– 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 β Limits at the same time (see Instruction Sheet for x-ray apparatus).

Preparing the PC-based measurement:

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

Carrying out the experiment

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_α and K_β 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° 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_a 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 10.2° (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 button 🗋 or the F4 key.
– Set the measuring time per angular step Δt = 10 s and the angular step width $\Delta\beta$ = 0.1°.
– Press the COUPLED key to activate 2ϑ coupling of target and sensor and set the lower limit of the target angle to 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 series under an appropriate name by pressing the button 🖫 or the F2 key.

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° 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_a 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° (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" or clear any existing measurement data using the button or the 🗋 F4 key.
– 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 24°.
– 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

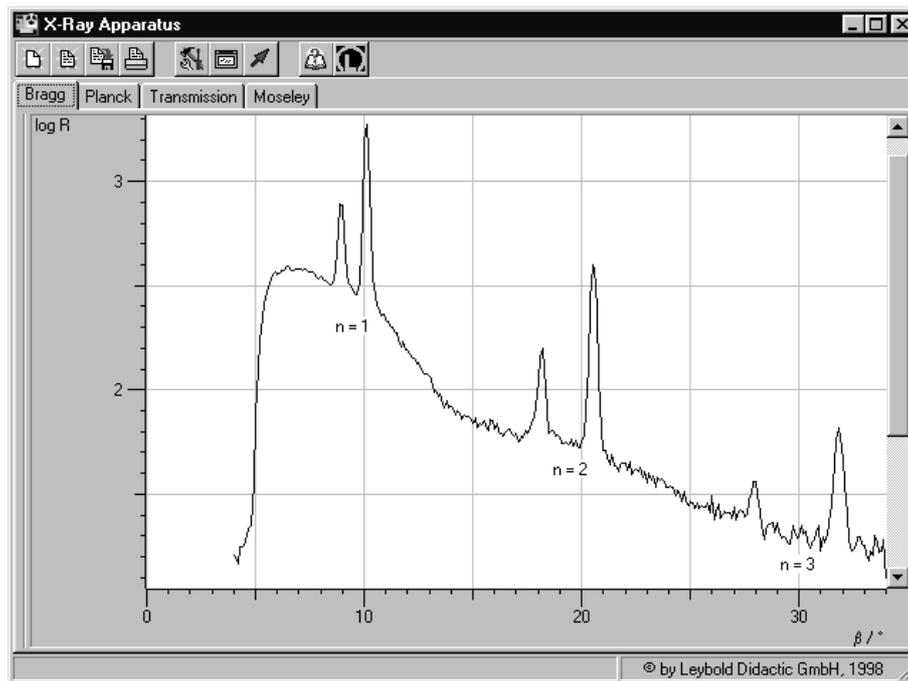**a) Bragg reflection at an LiF monocrystal:**

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:
$U = 35$ kV, $I = 1$ mA

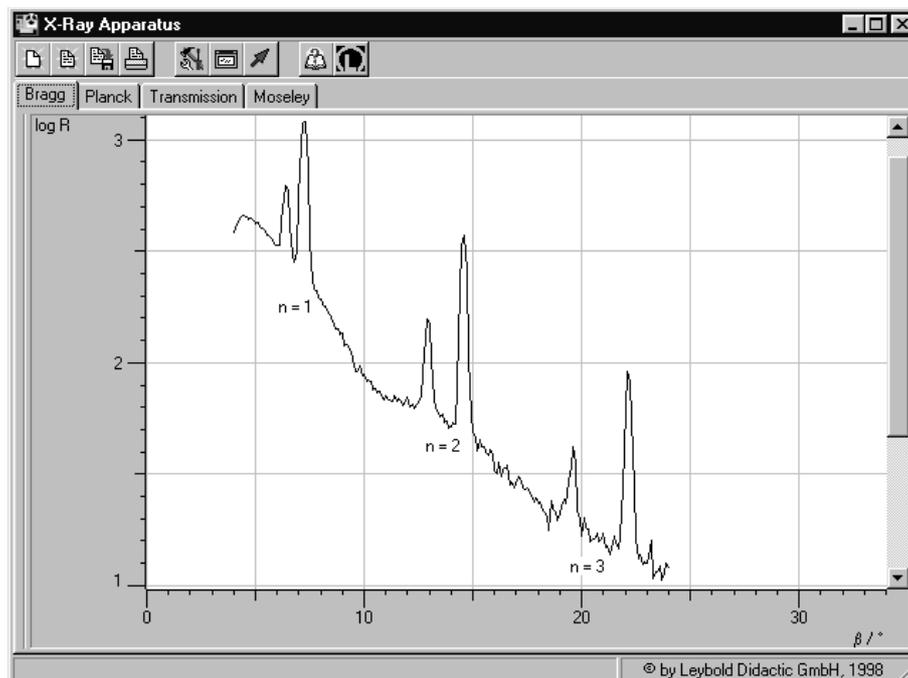**b) Bragg reflection at an NaCl monocrystal:**

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:
$U = 35$ kV, $I = 1$ 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 ϑ, calculate the values sin ϑ 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.
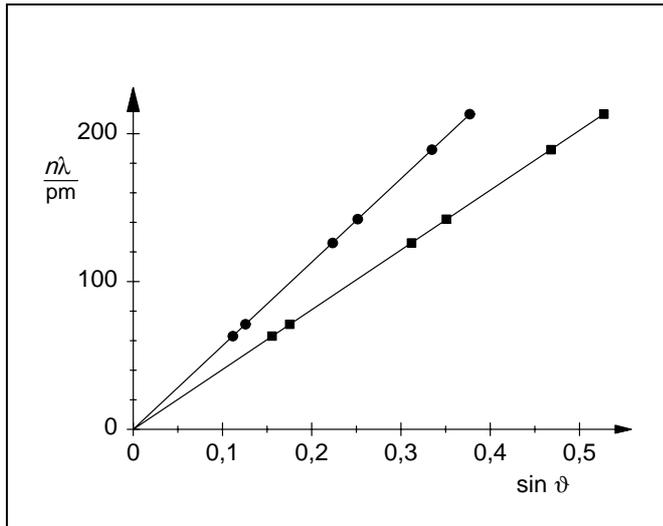
Fig. 6   Value pairs as a function of sin ϑ
LiF: squares, slope of line = 404.5 pm
NaCl: circles, slope of line = 565.2 pm

Table 3: Glancing angle ϑ of the NaCl crystal

ϑ	sin ϑ	Line	n	$\dfrac{n \cdot \lambda}{pm}$
6.41°	0.112	K_β	1	63.06
7.23°	0.126	K_α	1	71.08
12.91°	0.223	K_β	2	126.12
14.57°	0.252	K_α	2	142.16
19.55°	0.335	K_β	3	189.18
22.15°	0.377	K_α	3	213.24

Results

a) LiF crystal:

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

b) NaCl crystal:

Measurement result:	a_0 = 565.2 pm
Literature value:	a_0 = 564.02 pm
Ion radii [3]:	98 pm (Na^+), 181 pm (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

Table 2: Glancing angle ϑ of the LiF crystal

ϑ	sin ϑ	Line	n	$\dfrac{n \cdot \lambda}{pm}$
8.95°	0.156	K_β	1	63.06
10.10°	0.175	K_α	1	71.08
18.17°	0.312	K_β	2	126.12
20.54°	0.351	K_α	2	142.16
27.91°	0.468	K_β	3	189.18
31.82°	0.527	K_α	3	213.24