

Monochromatisation of molybdenum X-rays



Physics

Modern Physics

Production & use of X-rays



Difficulty level

hard



Group size

2



Preparation time

45+ minutes



Execution time

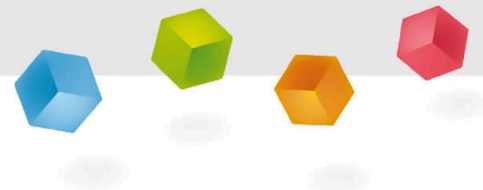
45+ minutes

This content can also be found online at:



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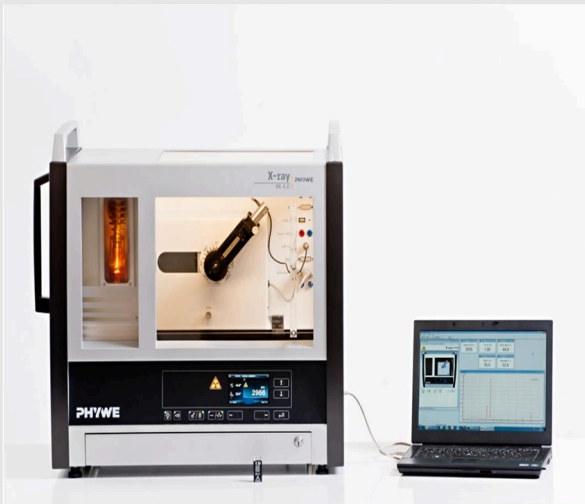
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General information

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 people becomes possible. This has wide usage in fields such as medicine or security.

Other information (1/2)

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**Prior****knowledge****Main****principle**

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

The X-rays that are generated by an X-ray tube are polychromatic. Numerous experiments (e.g. Debye-Scherrer experiments concerning crystal structures), however, require monochromatic X-radiation, which can be generated by filtering the X-rays with monocrystals or with the aid of metal foils.

Other information (2/2)

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**Learning
objective****Tasks**

The goal of this experiment is to get to investigate the monochromatic characteristic X-radiation of molybdenum.

1. Analyse the intensity of the X-radiation of molybdenum as a function of the Bragg angle and with the aid of LiF and KBr monocrystals. Determine the energy of the characteristic X-ray lines.
2. Use the LiF monocrystal to filter a characteristic line out of the polychromatic spectrum.
3. Monochromatise the X-ray spectrum with the aid of the zirconium foil.

Theory (1/2)

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The X-rays that are generated by an X-ray tube are polychromatic. Lines whose energies are not dependent on the anode voltage but on the anode material are superimposed on the bremsstrahlung spectrum (see experiment P2540205). Since some experiments (Debye-Scherrer experiments concerning crystal structures) require monochromatic X-radiation, this radiation is generated by filtration with monocrystals or with the aid of metal foils.

When X-rays of the wavelength λ impinge on the lattice planes of a monocrystal under the glancing angle θ , the rays that are reflected on the lattice planes interfere with each other in a constructive manner provided that their path difference corresponds to an integral multiple of the wavelength. This situation is explained by Bragg's law:

$$2d \sin(\theta) = n\lambda \quad (1) \quad (d: \text{interplanar spacing}; n = 1, 2, 3, \dots)$$

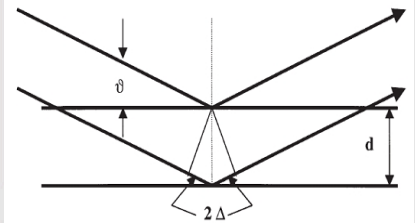


Fig. 1: Bragg scattering on a pair of lattice planes

Theory (2/2)

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

$$E = h \cdot f = \frac{hc}{\lambda} \quad (2)$$

When combining (1) and (2), we obtain:

$$E = \frac{n \cdot h \cdot c}{2d \cdot \sin(\theta)} \quad (3)$$

Note:

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

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

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

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

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 Mo tube	09057-61	1
4	XRC 4.0 X-ray characteristics upgrade set	09135-88	1

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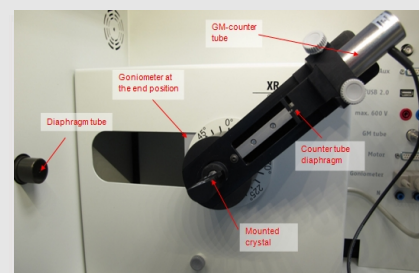
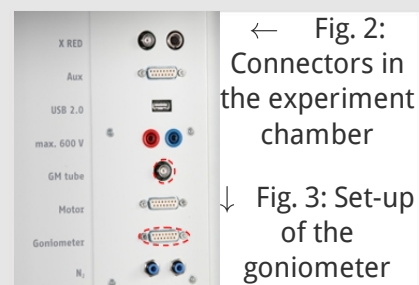
Setup and Procedure

Setup

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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 mm into the beam outlet of the tube plug-in unit.

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.



Procedure (1/4)

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



Fig. 4: Connection of the computer

Procedure (2/4)

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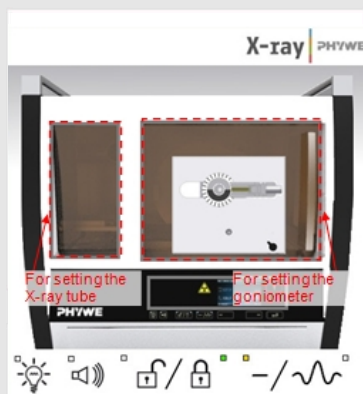


Fig. 5: Part of the user interface of the software

- Click the experiment chamber (see the red marking in Figure 5) to change the parameters for the experiment. Select a start angle of 3° and a stop angle of 65° for the LiF crystal. If you use the KBr crystal, select a start angle of 3° and a stop angle of 75° .
- If you click the X-ray tube (see the red marking in Figure 5), you can change the voltage and current of the X-ray tube. Select the parameters as shown in Fig. 6.

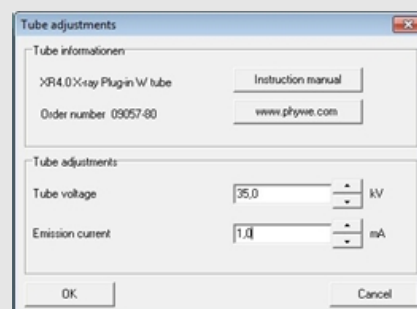
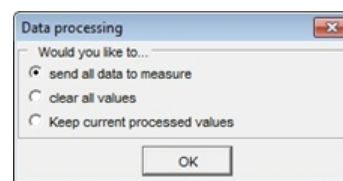
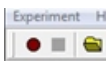


Fig 6: Voltage and current settings

Procedure (3/4)

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- 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.

Overview of the goniometer and X-ray unit settings for task 1:

- 1:2 coupling mode
- Gate time 2 s; angle step width 0.1°
- Scanning range 3°-65° (LiF monocrystal) and 3°-75° (KBr monocrystal)
- Anode voltage $U_A = 35$ kV; anode current $I_A = 1$ mA

Procedure (4/4)

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Task 2: Monochromatisation with the aid of monocrystals

If only a narrow range of the polychromatic spectrum (e.g. the characteristic K_α line) is required, the goniometer settings must be changed as follows: The analyser crystal is set to its glancing angle position $\theta = 10.3^\circ$ ($n = 1$) in a fixed manner. The Geiger-Müller detector turns around the analyser crystal, e.g. around the following scanning range: start angle $10^\circ < 2\theta < \text{stop angle } 30^\circ$. The corresponding goniometer settings can be found in Figure 7.

Task 3: Monochromatisation with the aid of filters

The procedure for this task corresponds to the procedure for task 1. In this case, however, the diaphragm tube (1 mm) must be replaced with the zirconium filter.

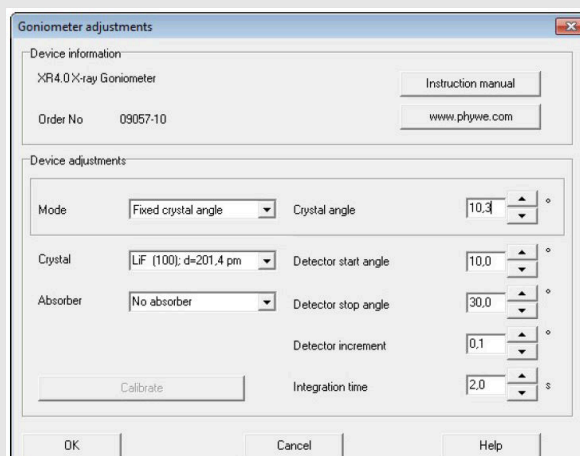
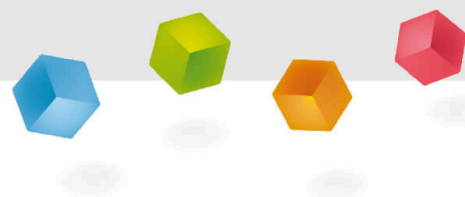


Fig 7: Goniometer settings; task 2

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Evaluation



Task 1

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Task 1: Analysis of the X-ray spectrum with LiF and KBr monocrystals

Figure 8 shows the intensity of X-ray spectrum of molybdenum as a function of the glancing angle θ , with the LiF crystal used as the analyser.

Table 1 shows the energy values for the characteristic X-ray lines that result from the glancing angles θ of the characteristic lines in Figure 8 and from the interplanar spacing ($d = 201.4 \text{ pm}$) of the analyser crystal in accordance with (3) (see also P2540205).

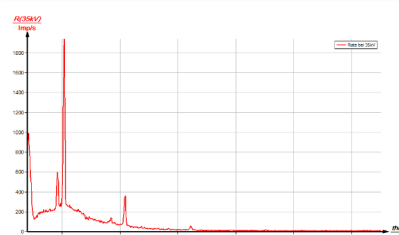


Fig. 8: X-ray spectrum of molybdenum with a LiF crystal as the analyser

Glancing angle θ Energy values

$\theta = 10.4^\circ; n = 1$	$E_{K\alpha} = 17.100 \text{ keV}$
$\theta = 20.9^\circ; n = 2$	$E_{K\alpha} = 17.244 \text{ keV}$
$\theta = 32.2^\circ; n = 3$	$E_{K\alpha} = 17.324 \text{ keV}$
$\theta = 9.2^\circ; n = 1$	$E_{K\alpha} = 19.525 \text{ keV}$
$\theta = 18.5^\circ; n = 2$	$E_{K\alpha} = 19.401 \text{ keV}$
$\theta = 28.4^\circ; n = 3$	$E_{K\alpha} = 19.399 \text{ keV}$

Task 2

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Task 2: Monochromatisation with the aid of monocrystals

Figure 9 shows that only around an angle of $2\theta = 20.6^\circ$ X-rays leave the analyser crystal.

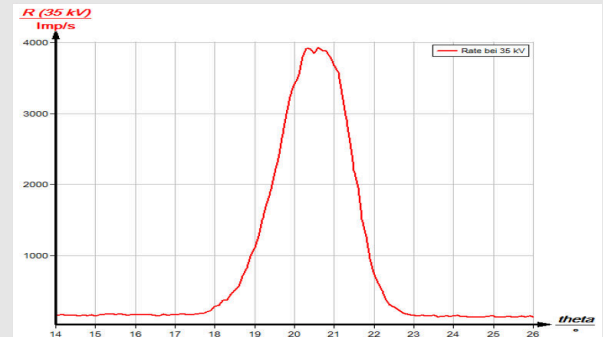


Fig. 9: Monochromatisation of molybdenum X-radiation with the aid of a monocrystal; filtered wavelength: λ_{K_α}

Task 3

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Task 3: Monochromatisation with the aid of absorption foils

If a thin metal foil of the thickness x is brought into the path of an X-ray beam of the energy E and intensity I_0 , the intensity behind the foil is as follows due to absorption:

$$I(E, x) = I_0 e^{-\mu(E) \cdot x} \quad (\mu[\text{cm}^{-1}] = \text{linear absorption coefficient}). \quad (4)$$

Although the absorption coefficient is dependent on the energy, it generally shows no dramatic change within an energy interval of several keV. This changes dramatically, however, when the energy of the X-ray quanta is sufficiently high to eject electrons from the lower energy levels of the absorber material. In such a case, the absorption course shows a so-called absorption edge. Zirconium, for example, is able to absorb the energy of the characteristic K_β line of molybdenum nearly completely, because the energy of the K level of zirconium is slightly smaller than the energy of the K_β line. The energy of the characteristic K_α line of molybdenum, on the other hand, is too small for ionising zirconium on the K shell. As a result, the intensity of the K_α line is reduced only slightly by the zirconium filter in accordance with (4).

Task 3 (part 2)

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Figure 10 shows the result of the analysis of the X-ray spectrum of molybdenum with a zirconium filter and a LiF monocrystal as the analyser. A comparison with the corresponding unfiltered spectrum (Fig. 8) shows a considerable intensity reduction of the K_{β} line of molybdenum, whereas the intensity of the K_{α} line of molybdenum has been hardly reduced by the thin foil at all.

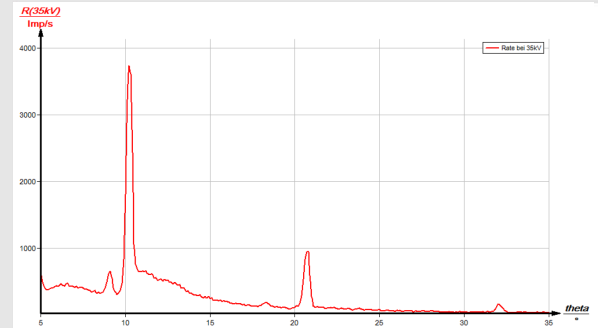


Fig 10: X-ray spectrum of molybdenum with a zirconium filter and a LiF crystal as the analyser

Task 3 (part 3)

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By approximation, the intensity of a spectral line is proportional to the intensity maximum of the line. Based on this approximation, the comparison of both spectra (for $n = 1$) shows that the intensity of the Mo - K_{β} line has been reduced by approximately 60% by the zirconium filter (thickness $d = 0.005$ mm). If a KBr monocrystal is used as the analyser, the results are the same (Figs. 11 and 12).

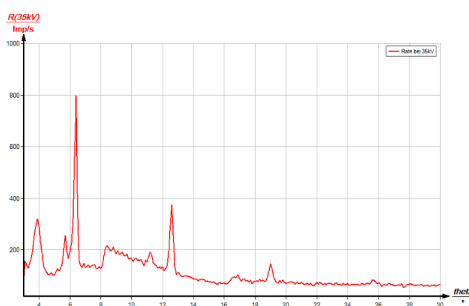


Fig. 11: X-ray spectrum of molybdenum with a KBr crystal as the analyser

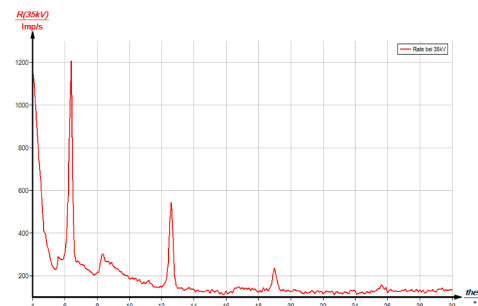




Fig. 12: X-ray spectrum of molybdenum with a zirconium K_{β} filter and a KBr crystal as the analyser

Note

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"measure" software

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

- Click the button  "Mark" and select the area for the peak determination.
- Click the button  "Peak analysis".
- The window "Peak analysis" appears (see Fig. 13).
- 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.

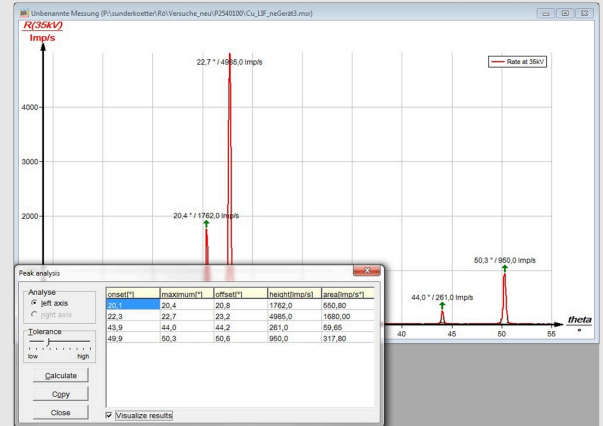


Fig. 13: Automatic peak analysis with "measure"