XRD

Experiment

PHYSICS LAB 403

TABLE OF CONTENT

i.

(P6.3.6.1)

Fine structure of the characteristic x- radiation of a molybdenum anode .

LEYBOLD
Physics
Leaflets

P6.3.6.1

Fine structure of the characteristic x-radiation of a molybdenum anode

Objects of the experiment

- Investigating the fine structure of the characteristic x-radiation of molybdenum by means of Bragg reflection at an NaCl monocrystal in the fifth diffraction order.
- Identifying the characteristic K_α, K_β and K_γ lines.
- Resolving the fine structure of the K_α line as a line doublet and determining the wavelength interval $\Delta\lambda$ within the doublet.

Principles

On closer examination, the characteristic K_α and K_β lines of the x-radiation prove to be line doublets. The two doublets can be resolved by means of Bragg reflection at an NaCl monocrystal when measured in a higher diffraction order. However, they differ in their physical nature.

The K_β doublet consists of the pure K_β line, i.e. transitions of excited atoms from the M-shell to the K-shell, and the K_γ line, i.e. transitions from the N-shell to the K-shell. The wavelength interval $\Delta\lambda$ between the two lines is just 1.2 pm (see table 1), so that they can only be resolved at a high resolution.

Table 1: Transition energies E, wavelengths λ and relative components of the characteristic K_α, K_β and K_γ lines of molybdenum (weighted mean values according to [1])

	$\frac{E}{\text{keV}}$	$\frac{\lambda}{\text{pm}}$	Relative proportion
K_α	17.44	71.08	1.000
K_β	19.60	63.26	0.170
K_γ	19.97	62.09	0.027
Doublet $K_\beta + K_\gamma$	19.65	63.09	

The fine structure of the K_α line arises from the fine structure of the L-shell, and thus ultimately from the spin-orbit characteristic of the electrons. The L-shell actually consists of three sub-shells, designated L_I, L_{II} and L_{III} in x-ray spectroscopy. The transitions from these sub-shells to the K-shell with emission of an x-ray is subject to the selection rules

$$\Delta l = \pm 1,\ \Delta j = 0, \pm 1 \qquad (I)$$

for the change of the orbital angular momentum l and the total angular momentum j on transition. Thus, two transitions from the L-shell to the K-shell are permitted, designated $K_{\alpha 1}$ and $K_{\alpha 2}$ (see Fig. 1). Table 2 shows the values generally found in the literature for molybdenum. According to these, the wavelength interval within the K_α doublet is $\Delta\lambda$ = 0.43 pm.

Fig. 1 Diagram of fine structure of the characteristic line K_α

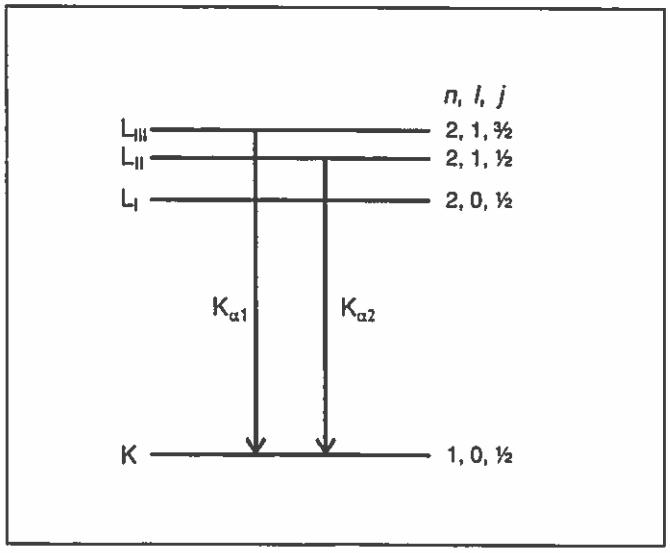


Table 2: Wavelengths λ (calculated from literature specifications [1] for transition energies) and relative proportions of K_α radiation of molybdenum

Line	$\frac{\lambda}{\text{pm}}$	Relative proportion
$K_{\alpha 1}$	70.93	1.000
$K_{\alpha 2}$	71.36	0.525

0308-Ste

Apparatus

1 X-ray apparatus	554 811
1 End-window counter for α, β, γ and x-ray radiation	559 01

additionally required:

1 PC with Windows 9x/NT

The object of the experiment is to resolve this fine structure by means of Bragg reflection at an NaCl monocrystal at higher diffraction orders.

According to Bragg's law of reflection, the following relationship exists between the wavelength λ of the incident characteristic radiation and the glancing angle ϑ at which we may expect an intensity maximum:

$n \cdot \lambda = 2 \cdot d \cdot \sin\vartheta$ (II)

n: diffraction order,
d = 282.01 pm: lattice plane spacing of NaCl

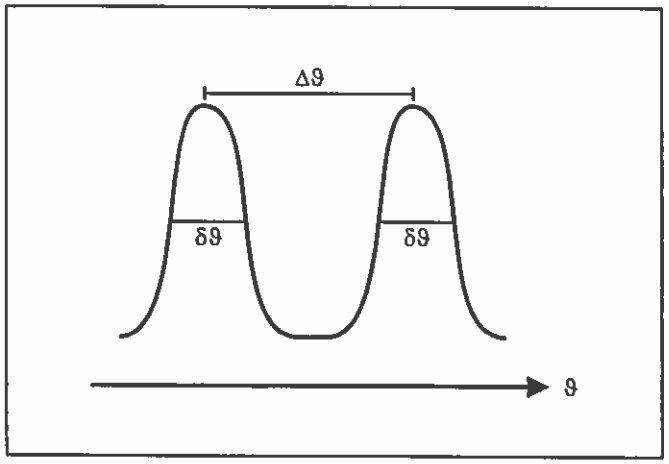


Fig. 2 Definition of the angular width δϑ and the angular spacing Δϑ of two intensity maxima.

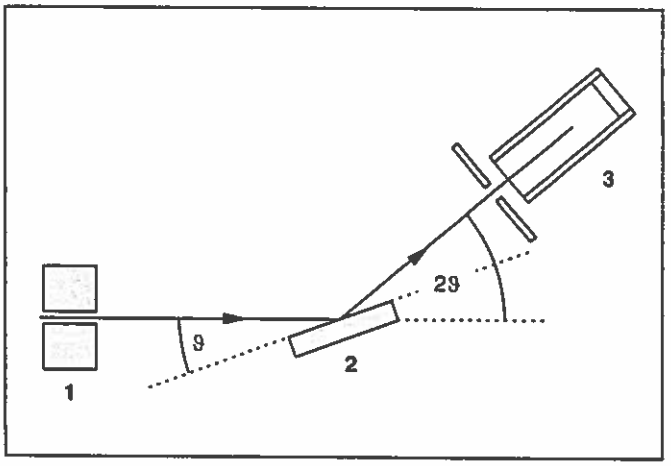


Fig. 3 Diagram showing the diffraction of x-rays at a monocrystal 1 collimator, 2 monocrystal, 3 counter tube

Safety notes

The x-ray apparatus fulfills all regulations governing an x-ray apparatus and fully protected device for instructional use and is type approved for school use in Germany (NW 807/97 Rö).

The built-in protection and screening measures reduce the local dose rate outside of the x-ray apparatus to less than 1 μSv/h, a value which is on the order of magnitude of the natural background radiation.

- Before putting the x-ray apparatus into operation inspect it for damage and to make sure that the high voltage is shut off when the sliding doors are opened (see Instruction Sheet for x-ray apparatus).
- Keep the x-ray apparatus secure from access by unauthorized persons.

Do not allow the anode of the x-ray tube Mo to overheat.

- When switching on the x-ray apparatus, check to make sure that the ventilator in the tube chamber is turning.

The goniometer is positioned solely by electric stepper motors.

- Do not block the target arm and sensor arm of the goniometer and do not use force to move them.

The wavelength interval Δλ of two lines thus corresponds to the angular spacing

$$\Delta\vartheta = \frac{n \cdot \Delta\lambda}{2 \cdot d \cdot \cos\vartheta} \quad \text{(III)},$$

which increases with the diffraction order. It is important to distinguish between the angular spacing Δϑ and the angular width δϑ of an intensity maximum. This latter should be smaller than the angular spacing so that the two lines can be observed separately (see Fig. 2). The angular width is determined by the opening slit of the counter tube (see Fig. 3), its distance from the crystal and the divergence of the incident x-ray beam, and remains constant even for higher diffraction orders. Thus, the K_α doublet can be resolved in the diffraction order $n = 5$.

Setup

Setup in Bragg configuration:

Fig. 4 shows some important details of the experiment setup. To set up the experiment, proceed as follows (see also the Instruction Sheet for the x-ray apparatus):

- Mount the collimator in the collimator mount (a) (note the guide groove).
- Attach the goniometer to guide rods (d) so that the distance s_1 between the slit diaphragm of the collimator and the target arm is approx. 5 cm. Connect ribbon cable (c) for controlling the goniometer.
- Remove the protective cap of the end-window counter, place the end-window counter in sensor seat (e) and connect the counter tube cable to the socket marked GM TUBE.
- By moving the sensor holder (b), set the distance s_2 between the target arm and the slit diaphragm of the sensor seat to approx. 6 cm.

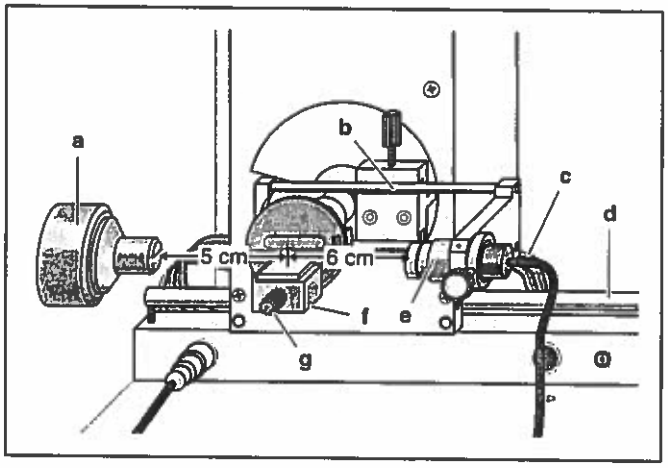


Fig. 4 Experiment setup in Bragg configuration

- Mount the target holder (f) with target stage.
- Loosen knurled screw (g), place the NaCl crystal flat on the target stage, carefully raise the target stage with crystal all the way to the stop and gently tighten the knurled screw (prevent skewing of the crystal by applying a slight pressure).
- If necessary, adjust the mechanical zero position of the goniometer (see Instruction Sheet for x-ray apparatus).

Notes:

NaCl crystals are hygroscopic and extremely fragile. Store the crystals in a dry place; avoid mechanical stresses on the crystal; handle the crystal by the short faces only.

If the counting rate is too low, you can reduce the distance s_2 between the target and the sensor somewhat. However, the distance should not be too small, as otherwise the angular resolution of the goniometer is no longer sufficient.

Preparing the PC-based measurement:

- Connect the RS-232 output and the serial interface on your PC (usually COM1 or COM2) using the 9-pin V.24 cable (supplied with x-ray apparatus).
- If necessary, install the software "X-ray Apparatus" under Windows 9x/NT (see Instruction Sheet for x-ray apparatus) and select the desired language.

Carrying out the experiment

- Start the software "X-ray Apparatus", check to make sure that the apparatus is connected correctly, and clear any existing measurement data using the button or the F4 key.
- Set the tube high voltage U = 35 kV, the emission current I = 1.00 mA and the angular step width $\Delta\beta$ = 0.1°.
- Press the COUPLED key for 2ϑ coupling of target and sensor.

a) First order of diffraction:

- To record the first diffraction order, set the lower limit of the target angle to 5.5° and the upper limit to 8.0°, and set the measuring time per angular step to Δt = 10 s.
- Start measurement and data transfer to the PC by pressing the SCAN key.
- When the measurement is finished, open the "Settings" dialog with the button or F5 and enter the lattice plane spacing for NaCl to show the wavelength-dependency of the counting rate.
- Save the measurement series under a suitable name using the button or by pressing F2.

b) Fifth order of diffraction:

- To record the fifth diffraction order, set the lower limit of the target angle to 32.5° and the upper limit to 40.5°, and
- Set the measuring time per angular step to Δt = 400 s.

Note: Due to the low counting rate to be expected, you need to set a relatively long measuring time to obtain a satisfactory statistical accuracy. In this setting, the total measuring time is 9 h.

- Start measurement and data transfer to the PC by pressing the SCAN key.
- When the measurement is finished, open the "Settings" dialog with the button or F5 and enter the lattice plane spacing for NaCl to show the wavelength-dependency of the counting rate.
- Save the measurement series under a suitable name using the button or by pressing F2.

Measuring example

Fig. 5 shows the diffraction spectrum measured in the first order, and Fig. 6 shows the spectrum for the fifth order of diffraction.

a) First order of diffraction:

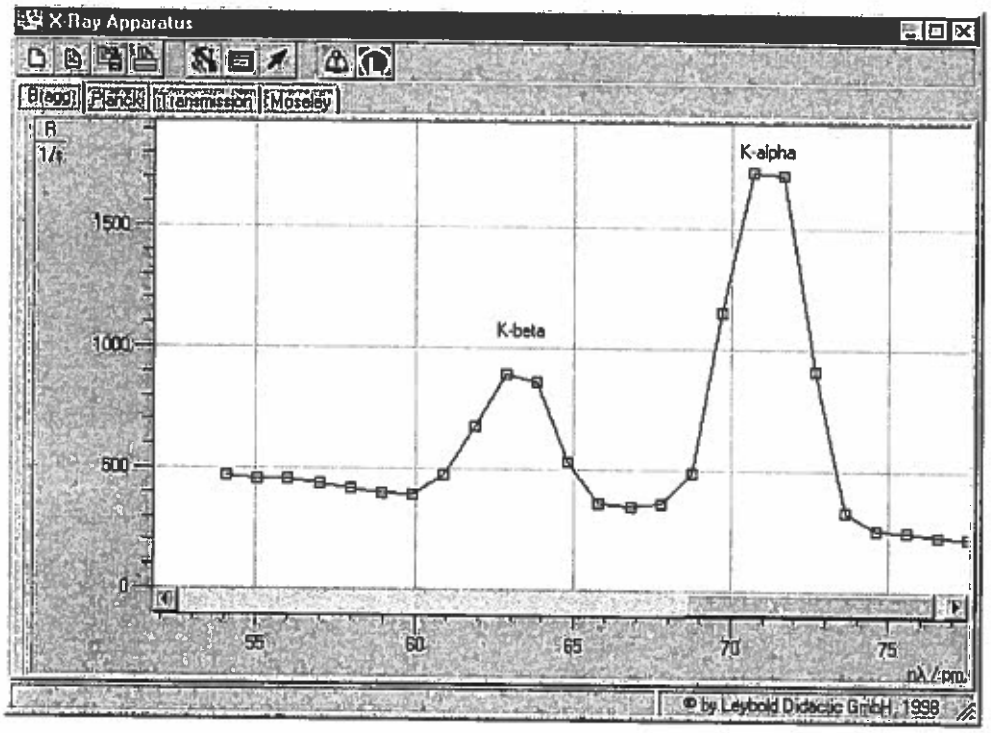


Fig. 5 Diffraction spectrum of x-rays in Bragg reflection in the first order at an NaCl monocrystal
Parameters: U = 35 kV, I = 1 mA, Δt = 10 s

b) Fifth order of diffraction:

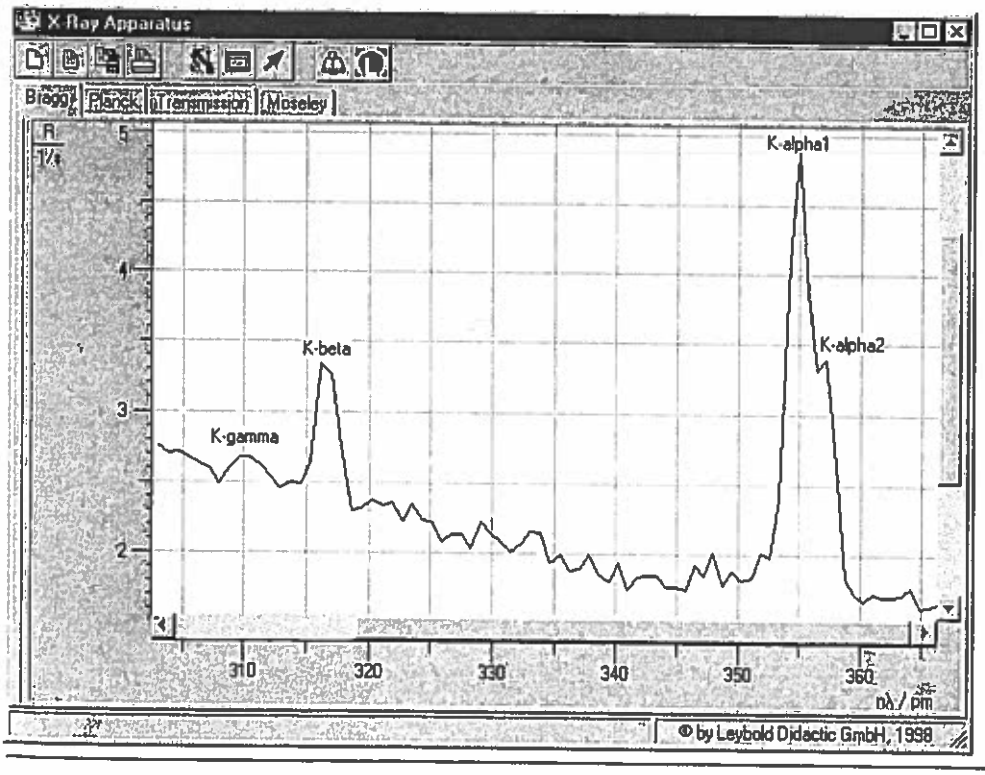


Fig. 6 Diffraction spectrum of x-rays in Bragg reflection in the fifth order at an NaCl monocrystal
Parameters: U = 35 kV, I = 1 mA, Δt = 400 s

Evaluation

- In the diagram, click the right mouse button to access the evaluation functions of the software "X-ray Apparatus" and select the command "Display Coordinates".
- Drag the mouse pointer across the peaks and read the corresponding $n \cdot \lambda$ values in the bottom left corner of the window.

a) First order of diffraction:

Table 3: Measuring results for the first diffraction order and literature value for the characteristic wavelengths (cf. table 1)

	Measurement result	Literature value
Line doublet	$\frac{\lambda}{\text{pm}}$	$\frac{\lambda}{\text{pm}}$
K_α	71.0	71.08
$K_\beta + K_\gamma$	63.1	63.09

b) Fifth order of diffraction:

Table 4: Measuring results for the fifth diffraction order and literature value for the characteristic wavelengths (cf. tables 1 and 2)

	Measurement result		Literature value
Lines	$\frac{5 \cdot \lambda}{\text{pm}}$	$\frac{\lambda}{\text{pm}}$	$\frac{\lambda}{\text{pm}}$
$K_{\alpha 1}$	355	71.0	70.93
$K_{\alpha 2}$	357	71.4	71.36
K_β	316.7	63.34	63.26
K_γ	310.3	62.06	62.09

Splitting of doublet K_α:

$\Delta\lambda$ = 0.4 pm Literature value: $\Delta\lambda$ = 0.43 pm

Splitting of doublet $K_\beta + K_\gamma$:

$\Delta\lambda$ = 1.28 pm Literature value: $\Delta\lambda$ = 1.17 pm

Results

The characteristic K_α und K_β lines we observe in the first diffraction order split into doublets. We can observe this split in the fifth diffraction order.

The fine structure of the K_α doublet is a consequence of the fine structure of the L-shell. The K_β doublet is composed of the pure K_β line and the K_γ line.

Additional information

Strictly speaking, the K_β and K_γ lines also show a fine structure due to the fine structure of shells M and N. However, this split is so slight that we cannot observe it with the means at hand. Table 1 shows the weighted mean values of the respective individual lines from this substructure.

Literature

[1] C. M. Lederer and V. S. Shirley, Table of Isotopes, 7th Edition, 1978, John Wiley & Sons, Inc., New York, USA.

ii.

(P6.3.3.3)

Duane – Hunt relation and determination of Planck's constant.

Duane-Hunt relation and determination of *Planck*'s constant

Objects of the experiment

- To determine the limit wavelength λ_{min} of the bremsstrahlung continuum as a function of the high voltage U of the x-ray tube.
- To confirme the Duane-Hunt relation.
- To determine Planck's constant.

Principles

The bremsstrahlung continuum in the emission spectrum of an x-ray tube is characterized by the limit wavelength λ_{min} (see Fig. 1), which becomes smaller as the tube high voltage increases (see experiment P6.3.3.2). In 1915, the American physicists *William Duane* and *Franklin L. Hunt* discovered an inverse proportionality between the limit wavelength and the tube high voltage:

$$\lambda_{min} \sim \frac{1}{U} \qquad \text{(I)}.$$

This Duane-Hunt relationship can be sufficiently explained by examining some basic quantum mechanical considerations: As the wavelength amda and the frequency ν for any electromagnetic radiation are related in the manner

$$\lambda = \frac{c}{\nu} \qquad \text{(II)}$$

$c = 2.9979 \cdot 10^8$ m s^{-1}: velocity of light

the minimum wavelength λ_{min} corresponds to a maximum frequency ν_{max} respectively a maximum energy

$$E_{max} = h \cdot \nu_{max} \qquad \text{(III)}$$

h: Planck's constant

of the emitted x-ray quanta. However, an x-ray quantum attains maximum energy at precisely the moment in which it acquires the total kinetic energy

$$E = e \cdot U \qquad \text{(IV)}$$

$e = 1.6022 \cdot 10^{-19}$ A s: elementary charge

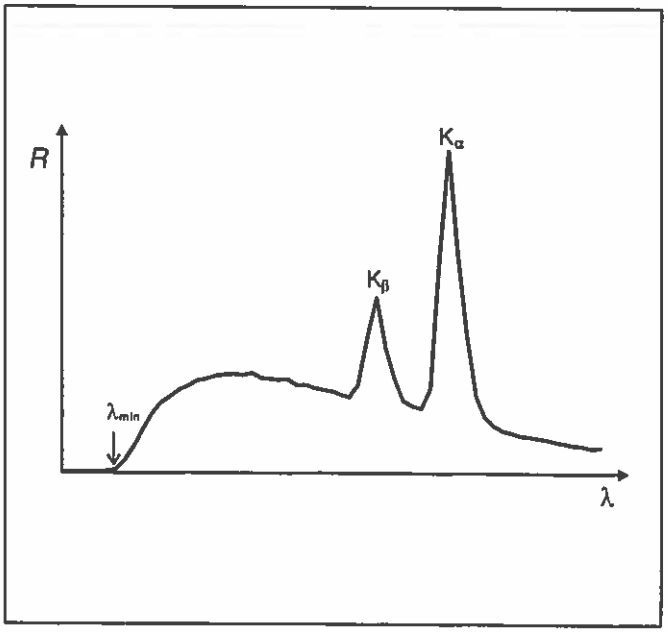
of an electrode decelerated in the anode. It thus follows that

$$\nu_{max} = \frac{e}{h} \cdot U \qquad \text{(V)}$$

respectively

$$\lambda_{min} = \frac{h \cdot c}{e} \cdot \frac{1}{U} \qquad \text{(VI)}$$

Fig. 1 Emission spectrum of an x-ray tube with the limit wavelength λ_{min} of the bremsstrahlung continuum and the characteristic K_α and K_β lines.



Apparatus

1 X-ray apparatus	554 811
1 End-window counter for α, β, γ and x-ray radiation	559 01

additionally required:

1 PC with Windows 95/98/NT

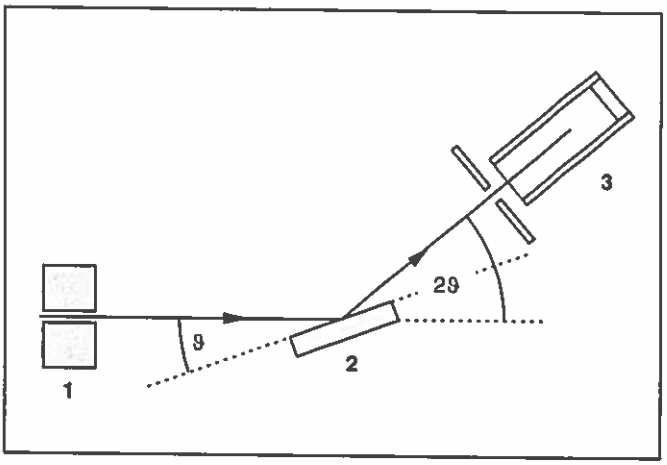


Fig. 2 Schematic diagram of diffraction of x-rays at a monocrystal and 2ϑ coupling between counter-tube angle and scattering angle (glancing angle)
1 collimator, 2 monocrystal, 3 counter tube

Equation (VI) corresponds to Duane and Hunt's law. The proportionality factor

$$A = \frac{h \cdot c}{e} \qquad \text{(VII)}$$

can be used to determine Planck's constant h when the quantities c and e are known.

A goniometer with NaCl crystal and a Geiger-Müller counter tube in the Bragg configuration together comprise the spectrometer in this experiment. The crystal and counter tube are pivoted with respect to the incident x-ray beam in 2ϑ coupling (cf. Fig. 2).

In accordance with Bragg's law of reflection, the scattering angle ϑ in the first order of diffraction corresponds to the wavelength

$$\lambda = 2 \cdot d \cdot \sin \vartheta \qquad \text{(VIII).}$$

d = 282.01 pm: lattice plane spacing of NaCl

Safety notes

The x-ray apparatus fulfills all regulations governing an x-ray apparatus and fully protected device for instructional use and is type approved for school use in Germany (NW 807/97 Rö).

The built-in protection and screening measures reduce the local dose rate outside of the x-ray apparatus to less than 1 µSv/h, a value which is on the order of magnitude of the natural background radiation.

- Before putting the x-ray apparatus into operation inspect it for damage and to make sure that the high voltage is shut off when the sliding doors are opened (see Instruction Sheet for x-ray apparatus).
- Keep the x-ray apparatus secure from access by unauthorized persons.

Do not allow the anode of the x-ray tube Mo to overheat.

- When switching on the x-ray apparatus, check to make sure that the ventilator in the tube chamber is turning.

The goniometer is positioned solely by electric stepper motors.

- Do not block the target arm and sensor arm of the goniometer and do not use force to move them.

Setup

Setup in Bragg configuration:

Fig. 3 shows some important details of the experiment setup. To set up the experiment, proceed as follows (see also the Instruction Sheet for the x-ray apparatus):

- Mount the collimator in the collimator mount (a) (note the guide groove).
- Attach the goniometer to guide rods (d) so that the distance s_1 between the slit diaphragm of the collimator and the target arm is approx. 5 cm. Connect ribbon cable (c) for controlling the goniometer.
- Remove the protective cap of the end-window counter, place the end-window counter in sensor seat (e) and connect the counter tube cable to the socket marked GM TUBE.
- By moving the sensor holder (b), set the distance s_2 between the target arm and the slit diaphragm of the sensor receptor to approx. 6 cm.
- Mount the target holder (f) with target stage.
- Loosen knurled screw (g), place the NaCl crystal flat on the target stage, carefully raise the target stage with crystal all

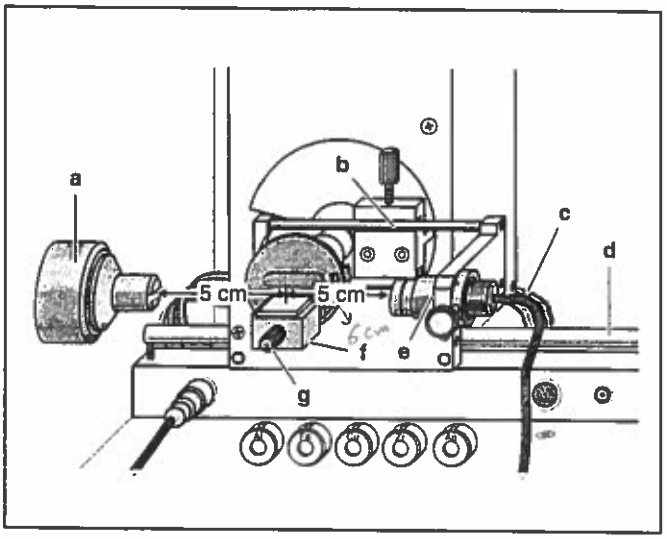


Fig. 3 Experiment setup in Bragg configuration

Tab. 1: Recommended parameters for recording the measurement series

$\frac{U}{kV}$	$\frac{I}{mA}$	$\frac{\Delta t}{s}$	$\frac{\beta_{min}}{grd}$	$\frac{\beta_{max}}{grd}$	$\frac{\Delta\beta}{grd}$
22	1.00	30	5.2	6.2	0.1
24	1.00	30	5.0	6.2	0.1
26	1.00	20	4.5	6.2	0.1
28	1.00	20	3.8	6.0	0.1
30	1.00	10	3.2	6.0	0.1
32	1.00	10	2.5	6.0	0.1
34	1.00	10	2.5	6.0	0.1
35	1.00	10	2.5	6.0	0.1

the way to the stop and gently tighten the knurled screw (prevent skewing of the crystal by applying a slight pressure).
- If necessary, adjust the mechanical zero position of the goniometer (see Instruction Sheet for x-ray apparatus).

Notes:

NaCl crystals are hygroscopic and extremely fragile. Store the crystals in a dry place; avoid mechanical stresses on the crystal; handle the crystal by the short faces only.

If the counting rate is too low, you can reduce the distance s_2 between the target and the sensor somewhat. However, the distance should not be too small, as otherwise the angular resolution of the goniometer is no longer sufficient.

Preparing the PC-based measurement:

- Connect the RS–232 output and the serial interface on your PC (usually COM1 or COM2) using the 9-pin V.24 cable (supplied with x-ray apparatus).
- If necessary, install the software "X-ray Apparatus" under Windows 95/98/NT (see Instruction Sheet for x-ray apparatus) and select the desired language.

Carrying out the experiment

- Start the software "X-ray Apparatus", check to make sure that the apparatus is connected correctly, and clear any existing measurement data using the button or the F4 key.
- Set the tube high voltage U = 22 kV, the emission current I = 1.00 mA, the measuring time per angular step Δt = 30 s and the angular step width $\Delta\beta$ = 0.1°.
- Press the COUPLED key to activate 2ϑ coupling of target and sensor and set the lower limit of the target angle to 5.2° and the upper limit to 6.2°.
- Start measurement and data transfer to the PC by pressing the SCAN key.
- Additionally record measurement series with the tube high voltages U = 24 kV, 26 kV, 28 kV, 30 kV, 32 kV, 34 kV and 35 kV; to save measuring time, use the parameters from table 1 for each series.
- To show the wavelength-dependency, open the "Settings" dialog with the button or F5 and enter the lattice plane spacing for NaCl.
- When you have finished measuring, save the measurement series under an appropriate name by pressing the button or the F2 key.

Measuring example and evaluation

Determining the limit wavelength λ_{min} as a function of the tube high voltage U:

For each recorded diffraction spectrum (see Fig. 4):

- In the diagram, click the right mouse button to access the evaluation functions of the software "X-ray Apparatus" and select the command "Best-fit Straight Line".
- Mark the curve range to which you want to fit a straight line to determine the limit wavelength λ_{min} using the left mouse button.
- Save the evaluations under a suitable name using the button or by pressing F2.

Confirming the Duane-Hunt relation and determining Planck's constant

- For further evaluation of the limit wavelengths λ_{min} determined in this experiment, click on the register "Planck".
- Position the pointer over the diagram, click the right mouse button, fit a straight line through the origin to the curve $\lambda_{min} = f(1/U)$ and read the slope A from the bottom left corner of the evaluation window (see Fig. 5).

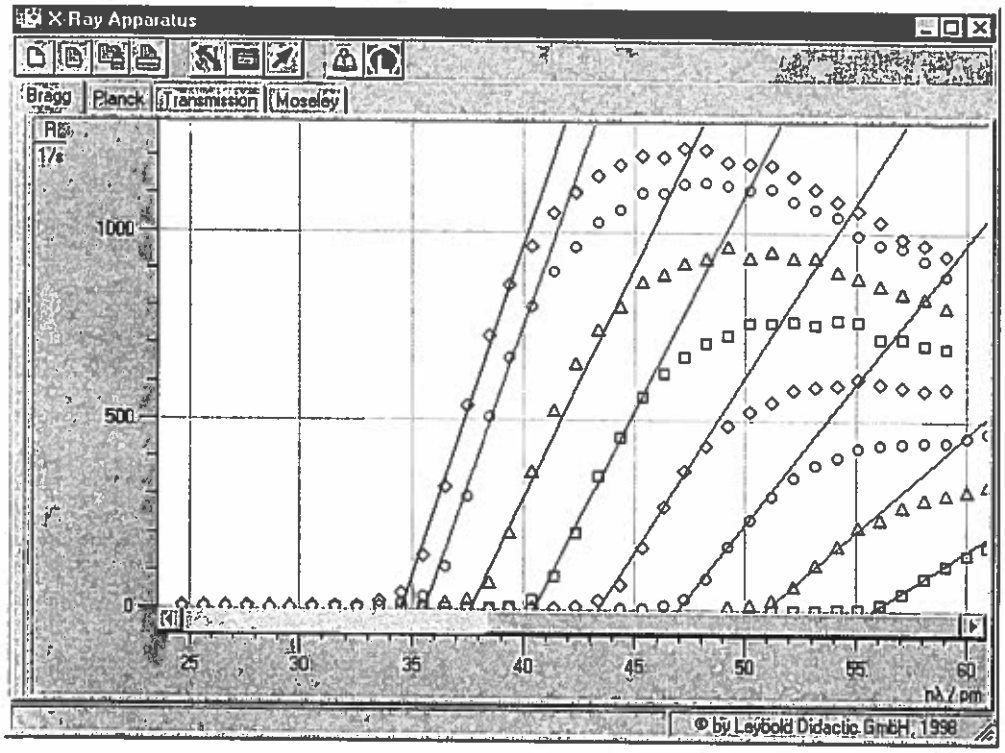


Fig. 4 Sections from the diffraction spectra of x-radiation for the tube high voltages U = 22, 24, 26, 28, 30, 32, 34 and 35 kV (from right to left) with best-fit straight line for determining the limit

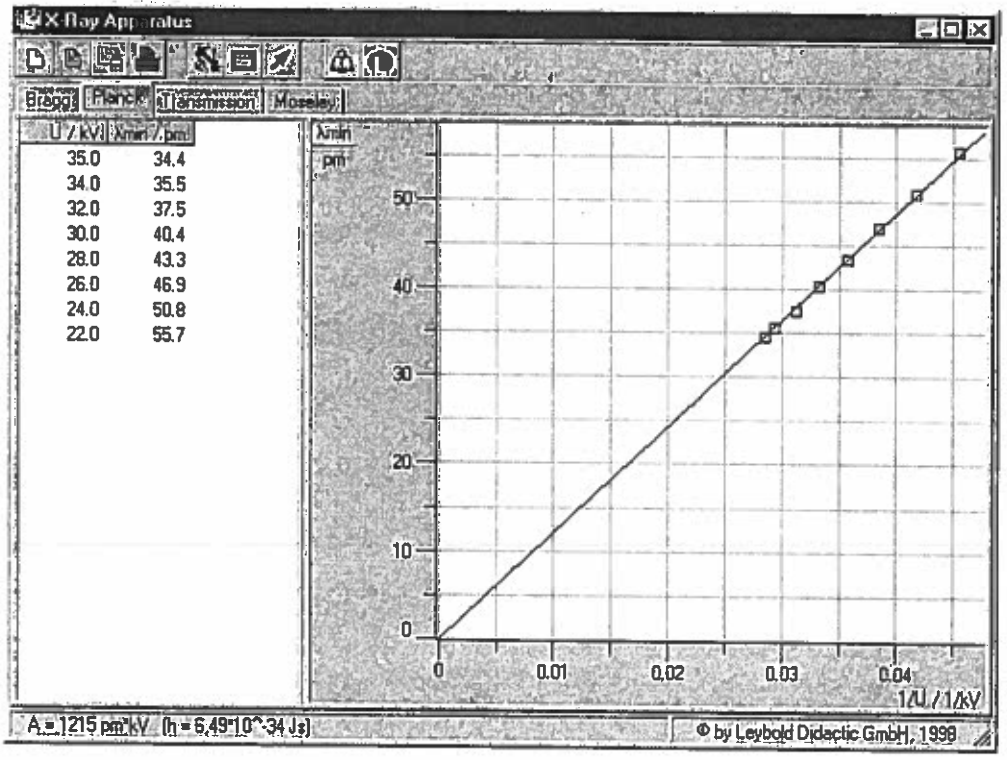


Fig. 5 Evaluation of the data $\lambda_{min} = f(1/U)$ for confirming the Duane-Hunt relation and determining Planck's constant

The best-fit straight line gives us

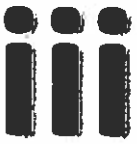
A = 1215 pm kV

When we insert this value in equation (VII), we can calculate Planck's constant as:

$h = 6.49 \cdot 10^{-34}$ J s

Literature value:

$h = 6.626 \cdot 10^{-34}$ J s



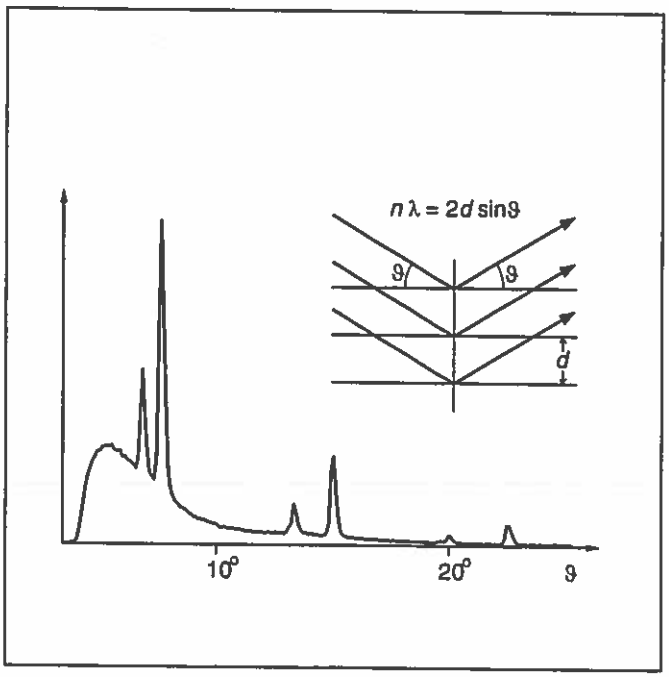
(P6.3.3.1)

Bragg reflection: diffraction of X-rays at monocrystal.

Bragg reflection: diffraction of x-rays at a monocrystal

Objects of the experiment

- To investigate Bragg reflection at an NaCl monocrystal using the characteristic x-ray radiation of molybdenum.
- To determine the wavelength for the characteristic K_α and K_β x-ray radiation of molybdenum.
- To confirm Bragg's law of reflection.
- To verify the wave nature of x-rays.



Principles

In 1913, *H. W.* and *W. L. Bragg* realized that the regular arrangement of atoms and/or ions in a crystal can be understood as an array of lattice elements on parallel lattice planes. When we expose such a crystal to parallel x-rays, additionally assuming that these have a wave nature, then each element in a lattice plane acts as a "scattering point", at which a spherical wavelet forms. According to *Huygens*, these spherical wavelets are superposed to create a "reflected" wavefront. In this model, the wavelength λ remains unchanged with respect to the "incident" wave front, and the radiation directions which are perpendicular to the two wave fronts fulfill the condition "angle of incidence = angle of reflection".

Constructive interference arises in the rays reflected at the individual lattice planes when their path differences Δ are integral multiples of the wavelength λ.

$\Delta = n \cdot \lambda$ with $n = 1, 2, 3, \ldots$ (I)

As Fig. 1 shows for two adjacent lattice planes with the spacing d, we can say for the path differences Δ_1 and Δ_2 of the incident and reflected rays with the angle ϑ:

$\Delta_1 = \Delta_2 = d \cdot \sin \vartheta$

so that the total path difference is

$\Delta = 2 \cdot d \cdot \sin \vartheta$. (II)

(I) and (II) give us Bragg's law of reflection:

$n \cdot \lambda = 2 \cdot d \cdot \sin \vartheta$ (III)

The angle ϑ is known as the glancing angle.

In this experiment, we verify Bragg's law of reflection by investigating the diffraction of x-rays at an NaCl monocrystal in which the lattice planes are parallel to the cubic surfaces of the unit cells of the crystal. The lattice spacing d of the cubic

Apparatus

1 X-ray apparatus	554 811
1 End-window counter for α, β, γ and x-ray radiation	559 01

additionally required:

1 PC with Windows 9x or Windows NT

face-centered NaCl crystal is half the lattice constant a_0. We can thus say [1]

$2 \cdot d = a_0 = 564.02$ pm

The measurements are conducted using the built-in goniometer of the x-ray apparatus (554 811). The x-rays are detected using a GM counter tube (end-window counter) which is swiveled in tandem with the NaCl crystal in a 2_ coupling with respect to the incident light; this means that the counter tube always advances by an angle which is twice that of the crystal (cf. Fig. 2).

The x-ray radiation consists of the bremsstrahlung continuum and several sharply defined lines which correspond to the characteristic x-ray radiation of the Mo anode and which originate in the K_α and K_β transitions of the molybdenum atoms. This characteristic radiation is particularly suitable for investigating Bragg's law. Its properties are known from the literature [2] and summarized in table 1. Table 2 shows the corresponding glancing angles at which the diffraction maxima of the characteristic radiation are to be expected for scattering at an NaCl monocrystal (d = 282.01 pm) up to the third diffraction order.

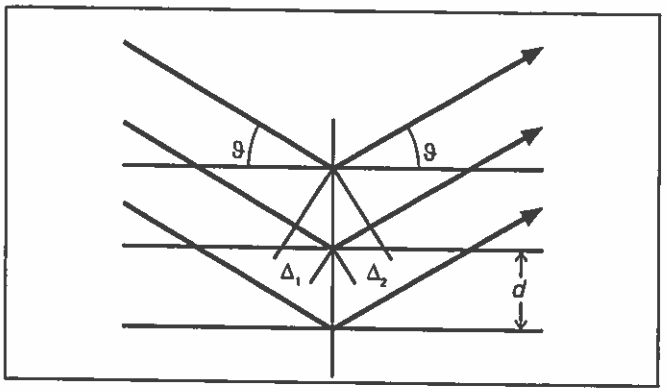


Fig. 1 Diagram of the reflection of x-rays at the lattice planes of a monocrystal.
Δ_1, Δ_2: path differences,
ϑ: glancing angle,
d: spacing of lattice planes

Table 1: Energy E, frequency ν and wavelength λ of the characteristic x-ray radiation of molybdenum (weighted mean values [1])

	$\frac{E}{\text{keV}}$	$\frac{\nu}{\text{EHz}}$	$\frac{\lambda}{\text{pm}}$
K_α	17.443	4.2264	71.080
K_β	19.651	4.8287	63.095

keV = 10^3 eV, EHz = 10^{18} Hz, pm = 10^{-12} m

Table 2: Glancing angle ϑ of the characteristic x-ray radiation of molybdenum for diffraction at an NaCl monocrystal up to the third order

n	$\vartheta(K_\alpha)$	$\vartheta(K_\beta)$
1	7.24°	6.42°
2	14.60°	12.93°
3	22.21°	19.61°

Safety notes

The x-ray apparatus fulfills all German regulations governing an x-ray apparatus and fully protected device for instructional use and is type approved for school use in Germany (NW 807/97 Rö).

The built-in protection and screening measures reduce the local dose rate outside of the x-ray apparatus to less than 1 μSv/h, a value which is on the order of magnitude of the natural background radiation.

- Before putting the x-ray apparatus into operation, inspect it for damage and check to make sure that the high voltage shuts off when the sliding doors are opened (see Instruction Sheet of x-ray apparatus).
- Keep the x-ray apparatus secure from access by unauthorized persons.

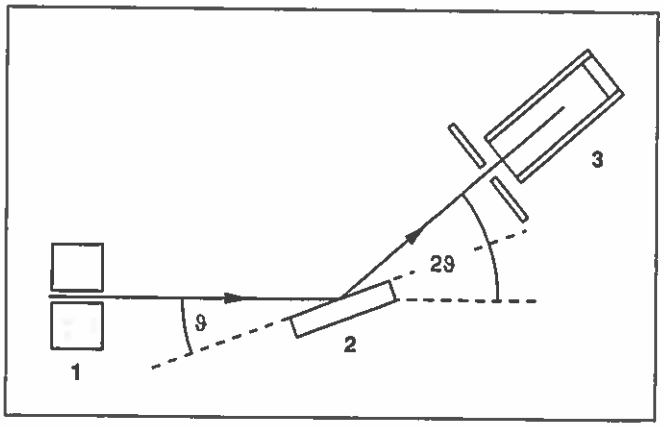
Do not allow the anode of the x-ray tube Mo to overheat.

- When switching on the x-ray apparatus, check to make sure that the ventilator in the tube chamber is turning.

The goniometer is positioned solely by electric stepper motors.

- Do not block the target arm and sensor arm of the goniometer and do not use force to move them.

Fig. 2 Diagram showing the principle of diffraction of x-rays at a monocrystal and 2ϑ coupling between counter-tube angle and scattering angle (glancing angle)
1 collimator, 2 monocrystal, 3 counter tube



General remarks

In principle, you can conduct measurements in both manual scan and autoscan modes of the x-ray apparatus (see the instruction Sheet of the x-ray apparatus). You can record the measured values manually by reading the values from the display field and writing them in a table, using a chart recorder or via a PC.

The fastest and most convenient measurement is in autoscan mode with simultaneous registration of measured values and subsequent evaluation on a Windows 9x/NT PC. This type of measurement is described in your instruction Sheet.

The data is transmitted to the PC via the RS–232 serial interface on the x-ray apparatus. The software "X-ray Apparatus", supplied with the device, enables you to record, display and evaluate the data stream supplied by the x-ray apparatus. The program contains detailed online help which you can access by pressing F1. Please refer to the instruction Sheet of the x-ray apparatus for details on installing the software.

The instruction Sheet also describes recording data under Windows 3.1.

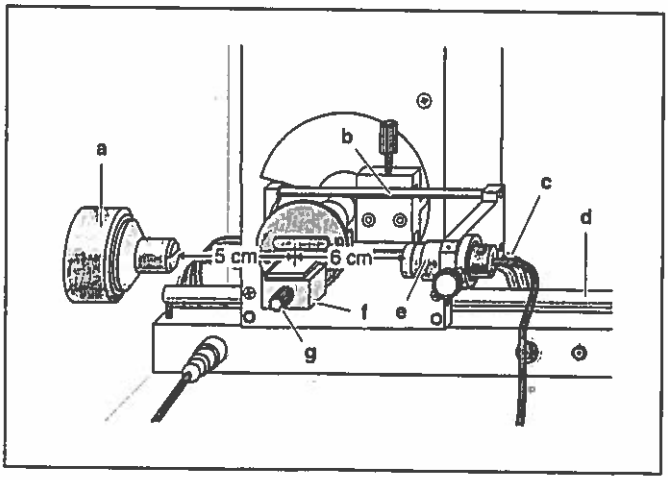
Setup

Setting up the Bragg configuration:

Fig. 3 shows some important details of the experiment setup. Specifically, you need to carry out the following steps (see also instruction Sheet of x-ray apparatus):

- Mount the collimator in the collimator mount (a) (note the guide groove).
- Attach the goniometer to the guide rods (d) in such a way that the distance s_1 between the slit diaphragm of the collimator and the target arm is approx. 5 cm. Connect the ribbon cable (c) for controlling the goniometer.
- Remove the cap of the end-window counter, insert the end-window counter in the sensor seat (e) and connect the counter tube lead to the socket marked GM-Tube.

Fig. 3 Experiment setup in Bragg configuration



- Adjust the sensor seat (b) until the distance s_2 between the target arm and the slit diaphragm of the sensor seat is approx. 6 cm.
- Attach the target holder with target stage (f).
- Loosen knurled screw (g), lay the NaCl crystal flat on the target stage, carefully raise the stage as far at it will go and then tighten the knurled screw with care (press against the screw lightly to prevent it from stripping).
- Adjust the zero position of the goniometer measuring system as necessary (see instruction Sheet of x-ray apparatus).

Notes:

NaCl crystals are hygroscopic and fragile. Store the crystals in a dry place. Avoid mechanical stresses on the crystal; handle the crystal by the short faces only.

If the counting rate is too low, you can reduce the distance s_2 between the target and the sensor somewhat. However, this distance must not be too small, as otherwise the angular resolution of the goniometer is no longer great enough to separate the characteristic K_α and K_β lines.

Preparing a PC-based measurement:

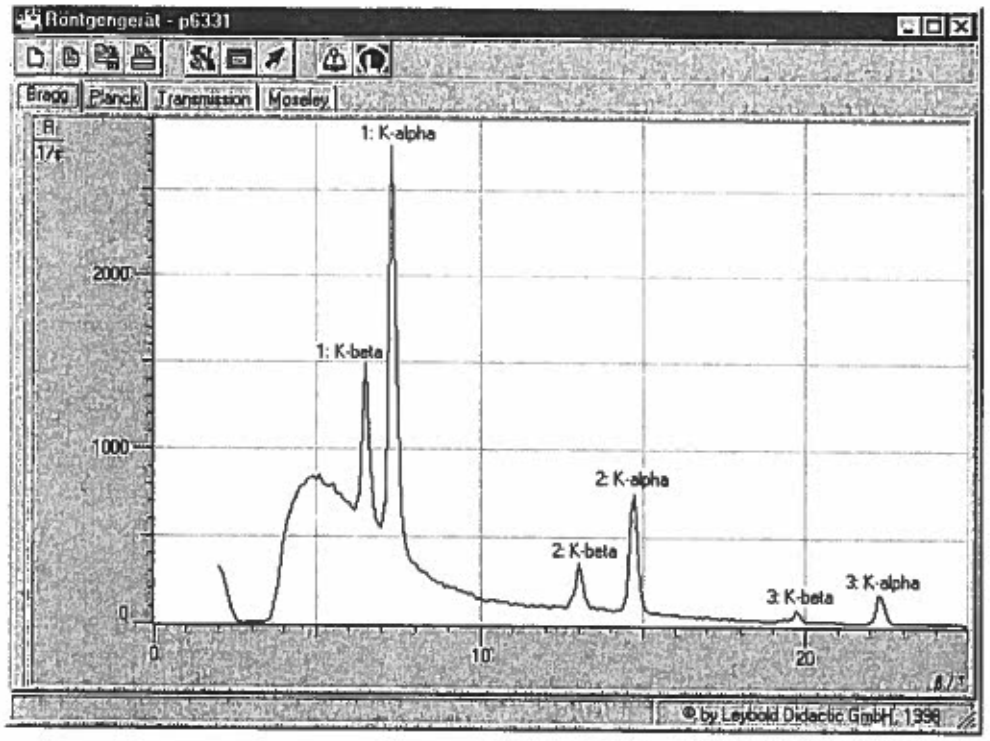
- Connect the RS–232 output to the serial interface on the PC (usually COM1 or COM2) using the 9-pin V.24 cable (included with the x-ray apparatus).
- If you have not already done so, install the software "X-ray Apparatus" under Windows 9x/NT (see instruction Sheet of x-ray apparatus) and select the desired language.

Carrying out the experiment

- Start the program "X-ray Apparatus", check to make sure that the x-ray apparatus is properly connected and delete any existing measurement data by clicking the button or pressing F4.
- Set the x-ray high voltage U = 35.0 kV, emission current I = 1.00 mA, measuring time per angular step Δt = 10 s and angular step width $\Delta\beta$ = 0.1°.
- Press the COUPLED key on the device to enable 2ϑ coupling of the target and sensor; set the lower limit value of the target angle to 2° and the upper limit to 25°.
- Press the SCAN key to start the measurement and data transmission to the PC.
- When the measurement is finished, save the measurement series to a file under a suitable name using the button or F2.

Measuring example

Fig. 4 shows the measured diffraction spectrum.



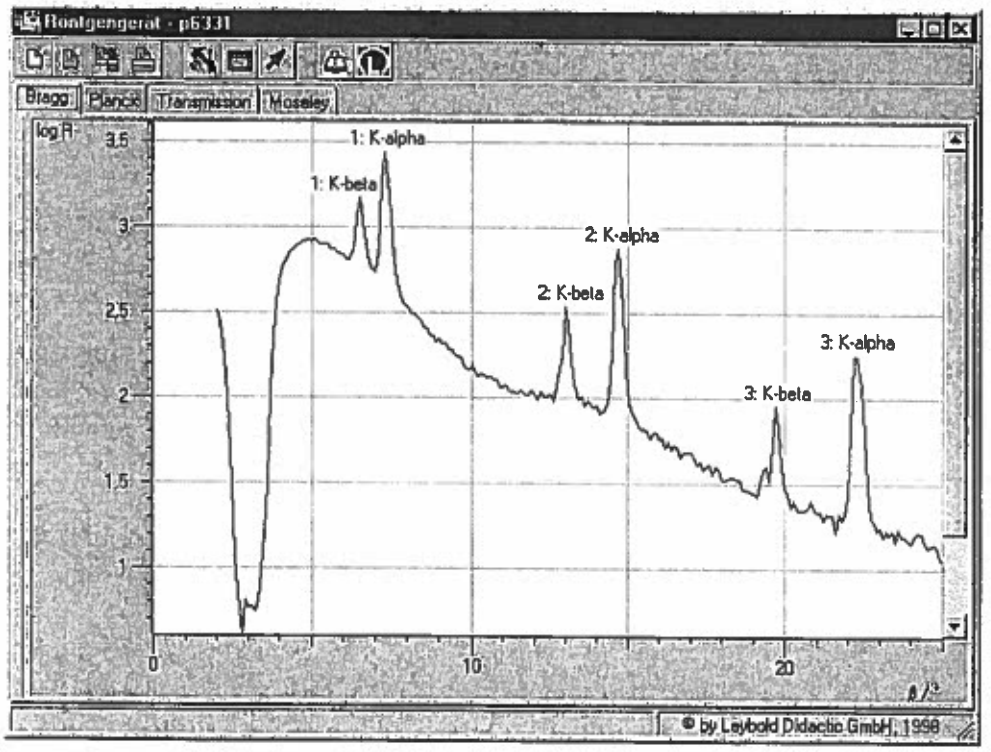


Fig. 4 Diffraction spectrum of x-ray radiation for *Bragg* reflection to the third order at an NaCl monocrystal
Top: linear representation of counting rate R
Bottom: logarithmic representation of counting rate R
Parameters of x-ray tube: U = 35 kV and I = 1 mA

Evaluation

- Access the evaluation functions of the software "X-ray Apparatus" by clicking the right-hand mouse button and select the command "Calculate Peak Center".
- Using the left mouse button, mark the "entire width" of the peaks; if desired, insert the calculated peak center β and the peak width σ in the diagram with Alt+T and note the center as the glancing angle in the measurement table (see tables 3 and 4).
- Save your measurements and evaluations to a suitably named file with the button or by pressing F2.
- Using the glancing angle ϑ and the lattice plane spacing d = 282.01 pm, calculate the wavelength λ using Bragg's law of reflection (IV) (see tables 3 and 4).
- Find the mean values for the individual diffraction orders of the measured wavelengths (see table 5).

Table 3: Measured glancing angles of the Mo K_α line and the calculated wavelengths λ for the first through third diffraction orders

n	$\vartheta(K_\alpha)$	$\frac{\lambda(K_\alpha)}{pm}$
1	7.24°	71.08
2	14.60°	71.09
3	22.20°	71.04

Table 4: Measured glancing angles of the Mo K_β line and the calculated wavelengths λ for the first through third diffraction orders

n	$\vartheta(K_\beta)$	$\frac{\lambda(K_\beta)}{pm}$
1	6.42°	63.07
2	12.94°	63.15
3	19.58°	63.01

Table 5: Mean value and literature value [2] for the characteristic wavelength λ

	$\frac{\lambda(K_\alpha)}{pm}$	$\frac{\lambda(K_\beta)}{pm}$
Mean value	71.07	63.08
Literature value	71.08	63.09

Results

The close agreement of the experimentally determined wavelengths for the characteristic lines with the literature values in table 5 verify the validity of Bragg's law. This simultaneously confirms the wave nature of x-rays, as this property was assumed in the process of deducing this law.

Additional information

The characteristic K_α and K_β lines actually consist of multiple, adjacent discrete lines, which can be observed separately at higher diffraction orders (see Physics Leaflet P 6.3.3.4). Table 1 shows the weighted mean values of the respective individual lines from this substructure.

Literature

[1] Handbook of Chemistry and Physics, 52nd Edition (1971–72), The Chemical Rubber Company, Cleveland, Ohio, USA.

[2] C. M. Lederer and V. S. Shirley, Table of Isotopes, 7th Edition, 1978, John Wiley & Sons, Inc., New York, USA.

iv

(P6.3.2.1)

Investigating the attenuation of x-rays as a function of the absorber material and absorber thickness.

Investigating the attenuation of x-rays as a function of the absorber material and absorber thickness

Objects of the experiment

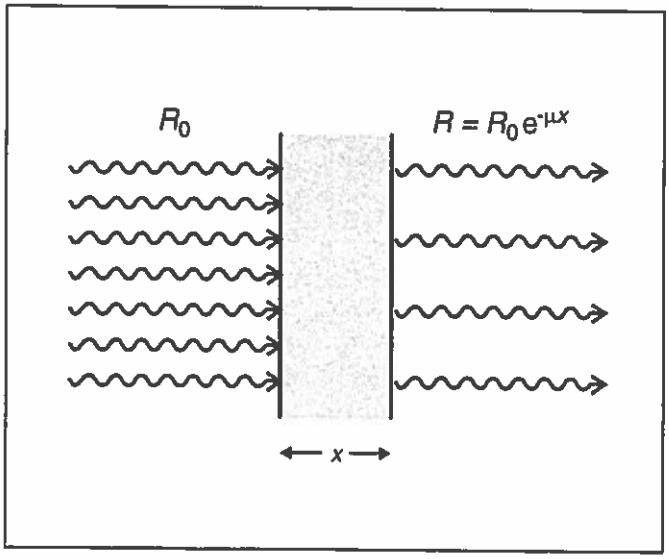
- To investigate the attenuation of x-rays as a function of the absorber thickness.
- To verify Lambert's law of attenuation.
- To investigate the attenuation of x-rays as a function of the absorber material.
- To confirme the wavelength-dependency of attenuation.

Principles

When we speak of attenuation of x-rays, we mean the decrease in intensity that occurs when the radiation passes through matter. This attenuation is caused mainly by two effects: scattering and absorption.

Although absorption and attenuation are different physical phenomena, the transilluminated object is often referred to —inaccurately— as an absorber; this should more properly be termed an attenuator. However, this description will follow the traditional usage in some places and refer to absorbers instead of attenuators.

The scattering of x-ray quanta at the atoms of the attenuator material causes a part of the radiation to change direction. This reduces the intensity in the original direction. This scattering can be either elastic or entail an energy loss or shift in wavelength, i.e. inelastic scattering.



In absorption, the entire energy of the x-ray quanta is transferred to the atoms or molecules of the irradiated material as excitation or ionizing energy.

If R_0 is the original counting rate in front of the attenuator and R is the counting rate behind it, we can quantify the transmission of the radiation to characterize the permeability of an attenuator using:

$$T = \frac{R}{R_0} \qquad \text{(I)}.$$

The greater the so-called transmittance of an attenuator is, the lower is its attenuating capacity.

The transmittance depends on the thickness of the attenuator. If we assume that the properties of the incident radiation remain unchanged in spite of attenuation, an increase in the thickness x by the amount dx will cause a decrease in the transmittance T by the amount dT. The relative reduction in transmission is proportional to the absolute increase in thickness:

$$-\frac{dT}{T} = \mu \cdot dx \qquad \text{(II)}.$$

The proportionality factor μ is referred to as the linear attenuation coefficient.

As the transmittance $T = 1$ for $x = 0$, integration of equation (II) gives us

$$T = e^{-\mu \cdot x} \qquad \text{(III)}$$

or

$$\ln T = -\mu \cdot x \qquad \text{(IV)}.$$

This relationship is known as Lambert's law of attenuation after *Johann Heinrich Lambert*, the 18th century scientist and philosopher.

The aim of this experiment is to verify Lambert's law of attenuation. It also demonstrates that the attenuation depends on the attenuating material and the wavelength of the x-rays.

Apparatus

1 X-ray apparatus	554 811
or	
1 X-ray apparatus	554 812
1 Goniometer	554 83
1 End-window counter for α, β, γ and x-ray radiation	559 01
1 Set of absorbers x-ray	554 834

Setup

Set up the experiment as shown in Fig. 1.

- Mount the collimator in the collimator mount (a) (note the guide groove).
- Attach the goniometer to guide rods (d) and connect ribbon cable (c) for controlling the goniometer.
- Remove the protective cap of the end-window counter, place the end-window counter in sensor seat (e) and connect the counter tube cable to the socket in the experiment chamber marked GM TUBE.
- Demount the target holder (g) of the goniometer and remove the target stage from the holder.
- Place the guide edge of the set of absorbers I (f) in the 90° curved groove of the target holder and carefully slide it into the target holder as far as it will go.

Safety notes

The x-ray apparatus fulfills all regulations governing an x-ray apparatus and fully protected device for instructional use and is type approved for school use in Germany (NW 807/97 Rö).

The built-in protection and screening measures reduce the local dose rate outside of the x-ray apparatus to less than 1 μSv/h, a value which is on the order of magnitude of the natural background radiation.

- Before putting the x-ray apparatus into operation inspect it for damage and to make sure that the high voltage is shut off when the sliding doors are opened (see Instruction Sheet for x-ray apparatus).
- Keep the x-ray apparatus secure from access by unauthorized persons.

Do not allow the anode of the x-ray tube Mo to overheat.

- When switching on the x-ray apparatus, check to make sure that the ventilator in the tube chamber is turning.

The goniometer is positioned solely by electric stepper motors.

- Do not block the target arm and sensor arm of the goniometer and do not use force to move them.

- Mount the target holder.
- Press the ZERO key to return the target and sensor arms to the zero position.
- Check the zero position of the empty diaphragm of the set of absorbers and the sensor and correct this if necessary (see "Adjusting the zero position of the measuring system" in the Instruction Sheet of the x-ray apparatus).
- By moving the goniometer, set a distance of approx. 5 cm between the collimator of the x-ray apparatus and the empty diaphragm, and set a distance of approx. 5 cm between the empty diaphragm and the sensor slit by moving the sensor holder (b).

Carrying out the experiment

a) Attenuation as a function of the absorber thickness:

a1) Without zirconium filter:

- Set the tube high voltage to U = 21 kV.
- Set the emission current I = 0.05 mA.

Note: The counting rate should not appreciably exceed 1500/s. This avoids having to correct for dead time.

- Press the key TARGET.
- Set the angular step width $\Delta\beta$ = 0° (see "Activating an exposure timer" in the Instruction Sheet of the x-ray apparatus).
- Set the measuring time Δt = 100 s.
- Using the ADJUST knob, set the angular positions of the absorbers (approx. 0°, 10°, 20°, 30°, 40°, 50° and 60°) one after another, start the measurement with the SCAN key and display the mean counting rate R after the measuring time elapses by pressing REPLAY. Write down your experiment results (see table 1).

a2) With zirconium filter:

- Mount the zirconium filter on the collimator (this suppresses the short-wave component of the bremsstrahlung radiation generated at U = 21 kV almost entirely).
- Set the emission current I = 0.15 mA and the measuring time Δt = 200 s.
- Using the ADJUST knob, set the angular positions of the absorbers (approx. 0°, 10°, 20°, 30°, 40°, 50° and 60°) one after another, start the measurement with the SCAN key, display the mean counting rate R after the measuring time elapses by pressing REPLAY and write down your results (see table 2).

b) Attenuation as a function of the absorber material:

b1) Without zirconium filter:

- Replace set of absorbers I (absorbers of different thicknesses) with set of absorbers II (absorbers of different materials, d = 0.05 cm).
- Remove the zirconium filter.
- Set the tube high voltage to U = 30 kV (this ensures that the radiation also penetrates the thick absorbers).
- Set the emission current I = 0.02 mA and the measuring time Δt = 30 s.

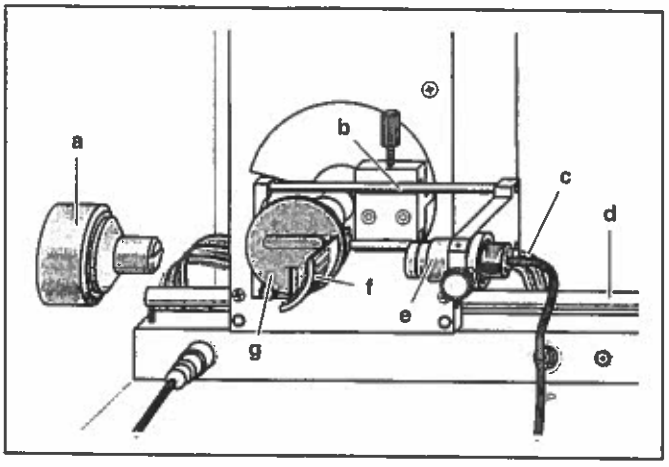


Fig. 1 Setup for investigating the attenuation of x-rays as a function of the thickness of the absorber material.

- Using the ADJUST knob, set the angular positions of the first three absorbers (approx. 0°, 10° and 20°) one after another, start the measurement with the SCAN key and display the mean counting rate R after the measuring time elapses by pressing REPLAY. Write down your results.
- Set the emission current I = 1.00 mA and the measuring time Δt = 300 s.
- Using the ADJUST knob, set the angular positions of the four remaining absorbers (approx. 30°, 40°, 50° and 60°) one after another, start the measurement with the SCAN key and display the mean counting rate R after the measuring time elapses by pressing REPLAY. Write down your experiment results (see table 3).

b2) With zirconium filter:

- Attach the zirconium filter and repeat the measurement as described for b1) (see table 4).

b3) Measuring the background effect:

- Set the parameters U = 0 kV and I = 0 mA and measure the counting rate R_1 of the background effect for a measuring time of Δt = 300 s.

Measuring example

a) Attenuation as a function of the absorber thickness:

Tab. 1: Counting rate R as a function of thickness d of the aluminum absorber (U = 21 kV, I = 0.05 mA, Δt = 100 s, without zirconium filter)

	$\frac{R}{s^{-1}}$
0	977.9
0.5	428.6
1.0	210.1
1.5	106.1
2.0	49.10
2.5	30.55
3.0	16.11

Tab. 2: Counting rate R as a function of thickness d of the aluminum absorber (U = 21 kV, I = 0.15 mA, Δt = 200 s, with zirconium filter)

$\frac{d}{mm}$	$\frac{R}{s^{-1}}$
0	969.4
0.5	426.1
1.0	197.3
1.5	84.29
2.0	40.51
2.5	19.48
3.0	9.52

b) Attenuation as a function of the absorber material:

Tab. 3: Counting rate R as a function of the absorber material (U = 30 kV, d = 0.05 cm, without zirconium filter)

Absorber	Z	$\frac{I}{mA}$	$\frac{\Delta t}{s}$	$\frac{R}{s^{-1}}$
none		0.02	30	1841
C	6	0.02	30	1801
Al	13	0.02	30	1164
Fe	26	1.00	300	93.3
Cu	29	1.00	300	16.63
Zr	40	1.00	300	194.3
Ag	47	1.00	300	106

Tab. 4: Counting rate R as a function of the absorber material (U = 30 kV, d = 0.05 cm, with zirconium filter)

Absorber	Z	$\frac{I}{mA}$	$\frac{\Delta t}{s}$	$\frac{R}{s^{-1}}$
none		0.02	30	718.3
C	6	0.02	30	698.4
Al	13	0.02	30	406.1
Fe	26	1.00	300	29.24
Cu	29	1.00	300	6.016
Zr	40	1.00	300	113.9
Ag	47	1.00	300	24.52

Background effect: $R_1 = 0.243\ s^{-1}$

Evaluation and results

a) Attenuation as a function of the absorber thickness:

When we insert the measurement data from tables 1 and 2 in equation 1, we obtain the transmittance T. Fig. 2 shows how this depends on the thickness d of the absorber. The plotted curve conforms to the exponential function to be expected from equation (III).

Fig. 3 shows a floating-point representation in accordance with equation (IV). In this representation, the attenuation of x-ray radiation (monochromatized using the zirconium filter) can be described very well using a straight line through the origin that has a slope which corresponds to the linear attenuation coefficient $\mu = 15.7\ cm^{-1}$.

For non-monochromatic (unfiltered) x-ray radiation, the slope of the straight line through the origin fitted according to equation (IV) gives us a slightly smaller value of $\mu = 14.2\ cm^{-1}$ for the attenuation coefficient. Also, we can note deviations from the linear curve. The attenuation cannot be described using a single attenuation coefficient; rather, the radiation has a larger high-energy component than the measurement with Zr filter, so that less attenuation occurs for the same absorber thickness.

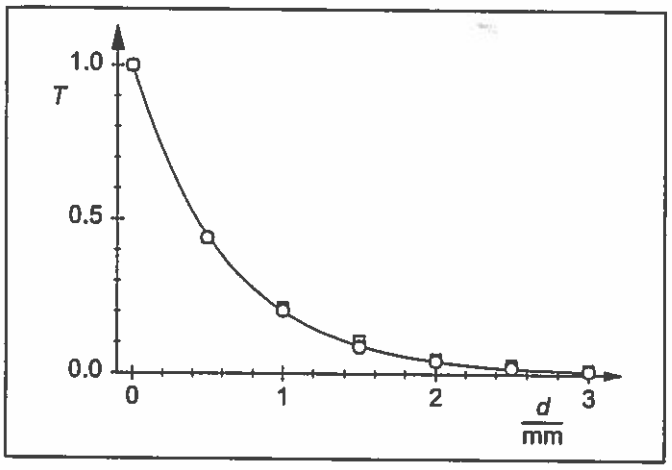


Fig. 2 Transmittance T as a function of the thickness d of the aluminum absorbers
Circles: measurement with zirconium filter
Squares: measurement without zirconium filter

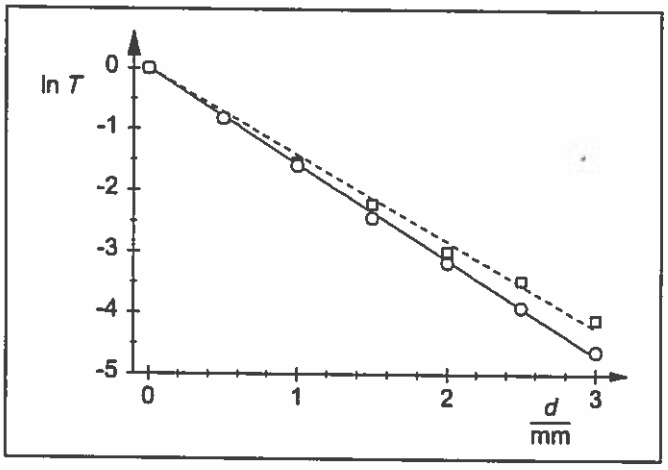


Fig. 3 Floating-point representation of transmission T as a function of the thickness d of the aluminum absorbers
Circles: measurement with zirconium filter
Squares: measurement without zirconium filter

b) Attenuation as a function of the absorber material:

Assuming that the counting rate is proportional to the emission current I, it is possible to scale the counting rates from tables 3 and 4 (after subtracting the background effect) to the emission current $I = 1.00$ mA.

Using the scaled data, equation (I) gives us the transmission T (see tables 5 and 6), which we can use to calculate the linear attenuation coefficient μ for $d = 0.05$ cm by means of equation (IV).

Fig. 4 shows the relationship between the linear attenuation coefficient μ and the atomic number Z. Below $Z = 40$ (Zr), the attenuation coefficient increases steeply as the atomic number rises. When Z reaches 40, we observe an abrupt decrease, which is more apparent for the filtered radiation. This reduction is due to the fact the certain excitations are no longer possible in Zr (binding energy of the K shell is too great, see experiment P6.3.4.5). The unfiltered radiation contains a high-energy component which can still generate this excitation, so that the decrease in μ is less.

Tab. 5: Counting rate R ($I = 1.00$ mA), transmittance T and linear attenuation coefficient μ as a function of the atomic number Z of the absorber material ($U = 30$ kV, $d = 0.05$ cm, without zirconium filter).

Z	$\frac{R}{s^{-1}}$	T	$\frac{\mu}{cm^{-1}}$
none	$92.0 \cdot 10^3$	1.000	0
6	$90.0 \cdot 10^3$	0.978	0.445
13	$58.3 \cdot 10^3$	0.634	9.11
26	93.1	$1.01 \cdot 10^{-3}$	138
29	16.4	$0.178 \cdot 10^{-3}$	173
40	194	$2.11 \cdot 10^{-3}$	123
47	106	$1.15 \cdot 10^{-3}$	135

Tab. 6: Counting rate R (I = 1.00 mA), transmittance T and linear attenuation coefficient μ as a function of the atomic number Z of the absorber material (U = 30 kV, d = 0.05 cm, with zirconium filter).

Z	$\frac{R}{s^{-1}}$	T	$\frac{\mu}{cm^{-1}}$
none	$35.9 \cdot 10^3$	1.000	0
6	$34.9 \cdot 10^3$	0.972	0.568
13	$20.3 \cdot 10^3$	0.565	11.4
26	29.0	$0.808 \cdot 10^{-3}$	142
29	5.77	$0.161 \cdot 10^{-3}$	175
40	114	$3.18 \cdot 10^{-3}$	115
47	24.3	$0.677 \cdot 10^{-3}$	146

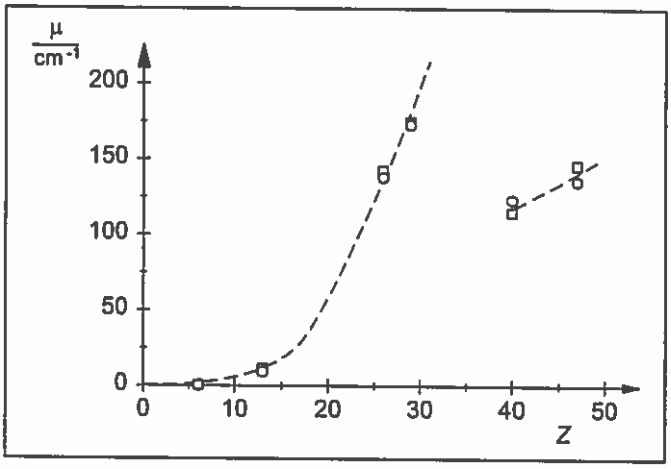


Fig. 4 Linear attenuation coefficient μ as a function of the atomic number Z of the absorber
Circles: measurement with zirconium filter
Squares: measurement without zirconium filter