

## Bragg reflection: determining the lattice constants of monocrystals

### Objects of the experiment

- Investigating and comparing Bragg reflection at an LiF and an NaCl monocrystal.
- Determining the lattice constant  $a_0$  of NaCl and LiF.

### Principles

Bragg's law of reflection describes the diffraction of plane waves at a monocrystal as the selective reflection of the waves at a set of lattice planes within the crystal. Due to the periodicity of the crystal, the lattice planes of a set have a fixed spacing  $d$ . An incident wave with the wavelength  $\lambda$  is reflected with maximum intensity when the Bragg condition

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

$n$ : diffraction order

$\lambda$ : wavelength

$d$ : spacing of lattice planes

is fulfilled (see experiment P6.3.3.1). The angle  $\vartheta$  shows the direction of the incident and reflected wave with respect to the

set of lattice planes and is often referred to as the glancing angle.

In a cubic crystal with NaCl structure (cf. Fig. 1), the lattice planes run parallel to the surfaces of the crystal's unit cells in the simplest case. Their spacing  $d$  corresponds to one half the lattice constant:

$$d = \frac{a_0}{2} \quad (II)$$

This lets us use (I) as an equation for determining the lattice constant  $a_0$ :

$$n \cdot \lambda = a_0 \cdot \sin\vartheta \quad (III)$$

In other words, to determine  $a_0$  we need to measure the glancing angle  $\vartheta$  for a known wavelength  $\lambda$  and diffraction order  $n$ . This method is more precise when the glancing angles are also measured in higher diffraction orders.

In this experiment, the molybdenum x-rays are used as radiation of a known wavelength. Table 1 shows its wavelengths  $\lambda$ .

Fig. 1 Three-dimensional representation of the structure of NaCl  
 $d$ : Spacing of lattice planes in [1,0,0]-direction  
 $a_0$ : lattice constant

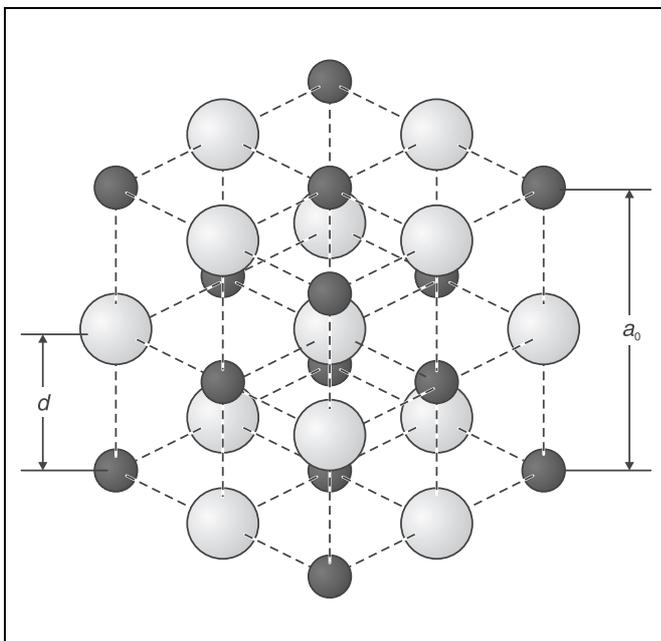


Table 1: Wavelengths of the characteristic x-ray radiation of molybdenum (weighted means [1])

Line	$\frac{\lambda}{\text{pm}}$
$K_{\alpha}$	71.08
$K_{\beta}$	63.09

A Geiger-Müller counter tube is used to detect the x-rays; this instrument and the crystal are both pivoted with respect to the incident x-ray beam in  $2\vartheta$  coupling – the counter tube is turned by twice the angle of the crystal (cf. Fig. 2). The zero point  $\vartheta = 0^\circ$  is characterized by the fact that the lattice planes and the

**Apparatus**

1 X-ray apparatus . . . . .	554 811
1 End-window counter for $\alpha$ , $\beta$ , $\gamma$ and x-ray radiation . . . . .	559 01
1 LiF monocrystal for Bragg reflection . . . . .	554 77
<i>additionally required:</i>	
1 PC with Windows 9x/NT	

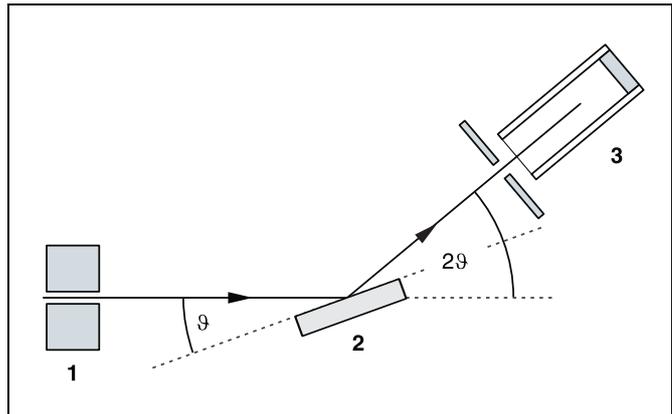


Fig. 2 Schematic diagram of diffraction of x-rays at a monocrystal and  $2\vartheta$  coupling between counter-tube angle and scattering angle (glancing angle)  
1 collimator, 2 monocrystal, 3 counter tube

axis of the counter tube are parallel to the incident x-ray beam. As the lattice planes are seldom precisely parallel to the surface of the crystal, the zero point of each crystal must be calibrated individually.

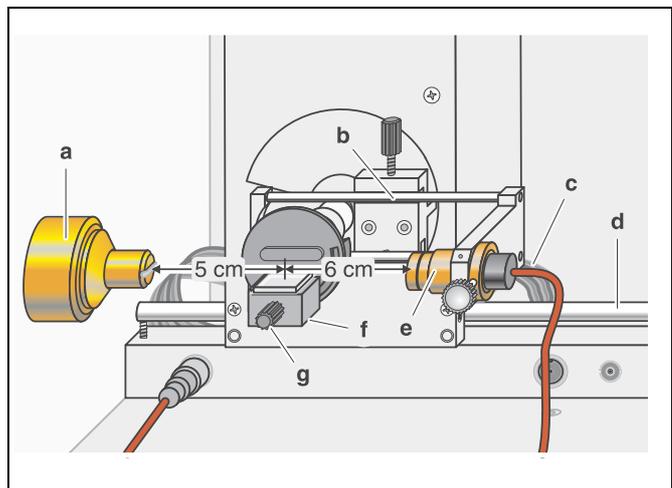
**Setup**

**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.

Fig. 3 Experiment setup in Bragg configuration



**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.

- 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 target stage.
- Manually align the target and sensor arm horizontally using the ADJUST knob and save these positions as the “zero position of the measuring system” by pressing TARGET, COUPLED and  $\beta$  Limits at the same time (see Instruction Sheet for x-ray apparatus).
- In coupled scanning mode, move the target back by  $10.2^\circ$  (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  $\beta$  LIMITS simultaneously.

#### Recording the diffraction spectrum:

#### 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.
- 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 measuring time per angular step  $\Delta t = 10$  s and the angular step width  $\Delta\beta = 0.1^\circ$ .
- Press the COUPLED key to activate 2 $\theta$  coupling of target and sensor and set the lower limit of the target angle to  $4^\circ$  and the upper limit to  $34^\circ$ .
- Start measurement and data transfer to the PC by pressing the SCAN key.
- When you have finished measuring, save the measurement series under an appropriate name by pressing the button  or the F2 key.

## Carrying out the experiment

### Notes:

*NaCl and LiF crystals are hygroscopic and extremely fragile. Store the crystals in a dry place; avoid mechanical stresses on the crystals; handle the crystals 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  $K_\alpha$  and  $K_\beta$  lines.*

#### a) Bragg reflection at an LiF monocrystal:

- 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 a slight pressure).
- Set the tube high voltage  $U = 35.0$  kV and the emission current  $I = 1.00$  mA.

#### Determining the zero position of the measuring system

- In coupled scanning mode, set the target to about  $10.2^\circ$  using the ADJUST knob.
- Switch on the tube high voltage with HV on/off.
- Leave the target position unchanged and, in sensor scanning mode, manually find the counting rate maximum for the first reflection maximum of the  $K_\alpha$  line.
- Leave the sensor unchanged in the maximum counting-rate position and manually find the maximum of the counting rate in target mode.
- Switch between sensor and target modes and check whether you have found the counting rate maximum.

#### a) Bragg reflection at an NaCl monocrystal:

- 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.

#### Determining the zero position of the measuring system

- In coupled scanning mode, set the target to about  $7.2^\circ$  using the ADJUST knob.
- Switch on the tube high voltage with HV on/off.
- Leave the target position unchanged and, in sensor scanning mode, manually find the counting rate maximum for the first reflection maximum of the  $K_\alpha$  line.
- Leave the sensor unchanged in the maximum counting-rate position and manually find the maximum of the counting rate in target mode.
- Switch between sensor and target modes and check whether you have found the counting rate maximum.
- In coupled scanning mode, move the target back by  $7.2^\circ$  (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  $\beta$  LIMITS simultaneously.

#### Recording the diffraction spectrum:

- Start the software “X-ray Apparatus” or clear any existing measurement data using the button or the  F4 key.
- Press the COUPLED key to activate 2 $\theta$  coupling of target and sensor and set the lower limit of the target angle to  $4^\circ$  and the upper limit to  $24^\circ$ .
- Start measurement and data transfer to the PC by pressing the SCAN key.
- When you have finished measuring, save the measurement series under an appropriate name by pressing the button  or the F2 key.

## Measuring example

## a) Bragg reflection at an LiF monocrystal:

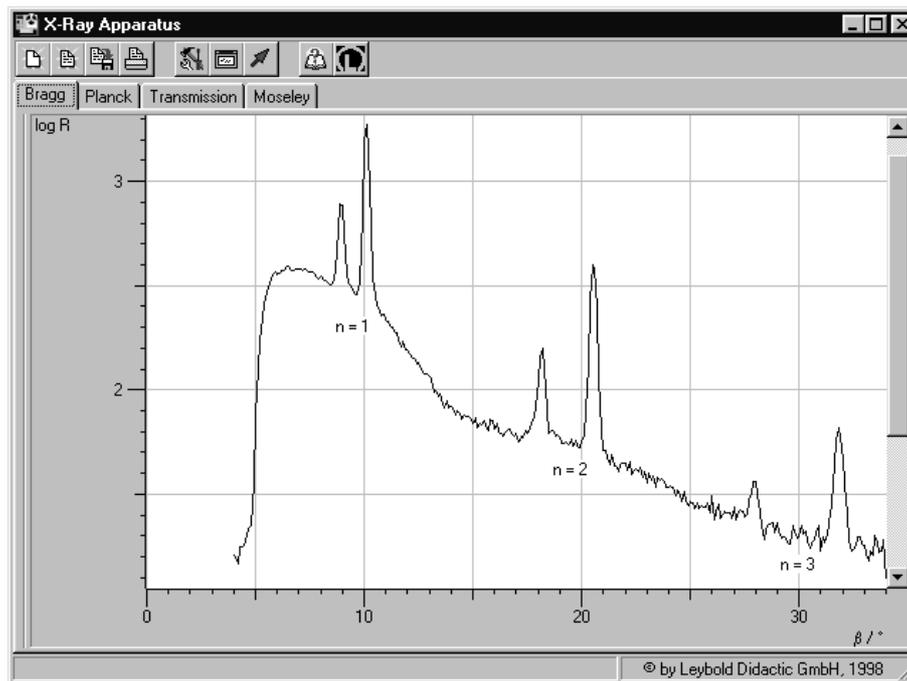


Fig. 4 Diffraction spectrum of x-rays in Bragg reflection to the third diffraction order at an LiF monocrystal with logarithmic display of counting rate  $R$ .  
Parameters of x-ray tube:  
 $U = 35 \text{ kV}$ ,  $I = 1 \text{ mA}$

## b) Bragg reflection at an NaCl monocrystal:

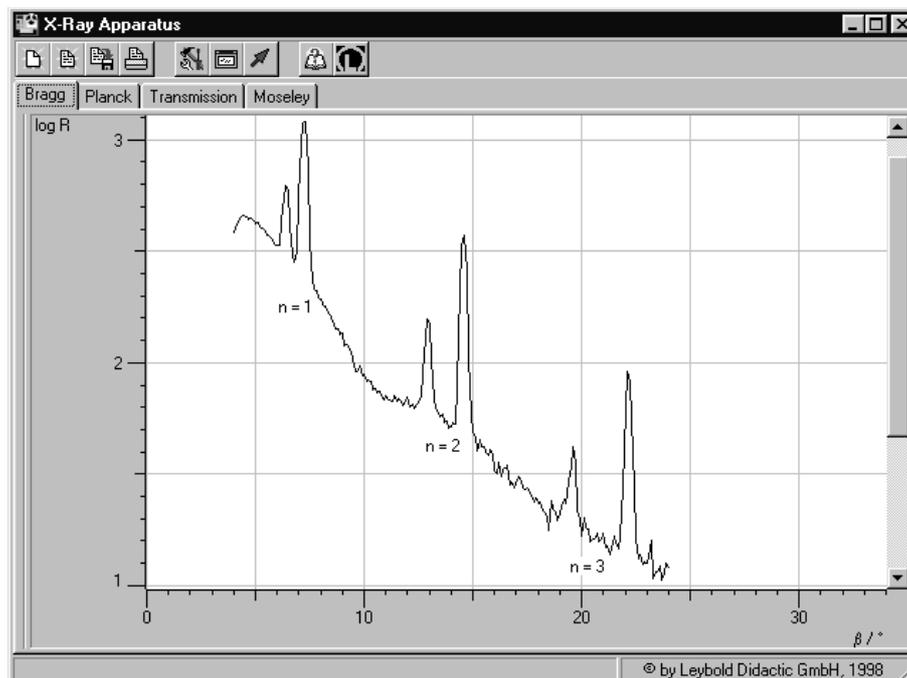


Fig. 5 Diffraction spectrum of x-rays in Bragg reflection to the third diffraction order at an NaCl monocrystal with logarithmic display of counting rate  $R$ .  
Parameters of x-ray tube:  
 $U = 35 \text{ kV}$ ,  $I = 1 \text{ mA}$

## Evaluation

- In each diagram, click the right mouse button to access the evaluation functions of the software “X-ray Apparatus” and select the command “Calculate Peak Center” to evaluate the diffraction spectra.
  - Using the left mouse button, mark the “full width” of each peak and write down the center values in a table as the glancing angle (see tables 2 and 3).
  - For each glancing angle  $\vartheta$ , calculate the values  $\sin \vartheta$  and and plot these value pairs in a diagram (see Fig. 6).
- In each case, the results lie along a straight line through the origin; in accordance with (III), its slope corresponds to the lattice constant  $a_0$ .

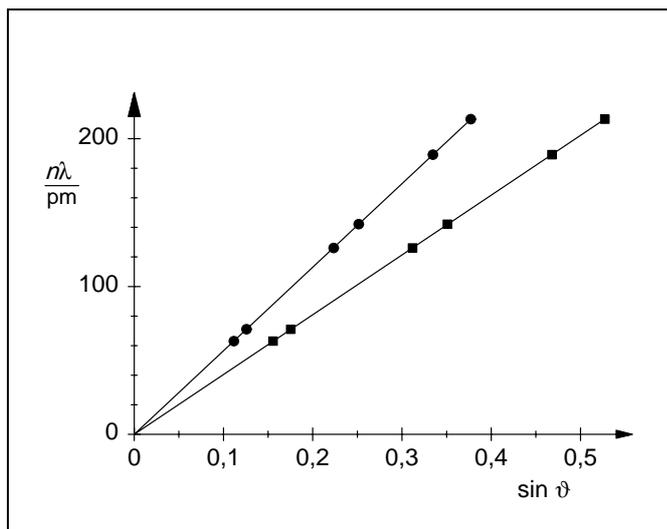


Fig. 6 Value pairs as a function of  $\sin \vartheta$   
 LiF: squares, slope of line = 404.5 pm  
 NaCl: circles, slope of line = 565.2 pm

Table 2: Glancing angle  $\vartheta$  of the LiF crystal

$\vartheta$	$\sin \vartheta$	Line	$n$	$\frac{n \cdot \lambda}{\text{pm}}$
8.95°	0.156	$K_{\beta}$	1	63.06
10.10°	0.175	$K_{\alpha}$	1	71.08
18.17°	0.312	$K_{\beta}$	2	126.12
20.54°	0.351	$K_{\alpha}$	2	142.16
27.91°	0.468	$K_{\beta}$	3	189.18
31.82°	0.527	$K_{\alpha}$	3	213.24

Table 3: Glancing angle  $\vartheta$  of the NaCl crystal

$\vartheta$	$\sin \vartheta$	Line	$n$	$\frac{n \cdot \lambda}{\text{pm}}$
6.41°	0.112	$K_{\beta}$	1	63.06
7.23°	0.126	$K_{\alpha}$	1	71.08
12.91°	0.223	$K_{\beta}$	2	126.12
14.57°	0.252	$K_{\alpha}$	2	142.16
19.55°	0.335	$K_{\beta}$	3	189.18
22.15°	0.377	$K_{\alpha}$	3	213.24

## Results

### a) LiF crystal:

Measurement result:  $a_0 = 404.5 \text{ pm}$   
 Literature value [2]:  $a_0 = 402.7 \text{ pm}$   
 Ion radii [3]: 68 pm ( $\text{Li}^+$ ), 133 pm ( $\text{F}^-$ )  
 Sum of ion radii: 201 pm

### b) NaCl crystal:

Measurement result:  $a_0 = 565.2 \text{ pm}$   
 Literature value:  $a_0 = 564.02 \text{ pm}$   
 Ion radii [3]: 98 pm ( $\text{Na}^+$ ), 181 pm ( $\text{Cl}^-$ )  
 Sum of ion radii: 279 pm

Conclusion: the LiF lattice shows a significantly smaller lattice constant than the NaCl lattice, as the radii of the ions involved are smaller.

## Literature

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

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

[3] Charles Kittel, Introduction to Solid State Physics, John Wiley & Sons, Inc. New York, USA