

## Diffraction patterns of powder samples with the three cubic Bravais lattices (Bragg-Brentano-geometry) (Item No.: P2542101)

### Curricular Relevance



#### Difficulty



Difficult

#### Preparation Time



1 Hour

#### Execution Time



2 Hours

#### Recommended Group Size



2 Students

#### Additional Requirements:

- PC

#### Experiment Variations:

#### Keywords:

Crystal lattices, crystal systems, Bravais-lattice, reciprocal lattice, Miller indices, structure factor, atomic scattering factor, Bragg scattering, characteristic X-rays, monochromatization of X-rays, Bragg-Brentano Geometry

## Overview

### Short description

#### Principle

Polycrystalline powder samples, which crystallize in the three cubic Bravais types, simple, face-centered and body-centered, are irradiated with the radiation from a X-ray tube with a copper anode. A swivelling Geiger-Mueller counter tube detects the radiation that is constructively reflected from the various lattice planes of the crystallites. The Bragg diagrams are automatically recorded. Their evaluation gives the assignment of the Bragg lines to the individual lattice planes, their spacings as well as the lattice constants of the samples, and so also the corresponding Bravais lattice type.



Fig. 1:P2542101

## Equipment

Position No.	Material	Order No.	Quantity
1	XR 4.0 expert unit, X-ray unit, 35 kV	09057-99	1
2	XR 4.0 X-ray goniometer	09057-10	1
3	XR 4.0 X-ray Plug-in Cu tube	09057-51	1
4	Geiger-Mueller counter tube, 15 mm (type B)	09005-00	1
5	XR 4.0 X-ray LiF crystal, mounted	09056-05	1
6	XR 4.0 X-ray Universal crystal holder for X-ray unit	09058-02	1
7	XR 4.0 X-ray Diaphragm tube w. nickel foil	09056-03	1
8	XR 4.0 X-ray Diaphragm tube d = 2 mm	09057-02	1
9	Ammonium chloride 250 g	30024-25	1
10	Potassium chloride 250 g	30098-25	1
11	Potassium bromide, 100 g	30258-10	1
12	Molybdenum, Powder, 99,7%, 100 g	31767-10	1
13	Microspoon, steel	33393-00	1
14	Vaseline 100 g	30238-10	1
15	Mortar w. pestle, 70ml, porcelain	32603-00	1
16	XR 4.0 Software measure X-ray	14414-61	1
17	Data cable USB, plug type A/B, 1.8 m	14608-00	1
18	XR 4.0 X-ray sample holder for powder samples (diffractometry)	09058-09	1

## Tasks

1. Record the intensity of the Cu-X-rays back scattered by the four cubic crystal powder samples with various Bravais lattice types as a function of the scattering angle.
2. Calculate the lattice plane spacings appropriate to the angular positions of the individual Bragg lines.
3. Assign the Bragg reflections to the respective lattice planes. Determine the lattice constants of the samples and their Bravais lattice types.
4. Determine the number of atoms in the unit cell.

## Set-up and procedure

### Set-up

Connect the goniometer and the Geiger-Müller counter tube to their respective sockets in the experiment chamber (see the red markings in Fig. 2). The goniometer block with the analyser crystal should be located at the end position on the right-hand side. Fasten the Geiger-Müller counter tube with its holder to the back stop of the guide rails. Do not forget to install the diaphragm in front of the counter tube.

Insert a diaphragm tube with a diameter of  $2\text{ mm}$  into the beam outlet of the tube plug-in unit.



Fig. 2: Connectors in the experiment chamber

### Note

Details concerning the operation of the X-ray unit and goniometer as well as information on how to handle the monocrystals can be found in the respective

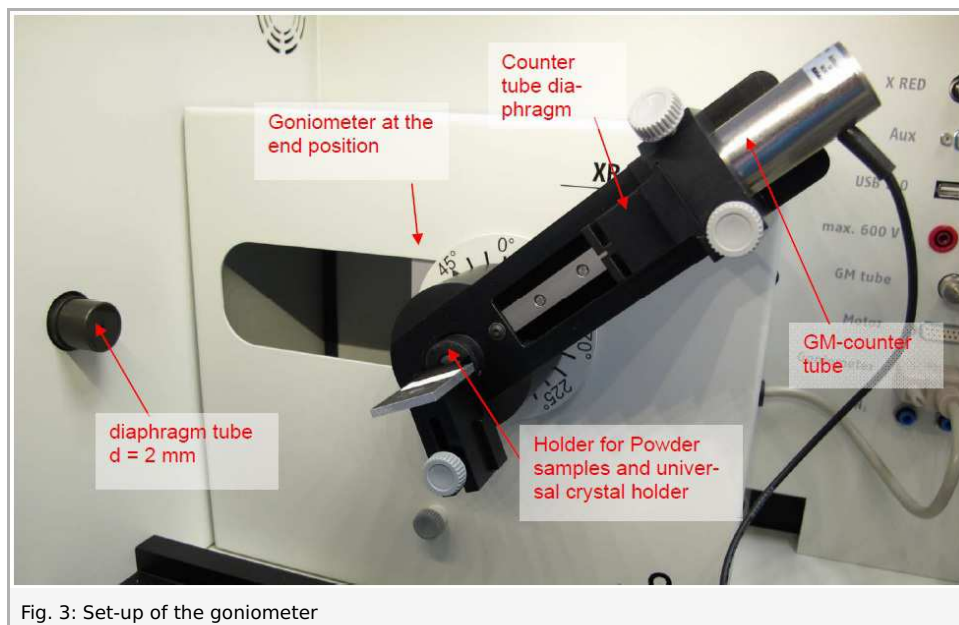


Fig. 3: Set-up of the goniometer

### Procedure

- Connect the X-ray unit via the USB cable to the USB port of your computer (the correct port of the X-ray unit is marked in Figure 4).
- Start the "measure" program. A virtual X-ray unit will be displayed on the screen.
- You can control the X-ray unit by clicking the various features on and under the virtual X-ray unit. Alternatively, you can

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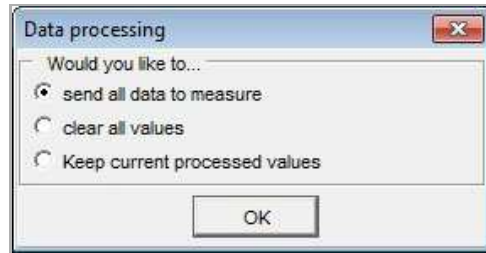
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also change the parameters at the real X-ray unit. The program will automatically adopt the settings.

- Click the experiment chamber (see the red marking in Figure 5) to change the parameters for the experiment. Select the parameters as shown in the text box.
- If you click the X-ray tube (see the red marking in Figure 5), you can change the voltage and current of the X-ray tube. Select the parameters as shown in the text box: Anode voltage  $U_A = 35 \text{ kV}$ ; anode current  $I_A = 1 \text{ mA}$ .
- Start the measurement by clicking the red circle:



- After the measurement, the following window appears:



- Select the first item and confirm by clicking OK. The measured values will now be transferred directly to the “measure” software.
- At the end of this manual, you will find a brief introduction to the evaluation of the resulting spectra.

## Note

Never expose the Geiger-Müller counter tube to the primary X-radiation for an extended period of time.



Fig. 4: Connection of the computer



Fig. 5: Part of the user interface of the software

### Sample preparation:

The sample must be so finely powdered that no grains can be felt when a little of it is rubbed between finger and thumb. A relatively high sample concentration can be obtained by mixing the powder with a little vaseline. To do this, transfer a small amount of the sample onto a sheet of paper and use a spatula to knead it to a firm paste. To achieve the highest concentration of material as possible, use very little vaseline (a spatula tip of it). Fill the relatively solid sample paste into the specimen for powder samples and smooth it flush. Use the universal crystal holder to hold the specimen.

### Calibration of the goniometer with the LiF single-crystal:

Exact angular positions of Debye-Scherrer reflections are only to be expected when the goniometer is correctly adjusted. Should the goniometer be out of adjustment for any reason whatever, this fault can be corrected either manually or by means of the autocalibration function:

- **Automatic calibration:**  
The anode material of the X-ray tube is automatically identified. The crystal must be manually set under "Menu", "Goniometer", "Parameter". For calibration, select "Menu", "Goniometer", "Autocalibration". The device now determines the optimal positions of the crystal and the goniometer to each other and then the positions of the peaks. The display shows the corresponding calibration curves. The newly configured zero position of the goniometer system is saved even after switch-off of the X-ray unit.
- **Manual calibration**  
The crystal for analysis must be manually brought to the theoretical Bragg angle  $\vartheta$  (counter tube correspondingly to  $2\vartheta$ ). Now search for the intensity maximum of the line by iterative turning of the crystal and counter tube by a few  $\pm 1/10^\circ$  around this angular position. Following this and in coupled mode, bring the crystal and counter tube to the particular zero position corrected by the error value and then confirm with "Menu", "Goniometer" and "Set to zero".

## Theory and evaluation

### Theory and Evaluation

When X-rays of wavelength  $\lambda$  strike a mass of lattice planes of a crystal of spacing  $d$  at a glancing angle of  $\vartheta$ , then the reflected rays will only be subject to constructive interference when Bragg's condition is fulfilled, i.e.:

$$2d\sin\vartheta = n\lambda; (n = 1, 2, 3, \dots) \quad (1)$$

Bragg's condition implies that all of the waves scattered at the atom are in phase and so amplify each other, whereas partial waves that are scattered in directions not fulfilling Bragg's conditions are of opposite phase and so extinguish each other. A more realistic way of looking at this must, however take the actual phase relationships of all of the partial waves scattered by the atom in a certain direction into consideration. When there are  $N$  atoms in a unit cell, then the total amplitude of the X-rays scattered by the cell is described by the structure factor  $F$ , which is calculated by summing up the atomic scattering factors  $f$  of the individual  $N$  atoms, taking their phases into account.

In general, the following is valid for  $F$ :

$$F_{hkl} = \sum_1^N f_n \cdot e^{2\pi i(hu_n + kv_n + hw_n)} \quad (2)$$

where  $h, k, l$  = Miller indices of the reflecting lattice planes and  $u_n, v_n, w_n$  are the coordinates of the atoms in fractions of the particular edge lengths of the unit cell.

As  $F$  is in general a complex number, the total scattered intensity is described by  $|F_{hkl}|^2$ .

A cubic simple unit cell contains only one atom with the coordinates 000. According to equation (2), therefore, the structure factor  $F$  for this lattice type is given by:

$$F = f \cdot e^{2\pi i(0)} = f; |F|^2 = f^2 \quad (3)$$

This means that  $F^2$  is independent of  $h, k$  and  $l$  and all Bragg reflections can therefore occur.

The unit cell of a cubic face-centered lattice has 4 atoms at 000,  $\frac{1}{2} \frac{1}{2} 0$ ,  $\frac{1}{2} 0 \frac{1}{2}$  and  $0 \frac{1}{2} \frac{1}{2}$ . The unit cell of a cubic body-centered lattice has in comparison only 2 atoms at 000 and  $\frac{1}{2} \frac{1}{2} \frac{1}{2}$ .

When the lattice only consists of one sort of atom, then the following conditions are valid for the structure factor:

fcc Lattice

$$\begin{aligned} |F|^2 &= 16f^2, \text{ with } hkl \text{ only even or only odd} \\ |F|^2 &= 0, \text{ with } hkl \text{ mixed} \end{aligned}$$

bcc Lattice

$$\begin{aligned} |F|^2 &= 4f^2, \text{ with } (h + k + l) \text{ even} \\ |F|^2 &= 0, \text{ with } (h + k + l) \text{ odd} \end{aligned} \quad (4)$$

The situation is somewhat different when a lattice is made up of different sorts of atoms.

When, for example, an fcc lattice consists of the atoms A and B, whereby the A atoms lie at 000,  $\frac{1}{2} \frac{1}{2} 0$ ,  $\frac{1}{2} 0 \frac{1}{2}$  and  $0 \frac{1}{2} \frac{1}{2}$ , and the B atoms at  $\frac{1}{2} \frac{1}{2} \frac{1}{2}$ ,  $0 0 \frac{1}{2}$ ,  $0 \frac{1}{2} 0$  and  $\frac{1}{2} 0 0$ , then the following additional condition is given for the structure factor  $F$ :

fcc Lattice with atoms A and B:

$$\begin{aligned} |F|^2 &= 16(f_A + f_B)^2, \text{ with } (h + k + l) \text{ even and} \\ |F|^2 &= 16(f_A - f_B)^2, \text{ with } (h + k + l) \text{ odd} \end{aligned} \quad (5)$$

In such fcc lattices, when the atomic scattering factors  $f$  of the two atoms are almost equal ( $f_A \approx f_B$ ), then 111 reflections will only be very weak, if they occur at all.

For the cubic crystal system, the spacing  $d$  of the individual lattice planes with the indices  $(hkl)$  is obtained from the quadratic form:

$$\frac{1}{d_{hkl}^2} = \frac{1}{a^2} (h^2 + k^2 + l^2) \quad (a =) \text{ lattice constant} \quad (6)$$

From this and equation (1), with  $n = 1$ , the quadratic Bragg equation is obtained:

$$\sin^2 \vartheta = \frac{\lambda^2}{4a^2} (h^2 + k^2 + l^2) \quad (7)$$

Examination of fcc lattices

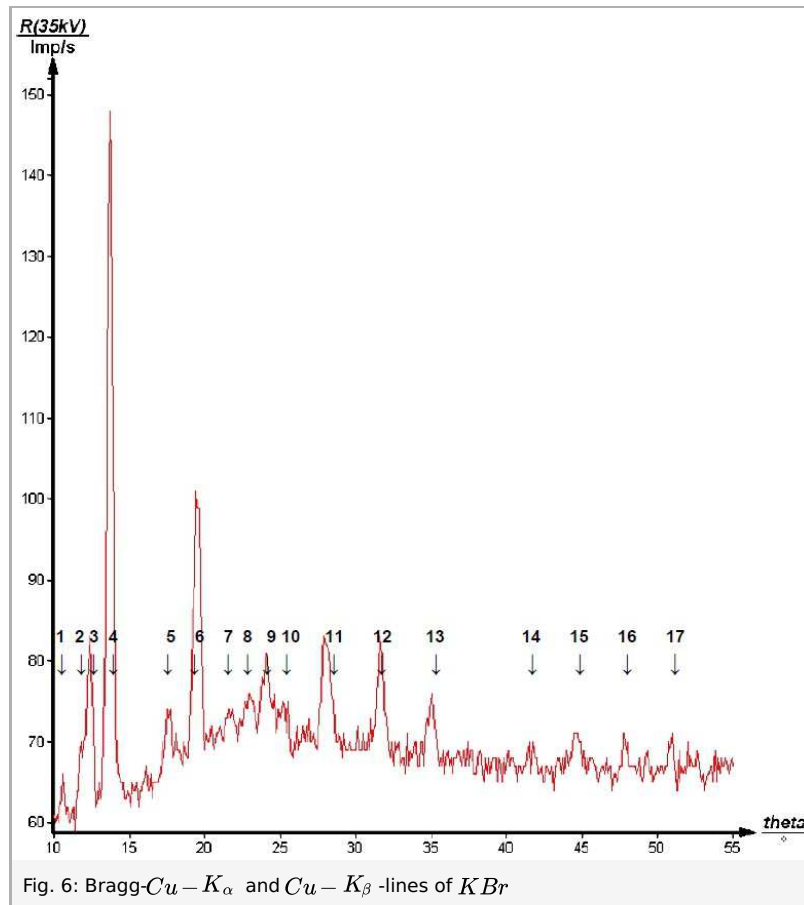
#### Potassium bromide

Fig. 6 shows the Debye-Scherrer spectrum of potassium bromide (KBr).

As no filter is used for the monochromatization of the X-rays, when individual lines are evaluated consideration must be given to the fact that the very intense lines that result from  $K\alpha$ -radiation are accompanied by secondary lines that result from the weaker

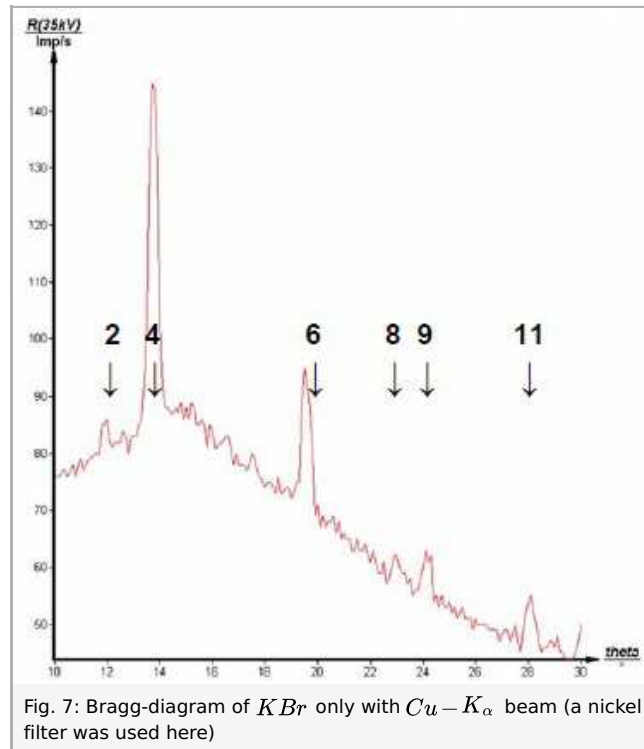
$K_\beta$  radiation. These pairs of lines can be identified by means of equation (1). It is namely approximately true with  $\lambda(K_\alpha) = 154.18 \text{ pm}$  and  $\lambda(K_\beta) = 139.22 \text{ pm}$ :

$$\frac{\lambda(K_\alpha)}{\lambda(K_\beta)} = \frac{\sin \vartheta_\alpha}{\sin \vartheta_\beta} = \frac{154.18 \text{ pm}}{139.22 \text{ pm}} \approx 1.1 \quad (8)$$



These values correspond to the quotients of the  $\sin \vartheta$  values (Fig. 6) of the pairs of lines 2-1, 4-3, 6-5 and 9-7, showing that the lines 1, 3, 5 and 7 originate from the  $CuK_\beta$  radiation.

The correctness of this conclusion can be shown by a control measurement (see Fig. 7) using the diaphragm tube with nickel foil to reduce the intensity of the  $K_\beta$  radiation. The reflexes in Fig. 6 that were previously assigned to the  $K_\beta$  lines are no longer to be seen. As the intensity of the  $K_\alpha$  - radiation is also somewhat weakened by the  $Ni$  foil, the detection of reflexes of weak intensity at larger glancing angles is made difficult when this is used.



The following method for evaluating the spectrum is given as an example, and is representative for that for the spectra of other samples. First determine the  $\sin \vartheta$  and  $\sin^2 \vartheta$  values for each individual reflex from the angle of diffraction  $\vartheta$  of the particular line. From equation (7), the ratios of the observed  $\sin^2 \vartheta$  values must be representable by the ratios of the sums of the squares of the three integer numbers ( $h, k, l$ ).

The ratios of the  $\sin^2$  values of the individual lines ( $n$ ) to the  $\sin^2$  value of the first line (2) are calculated as in column  $J$  of Table 1. The numbering in column  $E$  relates to the reflex lines indicated in Fig. 6. In column  $A$ , all of the possible  $h, k, l$  numbers are listed. Columns  $B, C$  and  $D$  show the individual ratios of the sums of squares of these numbers.



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A	B	C	D	E	F	G	H	I	J	K	L
<i>hkl</i>	$\frac{h^2+k^2+l^2}{k^2+l^2}$	$\frac{h^2+k^2+l^2}{(h^2+k^2+l^2)_{011}}$	$\frac{h^2+k^2+l^2}{(h^2+k^2+l^2)_{111}}$	Reflex no.	Intensity	$\vartheta^\circ$	$\sin\vartheta$	$\sin^2\vartheta$	$\frac{\sin^2\vartheta(n)}{\sin^2\vartheta(2)}$	<i>d</i> / pm	<i>a</i> / pm
100	1										
110	2	1									
111	3	1,5	1	2	w	11,80	0,20449	0,04182	1,00	377,0	652,9
200	4	2	1,33	4	vs	13,72	0,23718	0,05625	1,34	325,0	650,1
210	5	2,5	1,67								
211	6	3	2								
220	8	4	2,67	6	vs	19,46	0,33315	0,11099	2,65	231,4	654,5
221/300	9	4,5	3								
310	10	5	3,33								
311	11	5,5	3,67	8	w	22,95	0,38993	0,15204	3,64	197,7	655,7
222	12	6	4	9	s	24,08	0,40801	0,16647	3,98	188,9	654,5
320	13	6,5	4,33								
321	14	7	4,67								
400	16	8	5,33	11	s	27,97	0,46901	0,21997	5,26	164,4	657,5
410/322	17	8,5	5,67								
441/330	18	9	6								
331	19	9,5	6,22								
420	20	10	6,67	12	s	31,69	0,52532	0,27596	6,60	146,8	656,3
421	21	10,5	7								
332	22	11	7,33								
422	24	12	8	13	s	35,03	0,57401	0,32948	7,88	134,3	657,9
500/430	25	12,5	8,33								
510/431	26	13	8,67								
511/333	27	13,5	9								
520/432	29	14,5	9,67								
521	30	15	10								
440	32	16	10,67	14	vw	41,61	0,66406	0,44097	10,54	116,1	656,7
522/441	33	16,5	11								
530/433	34	17	11,33								
531	35	17,5	11,67	15	w	44,56	0,70166	0,49232	11,77	109,9	650,0
600/442	36	18	12								
610	37	18,5	12,33								
611/532	38	19	12,67								
620	40	20	13,33	16	w	47,86	0,74151	0,54983	13,15	104,0	657,5
621/540/443	41	20,5	13,67								
541	42	21	14								
533	43	21,5	14,33								
622	44	22	14,67	17	w	50,91	0,77656	0,60242	14,40	99,3	658,5

Table 1: Evaluation of the  $K_\alpha$ -Debye-Scherrer lines of *KBr*

When an attempt is made to allot the indices 100 or 110 to the first reflexes, then no agreement with the ratios of the  $\sin^2\vartheta$  values is found. When the index 111 is assigned to the first line, however, then all of the other lines can be assigned *hkl* index triplets with a certain accuracy.

Only even or odd numbers are now given, no mixed indices *hkl* triplets. According to this, *KBr* forms an fcc lattice. The corresponding lattice plane spacings *d*, calculated using equation (1), are given in column *K*. Values for the lattice constant *a* determined from equation (6) are given in column *L*. Taking both the  $K_\alpha$  lines and the  $K_\beta$  lines into consideration, the mean value of the lattice constant *a* is found to be:

$$a = (655.1 \pm 2.9) \text{ pm}; \Delta(a)/a < 0.5 \%;$$

(literature value:  $a = 658.0 \text{ pm}$ )

On dividing the total mass *M* of a unit cell by its volume *V*, the density  $\rho$  is given, so that:

$$\rho = \frac{M}{V} = n \cdot m \cdot \frac{1}{a^3} \quad \text{with } m = \frac{m_A}{N} \rightarrow n = \frac{\rho \cdot N \cdot a^3}{m_A} \quad (9)$$

where *n* = the number of atoms or molecules in the unit cell; *m* = atomic/molecular mass;  $m_A$  = atomic/ molecular weight;  $N = 6.022 \cdot 10^{23}$  = Avogadro's number.

On entering the appropriate values for *KBr*,  $\rho = 2.75 \text{ g} \cdot \text{cm}^{-3}$  and  $m_A = 119.01 \text{ g}$ , in equation (9), it follows that  $n = 3.91 \approx 4$ , i.e. the unit cell contains 4 atoms.

The  $K_\beta$  lines 1, 3, 5 and 7 that occur in Fig. 6 are evaluated in Table 2.

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A	B	C	D	E	F	G	H	I	J	K	L
<i>hkl</i>	$\frac{h^2+k^2}{k^2+l^2}$	$\frac{h^2+k^2+l^2}{(h^2+k^2+l^2)_{011}}$	$\frac{h^2+k^2+l^2}{(h^2+k^2+l^2)_{111}}$	Reflex no.	Intensity	$\vartheta/^\circ$	$\sin\vartheta$	$\sin^2\vartheta$	$\frac{\sin^2\vartheta(n)}{\sin^2\vartheta(2)}$	<i>d</i> / pm	<i>a</i> / pm
100	1										
110	2	1									
111	3	1,5	1	1	s	10,61	0,18412	0,03390	1,00	378,1	652,9
200	4	2	1,33	3	vs	12,38	0,21439	0,04596	1,36	324,7	650,1
210	5	2,5	1,67								
211	6	3	2								
220	8	4	2,67	5	s	17,61	0,30254	0,09153	2,70	230,1	654,5
221/300	9	4,5	3								
310	10	5	3,33								
311	11	5,5	3,67								
222	12	6	4	7	w	21,73	0,37023	0,13707	4,04	188,0	651,3

Table 2: Evaluation of the  $K_\beta$ -Debye-Scherrer lines of  $KBr$

## Potassium chloride

The Debye-Scherrer pattern for potassium chloride ( $KCl$ ) is shown in Fig. 8, and the evaluation of this with respect to the  $K_\alpha$ -radiation is given in Table 3.

The quotients of the sine values of the pairs of lines 2-1, 4-3, 6-5 and 8-7 again give approximately 1.1, so that the lines 1, 3, 5 and 7 can again be assigned to the  $K_\beta$  radiation.

Although  $KCl$  forms an fcc lattice, in contrast to  $KBr$  it gives exclusively even numbered  $h, k, l$  values and no, as expected for an fcc lattice, odd numbered  $h, k, l$  index triplets (see Tables 3 and 4). This is understandable when one considers that the atomic scattering factor  $f$  is correlated directly with the number of electrons of an atom, among others. As  $KCl$ , in contrast to  $KBr$ , contains atoms with almost the same scattering power ( $Z = 19$  for  $K$  and  $17$  for  $Cl$ ), it follows from equation (5) that reflexes with odd numbered  $h, k, l$  index triplets should not occur.

The mean value found for lattice constant  $a$  in the experiment is:  $a = (631.3 \pm 1.1) \text{ pm}$ ;  $\Delta(a)/a < 0.2\%$ ; (literature value:  $a = 629.3 \text{ pm}$ ).

From the experimentally determined average value for  $a$  and the known values for  $KCl$  ( $\rho = 1.984 \text{ g} \cdot \text{cm}^{-3}$  and  $m_A = 74.56 \text{ g}$ ), it follows from equation (9) that:  $n = 4.04 \approx 4$ , i.e. that the unit  $KCl$  cell contains 4 atoms.

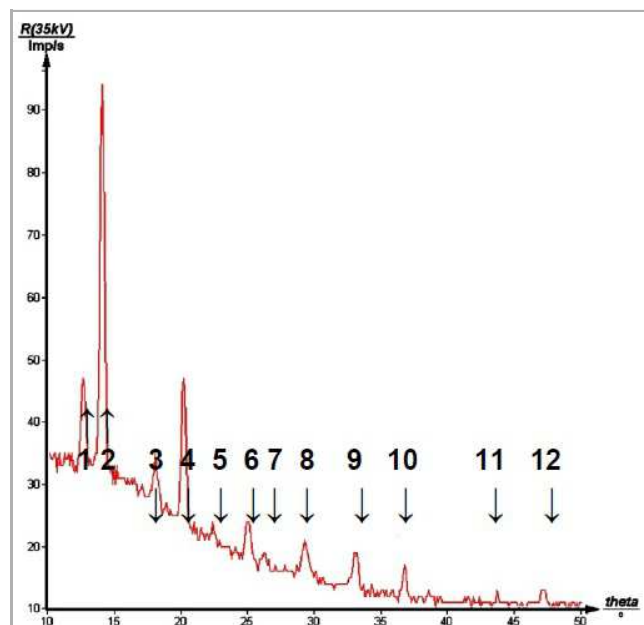


Fig. 8: Bragg- $Cu - K_\alpha$  and  $Cu - K_\beta$ -lines of  $KCl$

A	B	C	D	E	F	G	H	I	J
$hkl$	$\frac{h^2+k^2+l^2}{(h^2+k^2+l^2)_{002}}$	Reflex no.	Intensity	$\vartheta/^\circ$	$\sin\vartheta$	$\sin^2\vartheta$	$\frac{\sin^2\vartheta(n)}{\sin^2\vartheta(2)}$	d/pm	a/pm
200	1	2	vs	14, 13	0, 24412	0, 05960	1	315, 8	631, 6
220	2	4	vs	20, 22	0, 34562	0, 11946	2,00	223, 0	630, 9
222	3	6	s	25, 02	0, 42293	0, 17887	3,00	182, 3	631, 4
400	4	8	s	29, 30	0, 48938	0, 23950	4,02	157, 5	630, 1
420	5	9	s	33, 10	0, 54610	0, 29823	5,00	141, 2	631, 5
422	6	10	s	36, 80	0, 59902	0, 35883	6,02	128, 7	630, 5
440	8	11	vw	43, 72	0, 69113	0, 47767	8,01	111, 5	630, 7
600/442	9	12	w	47, 16	0, 73326	0, 53766	9,02	105, 1	630, 6

Table 3: Evaluation of the  $K_\alpha$  - Debye-Scherrer lines of  $KCl$

A	B	C	D	E	F	G	H	I	J
$hkl$	$\frac{h^2+k^2+l^2}{(h^2+k^2+l^2)_{002}}$	Reflex no.	Intensity	$\vartheta/^\circ$	$\sin\vartheta$	$\sin^2\vartheta$	$\frac{\sin^2\vartheta(n)}{\sin^2\vartheta(2)}$	d/pm	a/pm
200	1	1	vs	12,71	0,22002	0,04841	1	316,4	632,8
220	2	3	s	18,12	0,31101	0,09673	2,00	223,8	633,1
222	3	5	vw	22,40	0,38107	0,14521	3,00	182,7	632,8
400	4	7	vw	26,25	0,44229	0,19562	4,04	157,4	629,6

Table 4: Evaluation of the  $K_\beta$  - Debye-Scherrer lines of  $KCl$

### Examination of bcc lattices

Fig. 9 shows the spectrum of molybdenum ( $Mo$ ).

The evaluation in Table 5 shows that agreement with the ratios of the  $\sin^2\vartheta$  - values can only be given when  $(h + k + l)$  is even, i.e. that molybdenum forms a bcc lattice.

The experiment gives an average value for lattice constant  $a$  of  $a = (314.22 \pm 0.58) pm$ ;  $\Delta(a)/a < 0.2\%$  (literature value:  $a = 314.05 pm$ ).

A bcc lattice should contain 2 atoms per unit cell.

From the experimentally determined average value for  $a$  and the known values for  $Mo(\rho = 10.2 g \cdot cm^{-3}$  and  $m_A = 95.94 g)$ , it follows from equation (9) that:  $n = 1.99 \approx 2$ , i.e. that the unit  $Mo$  cell does actually contain 2 atoms.

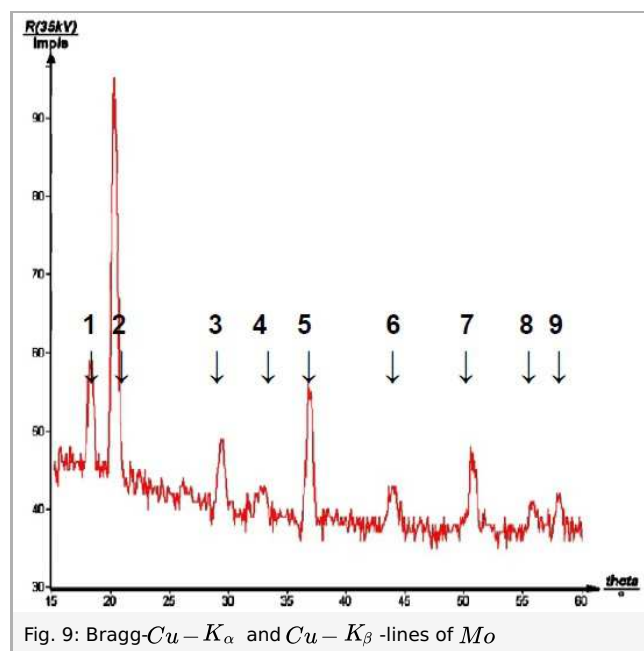


Fig. 9: Bragg-Cu -  $K_\alpha$  and Cu -  $K_\beta$  -lines of  $Mo$

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A	B	C	D	E	F	G	H	I	J	K
$hkl$	$h^2+k^2+l^2$	$\frac{h^2+k^2+l^2}{(h^2+k^2+l^2)_{011}}$	$\frac{h^2+k^2+l^2}{(h^2+k^2+l^2)_{111}}$	Reflex no.	$\vartheta/^\circ$	$\sin\vartheta$	$\sin^2\vartheta$	$\frac{\sin^2\vartheta(n)}{\sin^2\vartheta(2)}$	$d/pm$	$a/pm$
110 ( $\beta$ )	2			1	18,33	0,31449	0,09890		221,34	313,03
110	2	1		2	20,33	0,34743	0,12071	1	221,89	313,78
200	4	2	1	3	29,41	0,49106	0,24114	1,99	156,99	313,98
211 ( $\beta$ )	6			4	32,87	0,54273	0,29456		128,26	314,17
211	6	3	1,67	5	36,89	0,60029	0,36034	2,99	128,42	314,57
220	8	4	2	6	43,95	0,69403	0,48168	3,99	110,08	314,17
310	10	5	2,67	7	50,79	0,77483	0,60037	4,97	99,49	314,62
222	12	6	3,33	9	58,05	0,84851	0,71997	5,96	90,85	314,73
321 ( $\beta$ )	14			8	55,80	0,82708	0,68406		84,16	314,91

Table 5: Evaluation of the  $K_\alpha$ - and  $K_\beta$ -Debye-Scherrer lines of  $Mo$

## Examination of cubic simple (pc)-lattices

Fig. 10 shows the Debye-Scherrer spectrum of ammonium chloride ( $NH_4Cl$ ), the evaluation of which is given in Table 6.

Line 2 of the spectrum at  $\vartheta = 14.83^\circ$  is not taken into consideration, as the quotient of the  $\sin^2\vartheta$  value of the pairs of lines 3 and 2 is namely  $\sin^2(16.45^\circ)/\sin^2(14.83^\circ) = 1.11$ . Line 2 must therefore be assigned to the  $K_\beta$  radiation (see equation (9)).

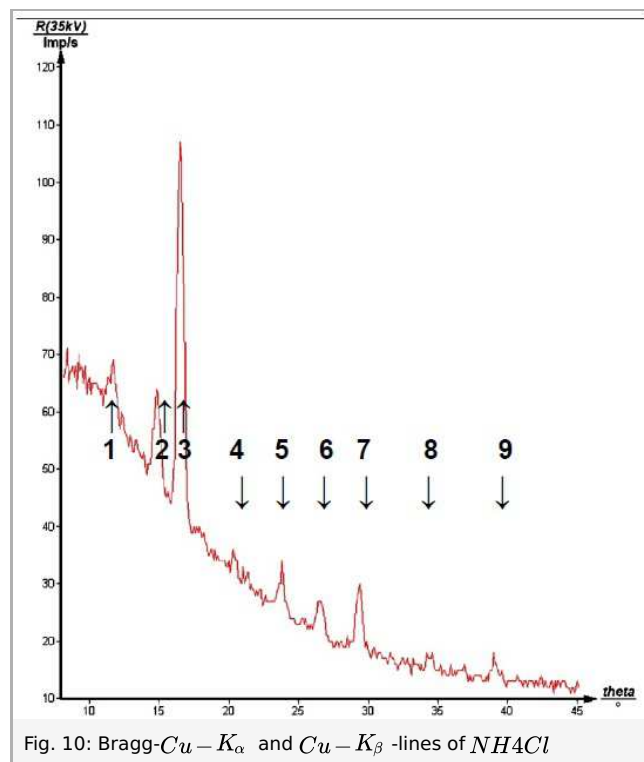


Fig. 10: Bragg- $Cu-K_\alpha$  and  $Cu-K_\beta$ -lines of  $NH_4Cl$

The quotient of the  $\sin^2\vartheta$  values in column  $I$  is always approximately even-numbered, and could so be assigned both to the bold face numbers in column  $B$  as well as to column  $C$ , so that it is not yet possible to make an unambiguous assignment to the reflex planes.

If assignment to column  $B$  should be correct, then mixed index  $(h, k, l)$  triplets and even-numbered  $(h + k + l)$  values would occur. This means that  $NH_4Cl$  has neither an fcc lattice nor a bcc lattice, but has rather a cubic simple (pc) cell with a mean lattice constant  $a$  (see column  $K$ ) of  $a = (384.5 \pm 1.7) \text{ pm}$ .

If assignment to column  $C$  should be correct, then only even-numbered  $(h + k + l)$  values would occur.

This would correspond to a bcc lattice with a mean lattice constant  $a$  (see column  $L$ ) of  $a^* = (543.7 \pm 2.2) \text{ pm}$ .

The following consideration helps to solve this dilemma.

The following values are given in Tables for  $NH_4Cl$ :  $\rho = 1.527 \text{ g} \cdot \text{cm}^{-3}$  and  $m_A = 53.49 \text{ g}$ .

Using these values and  $a = 384.5 \text{ pm}$  in equation (9),  $n = 0.977 \approx 1$ , i.e. only one molecule is present in the cell. According to this,  $NH_4Cl$  crystallizes cubic, simple.

On repeating this same procedure, but with  $a^* = (543.7 \pm 2.2) \text{ pm}$ , then  $n = 2.75$  is obtained.

The number of  $2\frac{3}{4}$  molecules in a unit cell can not be brought into accordance with a bcc lattice, as this contains only 2 atoms

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or molecules. From this it is clear that  $NH_4Cl$  forms a cubic simple lattice with the lattice constant  $a = (384.5 \pm 1.7) \text{ pm}$ ;  $\Delta(a)/a \leq 0.5 \%$ .

(Literature value:  $a = 386.0 \text{ pm}$ ).

<i>A</i>	<i>B</i>	<i>C</i>	<i>D</i>	<i>E</i>	<i>F</i>	<i>G</i>	<i>H</i>	<i>I</i>	<i>J</i>	<i>K</i>	<i>L</i>
<i>hkl</i>	$\frac{h^2+k^2+l^2}{k^2+l^2}$	$\frac{h^2+k^2+l^2}{(h^2+k^2+l^2)_{011}}$	$\frac{h^2+k^2+l^2}{(h^2+k^2+l^2)_{111}}$	Reflex no.	$\vartheta/^\circ$	$\sin\vartheta$	$\sin^2\vartheta$	$\frac{\sin^2\vartheta(n)}{\sin^2\vartheta(2)}$	<i>d</i> / pm	<i>a</i> / pm	<i>a</i> */ <i>l</i> pm
100	1			1	11,61	0,20125	0,04050	1,00	383,0	383,0	
110	2	1		3	16,45	0,28468	0,08105	2,00	270,8	383,0	541,6
111	3	1,5	1	4	20,34	0,34759	0,12082	2,98	221,8	384,1	543,3
200	4	2	1,33	5	23,79	0,40338	0,16272	4,02	191,1	382,2	541,6
210	5	2,5	1,67	6	26,51	0,44635	0,19923	4,92	172,7	386,2	
211	6	3	2	7	29,36	0,49030	0,24039	5,93	157,2	385,1	543,3
220	8	4	2,67	8	34,40	0,56497	0,31919	7,88	136,4	385,9	540,5
221/300	9	4,5	3								
310	10	5	3,33	9	39,06	0,63013	0,39707	9,80	122,3	386,8	546,1
311	11	5,5	3,67								
222	12	6	4								544,6
320	13	6,5	4,33								
321	14	7	4,67								
400	16	8	5,33								545,6
410/322	17	8,5	5,67								
441/330	18	9	6								
331	19	9,5	6,22								
420	20	10	6,67								546,9

Table 6: Evaluation of the  $K_\alpha$  - Debye-Scherrer lines of  $NH_4Cl$