

X-ray Diffraction

In this series of experiments, fundamental basics of x-ray physics will be acquired. For this purpose, properties of x-rays and their absorption in different materials will be examined. The application of different x-ray techniques will enable you to analyze structural properties of single crystals as well as of crystalline powders¹. X-ray diffraction techniques play more than ever a central role in research and technology. This fact is mirrored in numerous Nobel prizes associated with this method and the realization of major x-ray research projects like the X-ray Laser (XFEL) at DESY that offer a new and extraordinary perspective for many areas in physics, chemistry, biology or medical sciences.

I. Required Knowledge

Experimental basics²:

Generation and detection of x-rays:

- Mode of operation of an x-ray tube and generation of x-rays.
- Mode of operation of a radiation counter.

Theoretical basics³:

- Definition of a crystal
- Miller Indices
- Interaction of x-rays with matter
- X-ray Diffraction, Bragg's Law, Laue-Condition
- Structure Factor, Atomic Form Factor, Reflection Conditions (Extinction Rules), X-ray Absorption)
- Ewald Construction, Reciprocal Space

II. Literature

- L.H. Schwartz and J.B. Cohen, Diffraction from Materials, 2nd Edition, Springer, 1987 ²
- W. Kleber, Einführung in die Kristallographie, Oldenburg, 1998 ²
- T. Hahn, Internat. Tab. for Cryst., Volume A, D. Reidel Publishing Comp., 1987
- C. Giacovazzo, Fundamentals of Crystallography, Oxford Science Pub.2002
- J. Als-Nielsen, Elements of Modern X-ray Physics, Wiley, 2001 ²
- L. Azaroff, Elements of X-ray Crystallography, McGraw-Hill, 1968
- C. Kittel: Einführung in die Festkörperphysik, Oldenbourg Verlag (2006) ²
- Web site for this experiment:
http://www.itap.physik.uni-stuttgart.de/lehre/vorlesungen/praktikum/index_english.php

¹ In part based on experiments by LD Didactic Handblätter Physik, Hürth

² Literature on these topics can be obtained through your supervising tutor.

³ See: http://www.itap.physik.uni-stuttgart.de/lehre/vorlesungen/praktikum/index_english.php

III. Experimental Goals and Tasks⁴

1. Detection of X-ray Radiation and Measurement of its Intensity with a Geiger-Müller-Tube.

- Determination of the counting rate N as a function of the emission current I .
- Determination of the counting rate N as a function of the acceleration voltage U of the tube.
- Estimation of the Geiger tube's dead time.

2. Bragg-Reflection: Determination of Lattice Constants in Single-Crystals

- Measurement of the diffraction spectrum of a LiF single-crystal.
- Measurement of the diffraction spectrum of a NaCl single-crystal.

3. Verification of the Duane-Hunt Relation and the Determination of the Planck's Constant

Determination of the critical wavelength λ_{\min} with reference to the tube high voltage U .

4. Edge Absorption: Filters for X-rays

Measurement of the diffraction spectrum of a NaCl-crystal with Mo-radiation in the range between 40 and 80 pm ($U=30$ kV).

- Without Zr-filter
- With Zr-filter

5. Investigating the Attenuation of X-rays as a Function of the Absorber Material and its Thickness

- Measurement of x-ray absorption as a function of the material thickness:
 - Without Zr-filter
 - With Zr-filter
- Measurement of x-ray absorption as a function of the absorbing material itself:
 - Without Zr-filter
 - With Zr-filter
 - Measurement of the background

6. Debye-Scherrer-Scans: Determination of Lattice-Plane Spacings and Lattice Constants for Polycrystalline (Powder) Samples

- NaCl
- KCl

7. Laue-Photography: Symmetry und Structure of Crystalline Matter

- NaCl
- LiF

⁴ Bring an USB-stick to save your experimental data.

IV. Safety Information for the Experimental Procedures

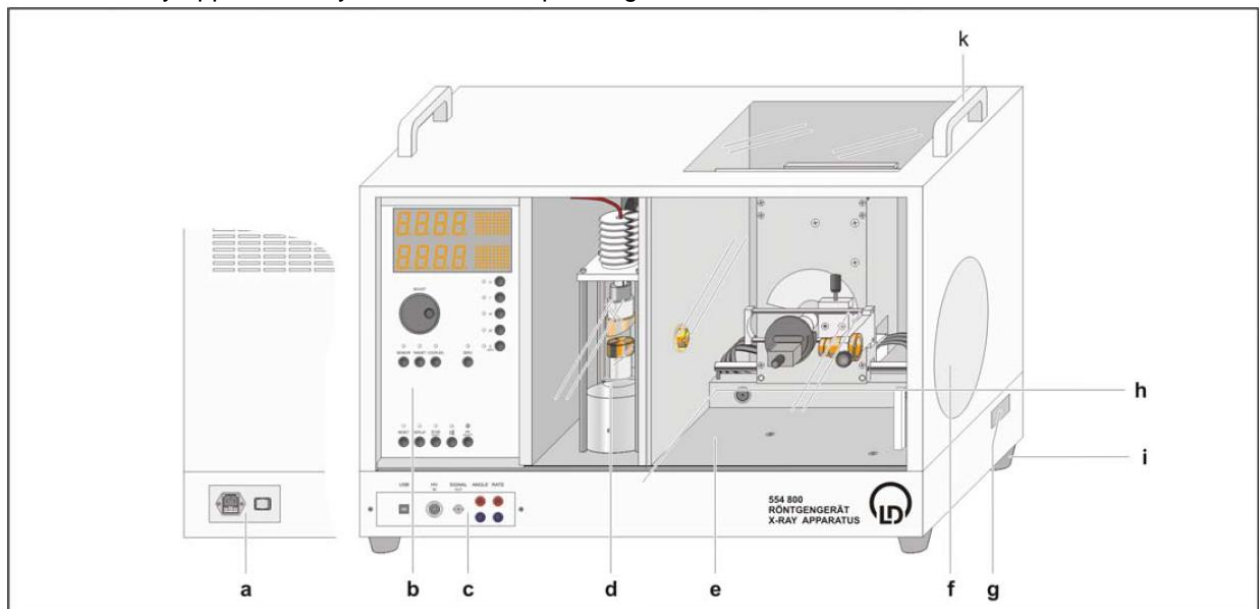
Admittance to this course on x-ray diffraction is only granted to those, who attended the radiation protection course in the present semester.

The design of the x-ray apparatus fulfills the safety regulations for teaching purposes. It has an inherent protection system that is licensed according to the atomic law regulations.

The implemented radiation protection of the x-ray apparatus reduces the local dose rate to less than 1 mSv/h. This value lies within the magnitude of the natural radiation background.

- Before you start working with the x-ray apparatus you need to check its integrity (e.g. damage of windows, case, cables; see also manual).
- Do not allow unauthorized people to operate the apparatus.
- 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.
- Do not block the target arm and sensor arm of the goniometer and do not use force to move them. Solely electric stepper motors position the goniometer.

Sketch of x-ray apparatus Leybold 554 800: Operating manual on the web site of the course



- | | | | |
|---------------------|---|----------------------|--------------------|
| a Mains power panel | d Tube chamber (with X-ray tube Mo) | f Fluorescent screen | i Feet |
| b Control panel | e Experiment chamber (here with goniometer) | g Free channel | k Carrying handles |
| c Connection panel | | h Lock | |

V. Preparation and Admittance to the Practical Course

Before the practical work, you must be able to answer questions to the following points:

- Knowledge of the theoretical and experimental basics according to point I on page 1.
- Having a general idea about the experiments and the principle mode of operation of the x-ray apparatus.
- Knowledge about the necessary basics of radiation protection according the regulations of the Radiation Control Act in the “Röntgenverordnung §36 RöV”.
(Will be acquired during the rad worker course prior to the experiments.)

VI. Experimental Procedure 1-7

(Evaluation of experimental results see pages 30ff.)

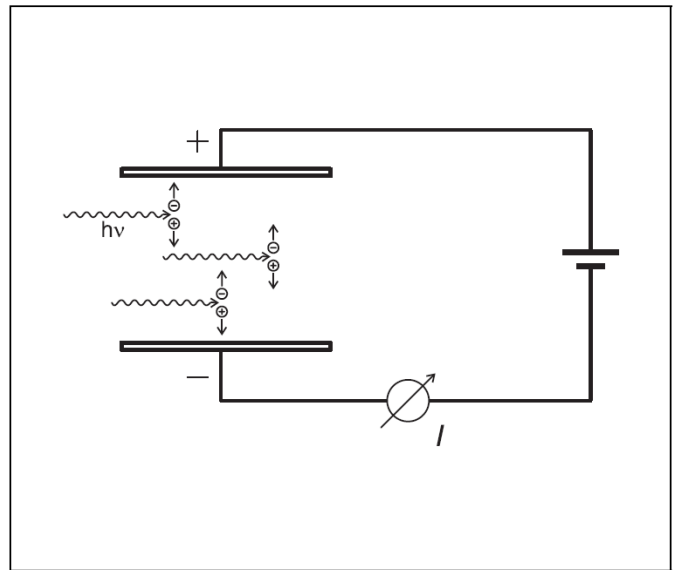
VI.1. Detection of X-ray Radiation and Measurement of its Intensity with a Geiger-Müller-Tube.

- Determination of the counting rate N as a function of the emission current I .
- Determination of the counting rate N as a function of the acceleration voltage U of the tube.
- Estimation of the Geiger tube's dead time.

X-radiation is detectable on account of its physical effects. For example, x-rays expose photographic films and cause air and other gases to become electrically conductive, the photo effect is observed at the surfaces of metals, and some fluorescent substances show luminescence. All these phenomena are caused by ionization of atoms or molecules of the transilluminated matter.

In the quantitative detection of x-rays, we can exploit this ionizing effect, e.g. by measuring the ionization current in a

plate capacitor filled with air or another gas. Due the way it is designed and built, this type of arrangement is called an ionization chamber. If the voltage on the capacitor is high enough each x-ray photon can cause a total ionization of the gas. This mode of operation is called a Geiger-Müller counter and will be used through out this experiment.



Setup

Figure 1.1 shows the set up of the experiment.

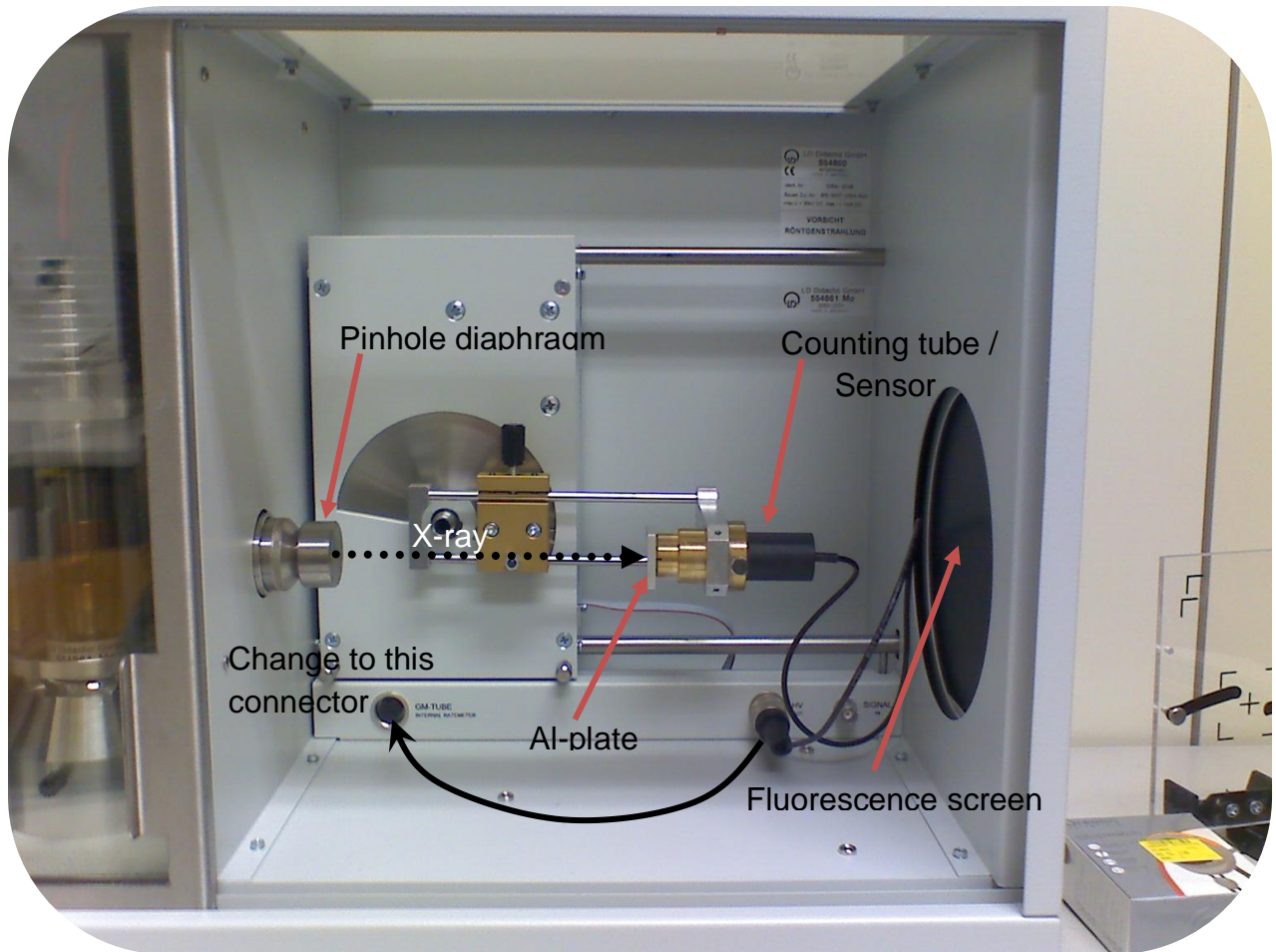


Fig. 1.1: Experimental setup

Experiment and Measurement

Please note the summary of the manual (poster on the wall) of the x-ray apparatus!

- Familiarize yourself with the controls of the apparatus. You find a short manual attached to the wall above the apparatus.
- Switch on the apparatus and computer.
- Set “Target” and “Sensor” to their zero-position. Press the button “Zero” for that purpose.
- Remove equipment not necessary for this experiment from the experimental chamber: target holder or target table of the goniometer (see Fig. 1.1).
- Attach the Al-plate in front of the detector slits with scotch tape (see Fig. 1.1).
- Attach the pinhole diaphragm on the exit slit of the x-rays (see Fig. 1.1).

a. Counting rate N as a function of the emission current I .

- Adjust high voltage of the x-ray tube U to 32 kV.
- Figure out appropriate settings for emission currents between 0 and 1 mA to measure their influence on the primary beam intensity.
- Set the measuring time to $\Delta t = 60$ s and $\Delta \beta = 0$.

- Press the button “Scan” after you have adjusted the measuring time Δt , $\Delta \beta$, high voltage U and the emission current I .
- The tube will switch off after this measurement. Press the button „Replay“ to display the average counting rate.
- Continue for other settings of I .

b. Counting rate N as a function of the acceleration voltage U of the tube.

- Increase the high voltage U in appropriate steps from 10 to 35 kV.
- Set the emission current I to 1.0 mA.
- Measure the primary beam intensity for each setting of U analogue to experiment a with the same values for Δt and $\Delta \beta$.

c. Estimation of the dead time T .

- Set the tube high voltage U to 26 kV.
- Remove the Al-plate in front of the detector.
- Remove the pinhole diaphragm on the exit slit of the x-rays.
- Determine the counting rate N for $I = 0$ mA bis 0.09 mA mit $\Delta I = 0.01$ mA, $\Delta t = 15$ s.
- Determine the counting rate N for $I = 0.1$ mA bis 0.18 mA mit $\Delta I = 0.02$ mA, $\Delta t = 15$ s.
- Determine the counting rate N for $I = 0.2$ mA bis 1.0 mA mit $\Delta I = 0.1$ mA, $\Delta t = 15$ s.

VI.2. Bragg-Reflection: Determination of Lattice Constants in Single-Crystals

- Measurement of the diffraction spectrum of a LiF single-crystal.
- Measurement of the diffraction spectrum of a NaCl single-crystal.

Table 1: Wave length of the characteristic x-ray radiation of molybdenum

Line	λ / pm
K_{α}	71.08
K_{β}	63.09

A Geiger-Müller counter tube is used to detect the x-rays. It will be moved together with the crystal in 2θ -coupling (mode “Coupled”) with respect to the incident x-ray beam. 2θ -coupling means that the counter tube is turned by twice the angle as the crystal (cf. Fig. 2.1). The zero point $\theta = 0^\circ$ is characterized by the lattice planes and the axis of the counter tube being parallel to the incident x-ray beam. As the lattice planes are not

necessarily parallel to the surface of the crystal, the zero point of each crystal must be calibrated individually.

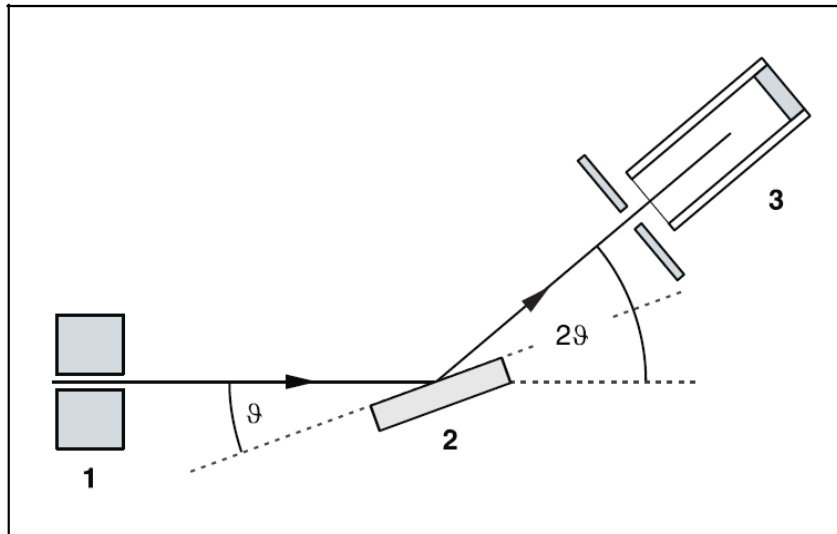


Fig. 2.1: Schematic diagram of diffraction of x-rays from a single crystal and 2θ coupling between counter-tube angle and scattering angle (glancing angle) 1 collimator, 2 single-crystal, 3 counter tube.

Setup of the Bragg configuration:

Fig. 2.2 shows some important details of the experimental setup. To set up the experiment, proceed as follows (see also the Instruction Sheet for the x-ray apparatus):

- Adjust the goniometer to the guide rods (**d**) so that the distance s_1 between the slit diaphragm of the collimator and the target arm is approx. 5 cm.
- 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.
- Mount the target holder (**f**) with its target stage.
- Align the target and sensor arm horizontally using the “Zero” button.

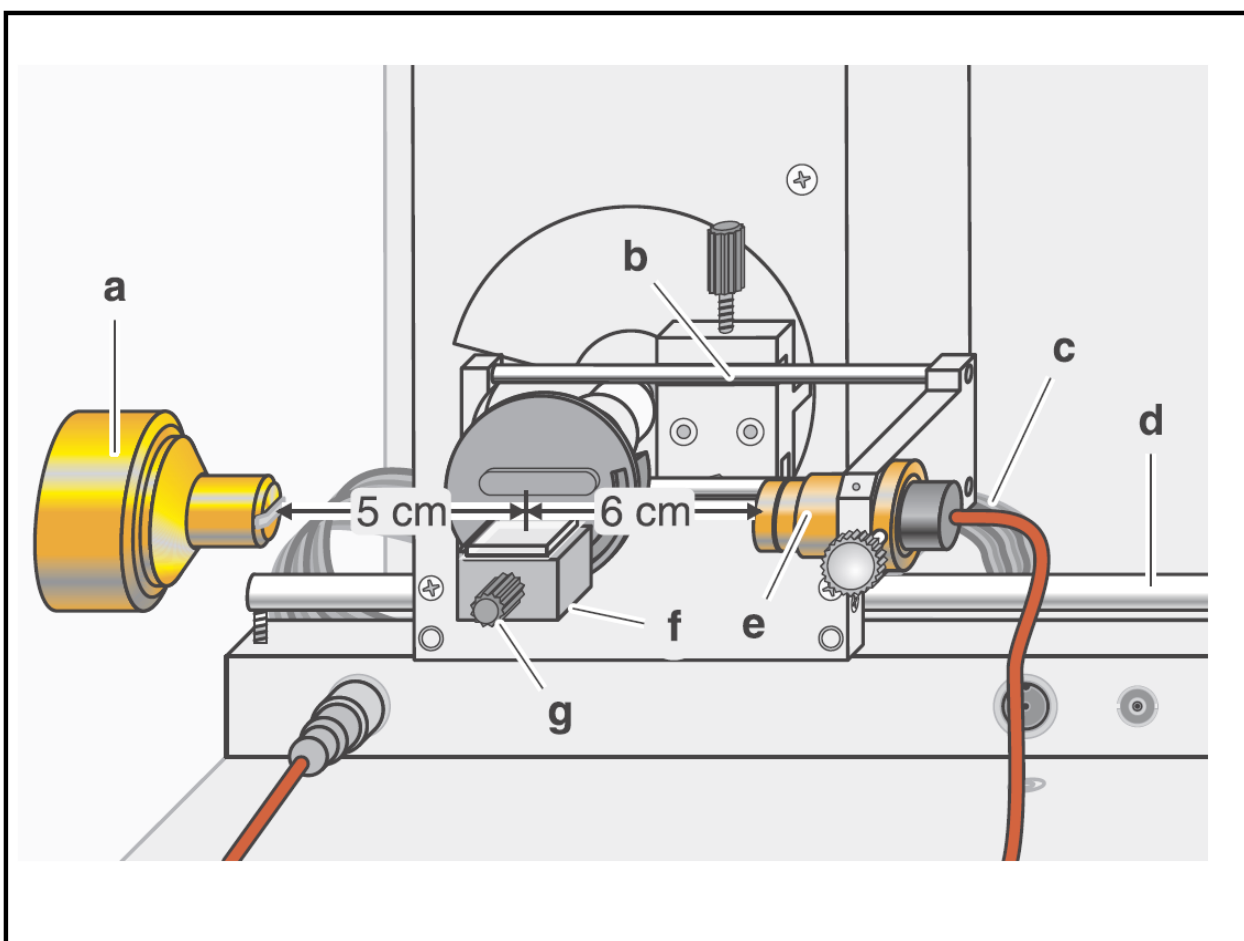


Fig. 2.2 Bragg's configuration

Experiment and Measurement

Note:

The NaCl and LiF crystals are hygroscopic and fragile. Store the crystals in a dry place. Avoid mechanical stress on the crystals. Handle the crystals by their short faces only. If the counting rate is too low, you can reduce the distance s_2 between the target and the sensor. 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.

a. Measurement of the diffraction spectrum of a LiF single-crystal

- Loosen knurled screw (**g**), place the LiF 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 only a slight pressure).
- Make a reasonable choice for the tube high voltage U and the emission current I . Discuss your choice with the tutor if you are uncertain.

Determination of the zero position of the detector and sample with respect to the x-ray incident angle:

- Calculate the Bragg angle of the (200) reflection of LiF, switch to coupled scanning mode and set the target to the calculated angle using the ADJUST knob.
- Switch on the tube high voltage with “HV on/off”.
- Leave the target position unchanged and manually find the maximum of the counting rate for the first reflection of the K_{α} line. Switch to “Sensor” scanning mode and move the detector to find its position for a maximum count rate.
- Leave the sensor fixed in the position of the maximum counting rate. Now look for the maximum of the counting rate in “Target” mode by turning the sample slightly forth and back.
- Switch between sensor and target mode and check iteratively whether you have found the maximum of the counting rate.
- Switch to the “Coupled” scanning mode. Move the target back by the calculated value of the angle for LiF (200) (even if this takes you into the negative range!).
- Save the positions of the target and the sensor as the “zero position of the measuring system” by pressing TARGET, COUPLED and β LIMITS simultaneously.

Recording the diffraction spectrum:

- Start the software “X-ray Apparatus”, check to make sure that the apparatus is connected correctly, and clear any existing measurement data using the key F4.
- Make suitable choices for the measuring time per angular step Δt [s] and the angular step width $\Delta \beta$ [°] for taking the Bragg spectrum.
- Press the “Coupled” key to activate 2θ coupling of target and sensor and set the lower limit of the target angle to 4° and the upper limit to 34°.
- Start measurement and data transfer to the PC by pressing the “Scan” key.
- When you have finished measuring, save the measurement under an appropriate name by pressing the key F2.

b. Measurement of the diffraction spectrum of a NaCl single-crystal

- Press the “Zero” key to return the target and sensor to the current zero position.
- Remove the LiF crystal and carefully mount the NaCl crystal in its place.
- Press the key “Coupled” to activate 2θ coupling of target and sensor and set the lower limit of the target angle to 4° and the upper limit to 24° .
- Proceed analogue to the measurement of LiF.

● Ask your supervising tutor for a copy of the software “X-ray Apparatus” which will help you to analyze our experimental data.

VI.3. Verification of the Duane-Hunt Relation and the Determination of the Planck’s Constant

Determination of the critical wavelength λ_{\min} with reference to the tube high voltage U .

The bremsstrahlung continuum of the emission spectrum of an x-ray tube is characterized by the critical wavelength λ_{\min} (see Fig. 3.1), which decreases as the tube high voltage is increased. In 1915, the American physicists *William Duane* and *Franklin L. Hunt* discovered an inverse proportionality between the limit wavelength and the tube high voltage:

$$(I) \quad \lambda_{\min} \sim 1/U$$

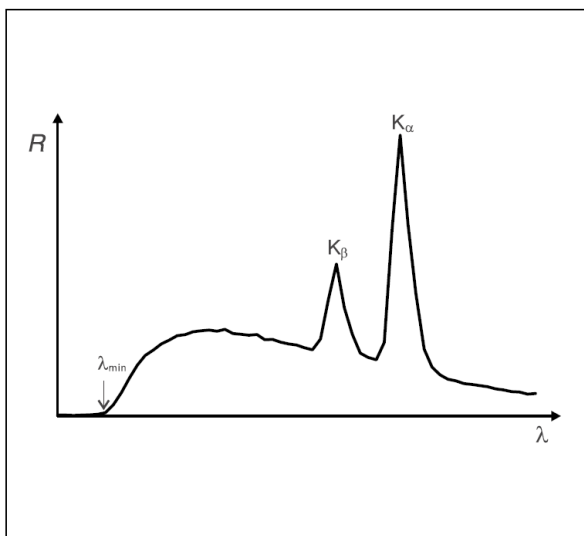


Fig. 3.1 Emission spectrum of an x-ray tube with the limit wavelength λ_{\min} of the bremsstrahlung continuum and the characteristic K_α und K_β lines.

The Duane-Hunt relationship can be sufficiently explained by examining some basic quantum mechanical considerations that you should take from the list of literature in Point II.

Setup of the Bragg configuration:

Please see same paragraph in Experiment VII.2, page 8.

Determination of the critical wave length λ_{\min} as a function of the in tube high voltage U :

- Loosen knurled screw **(g)** (see Fig. 2.2, Exp. VII.2), place the NaCl crystal flat on the target stage **(f)**, 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 page 10, top).

Experiment and Measurement

- Start the software “X-ray Apparatus” and clear any existing measurement data using the key F4.
- Set the tube high voltage to $U = 22 \text{ kV}$ and the emission current to $I = 1.00 \text{ mA}$.
- Set the measuring time per angular step Δt and the angular step width $\Delta\beta$ appropriately in order to obtain small statistical errors for your data.
- Press the “Coupled” key to activate 2θ coupling of target and sensor and set the lower limit β of the target angle to 5.2° and the upper limit of β to 6.2° .
- Start measurement and data transfer to the PC by pressing the “Scan” button.
- In addition, record a series of measurements with the tube high voltages $U = 24 \text{ kV}, 26 \text{ kV}, 28 \text{ kV}, 30 \text{ kV}, 32 \text{ kV}, 34 \text{ kV}$ and 35 kV . You will need to adjust the scan range β for increasing U accordingly.
- To show the wavelength-dependency, open the “Settings” dialog of the software with F5 and choose the lattice constant for NaCl in the top window.
- When you have finished measuring, save the measurement series under an appropriate name by pressing the F2.

VI.4. Edge Absorption: Filters for X-rays

Measurement of the diffraction spectrum of a NaCl-crystal with Mo-radiation with wavelengths between 40 and 80 pm ($U=30$ kV).

- a. Without Zr-filter
- b. With Zr-filter

When x-rays pass through matter, they are attenuated by absorption and scattering of the x-ray quanta. The absorption effect often predominates. Essentially, this is due to the ionization of atoms, which release an electron from an inner shell, e.g. from the K-shell. This can only occur when the quantum energy

(I) $E = h \cdot c / \lambda$, h : Planck's constant, c : velocity of light

is larger than the binding energy E_K of the shell. The transmission

(II) $T = R / R_0$, R : intensity behind attenuator, R_0 : intensity in front of attenuator

of the material thus increases abruptly as a function of the wavelength at

(III) $\lambda_K = h \cdot c / E_K$

This abrupt change is known as the absorption edge, here: the K-absorption edge.

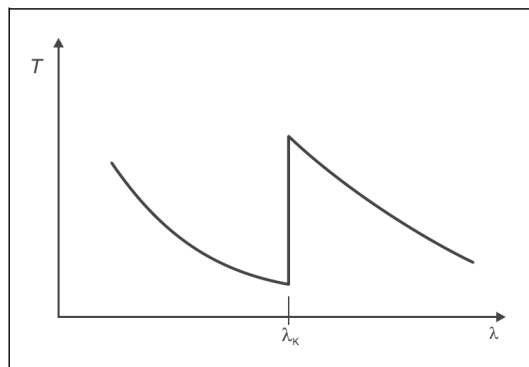


Fig. 4.1: Schematic diagram of the transmission of an attenuator as a function of the x-ray wavelength

We must distinguish between the K-absorption edge and the characteristic x-ray radiation K_α and K_β that is emitted by the transition of an electron from a higher shell to the K-shell. The relationship

(IV) $\lambda(K_\alpha) = h \cdot c / (E_K - E_L)$ und $\lambda(K_\beta) = h \cdot c / (E_K - E_M)$

applies. λ_K is smaller than $\lambda(K_\alpha)$ und $\lambda(K_\beta)$. All three quantities depend on the atomic number Z of the absorbing (and emitting) atoms. Tab. 1 contains a selection of theses quantities taken from literature⁵.

Table 1: Wavelengths $\lambda(K_\alpha)$ and $\lambda(K_\beta)$ and λ_K for atomic numbers $Z = 40-42$.

Element	Z	$\frac{\lambda(K_\alpha)}{\text{pm}}$	$\frac{\lambda(K_\beta)}{\text{pm}}$	$\frac{\lambda_K}{\text{pm}}$
Zr	40	78,74	70,05	68,88
Nb	41	74,77	66,43	65,31
Mo	42	71,08	63,09	61,99

Setup of the Bragg configuration:

- To set up the Bragg geometry, please proceed according to Experiment VII.2 (page 8) und Fig. 4.2 on the following page.

Additional steps:

- 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. 5 cm.
- If necessary, adjust the mechanical zero position of the goniometer (see page 10).

Note:

If the counting rate is too low, you can reduce the distance s_2 between the target and the sensor. 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.

⁵ C. M. Lederer and V. S. Shirley, Table of Isotopes, 7th Edition, 1978, John Wiley & Sons, Inc., New York, USA.

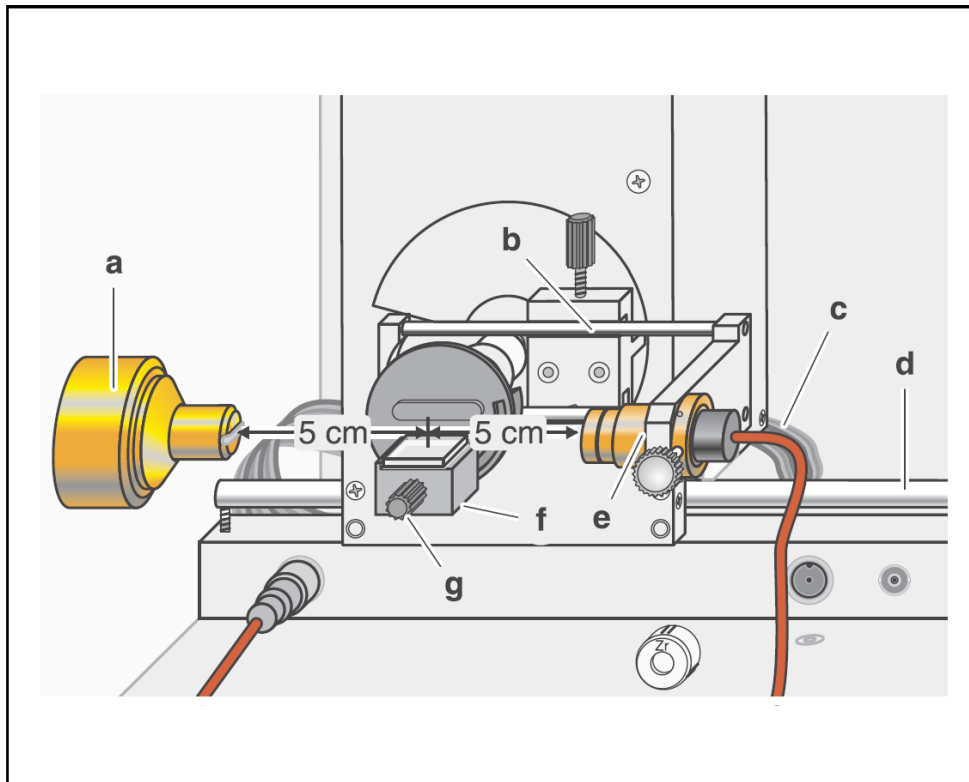


Fig.4.2 Experimental setup

Experiment and Measurement

a. Without Zr-Filter

- Start the software “X-ray Apparatus”.
- Press the key “Coupled” to activate 2θ - coupling of target and sensor and set the lower limit of the target angle to 4.2° and the upper limit to 8.3° .
- Please take time to think about reasonable choices for
 - tube high voltage U ,
 - emission current I ,
 - measuring time per step Δt and
 - step width $\Delta\beta$.

b. With Zr-Filter

- When the scan is finished, mount the zirconium foil on the sensor seat (**e**) of the goniometer and start a new measurement by pressing the key “Scan”.
- When you have finished measuring, save the measurement series under an appropriate name by pressing the key F2.
- To display the measured data as a function of the wavelength λ , open the “Settings” dialog with F5, and click on the button “Enter NaCl” in the tab “Crystal”.

VI.5. Investigating the Attenuation of X-rays as a Function of the Absorber Material and its Thickness

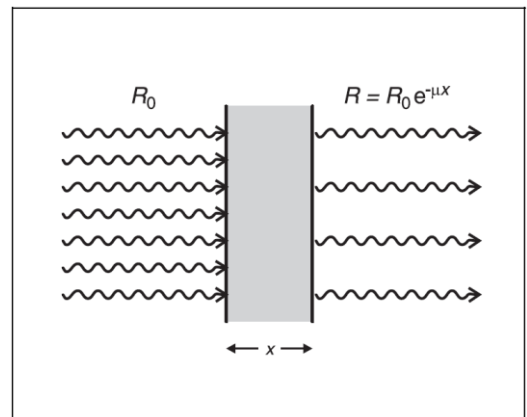
- a. Measurement of x-ray absorption as a function of the material thickness:
 - i. Without Zr-filter
 - ii. With Zr-filter
- b. Measurement of x-ray absorption as a function of the absorbing material itself:
 - i. Without Zr-filter
 - ii. With Zr-filter
 - iii. Measurement of the background

When we speak of attenuation of x-rays, we mean the decrease in intensity that occurs when the radiation passes through matter. Mainly two effects cause this attenuation: scattering and absorption.

The scattering of x-ray quanta by atoms in the attenuator material causes a part of the radiation to change direction. This reduces the intensity of the radiation in the original direction. This scattering can be either elastic or inelastic which would be characterized by an energy loss or change in wavelength.

In the absorption process, the entire energy of the x-ray quanta is transferred to the atoms or molecules of the irradiated material in form of 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 which characterizes the permeability of an attenuator by using:

$$(I) \quad T = R / R_0$$



Lambert's law of attenuation expresses the transmission as a function of wave length and material properties. Please read more on the absorption of x-rays in the listed literature under paragraph II.

Setup

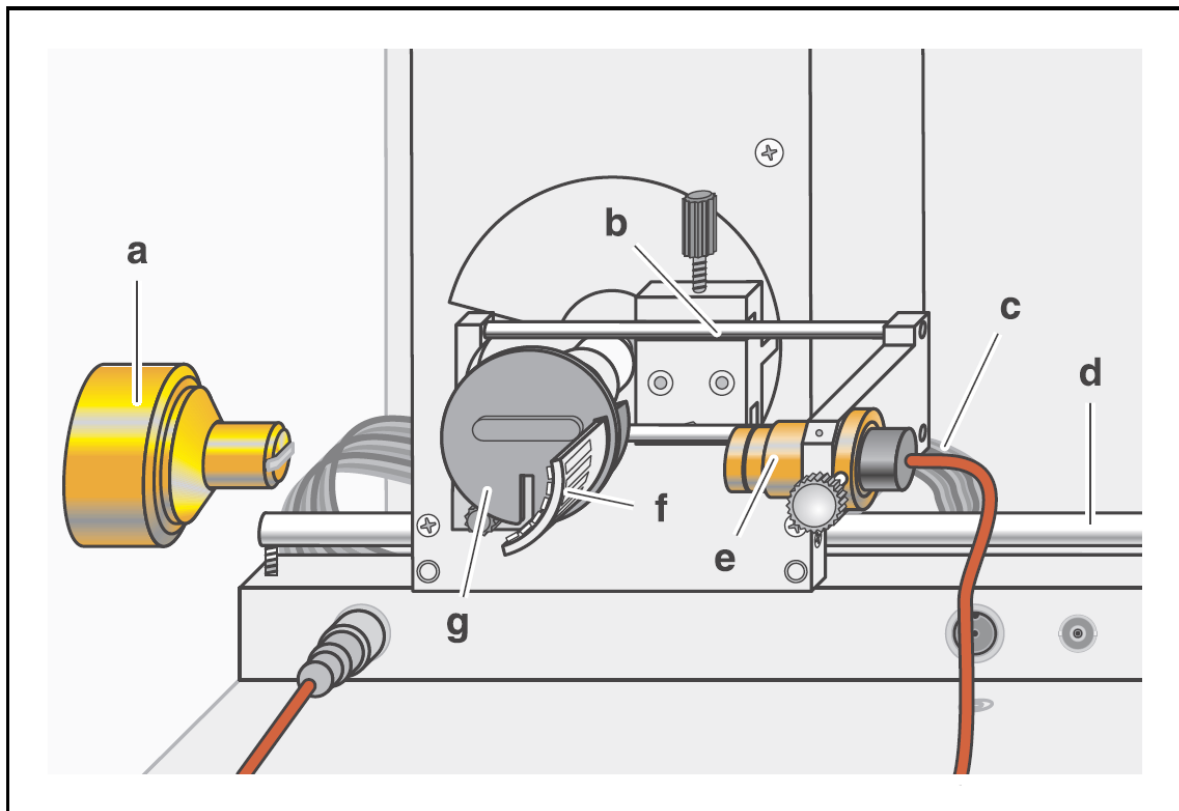


Fig. 5.1 Setup for investigating the attenuation of x-rays as a function of the thickness of the absorber material.

- Dismount the target table from target holder **(g)** of the goniometer.
- Place the set of absorbers **(f)** in the groove (shaped like a quarter of a circle) of the target holder and carefully slide it into the target holder as far as possible.
- Press the key “Zero” to return the target and sensor arms to the absolute zero position of the target holder and detector.
- Set a distance of approx. 5 cm between the exit slit of the primary beam and the empty diaphragm by moving the whole goniometer block. Also set a distance of approx. 5 cm between the empty diaphragm and the sensor slit by moving the sensor holder **(b)**.

Experiment und Measurement

a. Attenuation as a function of the thickness of the absorber:

i. without Zirconium filter:

- The counting rate should not significantly exceed 1500 counts/s. This avoids corrections for dead time. Adjust the tube high voltage U and the emission current I accordingly.
- Optimize the zero position of the empty diaphragm on the set of absorbers by manually scanning the target angle for a maximum countrate. Set adjusted angle position to 0° by pressing TARGET, COUPLED and β LIMITS simultaneously.
- $d_{Al} = 0, 0.5, 1.0, 1.5, 2.0, 2.5, 3.0$ [mm]
- Press the TARGET key.
- Set the angular step width $\Delta\beta = 10^\circ$.
- Set the measuring time $\Delta t = 100$ s.
- Set β limits between 0° and 60° .
- Start the measurement with the key "Scan".

ii. 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).
- Adjust the emission current I accordingly to about 1500 counts/s for the empty absorber slot.
- Repeat the measurement from experiment a.i.

b. Attenuation as a function of the absorber material:

i. without Zirconium filter:

- Replace absorber set I (absorbers of different thicknesses) with the absorber set II (absorbers of different materials, all with $d = 0.05$ cm).
- Evaluate an appropriate choice for the high voltage U setting to ensure that the radiation also penetrates the heavy absorbers.
- Derive suitable settings for the emission current I and for the measuring Δt time from the sequence of the different absorbing materials.
- Set the angular step width $\Delta\beta$ and β limits to 0° .
- Using the knob ADJUST, set the angular positions of the absorbers one after another.
- Start the measurement with the key SCAN and display the mean counting rate N by pressing REPLAY after the measuring time has elapsed.

Material	Z
empty	
C	6
Al	13
Fe	26
Cu	29
Zr	40
Ag	47

ii. with Zirconium filter:

- Attach the zirconium filter and repeat the measurement analogue to b.i.

iii. Background measurement:

- Set the parameters $U = 30$ kV and $I = 0$ mA and measure the counting rate R_1 of the background effect for a measuring time of $\Delta t = 300$ s.

VI.6. Debye-Scherrer-Scans: Determination of Lattice-Plane Spacings and Lattice Constants for Polycrystalline (Powder) Samples

- c. NaCl
- d. KCl

Basics

Debye-Scherrer-Method:

The Debye-Scherrer method uses a crystalline powder and illuminates it with monochromatic x-rays. The diffracted signal is detected by a Geiger-Müller-Tube. The powder sample contains small single-crystals of about 5 - 50 μm in diameter, so-called crystallites. A set of parallel lattice planes (hkl) in a crystallite leads to constructive interference of reflected x-rays if its alignment fulfills the Bragg condition

$$(I) \quad n \cdot \lambda = 2 \cdot d_{hkl} \cdot \sin \theta \quad (\text{see list of literature in paragraph II})$$

n : diffraction order, λ : wave length, d_{hkl} : spacing of the lattice plane (hkl), θ : Bragg angle. The angle between the diffracted beam and the incident x-ray is 2θ (see Fig. 6.2).

In general, the crystallites are randomly oriented without any privileged direction. If Bragg's law is satisfied for a certain lattice plane (hkl) of these crystallites their diffracted beam describes a cone with an aperture angle of 4θ around the primary beam (see Fig. 6.1). The radius R of the cone in the distance L from the sample is given by:

$$(II) \quad R = L \cdot \tan 2\theta, \quad L: \text{Distance between sample and film.}$$

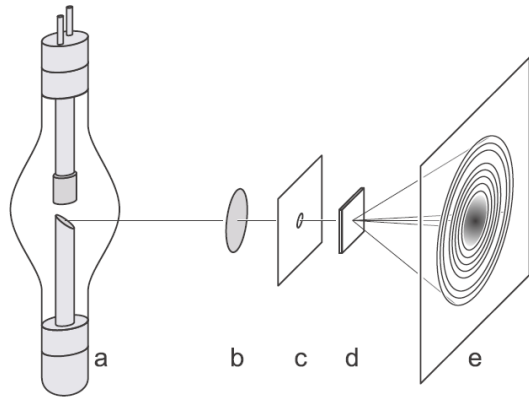


Fig. 6.1 Scheme of the setup for the Debye-Scherrer method.

a: x-ray tube,
b: Zr-filter,
c: collimator,
d: sample,
e: film.

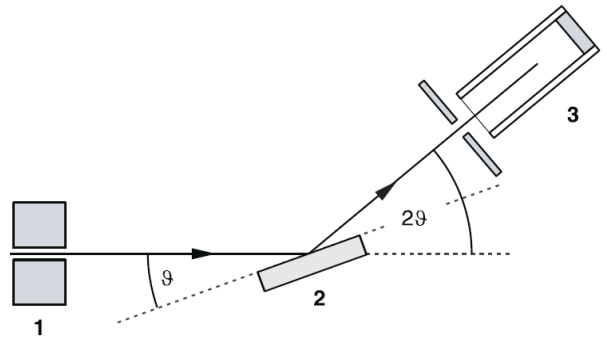


Fig. 6.2 Schematic diagram of x-ray diffraction from a crystalite in a powder sample:

1: collimator,
2: powder sample,
3: counter tube.

The angle between the diffraction peak and the primary ray is 2θ . The detector makes a line scan through the individual rings and records their intensity.

The finer the power, the more uniformly the individual reflections of the crystallites will be distributed around the circle of diffraction. The complete diffraction pattern is a family of concentric circles (see Fig. 6.1 e). Each radius R belongs to a certain interplanar spacing d according to (I) and (II) with a certain order of diffraction n .

Cubic crystals:

If your sample belongs to a cubic crystal system the lattice plane spacing can be expressed in the form:

$$(III) \quad d = a_0 / (h^2 + k^2 + l^2)^{1/2} \quad , \quad a_0: \text{lattice constant}$$

The integers h, k, l are the Miller indices of the set of lattice planes under consideration. If equation (III) is inserted into equation (I), the quadratic form

$$(IV) \quad \sin^2\theta = (\lambda / 2a_0)^2 \cdot [(n \cdot h)^2 + (n \cdot k)^2 + (n \cdot l)^2]$$

is obtained. Formula (IV) can be written in a short form:

$$(V) \quad \sin^2\theta = F \cdot Z \quad \text{with}$$

$$(VI) \quad F = (\lambda / 2a_0)^2 \quad \text{and}$$

$$(VII) \quad Z = (n \cdot h)^2 + (n \cdot k)^2 + (n \cdot l)^2$$

Due to its integer components, Z stays an integer in (VII). For simple cubic crystals, every combination of integers n, h, k and l is generally allowed, however, the intensity of the

diffraction reflections is weaker for higher diffraction orders n and for greater Miller indices h, k, l .

Crystals with NaCl-structure:

In the case of crystals with NaCl structure, the diffraction conditions for h, k , and l become more specific because alkali (here: Na, K) and halogenide (here Cl) atoms take each the positions of a cubic face centered lattice. We obtain the NaCl-structure when we superimpose the two lattices of Na and Cl and translate one of them by a vector $\mathbf{t} = (\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$ with reference to the lattice unit vectors \mathbf{a}_0 (see Fig. 6.3).

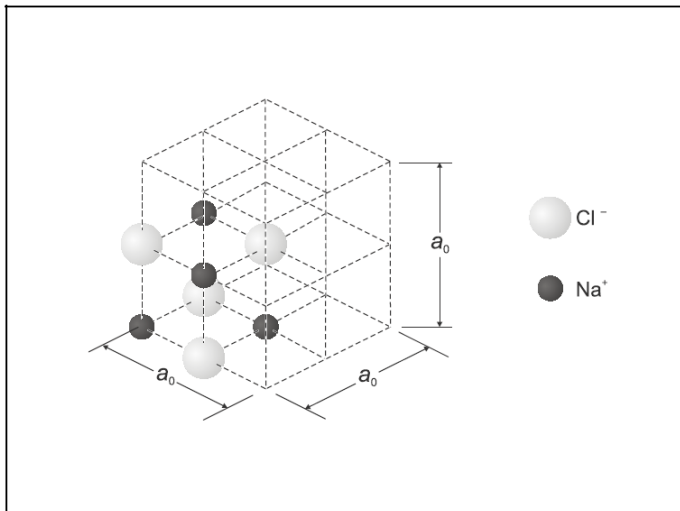


Fig. 6.3: Unit cell of NaCl

Every unit cell contains four alkali atoms with the relative coordinates in units of a_0 . Please write the components of their position vectors in the brackets below:

$\mathbf{r}_1 = (\quad , \quad , \quad)$, $\mathbf{r}_2 = (\quad , \quad , \quad)$, $\mathbf{r}_3 = (\quad , \quad , \quad)$, $\mathbf{r}_4 = (\quad , \quad , \quad)$,

The four halogenide-atoms have then the relative coordinates:

$\mathbf{r}_5 = (\quad , \quad , \quad)$, $\mathbf{r}_6 = (\quad , \quad , \quad)$, $\mathbf{r}_7 = (\quad , \quad , \quad)$, $\mathbf{r}_8 = (\quad , \quad , \quad)$.

An incoming x-ray is scattered by each atom of the unit cell, whereby the amplitudes of the scattered partial waves depend on the atomic number of the atom. The differences in the path Δ_i for each wave diffracted from atoms at sites \mathbf{r}_i can be calculated from the coordinates \mathbf{r}_i of the atoms:

(VIII) $\Delta_i = (\mathbf{s}_1 - \mathbf{s}_2) \cdot \mathbf{r}_i$
 \mathbf{s}_1 : unit vector in the direction of the primary ray
 \mathbf{s}_2 : unit vector in the direction of the diffracted x-ray

The partial waves scattered from all alkali atoms A and from all halogenide atoms H interfere with each other to form a resulting wave that represents the total scattering from the unit cell. The amplitude of this wave has the form

$$(IX) \quad A = A_A + A_H \quad \text{with}$$

$$A_A = f_A \cdot [\cos(2\pi \cdot \Delta_1 / \lambda) + \cos(2\pi \cdot \Delta_2 / \lambda) + \cos(2\pi \cdot \Delta_3 / \lambda) + \cos(2\pi \cdot \Delta_4 / \lambda)]$$

$$A_H = f_H \cdot [\cos(2\pi \cdot \Delta_5 / \lambda) + \cos(2\pi \cdot \Delta_6 / \lambda) + \cos(2\pi \cdot \Delta_7 / \lambda) + \cos(2\pi \cdot \Delta_8 / \lambda)]$$

Moreover, the scattered waves from all unit cells interfere constructively with each other if the *Bragg* condition (I) is fulfilled. This is equivalent to the so-called *Laue* condition, which can be written for crystals with cubic lattices in the form

$$(X) \quad \mathbf{s}_1 - \mathbf{s}_2 = \lambda \cdot \mathbf{G} \quad \text{with} \quad \mathbf{G} = (h, k, l) \cdot 1/a_0$$

By inserting Equations (X) and (VIII) into (IX) one obtains

$$A_A = f_A \cdot \{ 1 + \cos[\pi \cdot (h+k)] + \cos[\pi \cdot (h+l)] + \cos[\pi \cdot (k+l)] \} \quad \text{and}$$

$$A_H = f_H \cdot \{ \cos(\pi \cdot h) + \cos(\pi \cdot k) + \cos(\pi \cdot l) + \cos[\pi \cdot (h + k + l)] \}.$$

Exercise:

Derive from the equations above: What combination of h , k and l leads to a scattering amplitude $A = 0$ and $A \neq 0$? From this result follows the relationship (XI) that you should insert below. It represents the reflection conditions of the NaCl-structure:

(XI)

Other crystal structures with atomic arrangements different from the NaCl-type could lead of course to other reflection conditions.

In this experiment, Debye-Scherrer scans of crystals with NaCl structure are taken. The Bragg angles for different h , k , l indices are recorded and analyzed.

There are two different ways to record the spectra with the x-ray apparatus:

- keeping the powder sample at a fixed angle and let only the sensor rotate
- moving powder sample and sensor in a coupled motion, similar to the ordinary single crystal scans

Rotating a powder sample should not make any difference at all, but for geometrical reasons of the non-circular sample the second way is preferred.

Setup and carrying out the experiment

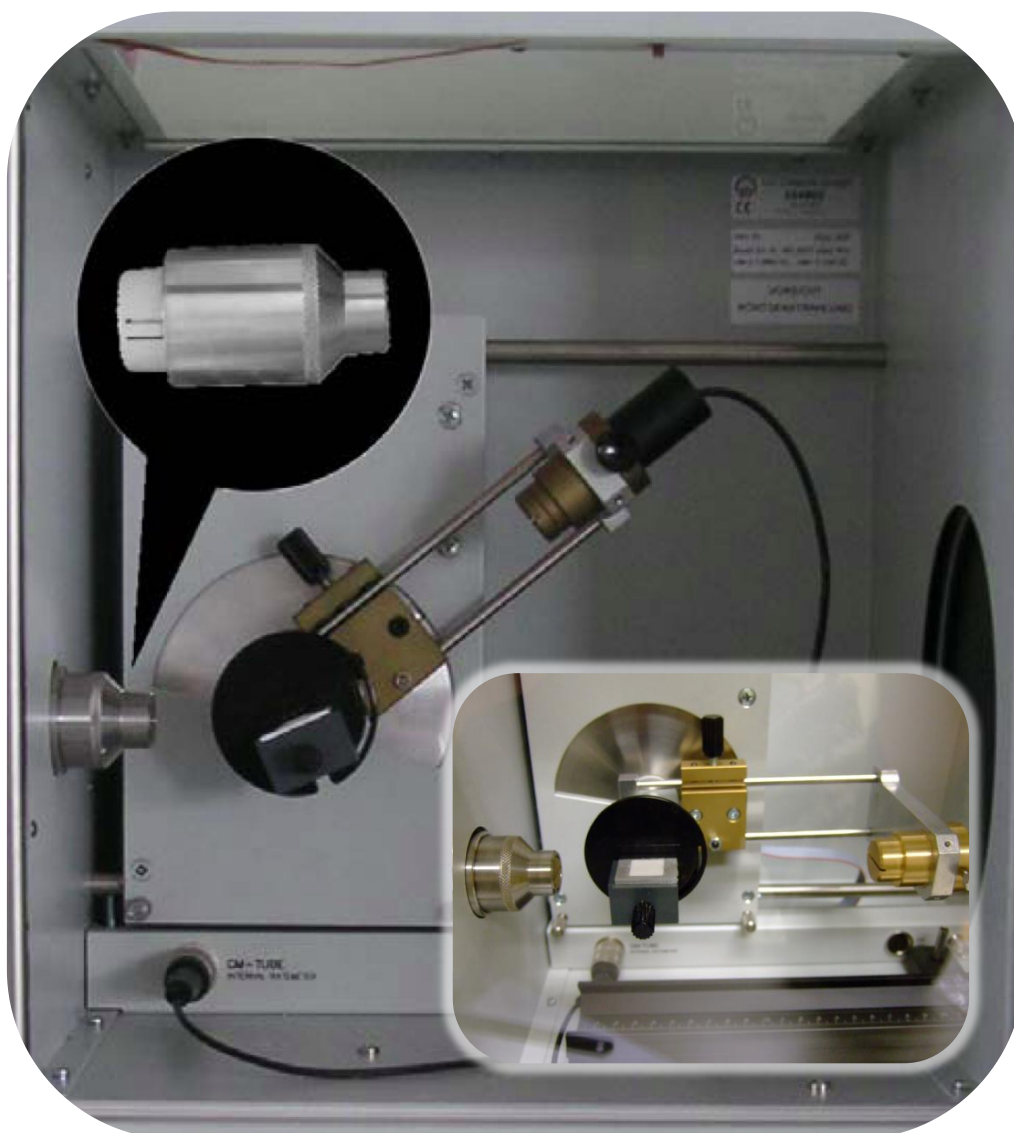


Fig. 6.4 Experimental setup for taking a scan through *Debye-Scherrer* rings of polycrystalline powder samples

- To reduce stray radiation detected by the counter tube, put the Goniometer block to the very left position and use the full length of the sensors arm. See Fig 6.4.
- Mount the Zr-filter on the collimator to produce monochromatic Mo- K_{α} x-rays. The filter must be mounted on the left side off the collimator, facing the tube (see Fig. 6.4 top left corner). This geometry reduces stray radiation into the experiment chamber.
- Prepare a powder sample by using the mortar and the pestle as described below. The NaCl (KCl) salt is grind in a mortar until no grains can be felt when rubbing the powder between the finger tips. Do not try to prepare too much powder at once, as it will take a lot of time. An amount of 2 times the volume of the powder holder will take approximately 5 minutes to pulverise.
- The powder holder consists of an aluminum frame. Attach strips of scotch-tape to the rear of the powder holder and fill the prepared powder into the frame (Fig. 6.5).

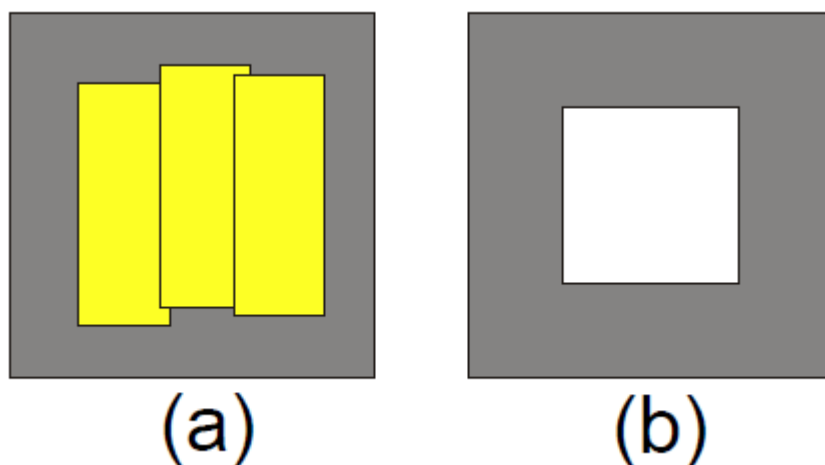


Fig. 6.5: Preparation of the powder holder frame schematically: (a) rear side with strips of scotch-tape (b) front side with squeezed powder

- Squeeze the powder into the frame with the spatula as dense as possible. The powder should form an even surface with the edges of the Al-frame.
- Put the frame onto the target holder inside the X-ray apparatus and slide it sideways onto the target table.

Experiment

a. Debye-Scherrer-Scan of NaCl:

- Set the tube high voltage $U = 35 \text{ kV}$, emission current $I = 1.0 \text{ mA}$, $\Delta\beta = 0.1^\circ$ and $\Delta t = 15 \text{ s}$.
- Select “Coupled” scan mode.
- Set β limits from 2° to 25° .
- Switch on the high voltage and press the “Scan” button for recording the spectra.
- To check for the existence of the weak (111) reflection, scan that region left of the 200 reflex (4.5° 8.5° for NaCl) with $\Delta t = 30 \text{ s}$ at least.

b. Debye-Scherrer-Scan of KCl:

- Exchange NaCl-powder for the carefully ground KCl-powder and repeat the measurement.
- Set β limits from 2° to 20° .
- To check for the existence of the weak (111) reflection, scan left of the 200 reflex (3.5° 7.5° for KCl) with $\Delta t = 30 \text{ s}$ at least.

VI.7. Laue-Photography: Symmetry und Structure of Crystalline Matter

- c. NaCl
- d. LiF

Background and basics

In 1912, *Max von Laue* suggested to prove the wave character of x-rays by diffracting them from a crystal's lattice. *Friedrich* and *Knipping* took up his proposal and exposed a crystal to a collimated ray from an x-ray tube. On a photographic plate behind the crystal they observed a diffraction pattern with discrete reflections (see Fig. 7.1). With this experimental result they also confirmed that crystalline substances have an ordered geometric structure.

Laue-Condition:

In his interpretation of these findings, *Max von Laue* considered the crystal to be a lattice built up of three groups of one-dimensional equidistant rows of points. When an x-ray is diffracted at a row of points (see Fig. 7.2),

$$(I) \quad \Delta = \Delta_1 - \Delta_2 = a_0 \cdot \cos \alpha_1 - a_0 \cdot \cos \alpha_2 \quad , \quad (\text{see also list of literature under II.})$$

a_0 : distance between points

α_1 : angle between *the incoming* x-ray and the row of points

α_2 : angle between *the diffracted* x-ray and the row of points

is the difference of path of x-rays scattered at two neighboring points (lattice elements, see Fig. 7.2). There is constructive interference between the diffracted x-rays if Δ is an integer multiple of the wave length λ . This condition has to be fulfilled in all three spatial directions.

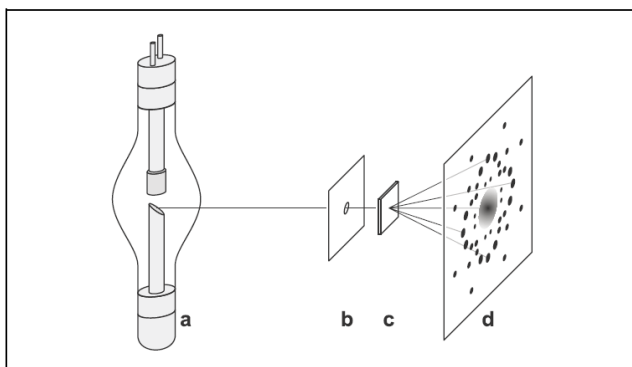


Fig. 7.1 Scheme of the setup for taking a *Laue* diagram of a single crystal: **a** x-ray tube, **b** Collimator, **c** Crystal, **d** x-ray film

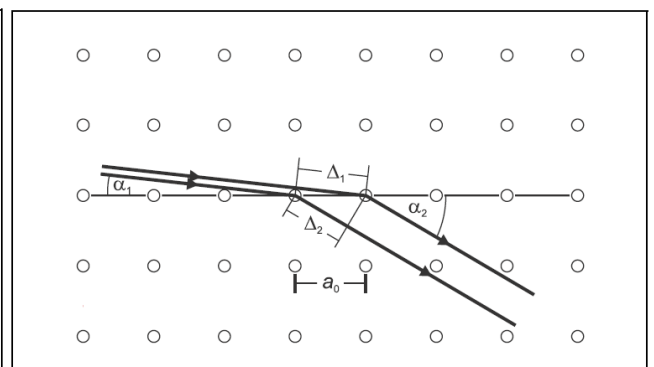


Fig. 7.2 Two-dimensional representation for calculating the difference of path $\Delta = \Delta_1 - \Delta_2$ between two neighbouring x-rays which are diffracted from a row of points in a cubic crystal.

In a cubic crystal, the rows of points associated with the three spatial directions are perpendicular to each other. The distance a_0 is the separation of neighboring points in a row. The *Laue* condition for constructive interference therefore reads:

$$(II) \quad \begin{aligned} a_0 \cdot \cos \alpha_1 - a_0 \cdot \cos \alpha_2 &= h \cdot \lambda \\ a_0 \cdot \cos \beta_1 - a_0 \cdot \cos \beta_2 &= k \cdot \lambda \\ a_0 \cdot \cos \gamma_1 - a_0 \cdot \cos \gamma_2 &= l \cdot \lambda \end{aligned} \quad , \quad \text{with integer values for } h, k, l$$

α_1, β_1 and γ_1 are the angles between the incoming ray and the rows of points whereas α_2, β_2 and γ_2 are the angles between the diffracted ray and the rows of points. The quantities h, k, l are integers and called *Laue* indices.

The unit vectors

$$(III) \quad \mathbf{s}_1 = (\cos \alpha_1, \cos \beta_1, \cos \gamma_1) \quad \text{und} \quad \mathbf{s}_2 = (\cos \alpha_2, \cos \beta_2, \cos \gamma_2)$$

point in the direction of the incoming x-ray and the diffracted x-ray, respectively. The *Laue* condition (II) then takes the form

$$(IV) \quad \mathbf{s}_1 - \mathbf{s}_2 = \lambda \cdot \mathbf{G} \quad \text{with } \mathbf{G} = (h, k, l) \cdot 1/a_0$$

\mathbf{G} is called the vector of the reciprocal lattice. The *Laue* condition is fulfilled if h, k and l are integers.

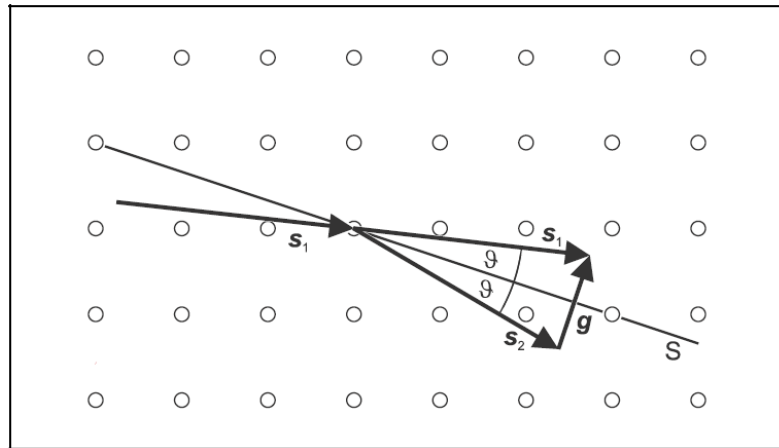


Fig. 7.3 Geometric relationship between the unit vectors \mathbf{s}_1 und \mathbf{s}_2 and the reciprocal lattice vector $\mathbf{g} = \lambda \mathbf{G}$.

Bragg-condition:

\mathbf{s}_1 and \mathbf{s}_2 are unit vectors with equal magnitudes. Therefore, the vector \mathbf{G} is perpendicular to the bisectrix S between the incoming and the diffracted x-ray (see Fig. 7.3). From this follows

$$\lambda \cdot |\mathbf{G}| = |\mathbf{s}_1 - \mathbf{s}_2| = 2 \cdot \sin \theta$$

and after inserting the magnitude of G

$$(V) \quad \lambda = 2 \cdot \sin \theta \cdot a_0 / (h^2 + k^2 + l^2)^{1/2}$$

Eq. (V) is identical with *Bragg's law* if the spacing of the lattice planes is

$$(VI) \quad d = a_0 / (h^2 + k^2 + l^2)^{1/2}$$

From *Bragg's* point of view the crystal lattice thus consists of a set of lattice planes that have the spacing d (see Fig. 7.4). These lattice planes are parallel to the bisectrix S and perpendicular to the vector \mathbf{G} of the reciprocal lattice.

The conditional equation for a set of lattice planes in the coordinate system that is spanned by the crystal axes is

$$(VII) \quad x \cdot h + y \cdot k + z \cdot l = m \cdot a_0, \quad m: \text{counter in a set of lattice planes}$$

h^{-1} , k^{-1} und l^{-1} are the axis sections of the first lattice plane ($m = 1$) measured in units of the lattice constant (see Fig. 7.5). Each set of lattice planes is unique and is assigned by the triplet $(h \ k \ l)$.

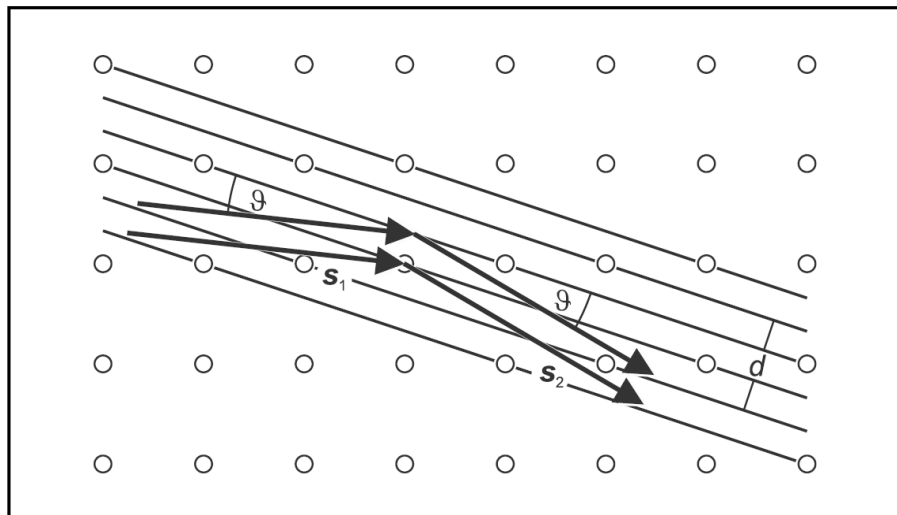


Fig. 7.4 Two-dimensional representation of the *Bragg* reflection of x-rays in a cubic crystal. The lattice planes are parallel to the bisectrix S between the incoming and the diffracted x-ray.

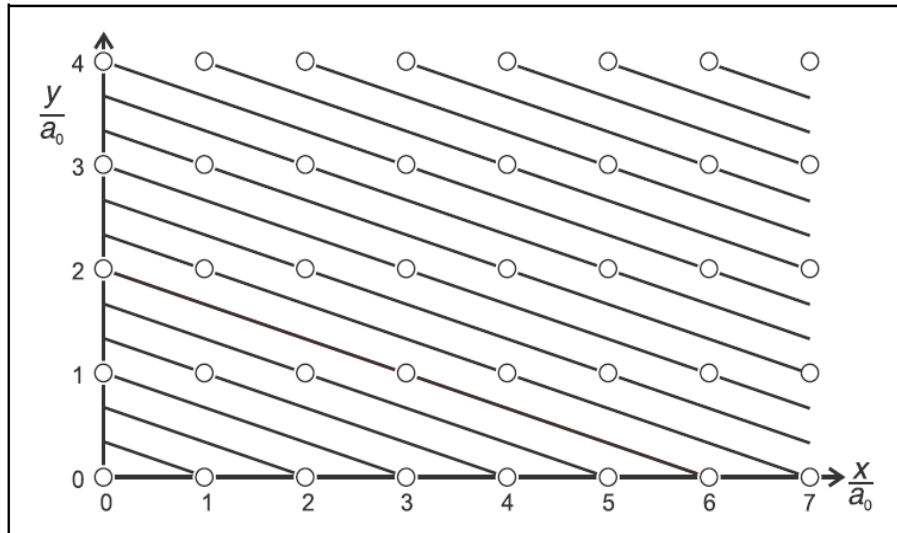


Fig. 7.5 Two-dimensional representation of the set of lattice planes (1 3 0) in a cubic crystal..

The calculation of the structure factor for NaCl or LiF is analogue to experiment VI.6.

Setup and experiment

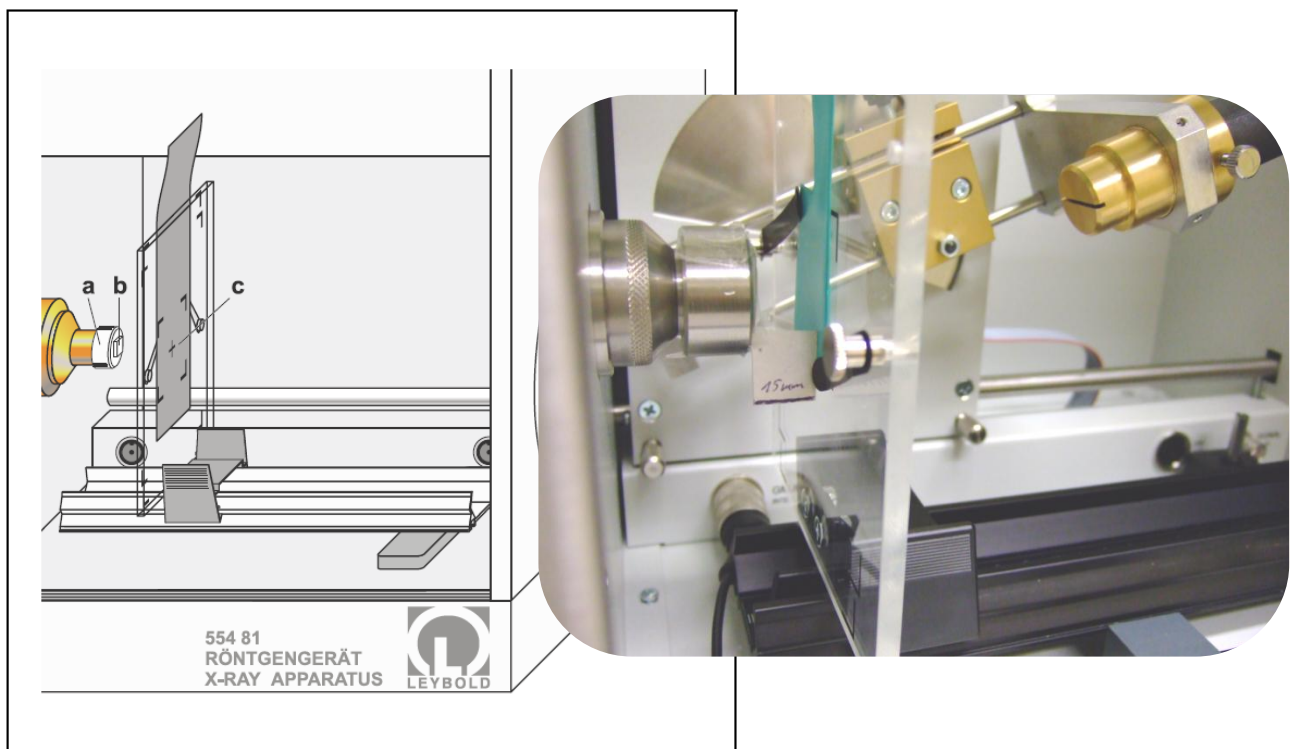


Fig. 7.7 Experimental setup for taking a *Laue* diagram from crystals

a. Laue-photograph from NaCl:

- Detach target holder completely from the goniometer to make room for the film holder in the experimental chamber.
- Carefully attach the NaCl crystal for Laue diagrams (b) to the pinhole diaphragm (a) with transparent adhesive tape.
- Attach the pinhole diaphragm to the collimator at the front end and align the crystal edges as horizontally and vertically as possible.
- Clamp the x-ray film (c) on the film holder so that its markings are centered. Take care that the entire surface of the film is planar.
- Clamp the film holder onto the rail, and mount the rail in the experimental chamber of the x-ray apparatus.
- Make a 15 mm long spacer from paper board and shift the film holder so that the distance between the single crystal and the film is 15 mm (by varying the distance between the crystal and the film the magnification of the picture is changed).
- Set the tube high voltage to $U = 35 \text{ kV}$, the emission current to $I = 1.0 \text{ mA}$ and $\Delta\beta = 0.0^\circ$.
- Select the measuring time $\Delta t = 1800 \text{ s}$, and start the exposure timer with the key "Scan".
- If the exposure time is longer, the reflections near the centre are blurred by the unscattered x-rays and background radiation, however, structures which are further away from the centre become more distinguishable.
- When the exposure time is over, take the film holder with the experiment rail out of the experiment chamber.
- Remove the x-ray film from the holder, and develop it according to the instruction sheet for the x-ray film:

<http://www.itap.physik.uni-stuttgart.de/lehre/vorlesungen/praktikum/Debye-Scherrer-Film-554895e.pdf>

b. Laue-photograph from LiF:

- Exchange the NaCl crystal with the LiF crystal, and align it.
- Clamp a new x-ray film in the film holder, and mount the experiment rail with the film holder once more.
- Distance between sample and film should equal to 11mm.
- Select the measuring time $\Delta t = 1200 \text{ s}$, and start the exposure timer with the key Scan.
- When the exposure time is over, take the film holder with the experiment rail out of the experiment chamber.
- Remove the x-ray film from the holder, and develop it according to the instruction sheet for the x-ray film:

<http://www.itap.physik.uni-stuttgart.de/lehre/vorlesungen/praktikum/Debye-Scherrer-Film-554895e.pdf>

VII. Evaluation and Interpretation of the Experimental Results

VII.1. Detection of X-ray Radiation and Measurement of its Intensity with a Geiger-Müller-Tube.

- a. Determination of the counting rate N as a function of the emission current I .
- b. Determination of the counting rate N as a function of the acceleration voltage U of the tube.
- c. Estimation of the Geiger tube's dead time.

Evaluation

- Put the experimental results from experiments a to c in a graphical representation.
- Explain the behavior of the counting rates from experiments a to c. Especially, discuss (= give arguments that support our assumptions) the non linear behavior of the counting rate from experiment c.
- The Geiger tube is operated at a voltage of 500 V. At this voltage, a photon can initiate an ionization avalanche by ionizing just a single atom. Decreasing the voltage prevents the avalanche effect and leads to a different behavior of the counting tube. The number of ions in the gas will then be proportional to the number and energy of the incoming x-ray photons. In this mode, the detector is called a proportional counter.

Review all operating modes of the ionization chamber setup (see literatur).

Exercise:

To what extend could a proportional counter be used to make accurate measurements of the intensity of x-ray radiation as a function of its energy? Estimate the detection threshold for x-rays with the energy 1 keV, 8 keV and 10 keV.

Hint: Ionization chamber filled with argon:
 Ionization energy for Ar-atoms amounts to 25 eV
 Detection limit for current measurements lies around 10^{-15} A.

VII.2. Bragg-Reflection: Determination of Lattice Constants in Single-Crystals

- a. Measurement of the diffraction spectrum of a LiF single-crystal.
- b. Measurement of the diffraction spectrum of a NaCl single-crystal.

Evaluation for a and b

- Use the software “X-ray Apparatus” to evaluate the Bragg angle θ from the center of mass of the their spectra.
- Calculate for each Bragg angle θ the values for $\sin \theta$ and $n \cdot \lambda$.
- Calculate the lattice constant a_0 of LiF and NaCl.
Literature value LiF: $a_0 = 402.7 \text{ pm}$; NaCl: $a_0 = 564.02 \text{ pm}$
- Discuss the various error sources (alignment, counting statistics, error propagation) of your measurement and calculate the error for all measurements and your result.
- Explain the differences in magnitude of the lattice constants of LiF and NaCl.

VII.3. Verification of the Duane-Hunt Relation and the Determination of the Planck's Constant

Determination of the critical wavelength λ_{\min} with reference to the tube high voltage U .

Evaluation

Determination of the critical wavelength λ_{\min} as a function of the tube high voltage U :

For each recorded diffraction spectrum:

- Evaluate the critical wave length λ_{\min} by fitting a line of regression to the measured spectra.
- Discuss the various error sources (alignment, counting statistics, fit error) of your measurement and calculate the error of your result.

Conformation of the Duane-Hunt relation and determination of Planck's constant:

- Evaluate Planck's constant from the data for $\lambda_{\min} = f(1/U)$.
Literature value: $h = 6.626 \times 10^{-34} \text{ J s}$

VII.4. Edge Absorption: Filters for X-rays

Measurement of the diffraction spectrum of a NaCl-crystal with Mo-radiation with wavelengths between 40 and 80 pm ($U=30$ kV).

- a. Without Zr-filter
- b. With Zr-filter

Evaluation

- Access the evaluation functions of the software “X-ray Apparatus” to evaluate the integral counting rate R_i of the characteristic peaks from the measured spectra.
- Calculate the ratio V of the K_β -radiation with reference to the total characteristic radiation.

$$V = R_i(K_\beta) / (R_i(K_\alpha) + R_i(K_\beta))$$

- Verify the wave length of the absorption edge for the Zr filter.
- Discuss the various error sources (alignment, counting statistics, fit error) of your measurement and calculate the error of your result.
- Summarize the most important results of this experiment and their meaning.

VII.5. Investigating the Attenuation of X-rays as a Function of the Absorber Material and its Thickness

- a. Measurement of x-ray absorption as a function of the material thickness:
 - i. Without Zr-filter
 - ii. With Zr-filter
- b. Measurement of x-ray absorption as a function of the absorbing material itself:
 - i. Without Zr-filter
 - ii. With Zr-filter
 - iii. Measurement of the background

Evaluation

a) Attenuation as a function of the absorber thickness:

The transmission T can be calculated from the measured data. Make a graphical representation of T as a function of the thickness d of the absorber.

The attenuation of the monochromatic (by use of a Zr filter) x-rays can be well described by a line through the origin in a semi logarithmic plot.

- Evaluate the absorption coefficient μ for Mo-radiation in Al.

- Discuss the various error sources (alignment of absorber, counting statistics, , error propagation) of your measurement and calculate the error of your result.
- Compare your result to the literature value (see link on webpage to this experiment under - Interesting or useful links -).

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 the experimental data (after subtracting the background effect) to the emission current $I = 1.00$ mA. The scaled data leads us to the transmission T which can be used to calculate the linear attenuation coefficient μ for $d = 0.05$ cm.

- Plot the attenuation coefficient μ as a function of the atomic number Z .
- Evaluate T and μ for all used materials.
- Discuss the various error sources (alignment of absorber, counting statistics, , error propagation) of your measurement and calculate the error of your result.
- Compare your result to the literature value (see link on webpage to this experiment under - Interesting or useful links -).
- Explain and discuss the behavior of the attenuation coefficient μ as a function of Z , especially for values around $Z = 40$.
- How does the attenuation coefficient behave if a Zr-filter is used? Explain!

VII.6. Debye-Scherrer-Scans: Determination of Lattice-Plane Spacings and Lattice Constants for Polycrystalline (Powder) Samples

- a. NaCl
- b. KCl

Evaluation

- Evaluate all Bragg angles θ and calculate $\sin 2\theta$.
- Deduce integer factor Z from $\sin^2\theta$ (comp. Eq. (V) and (VII)). Assign the Miller indices to the diffraction peaks and calculate from Eq. (V) factor F .
- Calculate the inter lattice spacings d_{hkl} for each peak of diffraction.
- Discuss the various error sources (sample alignment and preparation, counting statistics, error propagation) of your measurement and calculate the error of your result.
- Summarize the most important results of this experiment and their meaning.

Debye-Scherrer-Scans from NaCl and KCl:

Table 1: Decomposition of $\sin 2\theta$ into the factors F and Z for NaCl and KCl

No.	$\theta / ^\circ$	$\sin^2\theta$	n	h	k	l	Z	F
1								
2								
...								

In Table 1, the decomposition of the experimental results for $\sin 2\theta$ into the factors F and Z , the associated *Miller* indices h , k , l and the diffraction order n are listed. The mean value of the factors F leads finally to the lattice constant of NaCl and KCl:

Table 2: Lattice plane spacings d contributing to the *Debye-Scherrer peaks* of NaCl and KCl

Nr.	h	k	l	d / pm
1				
2				
...				

VII. 7. Laue-Photography: Symmetry und Structure of Crystalline Matter

- a. NaCl
- b. LiF

Evaluation of a *Laue*-diagram:

The object of the evaluation of a *Laue*-diagram is to assign the set of lattice planes with its spacing d_{hkl} to one of the reflections observed on the x-ray film. For this purpose, the origin of the coordinate system of the diagram corresponds to the space point of the incoming x-ray on the x-ray film.

The x-ray film is perpendicular to the path of the x-ray, i. e. it lies in the x-y-plane of the diffraction pattern (see Fig. 7.8). The orientation of the z-axis is antiparallel to the propagation direction of the x-ray. The x-ray penetrates the flat crystal at the point K; its undiffracted part impinges on the x-ray film at the point O.

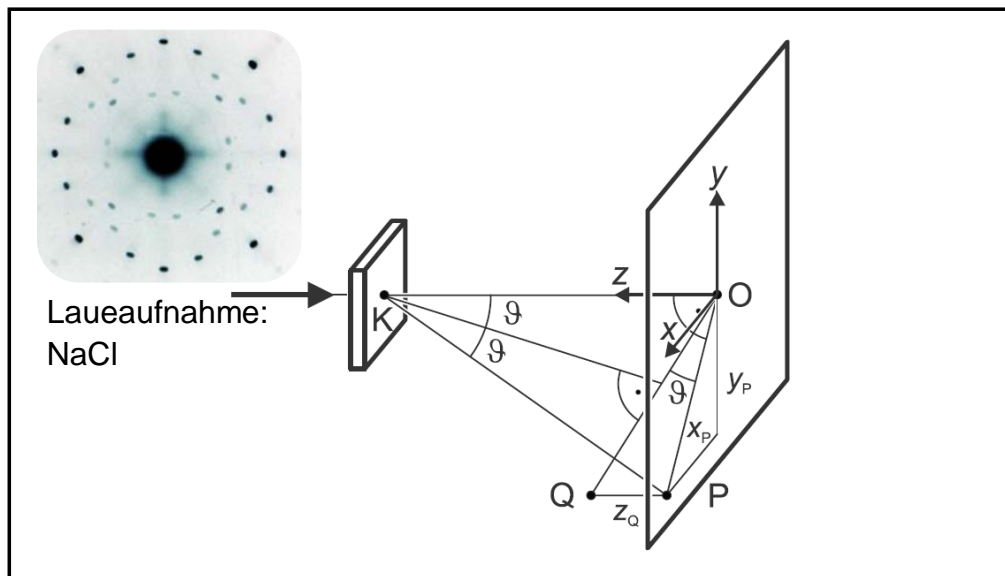


Fig. 7.8 Geometrical description of an x-ray that is diffracted at a point K of the crystal and that penetrates the film plane in the point P.

The part of the scattered x-ray at K and which fulfils the *Laue* condition (II) leaves the crystal with an angle 2θ relative to the direction of the primary ray and impinges on the x-ray film at the point $P = (x_P, y_P, 0)$. Therefore:

$$(XII) \quad \tan 2\theta = (x_P^2 + y_P^2)^{1/2} / L, \quad L: \text{distance between crystal and x-ray film}$$

The orientation of the set of lattice planes $(h \ k \ l)$, which leads to the reflection, is given by the bisectrix of the angle 2θ (see Fig. 7.3). The angle between the perpendicular on the bisectrix which passes through O and the straight line OP is θ . This perpendicular intersects a parallel to the z-axis through P in the point Q. The vector \mathbf{OQ} has the coordinates (x_Q, y_Q, z_Q) with

$$(XIII) \quad \tan \theta = z_Q / (x_Q^2 + y_Q^2)^{1/2}$$

and is parallel to the vector **G** of the reciprocal lattice.

Because of $x_P = x_Q$, $y_P = y_Q$ and Eq. (XI),

$$(XIV) \quad z_Q = (x_Q^2 + y_Q^2 + L^2)^{1/2} - L$$

The crystals used in this experiment are cut parallel to the (1 0 0) plane. Their crystal axes thus coincide with the axis of the chosen laboratory system. From the fact that the vectors **G** and **OQ** are parallel follows:

$$(XV) \quad h : k : l = x_Q : y_Q : z_Q$$

The (unknown but wanted) indices h , k , l are the smallest unmixed triple of integers which fulfill Eq. (XV). The spacing of lattice planes d is obtained from Eq. (VI), the wavelength λ from Eq. (V) and the Bragg angle θ is, according to Eqs. (XIII) and (XV):

$$(XVI) \quad \theta = \arctan [l / (h^2 + k^2)^{1/2}]$$

Evaluation

- Lay a piece of millimeter-squared paper on the x-ray film, read the x_Q - and y_Q - coordinates of the reflections, and calculate the z_Q -coordinate according to Eq. (XIV).
- Calculate the spacing of the lattice planes d according to Eq. (VI), the Bragg angle θ according to Eq. (XVI) and the wavelength λ according to Eq. (V).
- Now apply Eqs. (XII) and (XV) to calculate the coordinates x_P , y_P and x_Q , y_Q , respectively, by comparing them with the measured values.
- Identify the indices h , k and l according to Eq. (XV).
- Discuss the various error sources (alignment of film, sample) of your measurement and their influence on the result.
- Describe the symmetry of the Laue diffraction pattern for NaCl. What conclusions can be drawn from this description about the symmetry of the NaCl structure?
- Summarize the most important results of this experiment and their meaning.

Tables:

Table 1: Coordinates and extended *Miller* indices of the reflections in the *Laue* diagram for NaCl

(* calculated)

[mm]						[mm]	[mm]
x_Q	y_Q	z_Q^*	h	k	l	x^*	y^*

Table 2: Spacing of lattice planes d , *Bragg* angle θ and wavelength λ associated with the sets of lattice planes of NaCl, $a_0 = 564.02$ pm und LiF, $a_0 = 402.80$ pm

			[pm]	[°]	[pm]
h	k	l	d	θ	λ

VIII. Specific Questions

1. How does a ionization chamber work? What modes of operation are used to detect ionizing radiation with it? What mechanisms limit its maximum count rate?
2. Explain the origin of the x-ray spectrum? What information does it carry about the atomic electron shell?
3. How does the x-ray spectrum of a Mo-radiation look like with and without a Zr-filter?
4. Describe the dependency of the attenuation of x-rays with respect to their energy E and the atomic number Z of the absorbing material?
5. Explain the existence of absorption edges in solids as a function of the x-ray energy? How can this be used to create monochromatic x-rays?
6. What is the ratio between scattered and absorbed x-rays in an x-ray diffraction experiment?
7. What role has the phenomenon of symmetry in physics and in particular for x-ray diffraction (3-4 sentences)?
8. How could you use the Laue method to obtain the crystallographic orientation of a single crystal (qualitatively)?