TASK 105	X-RAY SPECTROSCOPY

Demonstration – Measurement procedure

1 The list of necessary equipment

a. An X-ray unit equipped with X-ray tube with a tungsten anode.
b. X-ray goniometer with lithium fluoride crystal.
c. A Geiger-Müller counter tube.
d. Computer.

2 Issues

- continuous bremsspectrum and characteristic X-radiation,
- energy levels in atom,
- crystal lattice constant,
- Bragg's law.

3 Experimental setup

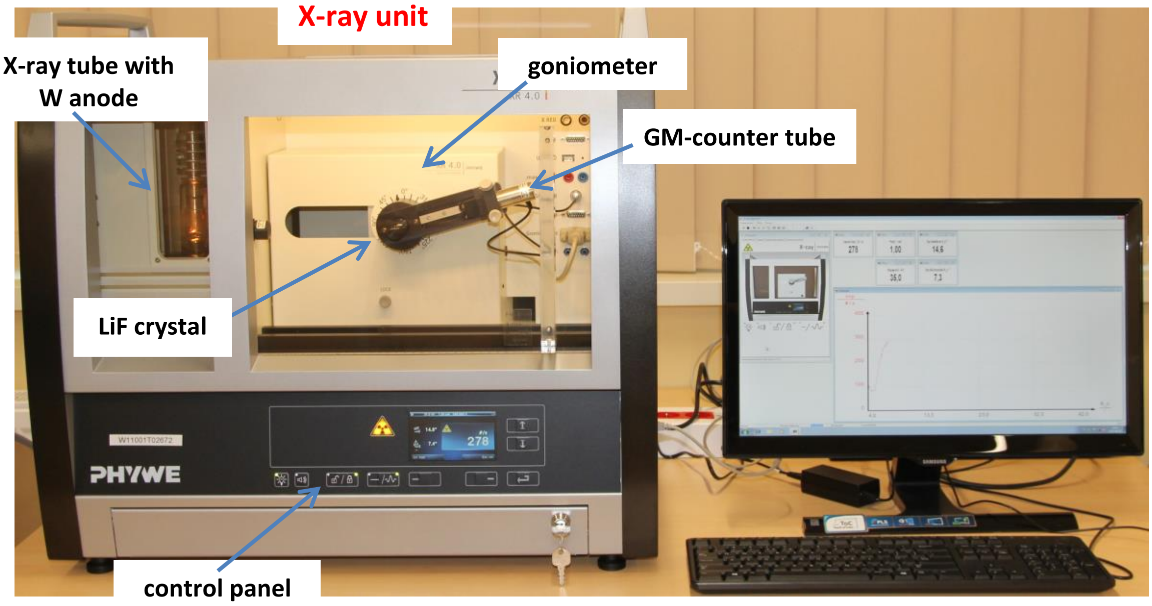


Fig. 1 Picture of experimental setup.

4 Measurement procedure

Turn on PC computer and X-ray unit (switch button is located on the rear panel of the device). The measuring chamber may be opened by gently moving the door to the right - if the door can't be opened in that way, it may be locked electromagnetically by button located on the control panel. The chamber can illuminated (darken) from the inside by pressing the button (first from the left) on the control panel.

Start ***Measure*** software – on the screen the window *Nawigator* showing virtual X-ray unit should appear (see Fig. 2). You can control the X-ray unit by clicking the various features on and under the virtual X-ray unit. If you click the display of the measuring chamber (see the right red marking in Fig. 2), you can change the parameters of the experiment – settings for the goniometer (Fig. 3a). Click the X-ray tube in order to change the voltage and current of the X-ray tube. Select the settings as shown in Fig. 3b.

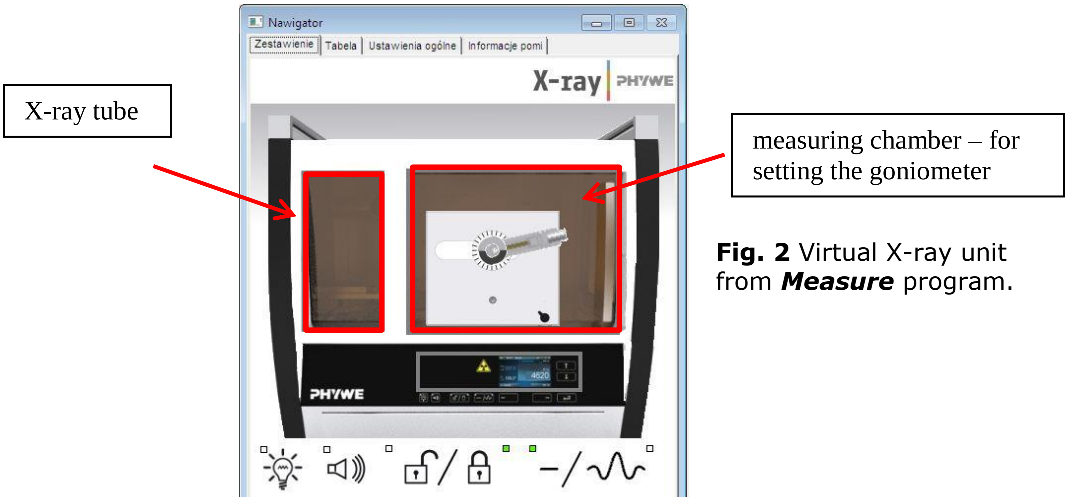
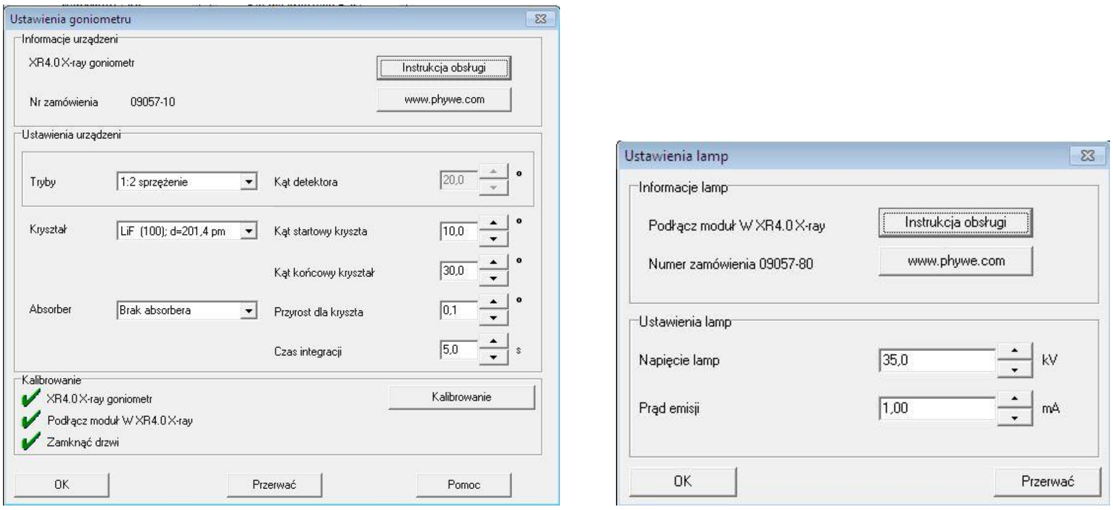


Fig. 2 Virtual X-ray unit from ***Measure*** program.

a)



b)

Fig. 3 Configuration panel for goniometer (a) and X-ray source (b).

Below a picture of an X-ray unit are shown an icons that correspond to buttons on the control panel of real unit, e.g.: on/off lighting (bulb icon), lock/unlock the main chamber door (the lock icon). Before starting the measurement, lock the door by pressing a button on the control panel X-ray unit. To run the measurement press the red circle in the upper left corner of ***Measure*** window. To stop the measurement press the black square. When the measurement is completed (or interrupted) message box *Dalsza obróbka danych* will appear – Fig. 4. To save the measurement select the first option from the top: *Wszystkie pomiary przenieś do measure*.

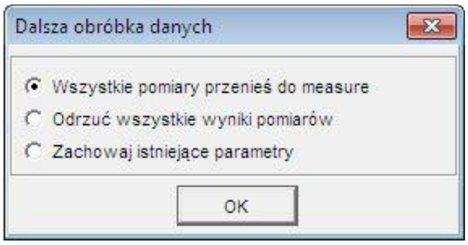


Fig. 4 Message box appearing after measurement.

Caution! Never expose the Geiger-Müller counter tube to the primary X-radiation (goniometer set to 0° when the crystal is not mounted).

4.1 Examination of characteristic X-radiation of tungsten

When electrons hit the metallic anode of the x-ray tube with a high kinetic energy, x-rays with a continuous energy distribution (bremsstrahlung) are generated. The bremstrahlung is superimposed by additional discrete lines. If an atom of the anode material is ionized, for example, in a deeper shell by an electron impact, an electron from a higher shell can take the now free place while emitting inter alia an x-ray quantum. The energy of this x-ray quantum corresponds to the energy difference of the two levels that are involved in this process. Since the energy difference is atom specific, the radiation that is generated by this process is also called the characteristic X-radiation.

The source of X-radiation is anode made of tungsten (W, atomic number Z = 74). X-ray is analyzed with use of lithium fluoride crystal (LiF). Geiger-Müller counter is used to measure the intensity of X-rays.

Polychromatic X-radiation produced in anode tube entering the measuring chamber through small slit. In measuring chamber polychromatic photons hits LiF crystal. The rays are reflected by the crystal lattice planes only interfere in a constructive manner and reaches GM counter tube. Both the LiF crystal and GM counter tube are mounted in the goniometer. The measurement is based on measuring the X-rays intensity depending on the angle at which the X-rays fall on the crystal.

The obtained spectrum, on a continuous background, presents maxima from which position we can determine the energies of the characteristic radiation of tungsten.

4.2 Measurement of the X-ray spectrum of tungsten

Measure the X-ray spectrum of tungsten in one of the two ranges: between 14° and 26° – which allows us to observe transitions for m = 1 or between 30° a 50° which allows us to observe transitions for m = 2 (measurements between 30° a 80° allows us to observe transition for m = 2 and 3).

Overview of the settings of the goniometer and X-ray unit:

Mode	– 1:2 coupling mode
Crystal	– LiF (100)
Absorber	– No absorber
Crystal start angle	– 14° (30°)
Crystal stop angle	– 26° (50° lub 80°)
Crystal angle increment	– 0,1°
Integration time	– np. 6 s (14-26° i 30-50°) i 4 s (30-80°)
Tube voltage	– U_A = 35 kV
Emission current	– I_A = 1 mA

After measurement the obtained spectrum should be made available to students. It is convenient to save the received spectrum in text format. Namely, in the tab *Pomiar* clicking *Eksport wartości pomiarowych* - opens the box *Eksportuj wartości pomiarowe*: for *Cel* - select *Zapisz do pliku*, for *Format* – *Eksportuj jako wartości liczbowe*. Write the file in the folder *Pomiary* (folder on the desktop), giving it the unique name and extension (e.g. .dat or .txt). Back to the window which enables measurements is after pressing the red dot icon.

4.3 Determination of the characteristic energy values of tungsten

Students read the angles ϑ corresponding to a few maxima in the measured spectrum (one may use a spreadsheet or just a text editor). Using the formula:

$$E = \frac{mhc}{2d\sin\vartheta} \qquad (1)$$

where: *m* – diffraction order (to check in the table),

h – Planck's constant ($h = 6{,}6261 \cdot 10^{-34}\,\mathrm{J\,s} = 4{,}1357 \cdot 10^{-15}\,\mathrm{eV\,s}$),

c – speed of light ($c = 2{,}9979 \cdot 10^{8}\,\mathrm{m/s}$),

d – interplanar spacing LiF ($d = 2{,}014 \cdot 10^{-10}\,\mathrm{m}$),

students calculate the energies corresponding to the chosen transitions (the number of transitions specifies teacher). They determine the accuracy of angle $u(\vartheta)$. Students calculate the complex uncertainty $u_c(E)$. The obtained energy values of internal transitions in tungsten students compare with the table (or energy levels diagram).

4.4 Determination of the lattice constant of LiF crystal

Students read the angles ϑ corresponding to a few maxima in the measured spectrum (the number of transitions specifies teacher) and identify chosen transitions with help of Fig. 5. Using the table (for transition energy determination) and after conversion formula (1):

$$d = \frac{mhc}{2E\sin\vartheta}, \qquad (2)$$

students can determine the lattice constant of LiF crystal. Students should calculate the complex uncertainty $u_c(d)$, mean value $\bar{d}$ and total uncertainty $u(d)$. The result can be compared with the real lattice constant of LiF crystal.

5 Additional content

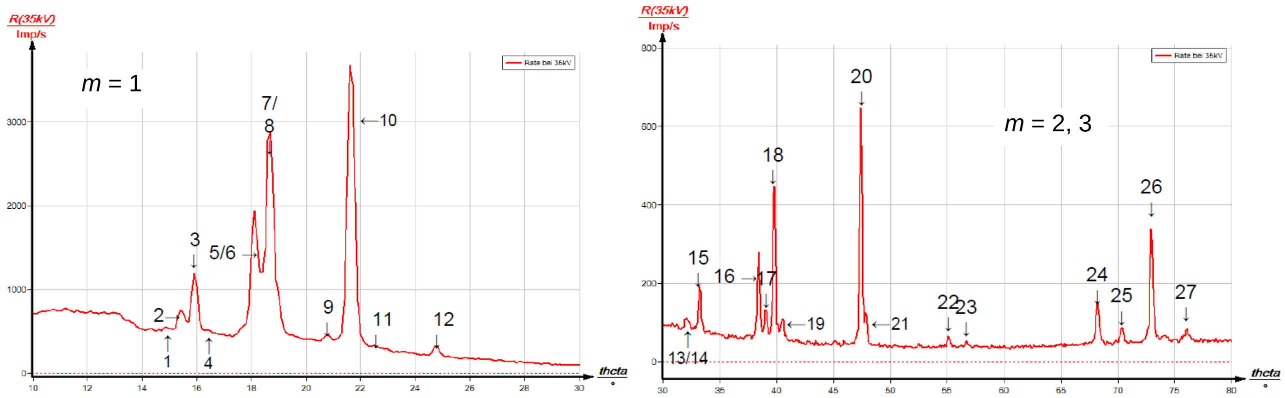


Fig. 5 Examples of X-ray intensity on angle dependence spectra for the tungsten (in the suggested ranges of angle change). Transition energies you can check in the table.

Table Characteristic lines and its energies for tungsten.

Number	*m*	Line	Transition	Energy [eV]
1	1	γ_4	L_1O_3	12063
2	1	$\gamma_{3/2}$	L_1N_3/L_1N_2	
3	1	γ_1	L_2N_4	11286
4	1	γ_5	L_2N_1	10949
5	1	β_2	L_3N_5	9961
6	1	β_3	L_1M_3	
7	1	β_1	L_2M_4	9673
8	1	β_4	L_1M_2	
9	1	η	L_2M_1	8725
10	1	$\alpha_{1/2}$	L_3M_5/L_3M_4	8397
11	1	Cu-K$\alpha_{1/2}$		
12	1	l	L_3M_1	7387
13	2	γ_3	L_1N_3	11674
14	2	γ_2	L_1N_2	11608
15	2	γ_1	L_2N_4	11286
16	2	β_2	L_3N_5	9961
17	2	β_3	L_1M_3	9818
18	2	β_1	L_2M_4	9673
19	2	β_4	L_1M_2	9525
20	2	α_1	L_3M_5	8397
21	2	α_2	L_3M_4	8335
22	3	γ_1	L_2N_4	11286
23	2	l	L_3M_1	7387
24	3	β_2	L_3N_5	9961
25	3	β_3	L_1M_3	9818
26	3	β_1	L_2M_4	9673
27	3	β_4	L_1M_2	9525

6 Measurement tables

Number	ϑ [°]	$u(\vartheta)$ [°]	Line	*m*	*E* [eV]	$u_c(E)$	E_{tab} [eV]
...	...	...	...	...	...	...	...

Number	ϑ [°]	$u(\vartheta)$ [°]	Line	*m*	E_{tab} [eV]	*d* [m]	$u_c(d)$ [m]
...	...	...	...	...	...	...	...
						$\bar{d}$ [m]	
						u(d) [m]	
						d_{tab} [m]	