

Estudo da radiação X e da sua interacção com a matéria

Para realizar este trabalho usa-se um difractor de Raios X, que se baseia no conjunto emissor-amostra-detector. O emissor é uma ampola de raios X com ânodo de molibdénio (Mo) e o detector um contador de Geiger-Müller. O suporte da amostra ocupa o centro de um goniómetro vertical. Para medições em função do ângulo, fazem-se varrimentos (*scans*) em que a amostra e o detector podem rodar independentemente (modos: TARGET ou SENSOR) ou solidariamente (modo COUPLED).

Cada grupo deve levar uma disquete para copiar os resultados brutos.

0. Estudo da colimação do feixe de raios X

Fazer a seguinte aquisição em modo SENSOR:

Fixar $U = 25$ kV e $I=0.02$ mA,

ângulo mínimo (início) de -2° e máximo de 2° ,

passos de 0.2° ,

tempos de 10 s.

1. Estudo do espectro de energia da ampola de raios X de Mo em função da alta tensão aplicada e em função da corrente de emissão (P 6.3.3.2)

Pedir ao docente para colocar a amostra de NaCl no goniómetro.

Fazer as seguintes aquisições, sempre em modo COUPLED:

A. Em função da tensão:

$U = 20, 25, 35$ kV, tendo fixado $I=1.00$ mA,

ângulo mínimo (início) de 2.5° e máximo de 12.5° ,

passos de 0.2° ,

tempos de 20 s para 20kV e de 10 s para 25 e 35 kV.

B. Em função da corrente:

$I = 0.40, 1.00$ mA (não repetir!), tendo fixado $U=35$ kV,

ângulo mínimo (início) de 2.5° e máximo de 12.5° ,

passos de 0.2° ,

tempos de 10 s.

Organizar este conjunto de espectros em gráficos, de modo a pôr em evidência as propriedades atómicas em questão, e comentar cada um. (Sugestões: espectro contínuo/característico, limiar de produção de raios X característicos, limites cinemáticos dos espectros.)

2. Estudo da atenuação de um feixe de raios X em função da espessura do material absorvedor e em função de diferentes materiais (P 6.3.2.1)

Pedir ao docente para retirar a amostra de NaCl do goniómetro.

Fazer as seguintes aquisições, sempre em modo TARGET:

A. *Em função da espessura do material:*

Fixar $U = 21$ kV e $I=0.05$ mA,

ângulo mínimo (início) de 10° e máximo de 60° ,

passos de 10° ,

ângulo do detector: 0° (usar o botão ADJUST)

tempos de 100 s.

Dados: Espessuras (montadas em conjunto) do absorvedor de Al: 0., 0.5, 1.0, 1.5, 2.0, 2.5, 3.0 mm

B. *Em função de diferentes materiais:*

★ Fixar $U = 30$ kV e $I=0.02$ mA,

ângulo mínimo (início) de 10° e máximo de 30° ,

passos de 10° ,

ângulo fixo do detector: 0° (usar o botão ADJUST)

tempos de 30 s.

★ Fixar $U = 30$ kV e $I=1.00$ mA,

ângulo mínimo (início) de 40° e máximo de 60° ,

passos de 10° ,

ângulo do detector: 0°

tempos de 300 s.

C. *Medida do ruído de fundo:*

Fixar $U = 0$ kV e $I=0$ mA,

tempo de 300 s.

Dados: Absorvedores (montados em conjunto): nenhum, C, Al, Fe, Cu, Zr, Ag ($Z=0, 6, 13, 26, 29, 40, 47$), todos de espessura 0.5 mm.

Em cada parte A e B organizar os dados numa tabela de contagens, brutas e calculadas (por subtracção do ruído de fundo); as contagens são acompanhadas dos seus erros (directos, ou bem propagados). Tabelas com ordenação correcta!

Fazer ajustes aos resultados e extrair:

- No caso A: O coeficiente de absorção linear. Obter o coeficiente de absorção de massa e compará-lo com valores tabelados.

- No caso B: O expoente n do ajuste a Z^n .

Comentar fisicamente os resultados.

3. Estudo da dependência do coeficiente de atenuação linear μ com o comprimento de onda λ da radiação emitida. Monocromadores (P 6.3.2.2)

Pedir ao docente para colocar a amostra de NaCl no goniómetro.

Fazer as seguintes aquisições, sempre em modo COUPLED:

Para cada absorvedor: nenhum, Cu, Zr:

Fixar $U = 30$ kV e $I=1.0$ mA,

ângulo mínimo (início) de 4.2° e máximo de 8.4° ,

passos de 0.1° ,

tempos de 15 s.

Dados: Absorvedores individuais: nenhum, Cu, Zr ($Z = 0, 29, 40$; $x_i = 0., 0.07, 0.05$ mm)

Organizar os dados brutos e os calculados numa tabela, como habitualmente. Determinar as transmitâncias em ambos os casos (Cu e Zr).

Investigating the energy spectrum of an x-ray tube as a function of the high voltage and the emission current

Objects of the experiment

- To record the energy spectra of an x-ray tube with Mo anode by means of Bragg reflection of the x-radiation at an NaCl crystal in the first diffraction order.
- To understand the energy spectra as a superposition of the continuum of bremsstrahlung radiation and the lines of the characteristic x-ray radiation of the anode material.
- To investigate how the bremsstrahlung radiation and the characteristic radiation depend on the high voltage and the emission current.

Principles

X-rays are created when fast-moving electrons are rapidly decelerated in matter. According to the laws of classical electrodynamics, this deceleration gives rise to electromagnetic radiation which is mainly radiated perpendicular to the direction of acceleration for energies below 50 keV, i.e. in this case perpendicular to the direction of the electrons striking the anode. For historical reasons, this x-ray component is referred to as "bremsstrahlung" after the German word for the deceleration process by which it occurs. The bremsstrahlung radiation has a continuous spectrum which extends to a certain maximum frequency ν_{\max} or a minimum wavelength λ_{\min} .

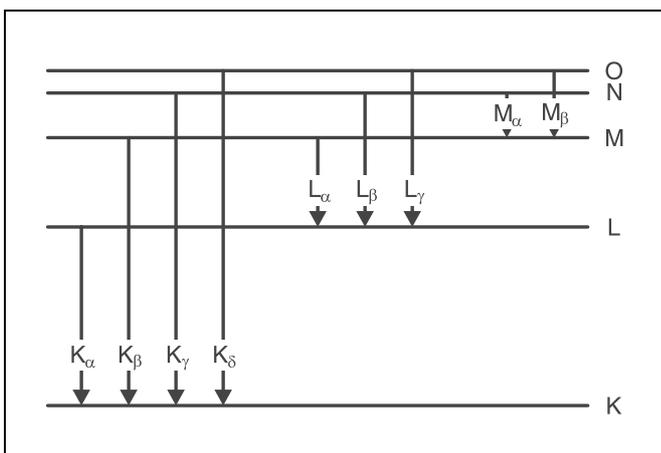
If the energy of the electrons exceeds a critical value, the characteristic x-radiation is generated, which appears in the spectrum as individual lines in addition to the continuous bremsstrahlung spectrum. These lines are generated when high-energy electrons penetrate deep into the atomic shells of the anode material and eject electrons from the innermost

orbitals by collision. The gaps created in this process are filled by electrons from the outer orbitals under emission of x-rays. The resulting x-radiation is characteristic of that anode material and is roughly comparable to the optical line spectrum of a material in a gaseous or vapor state. Solid bodies also emit individual, sharply defined lines in the x-ray range; unlike the visible light excited in the outer orbitals of the electron shell, their position is virtually independent of the chemical situation of the emitting atoms or the aggregate state of the material.

Fig. 1 serves to illustrate the nomenclature adopted for the orbital model of the atomic shell for the lines of the characteristic x-radiation: the individual orbitals are characterized by a particular binding energy and are designated from the innermost to the outermost with the letters K, L, M, N, etc. Electrons can move between the orbitals in accordance with the laws of quantum mechanics; these transitions entail either the absorption or emission of radiation, depending on the direction. For example, radiation from transitions to the K-orbital occur as a series of sequential lines designated K_{α} , K_{β} , K_{γ} , etc. Starting from K_{α} , the energy of the transitions increases and the corresponding wavelength decreases.

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

Fig. 1 Simplified term diagram of an atom and definition of the K, L and M series of the characteristic x-ray radiation



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 or Windows NT

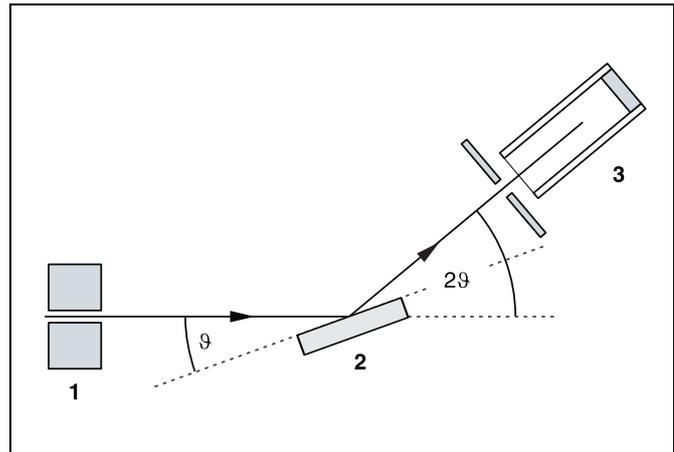


Fig. 2 Schematic diagram of diffraction of x-rays at a monocrystal and 2θ coupling between counter-tube angle and scattering angle (glancing angle)
1 collimator, 2 monocrystal, 3 counter tube

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

$$\lambda = 2 \cdot d \cdot \sin \vartheta \quad (I).$$

$d = 282.01$ pm: lattice plane spacing of NaCl

Together with the relationships valid for electromagnetic radiation

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

ν : frequency, c : velocity of light

and

$$E = h \cdot \nu \quad (III)$$

E : energy, h : Planck's constant

equation (I) gives us the energy of the x-radiation. The spectrometer thus provides the wavelength, frequency or energy spectrum of the radiation, depending on the selected representation mode.

This experiment investigates the effect of the tube high voltage U and the emission current I on the energy spectrum of the x-ray tube. The high voltage U is applied as the accelerating voltage for the electrons between the cathode and the anode (see Fig. 3). The emission current I , i.e. the current flowing between the anode and the cathode, can be controlled by changing the heating voltage U_K of the cathode.

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 $1 \mu\text{Sv/h}$, 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.

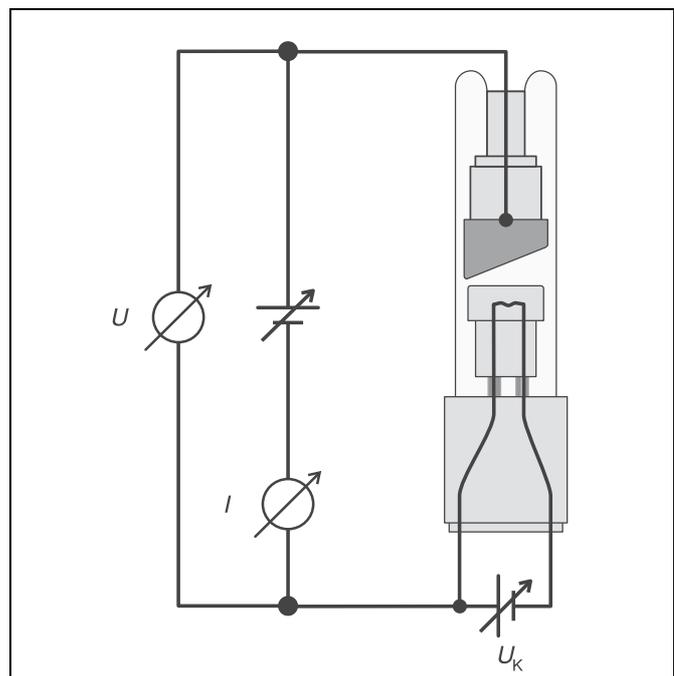
Do not allow the anode of the x-ray tube Mo to overheat.

- 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.

Fig. 3 Schematic diagram showing the structure of the x-ray tube



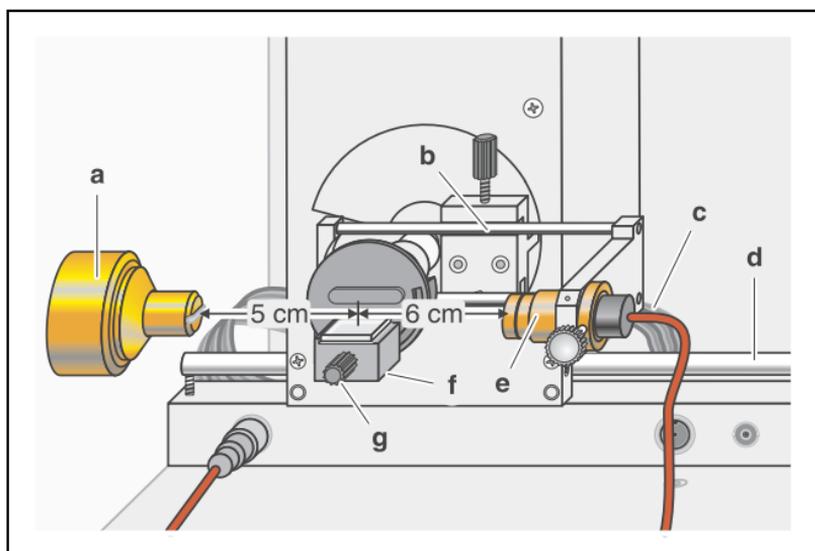


Fig. 4 Experiment setup for investigating the energy spectrum of an x-ray tube

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 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 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 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 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 to separate the characteristic K_α and K_β lines.

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 confirm 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:

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

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:

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

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

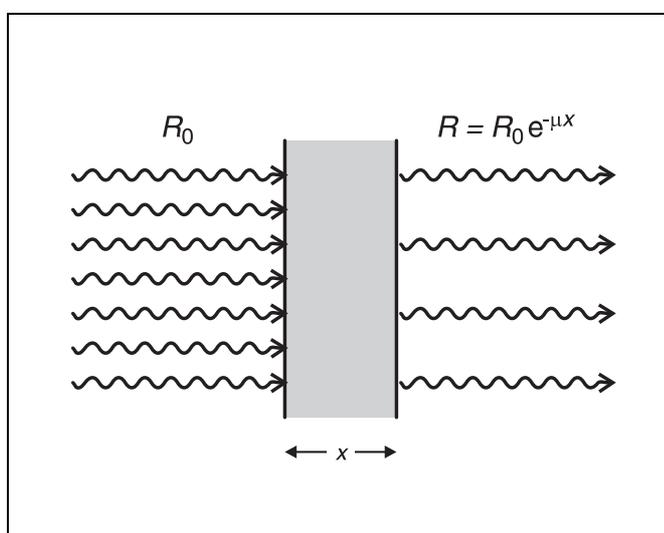
$$T = e^{-\mu \cdot x} \quad (III)$$

or

$$\ln T = -\mu \cdot x \quad (IV).$$

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

- 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).

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 (f) in the 90° curved groove of the target holder and carefully slide it into the target holder as far as it will go.

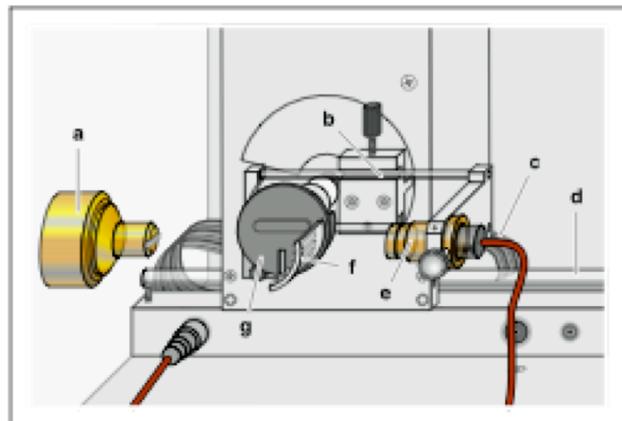


Fig. 1 Setup for investigating the attenuation of x-rays as a function of the thickness of the absorber material.

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 $1 \mu\text{Sv/h}$, 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.

Investigating the wavelength dependency of the coefficient of attenuation

Objects of the experiment

- To measure the transmittance T of a copper foil and a zirconium foil for x-rays as a function of the wavelength λ between 30 and 120 pm.
- To investigate the wavelength-dependency of the attenuation coefficient μ outside of the absorption edges.
- To confirm the λ^3 law as a function of the absorption coefficient τ .

Principles

The attenuation of x-rays passing through matter is described by Lambert's law (see experiment P6.3.2.1):

$$R = R_0 \cdot e^{-\mu x} \quad (I)$$

Here, R_0 is the intensity of the x-ray radiation in front of the attenuator, R is the intensity behind the attenuator, μ is the linear attenuation coefficient and x is the thickness of the attenuator.

Absorption and scattering both contribute to attenuation. The linear attenuation coefficient μ is thus composed of the linear absorption coefficient τ and the linear scattering coefficient σ .

$$\mu = \tau + \sigma \quad (II)$$

These coefficients are proportional to the mass and the density ρ of the irradiated material respectively. That is why we often use the so-called mass coefficients

$$\mu_m = \frac{\mu}{\rho}, \tau_m = \frac{\tau}{\rho}, \sigma_m = \frac{\sigma}{\rho} \quad (III)$$

or – for the pure metals observed here – the atomic coefficients or cross-sections

$$\mu_a = \mu_m \frac{A}{N_A}, \tau_a = \tau_m \frac{A}{N_A}, \sigma_a = \sigma_m \frac{A}{N_A} \quad (IV)$$

A : atomic weight

$$N_A = 6.022 \cdot 10^{23} \frac{1}{\text{mol}}, \text{ Avogadro's number}$$

Analogously to equation (II), we can say that

$$\mu_m = \tau_m + \sigma_m \quad (V)$$

and

$$\mu_a = \tau_a + \sigma_a \quad (VI)$$

The absorption of x-rays is essentially due to the ionization of atoms, which release an electron from an inner shell. The absorption cross-section is thus strongly dependent on the quantum energy $h\nu$ resp. the wavelength λ of the x-ray radiation as well as on the excitation energy of the atoms and thus the atomic number Z . For ionization to occur, the quantum energy of the x-rays must be greater than the binding energy E of the electrons of the respective shell. The absorption cross-section is thus very small once the quantum energy is just slightly below the binding energy. The limit wavelength at which the quantum energy is just sufficient is called the absorption edge (see Fig. 1).

Outside of the absorption edges, absorption is described to within a close approximation by the relationship

$$\tau_a = C \cdot \lambda^3 \cdot Z^4 \quad (VII).$$

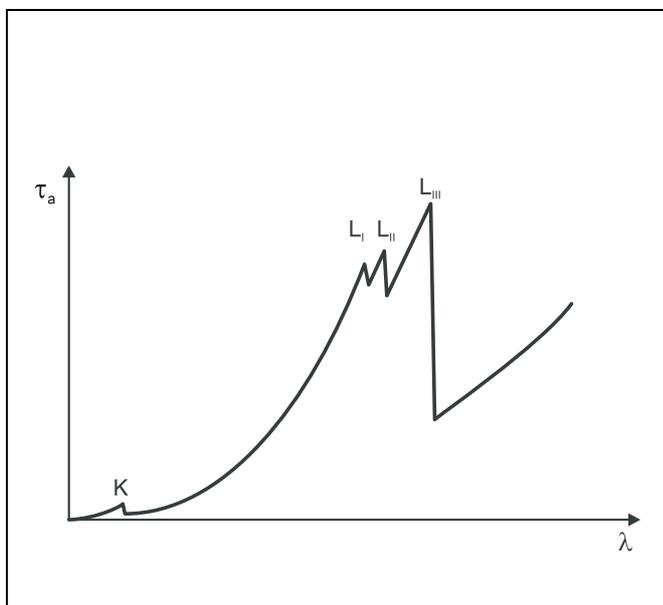


Fig. 1 Absorption coefficient as a function of the x-ray wavelength (schematic)

K: absorption edge of K shell

L_I, L_{II}, L_{III} : absorption edges of L shell

Apparatus

1 X-ray apparatus	554 811
1 End-window counter for α , β , γ and x-ray radiation	559 01
1 Set of absorber foils	554 832

additionally required:

1 PC with Windows 95/98 or Windows NT

This experiment verifies the dependence on the wavelength λ for two different metals, i.e. two different atomic numbers Z .

The evaluation exploits the fact that the scattering cross-section σ_a in the wavelength range $\lambda = 35\text{--}100$ pm is significantly less than the absorption cross-section and can be estimated approximately using

$$\sigma_a = 0.2 \frac{\text{cm}^2}{\text{g}} \cdot \frac{A}{N_A} \quad (\text{VIII})$$

By applying a series of transformations to equations (III)-(VII), we can calculate the absorption cross-section as:

$$\tau_a = \frac{\mu}{\rho} \cdot \frac{A}{N_A} = 0.2 \frac{\text{cm}^2}{\text{g}} \cdot \frac{A}{N_A} \quad (\text{IX})$$

This experiment measures the transmittance

$$T = \frac{R}{R_0} \quad (\text{X})$$

of the transilluminated material as a function of the wavelength of the x-ray radiation. When we apply Lambert's law of attenuation

$$T = e^{-\mu x} \quad (\text{XI})$$

we can calculate the linear attenuation coefficient μ and, using this value and equation (IX), the absorption cross-section τ_a :

$$\tau_a = \frac{-\ln T}{\rho \cdot x} \cdot \frac{A}{N_A} = 0.2 \frac{\text{cm}^2}{\text{g}} \cdot \frac{A}{N_A} \quad (\text{XII})$$

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 $1 \mu\text{Sv/h}$, 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.

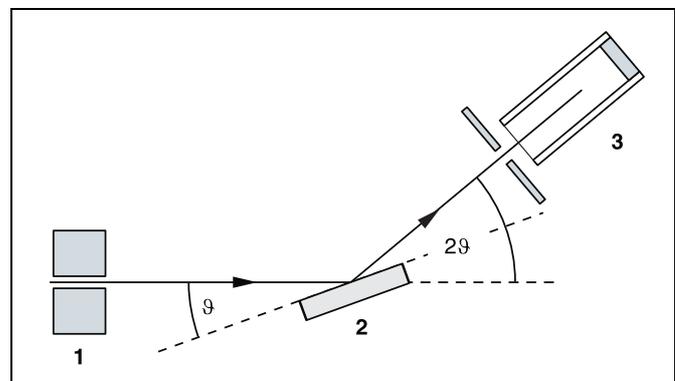


Fig. 2 Diffraction of x-rays at a monocrystal and for 2ϑ coupling between counter-tube angle and scattering angle (glancing angle)
1 collimator, 2 monocrystal, 3 counter tube

A goniometer with NaCl crystal and a Geiger-Müller counter tube in the Bragg configuration are used to record the intensities as a function of the wavelength. The crystal and counter tube are fixed with respect to the incident x-ray beam in 2ϑ coupling, i.e. the counter tube is turned at an angle twice as large as the crystal (see Fig. 2).

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

$$\lambda = 2 \cdot d \cdot \sin \vartheta \quad (\text{XIII})$$

$d = 282.01$ pm: lattice plane spacing of NaCl

Setup

Setup in Bragg configuration:

Set up the experiment as shown in Fig. 3. To do this, 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. 5 cm.
- Mount the target holder with target stage.
- Loosen knurled screw **(g)**, place the NaCl crystal flat on the target stage **(f)**, carefully raise the target stage with crystal all the way to the stop and carefully 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 to separate the K_α and K_β lines.

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 V24 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.

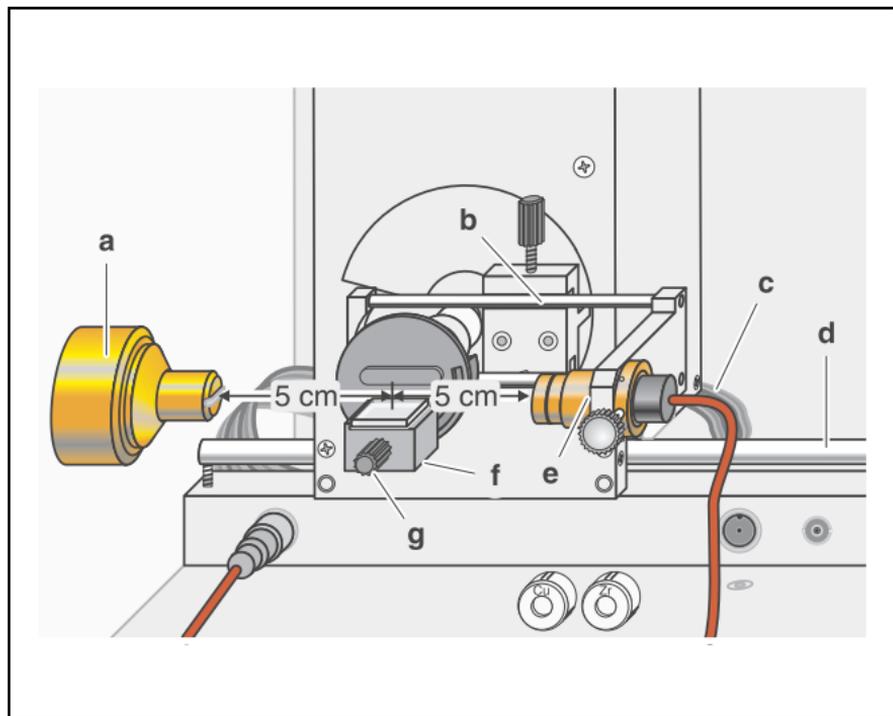


Fig. 3 Experiment for investigating the wavelength-dependency of the attenuation coefficient