1. Assignment of tasks

1.Task: With the X-ray machine, the intensity (pulse rate R of the Geiger Müller counter tube) of x-rays reflected on a LiF single crystal has to be determined depending on the refraction angle θ for five different anode voltages U_{AS}. The refraction angle has to be transformed into the wavelength λ by bragg's law taking into account the lattice distance of the Li F crystal (d_{LiF} = 201.4 pm).

From the curves $R(\lambda)$, the short-wave limit λ_{min} of the X-ray spectrum has to be determined graphically. With these values and the linear equation:

 $e U_{AS} = h^* f_{max}$

the Planck constant h should be derived from the slope of the plotted equation.

2.Task: With the X-ray machine (Mo-Anode and included Zr-filter: λ_{Ka} = 71.1 pm) the Bragg angles have to be determined for a NaCl single crystal.

Calculate the lattice distance d of NaCl and compare it with the literature value $d_{NaCl} = 282.5$ pm.

2. Experimental set-up



Fig. 2.1:

X-ray machine with X-ray tube (R), experimental chamber (E), aperture (B), crystal (K), counting tube (Z), pointer 1 (Z₁), pointer 2 (Z₂), motor drive (M)

Fig. 2.1 shows the X-ray machine used in the experiment, Fig. 2.2 shows the entire experimental setup schematically. The X-ray radiation generated by the Molybdenum X-ray tube enters the experimental chamber through an aperture (collimator) into which a filter (e.g. Zr-film) can be inserted. The filtered X-ray hit the crystal (LiF or NaCl), which is attached to a vertical axis that rotates to the beam direction. The angular position θ of the crystal surface (=crystal planes) to the incident X-ray beam is indicated with a pointer Z₁ on an angular scale. Around the same axis of rotation, the counting tube for the intensity measurement of the reflected X-ray radiation can be rotated, whose angular position 2θ to the incident X-ray beam is indicated with a second longer pointer Z₂.

Both angle positions are adjustable. Via a mechanical coupling it is ensured: when adjusting one the other is tracked accordingly. At the position $2\theta = \theta = 0$, both pointer Z_1 and Z_2 are on top of each other. Note that in this position there is no reflection of the crystal and the direct Xray beam hits partially directly onto the Geiger-Müller counter which should be avoided during experiment. Therefore all experiments will start with a minimum angle of $\theta = 3^{\circ}$. Note! The motor drive is powered by a 12 V voltage source.

According to the angular position of the motor, a voltage in the range <u>+</u>3V is picked up at a potentiometer, which is connected to the motor axis by means of a slip clutch, which is located at the input C of the interface.

The pulse rate R of the Geiger-Müller-counter (proportional of the radiation intensity) is registered via the input F of this interface, so that an automatic recording of the counting rate $R(\theta)$ or $R(\lambda)$ (at known network plane distance using the Bragg equation) is possible.



The use of a computer also makes it possible to immediately graphically display and assess the rate $R(\theta)$ during the measurement on the monitor.

In addition, it should be noted that the emission current of the X-ray tube remains constant during the measurement (approx. 1 mA). This is controlled with a DC meter. An AC volt meter is used to determine the gradually adjustable (1,...,8) anode voltage U_A. This is determined by the measured effective value of the AC voltage U over the relationship

$$U_A = \sqrt{2} \times 10^3 U$$

To shield the environment from the X-ray radiation of the tube, the X-ray tube itself and the experimental chamber are enclosed by steel plates and lead glass windows. The lateral window can only be opened when the anode voltage is switched off, e.g. for mounting the crystal. The operation of the X-ray tube (switched on anode voltage) is indicated by a red warning lamp on the top of the device.

3. Experimentation and evaluation

3.1 Start of experiment, angle calibration and angle correction

First, the X-ray machine (initially without high voltage), the rate meter, and finally the power supply of the actuator must be adjusted.

In general, a calibration of the angle adjustment of the motor is necessary before the start of the experiment. For this purpose, the menu item *Calibration/Ranges (Kallibrieren/Bereiche)* is selected, then *angle calibration (Winkelkalibrierung)*, then *start/stop*, after which the voltage U of the motor potentiometer shown on the screen is driven to the value of approx. -2.95 V, which should correspond to the angular position $2\theta = \theta = 0$. A slight adjustment of the hands is necessary by the keys +/-/S (larger / smaller angle / stop). The zero position is confirmed by F2, i.e. the set voltage value is assumed to be $2\theta = \theta = 0$.

The angle 2 θ = 60⁰ is then approached with the +/-/S keys. The voltage now displayed in the screen corresponds to θ = 30⁰, which is confirmed again by *F2*. After that, the calibration is finished and can be exited with *ESC*.

In the evaluation of the measured angles, it must be taken into account that the spectra have an offset of an angle Δ . This angular error Δ depends strongly on the used crystal surface and its position in the crystal holder and is provided for the 1st task, since no filter (monochromator) can be used here. The angular error Δ for the 2nd task can be determined using the Bragg equation. If θ_1^m and θ_2^m are the measured angles for the maximum intensity in 1st and 2nd order, the angle correction is $\Delta = \theta_1 - \theta_1^m = \theta_2 - \theta_2^m$. With the boundary condition, that the corrected angles θ_1 and θ_2 result in the same mesh plane spacing for NaCl ($d_{NaCl} = 282.5 \ pm$), one ends up with the relation to calculate Δ :

$$\tan \Delta = \frac{\sin \theta_2^m - 2\sin \theta_1^m}{2\cos \theta_1^m - \cos \theta_2^m}$$

3.1.1. 1st Task:

The measurement is carried out with the LiF crystal **without** filter for 5 different anode voltages (levels 4 - 8 on the scale). The respective voltages U_{eff} have to be noted and the high voltage U_A has to be calculated (see equation above)

Before the 1st measurement:

parameters:	Angle range: θ : 3-10 ⁰ , Δ : 0,1 ⁰
	Torzeit: t: 1s
	d _{LiF} = 201.4 pm
Measurement: start:	F1
After each measurement series:	
Print screen:	Determine minimum angle $ heta_{min}$ Substract Δ

After all series of measurements of the 1st task:

Derive λ_{min} with Bragg's law ($\lambda_{min} = 2d_{LiF} \sin(\theta_{min})$).

Calculate $f_{max} = c * \lambda_{min}$

Plot the linerar equation following from energy conservation

$$U * e = h * f_{max}$$

Determine the Planck constant h from the slope of the graph using linear regression and compare to literature.

3.1.2. 2nd Task:

The measurement is to be carried out with NaCl crystal and **with** Zr filter, voltage level 8 to determine the lattice distance of NaCl

Before measuring:

parameters:	Angle range θ : 3-25°,
	Torzeit: t: 1s
Measurement: start:	F1

After the measurement:

Print screen: Calculate Δ	Determine the peak angles for n=1,2,3 from the first two maxima with the equation given above
Derive d _{NaCl} for all 3 Maxima	Get an average and compare it with the literature value

4. Questions for Self-checking

- 1) How does an X-ray tube work?
- 2) Which different X-ray spectra are distinguished (drawing)?
- 3) Why is there a short-wave limit of the *Bremsstrahlung*?
- 4) What is the origin of the X-ray *characteristic spectrum*?
- 5) How do we prove that X-ray are electromagnetic waves?
- 6) What is *Bragg's Law*?
- 7) How does a counter tube work?