Vilnius University

Faculty of Physics

Laboratory of Atomic and Nuclear Physics

Experiment No. 4

STUDY OF CHARACTERISTIC X-RAYS OF VARIOUS MATERIALS

Measurement Procedure

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This is only a detailed description of the measurement procedure. When preparing for the experiment, you must use the other manual, which presents not only the experimental methodology, but also the theory. The description of the experimental part must remain next to the measurement equipment at all times; it may not be taken out of the laboratory.

Equipment

This experiment uses an educational X-ray apparatus manufactured by the German company "Phywe Systeme". The experimental setup consists of:

1) an X-ray apparatus with a molybdenum (Mo, $Z = 42$) X-ray tube (X-ray tube anode voltage adjustment range: 0 to 35 kV; anode current adjustment range: 0 to 1 mA),
2) a goniometer (an accessory of the X-ray apparatus designed for investigating X-ray spectra and scattering),
3) a computer with the "*MCA Lab*" software, designed for displaying and analyzing spectra,
4) a Si-PIN detector with a multichannel analyzer (in a single housing), connected to the computer via a USB port,
5) a lithium fluoride (LiF) crystal with a holder,
6) a set of X-ray fluorescence samples (plates of various metals and plastic vials containing powders of various inorganic compounds) and a sample holder.

Fig. 1 shows a general view of the measurement equipment. On the left side of the X-ray apparatus is the X-ray tube unit with a collimator aperture. When the X-ray tube anode voltage and current are turned on, the light emitted by the heated cathode is visible through that window (see Fig. 1). Inside the chamber of the X-ray apparatus is the goniometer. The plug-in sockets at the center of the goniometer axis are intended for mounting the sample holder or the crystal. The detector is mounted on guide rods that can rotate around the goniometer axis.

To open the chamber of the X-ray apparatus, slide the sliding door made of leaded acrylic glass to the left. The door can be opened only after unlocking the corresponding lock. To do this, press the red locking knob all the way in and, while holding it pressed, turn it a quarter turn to the right so that it locks in place. The X-ray apparatus will operate only when the door is locked. To lock the door, turn the locking knob a quarter turn to the left.

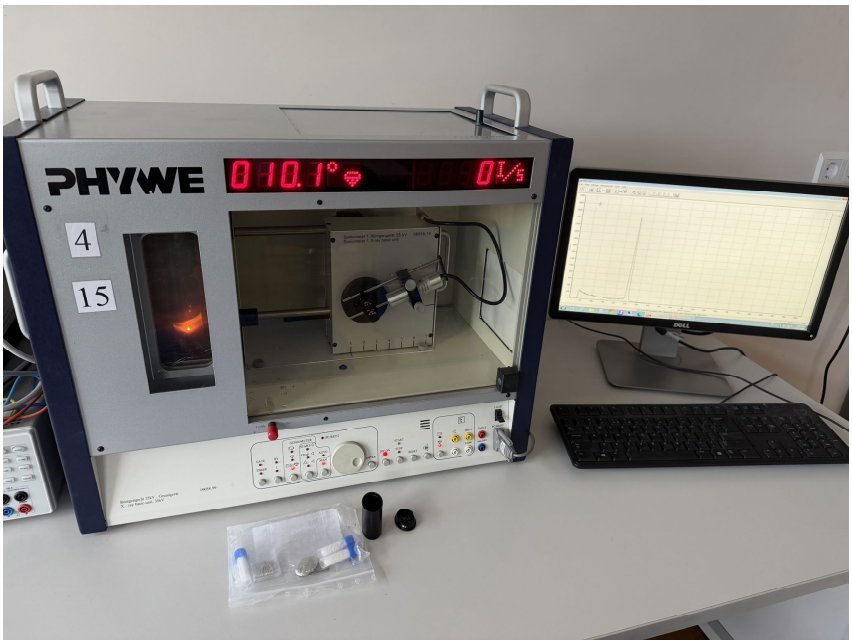


Fig. 1. General view of the measurement equipment.

Measurement Procedure

1. Ensure that there is no stand for vials inside the chamber of the X-ray apparatus. If it is present, remove it from the chamber (opening and closing the chamber door is explained on the first text page of this manual). In addition, if the sample holder or the LiF crystal is mounted in the goniometer, remove it as well.

2. Turn on the X-ray apparatus and the computer (the power switch of the X-ray apparatus is located on the rear panel of the instrument). When the X-ray apparatus is turned on, the goniometer automatically rotates the detector to the initial position corresponding to a zero glancing angle θ.

3. Using the controls of the X-ray apparatus located at the bottom of its front panel (see Fig. 2), set the X-ray tube anode voltage to **35** keV and the anode current to **0.1** mA. To do this, use the "HV-I" button (Fig. 2, left), the adjustment knob (Fig. 2, right), and the digital display at the top of the instrument (see Fig. 1). Pressing the "HV-I" button alternately activates the adjustment of the X-ray tube high voltage or anode current. The corresponding light-emitting diode (LED) and the digital display indicate which of the two operating quantities—"HV" or "I"—is selected. The value of the selected quantity is shown on the left side of the display. To change it, turn the adjustment knob. **After setting the required value, you must confirm it by pressing the "ENTER" button** (Fig. 2, right).

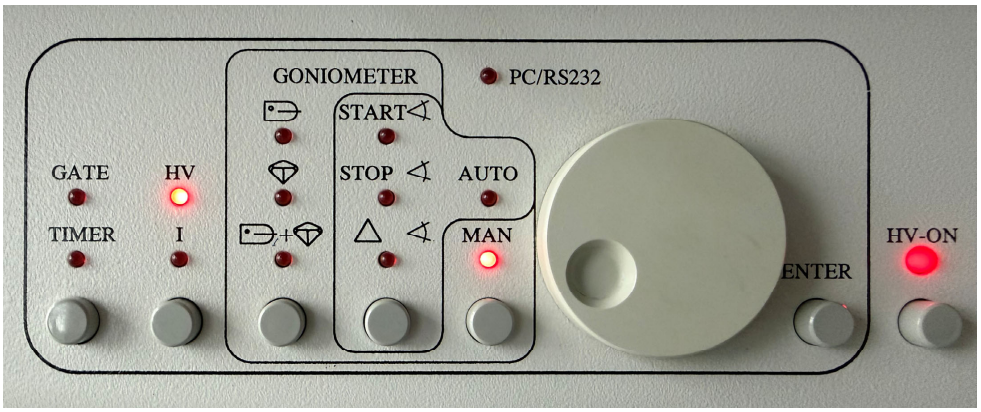


Fig. 2. Control panel of the X-ray apparatus.

4. Even such a low anode current is too high to direct the X-ray beam straight into the detector. A Si-PIN detector can record spectra without distortion only up to approximately one thousand photons per second, whereas in the primary radiation beam several hundred thousand photons per second would strike the detector window. Moreover, such a high radiation intensity can damage the detector. Therefore, place a 3 mm thick aluminum plate in front of the X-ray tube aperture, which will reduce the radiation flux by two orders of magnitude. Place the aluminum plate on a stand (a plastic box) and lean it against the X-ray tube aperture (see Fig. 3).

5. Launch the "*MCA Lab*" program. The channel number ("*MCA Channel*") must be plotted on the *X* axis. If the *X*-axis label is "*Energy E, keV*", click the toolbar button n (see Fig. 4).

6. Turn on the X-ray tube anode voltage (35 kV). If the high voltage (i.e., anode voltage) is being turned on for the first time after switching on the apparatus, or if the door of the X-ray apparatus was previously opened and closed, then before turning on the high voltage you must press the locking knob in once more (without turning it) all the way and release it so that it pops back out (if you do not do this, the high voltage will not turn on). To turn on the X-ray tube accelerating voltage, press the "HV-ON" button (Fig. 2, right). The X-ray tube will then begin emitting X-rays, and the LED above the "HV-ON" button will light up red. **Caution!** If the primary X-ray beam is directed into the detector window, the X-ray tube anode voltage may be turned on only when the aluminum filter is placed between the detector and the aperture (see step 4). An excessively high incident radiation intensity can damage the detector.

7. In the "*MCA Lab*" program, set the spectrum measurement duration (**3** min). To do this, select the menu command "*Measurement* > *Parameter*", then in the "*Settings*" dialog box that opens, enter the

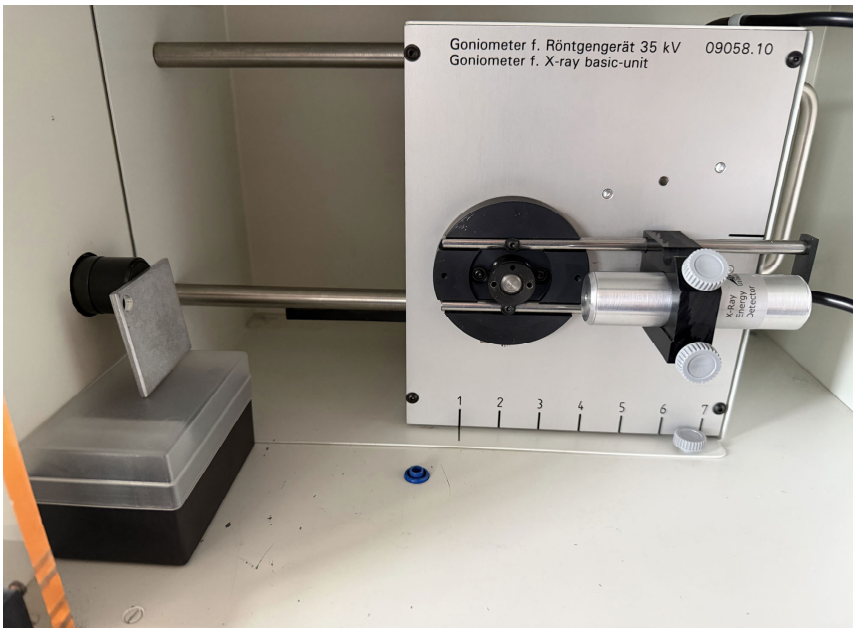


Fig. 3. Position of the detector and aluminum plate when measuring the spectrum of radiation from the X-ray tube with a molybdenum anode transmitted through a 3 mm thick aluminum layer.

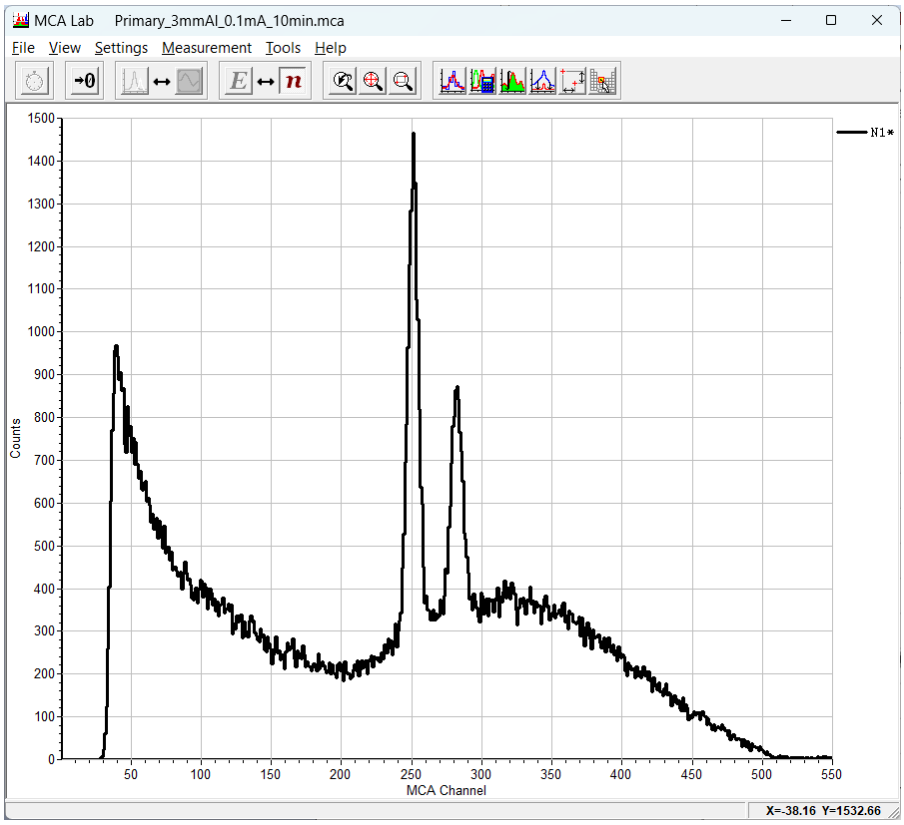


Fig. 4. Example of the primary spectrum measured using a 3 mm thick aluminum filter (measurement duration 10 min).

number of seconds (**180**) in the "*Standard duration of measurement, s*" text field and click the "*Close*" button.

8. Click the first toolbar button "". The "*MCA Lab*" program then begins recording the detector pulse height spectrum and displaying it in real time on the graph. During the measurement, wait until the program finishes acquiring the spectrum. The elapsed measurement time is displayed in the status bar (at the bottom of the program window) after the word "*Measured:*" (e.g., see Fig. 5). **Note:** While measuring a spectrum, the program does not update the graph axis limits automatically. The limits of the *X* or *Y* axis can be changed by right-clicking on the *X* or *Y* axis. A dialog box then opens in which you enter the desired limit values in the "*Minimum*" and "*Maximum*" fields and click the "*Close*" button. To set the "optimal" limits, i.e., *Y*-axis limits corresponding to the largest count in the channels, select the menu command "*View* > *Zoom All*", click the toolbar button , or use the key combination "*Alt*" + "*A*".

9. After completing the measurement, turn off the X-ray tube accelerating voltage. To do this, press the "HV-ON" button on the X-ray apparatus so that the LED above it turns off.

10. Using the "*MCA Lab*" menu command "*File* > *Save As...*", save the spectrum data to a file with the extension ".mca" (this file can later be opened in "*MCA Lab*"). **Note:** Lithuanian characters may not be used in the names of `*.mca` files or the folders containing them (otherwise "*MCA Lab*" will not be able to open them).

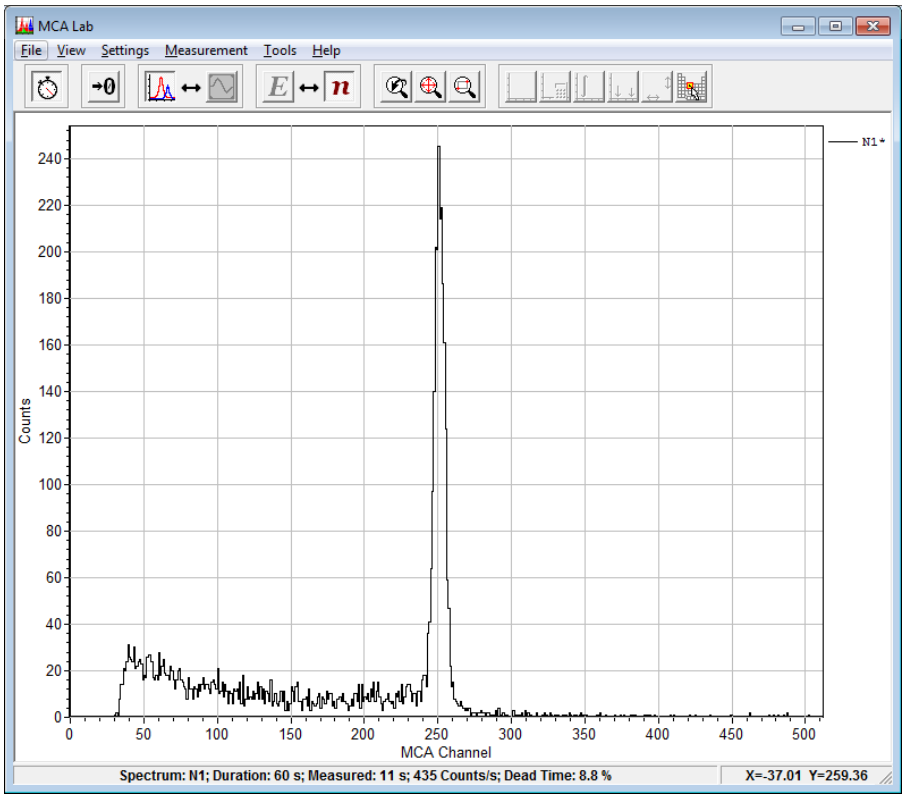


Fig. 5. Example of a measured spectrum of radiation from an X-ray tube with a Mo anode reflected from a LiF crystal.

11. Remove the aluminum filter. Enable manual goniometer angle adjustment. To do this, ensure that the "MAN" LED is lit above the fifth button ("AUTO-MAN") on the control panel (if "AUTO" is lit, press that button), and then press the third button on the control panel (see Fig. 2). Above that button there are three light-emitting diodes (LEDs), each of which indicates one of the three manual goniometer angle adjustment modes: (a) only the detector is rotated (symbol ""), (b) only the crystal is rotated (symbol ""), (c) the detector and the crystal are rotated simultaneously so that the rotation angle of the detector is twice the rotation angle of the crystal (symbol ""). In this experiment, only the last mode is required. If the corresponding LED is not lit and one of the other two LEDs is lit instead, press the same (third) button again.

12. Mount the LiF crystal in the goniometer. The crystal is kept in a black plastic box, which, together with other small accessories, is stored in the accessory compartment located on top of the X-ray apparatus (see Fig. 6a–d). After mounting the crystal in the goniometer, turn the adjustment knob until the crystal and the detector are in the position corresponding to a glancing angle θ = **9.5**°. The angle is shown at the top of the digital display (see Fig. 1). The crystal must be mounted in the goniometer as shown in Fig. 7.

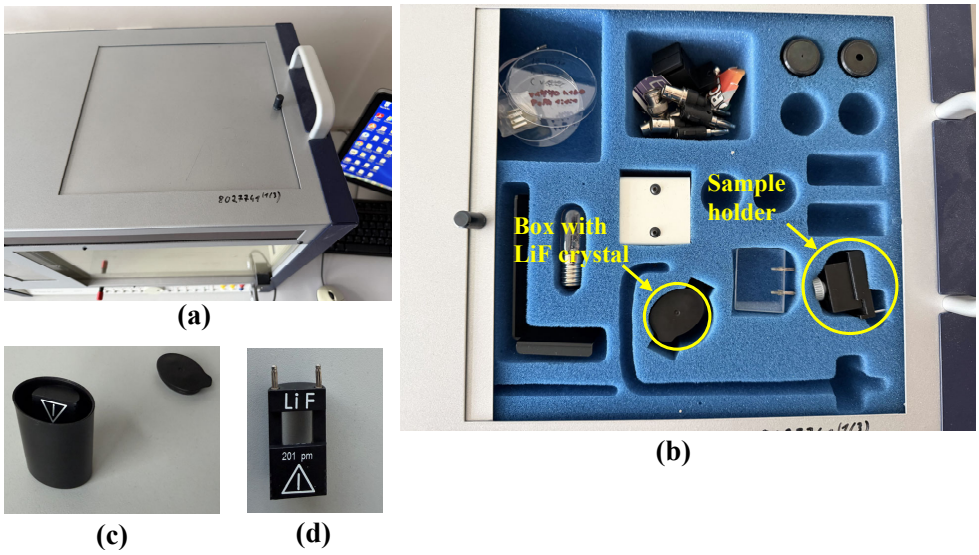


Fig. 6. Accessory compartment and lithium fluoride crystal: (a) cover of the accessory compartment, (b) location of the LiF crystal and sample holder in the accessory compartment, (c) opened box of the LiF crystal with the crystal inside, (d) LiF crystal.

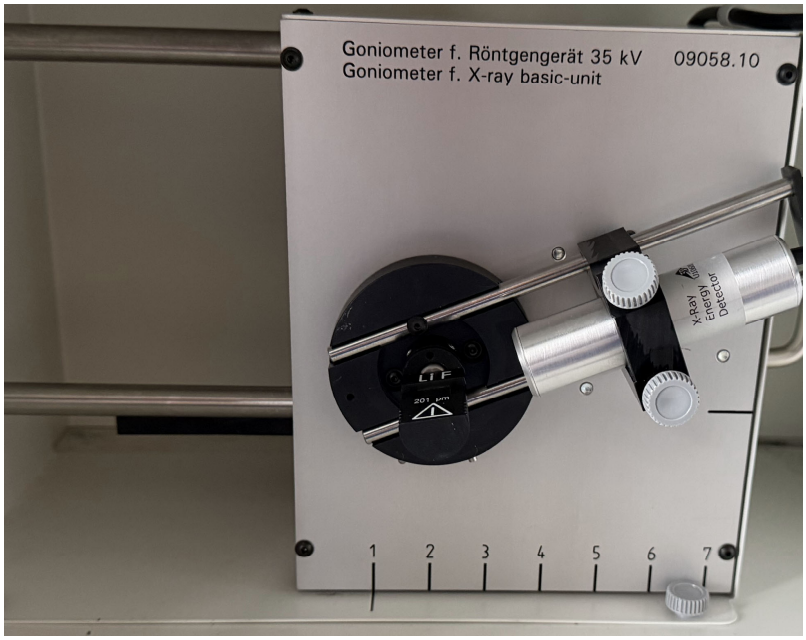


Fig. 7. Working position of the LiF crystal and detector.

13. Turn on the X-ray tube anode voltage. Start pulse counting in the "*MCA Lab*" program. Increase the glancing angle θ from **9.5**° to **10.5**° in steps of 0.1° and find the value of θ corresponding to the highest count rate. The current count rate is displayed in the "*MCA Lab*" window below the graph, before the words "*Counts/s*" (e.g., see Fig. 5). Record the above-mentioned value of θ, return the crystal and detector to that position, clear the current spectrum (using the menu command "*File* > *New*", declining to save the current spectrum), and perform a single spectrum measurement of **3** min duration. Since only radiation of a single wavelength is reflected from the crystal (Bragg condition), a single peak corresponding to the first-order reflection of the Mo K_α line from the LiF crystal should

be visible in the spectrum (e.g., see Fig. 5). After completing the measurement, turn off the anode voltage. Save the spectrum data to a `*.mca` file (see step 10).

14. Remove the LiF crystal from the goniometer. Place the crystal back into its box as shown in Fig. 6c, then close the box and put it back into the accessory compartment of the X-ray apparatus (see Fig. 6b). In place of the crystal, mount the X-ray fluorescence ("XRF") sample holder in the goniometer. The sample holder is stored in the accessory compartment (see Fig. 6b). The holder is shown in Fig. 8a. Samples are clamped by the sliding (upper) part of the holder, which must then be secured with a screw (see Fig. 8b–d). The silver and tin samples are not specifically adapted to this holder, so mounting them requires greater care. Most importantly, clamp the samples firmly enough so that they do not slip out of the holder while measuring the spectra.

15. Rotate the sample holder and the detector so that the glancing angle θ is equal to 75° (see Fig. 9). In this position, the angle between the primary radiation beam and the direction of the recorded radiation is equal to $2\theta = 150°$.

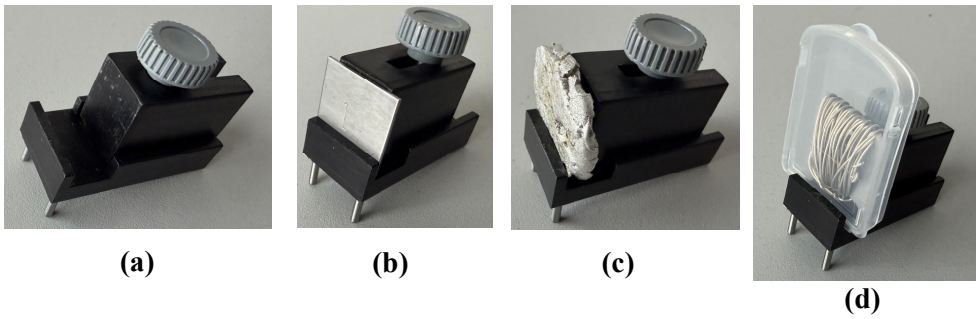


Fig. 8. X-ray fluorescence sample holder and examples of mounting various samples in it: (a) holder without a sample, (b) holder with a mounted stainless steel plate, (c) holder with a tin solder sample, (d) holder with a silver wire sample (the wire coil is inside a flat transparent plastic box).

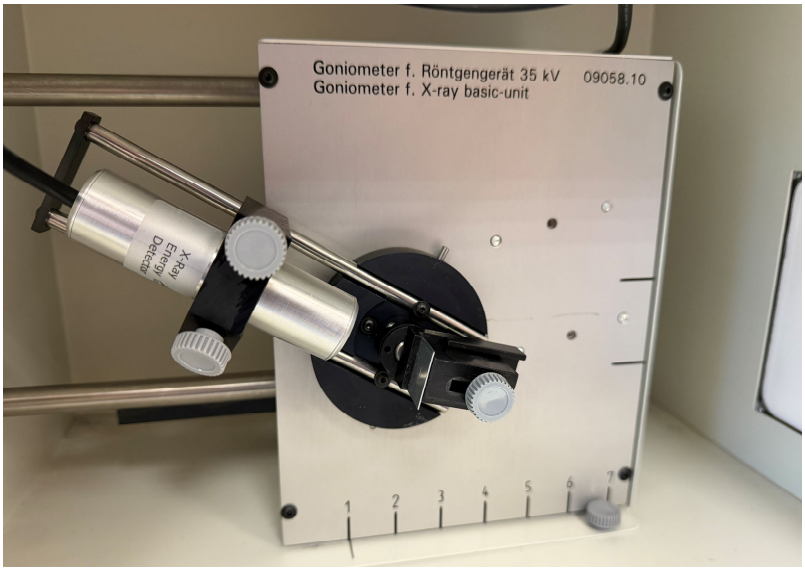


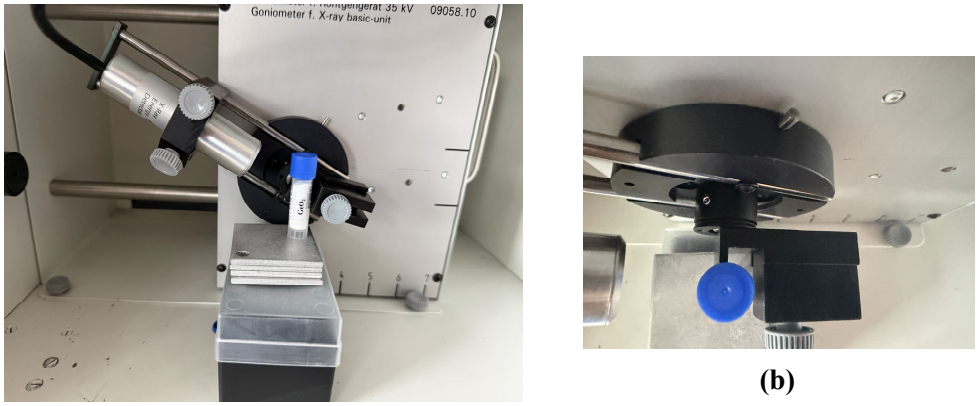
Fig. 9. Working position of the detector and sample when measuring an X-ray fluorescence spectrum with the sample clamped in the XRF sample holder (in this example, the sample is a zinc plate).

16. Place the XRF samples one after another into the holder and perform one XRF spectrum measurement of 3 min or 10 min duration with each of them. If a sample is in powder form, place the plastic vial with the powder onto the stand right next to the sample holder as shown in Fig. 10 (a plastic box with four 3 mm thick aluminum plates stacked on top serves as the stand). Using the "*MCA Lab*" menu command "*File > Save As...*", save each spectrum's data to a separate `*.mca` file (see step 10). The anode currents and measurement durations corresponding to the different samples are listed in Table 1 (after changing the anode current with the adjustment knob, always press the "ENTER" button on the

X-ray apparatus, as explained in step 3; otherwise, the new value will not be set). All samples are shown in Fig. 11. In that photograph, the samples are arranged in the same order as in Table 1. The stainless steel, copper, and zinc plates are labeled with the numbers "1", "2", and "7", respectively.

Table 1. XRF samples and spectrum measurement conditions

No.	Sample	Anode current (mA)	Measurement duration (min)
1	Stainless steel plate (≈ 70 % Fe + Cr)	0.75	3
2	Copper (Cu) plate	0.5	3
3	Zinc (Zn) plate	0.5	3
4	Germanium dioxide (GeO_2) powder	0.75	3
5	Potassium bromide (KBr) powder	1	3
6	Strontium sulfate ($SrSO_4$) powder	1	3
7	Silver wire (92.5 % Ag + 7.5 % Cu)	1	10
8	Tin solder (> 97 % Sn + Cu)	1	10



(a) **(b)**

Fig. 10. Position of the plastic vial with powder and the sample holder when the XRF sample is in powder form: (a) front view, (b) top view.

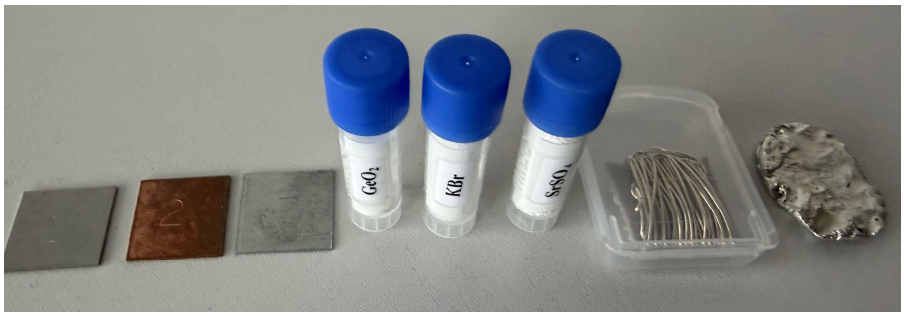


Fig. 11. Samples for the X-ray fluorescence investigation.

17. Turn off the X-ray apparatus. Place the sample holder back into its spot in the accessory compartment of the X-ray apparatus (see Fig. 6b). Put the four aluminum plates into their box (it must be left on the table next to the X-ray apparatus). Save all ten `*.mca` files created while performing steps 10, 13, and 16 to a USB flash drive or store them in cloud storage (e.g., via e-mail) for later analysis. In addition, open each of those files with the "*MCA Lab*" program (they must be opened not from Windows File Explorer, but using the "*MCA Lab*" menu command "*File* > *Open*" or the "*F3*" key, because otherwise only the initial "*MCA Lab*" window without data would open), optimize the axis limits (the maximum channel number should be approximately 500 or smaller), and, using the "*MCA Lab*" menu command "*File* > *Export Graph* > *Save as type*", save each graph as a "Windows Metafile" format file (`*.wmf`). Insert these files into a single Microsoft Word document (at least two graphs per page) using the Word menu command "*Insert* > *Picture* > *From File...*". Add brief figure captions so that it is clear what is shown in each of the ten graphs. Print the Word document. **Note:** The printer currently used in the

laboratory is not a network printer. That is, it is connected not directly to the computer network, but to a computer that is connected to the computer network. Before printing, make sure that this computer is turned on.

18. Place the XRF samples back into their original packaging and return them to the lab supervisor or laboratory assistant.

19. Have the sheets with the printed graphs signed by the lab supervisor or laboratory assistant.