum as a function of the glancing angle  $\vartheta$ ; analyser crystal :KBr

**Task 3:** Determine the energy values of the characteristic X-rays of molybdenum based on the spectra and compare them with the values that were determined based on the corresponding energy-level diagram.

Table 1 shows the glancing angles  $\vartheta$  that were determined with the aid of Figs. 10 and 11 and also the energy values for the characteristic X-ray lines of molybdenum that were calculated with the aid of equation (4).

Based on the energy values of the characteristic lines of Tasks 1 and 2, the following mean values result:  $E_{K_\alpha} = 17.244 \text{ keV}$  and  $E_{K_\beta} = 19.338 \text{ keV}$ . A comparison with the corresponding values of (1) shows good correspondence.

### Note

The evaluation of the two spectra can be varied as follows: Use the energy values of the characteristic lines that were

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determined for one of the spectra in order to determine the interplanar spacing  $d$  of the analyser crystal that was used for the other spectrum with the aid of equation (4).

|                    | $\theta/^\circ$ | Line       | $E_{\text{exp}}/\text{keV}$ |
|--------------------|-----------------|------------|-----------------------------|
| <b>LiF crystal</b> |                 |            |                             |
| $n=1$              | 10.4            | $K_\alpha$ | 17.100                      |
|                    | 9.2             | $K_\beta$  | 19.525                      |
| $n=2$              | 20.9            | $K_\alpha$ | 17.244                      |
|                    | 18.5            | $K_\beta$  | 19.401                      |
| $n=3$              | 32.2            | $K_\alpha$ | 17.324                      |
|                    | 28.4            | $K_\beta$  | 19.399                      |
| $n=4$              | 45.1            | $K_\alpha$ | 17.390                      |
| <b>KBr crystal</b> |                 |            |                             |
| $n=1$              | 6.4             | $K_\alpha$ | 16.823                      |
|                    | 5.7             | $K_\beta$  | 18.942                      |
| $n=2$              | 12.6            | $K_\alpha$ | 17.286                      |
|                    | 11.2            | $K_\beta$  | 19.425                      |
| $n=3$              | 19.0            | $K_\alpha$ | 17.339                      |
| $n=4$              | 25.6            | $K_\alpha$ | 17.446                      |

Table 1: Results

## “measure” software

With the “measure” software, the peaks in the spectrum can be determined rather easily:

- Click the button  and select the area for the peak determination.
- Click the button  “Peak analysis”.
- The window “Peak analysis” appears (see Fig. 12).
- Then, click “Calculate”.
- If not all of the desired peaks (or too many of them) are calculated, readjust the error tolerance accordingly.
- Select “Visualise results” in order to display the peak data directly in the spectrum.

Refer to the Help of the “measure” software for additional, more detailed explanations concerning the program features.

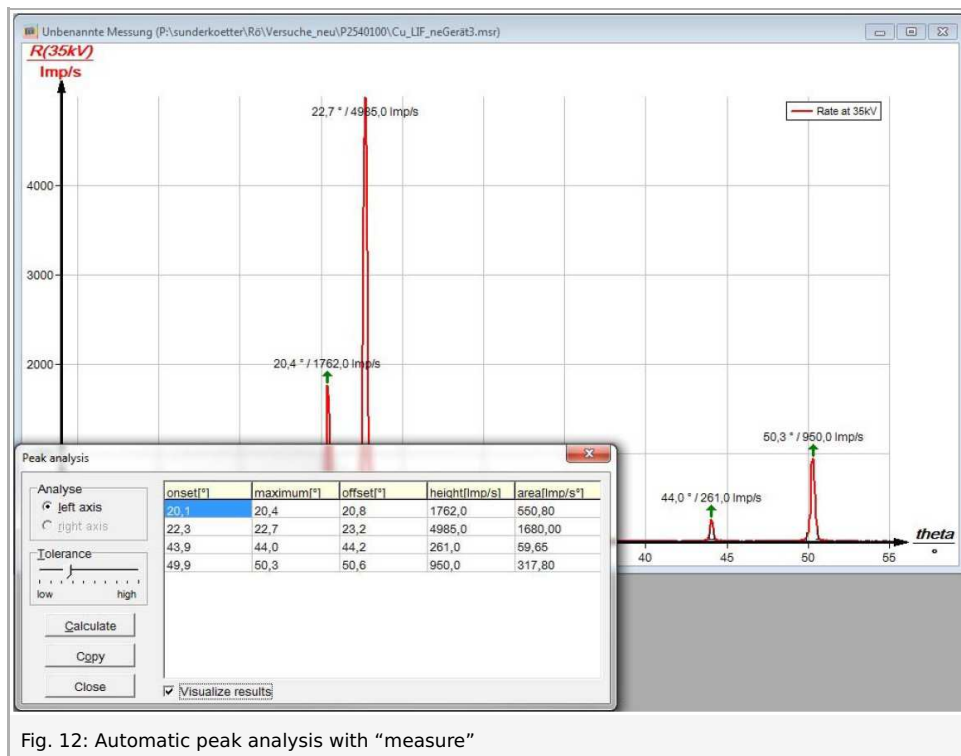


Fig. 12: Automatic peak analysis with "measure"