

ADVANCED PHYSICS LABORATORY

XRF

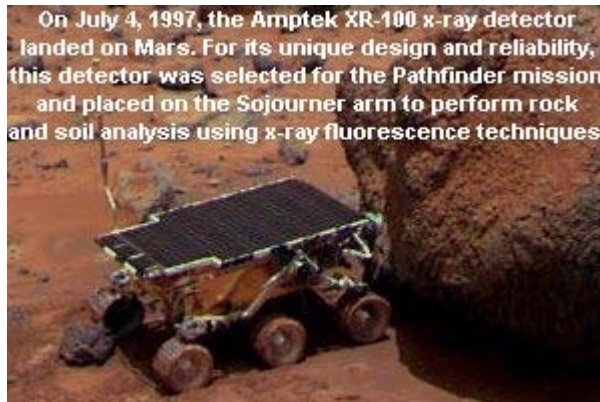
X-Ray Fluorescence: Energy-Dispersive Technique (EDXRF)

PART I

Introduction and Compulsory Exercises

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On July 4, 1997, the Amptek XR-100 x-ray detector landed on Mars. For its unique design and reliability, this detector was selected for the Pathfinder mission and placed on the Sojourner arm to perform rock and soil analysis using x-ray fluorescence techniques.



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Objectives

1. To study the excitation of x-rays in an x-ray tube.
2. To study production of an x-ray fluorescence spectrum in a solid sample with primary x-rays, emitted by an anode of the x-ray tube.
3. To study physical processes in a Si-PIN photodiode used as a detector of the x-ray spectrum.
4. To study functioning of a multichannel analyzer of the x-ray spectrum and spectra processing.
5. To study foundations of quantitative element analysis of a solid sample.

All the above is very difficult to cover completely during the time assigned for one experiment in the labs. Instead, we offer you a number of optional experiments from which you can choose at least one and the most interesting for you after the discussion with your supervisor. However, the fundamentals of the x-ray fluorescence phenomenon as well as the basic knowledge of the spectrum excitation, acquisition and interpretation must be studied by all students taking this experiment. *The two compulsory exercises with all questions answered should be accomplished by the date of the Progress Check.*

Glossary

Attenuation coefficient – a natural logarithm of the ratio of the emergent, I , and incident, I_0 , radiation intensities I / I_0 divided by either the depth of the radiation penetration (linear attenuation coefficient) or the surface density (mass absorption coefficient).

Bremsstrahlung – continuous spectrum produced by a charged particle moving with deceleration.

Continuous spectrum – a spectrum formed by photons with unresolved energies in a wide range.

Detection limit – a lowest amount of chemical element that can be found with probability of 99%.

Detector resolution – possibility to distinguish two overlapping peaks in the spectrum; depends on the ratio of the distance between the two peaks and FWHM; usually accepted as a value of FWHM.

Efficiency of a detector – the ratio of the number of photons participated in creation of a useful signal in the detector to the total number of photons incident on the detector surface.

Energy-dispersive technique – the technique used to simultaneously detect photons of the line spectrum in a wide range of the photons' energies.

Fluorescence – emission of photons by a substance induced by absorption of primary photons.

FWHM – full width at half maximum of the peak usually measured in electronvolts.

Ionizing radiation – particles or electromagnetic waves whose energy is sufficient to ionize a neutral atom or a molecule.

Line spectrum – a spectrum formed by photons with specific quantized energies only.

Matrix effects – The combined effect of all components of the sample other than the analyte on the measurement of the quantity of the analyte. The two main matrix effects are: (a) the attenuation of characteristic peak intensity due to inelastic scattering of photons, emitted by atoms of one chemical element, on atoms and electrons of other components; and (b) the enhancement of characteristic peak intensity due to additional excitation of atoms of one element by photons, emitted by other components.

Peak intensity – a value proportional to the total number of photons with same energy registered by a spectrometer and exposed as a bell-shaped curve called the *peak*.

Quantitative analysis – determination of amount of each chemical element of a sample.

Spectral series – series of spectral peaks produced by electron transitions from different energy levels to one specific energy level; K-series corresponds to all transitions to the ground state.

Spectrum – a function of a number of photons versus their energy, or versus their wavelength.

Spectrum background – a component of a spectrum which does not belong to the peak of interest; may be formed by bremsstrahlung radiation or by the tails of adjacent peaks.

X-ray tube – a kind of a vacuum tube with a filament in a cathode emitting electrons, and a pure metal plate as an anode, producing radiation in the x-ray range of electromagnetic spectrum.

I. Introduction and theoretical background

Electromagnetic radiation with energies in the range from hundreds of electronvolts to several megaelectronvolts (see Fig.1) is called x-ray radiation, or simply x-rays. X-rays appear in an experiment as a result of either the accelerated motion of the charged particles (x-ray tube, synchrotron radiation), or as a result of a transition of an atom from one of the excited states to the state with lower energy. X-rays, produced by the decelerated charged particle, form a continuous spectrum, or bremsstrahlung (see the Glossary).

The transition of the atom to a state with lower energy is accompanied by emission of a photon with the energy equal to the difference of energies of the initial and the final state. All transitions, permitted by selection rules for the specific atom produce a line spectrum, unique to this atom. This line spectrum is called *characteristic spectrum* of the chemical element. Intensity of a characteristic peak is proportional to the probability of the corresponding transition.

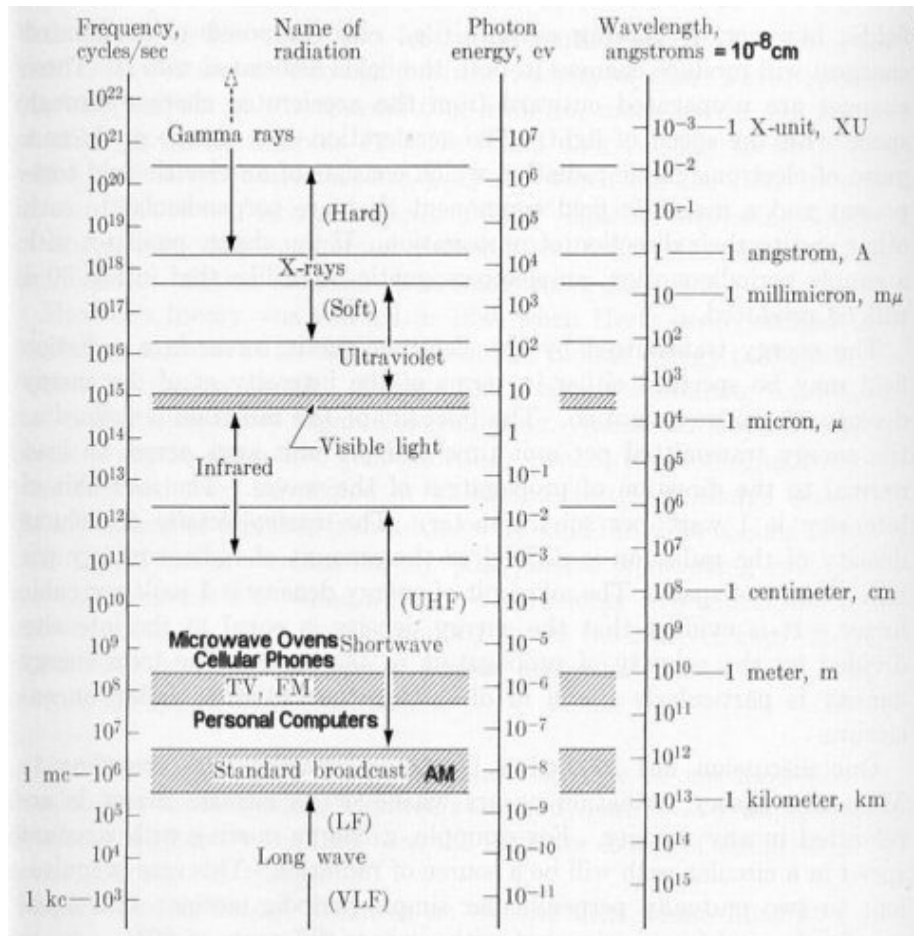
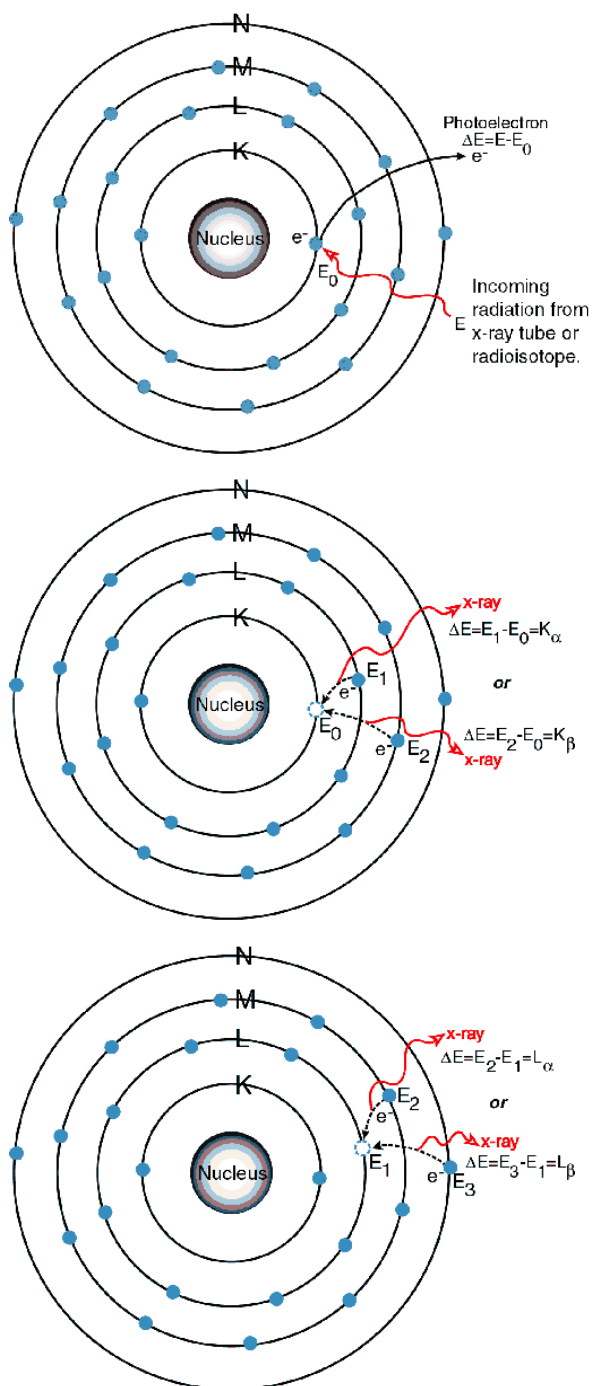


FIG.1. Electromagnetic spectrum

According to the principal quantum number n , electron shells in x-ray spectroscopy are named as follows: $n = 1$ indicates the K-shell; $n = 2$ indicates the L-shell; $n = 3$ indicates the M-shell, and so on. The x-ray fluorescence emission induced by photoelectric effect is shown in Fig.2 for an atom of titanium ($Z = 22$), whose K-shell electron acquires sufficient energy to escape from the atom.

All spectral lines that appear as a result of the electron transition to some particular shell are called *spectral series*. Transitions to the K-shell form the K-series; transitions to the L-shell form the L-series, etc. The electron shell with $n > 1$ contains subshells with different quantum numbers l (orbital) and j (total angular momentum). The subscript Greek letter with a number (α_1, β_2) indicates the atomic subshells participating in the electron transition (see Fig.3). Ionization of the K-shell, which has the highest absolute value of the potential energy, compared to the other shells in the atom, may, with different probability, result in a variety of electron transitions and consequently cause the emission of photons of all spectral series in a sample with lots and lots of identical atoms.

Energies of emitted photons for all chemical elements are listed in APPENDIX A. Uniqueness of the characteristic spectrum of an atom makes it a powerful tool to determine presence of the particular atoms in a sample or to study the interaction between external fields and the atom.



Photoelectric effect on the K-shell

An electron in the K-shell absorbs a photon of the primary x-ray beam and becomes free, while the atom gains a vacancy in the K-shell.

The K lines production

An electron from the L or M shell “jumps in” to fill the vacancy and in turn, produces a vacancy in the L or M shell. In the process, the atom emits a characteristic photon from the x-ray range of electromagnetic spectrum, unique to this chemical element,

The L lines production

After a vacancy is created in the L shell by either the primary beam photon or by the previous event, an electron from the M or N shell “jumps in” to occupy the vacancy. In this process, the atom emits a characteristic photon, unique to this chemical element, and a vacancy in the M or N shell is produced.

FIG.2. Ionization of the K-shell electron in the atom of Ti by photoelectric effect and emission of characteristic photons of different spectral series as a result of electron transitions.

X-ray fluorescence provides a rapid non-destructive means for both qualitative and quantitative analysis. A wide range of materials varying in size and shape can be studied with minimal requirements for sample preparation. Detection sensitivities as low as one part in a million can be obtained with this technique.

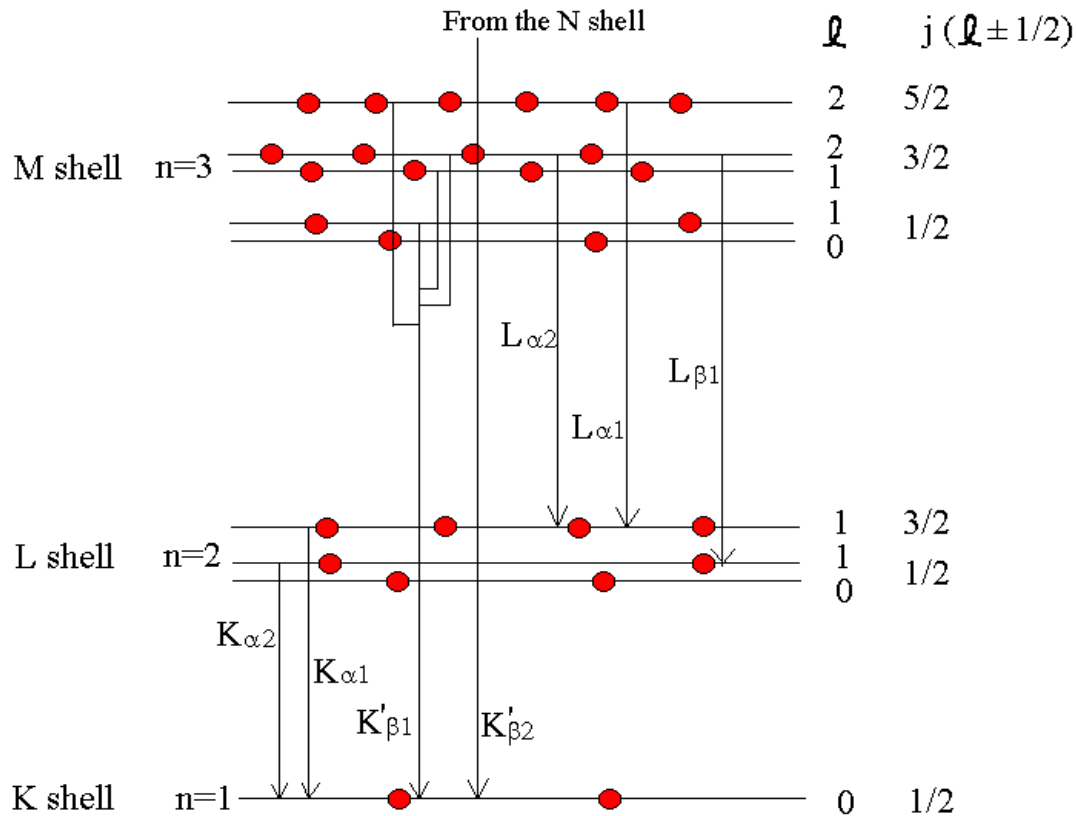


FIG.3. Electron transitions and emitted spectral lines in the atom after the K-shell ionization.

II. Experimental setup

X-ray fluorescence is the emission of characteristic x-rays following the excitation of atoms by ionizing radiation. Hence, the apparatus producing the x-ray fluorescence must include a source of ionizing radiation (an x-ray tube or radioactive source), a sample whose x-ray spectrum is studied, and the detection system to acquire and record the spectrum, or a spectrometer.

Six different processes of x-ray excitation can be considered in our experiment:

- 1) Production of continuous spectrum by electrons decelerating in an anode of the x-ray tube.
- 2) Excitation of characteristic spectrum of the anode of the x-ray tube by direct ionization of its atoms by the incident electrons.
- 3) Excitation of characteristic spectrum of the target (a sample under investigation) by the primary x-rays emitted by the anode of the x-ray tube (photoelectric effect).
- 4) Excitation of characteristic spectrum of the target by the primary x-rays from the x-ray tube or from a radioactive isotope used as a primary source (photoelectric effect).
- 5) Characteristic x-ray fluorescence of atoms of one component of the sample generated by the characteristic spectrum of the other components (secondary excitation, matrix effects).
- 6) Coherent and incoherent scattering of photons, produced in the x-ray tube and in the sample, on the atoms of the sample (Compton and Rayleigh components of the background spectrum).

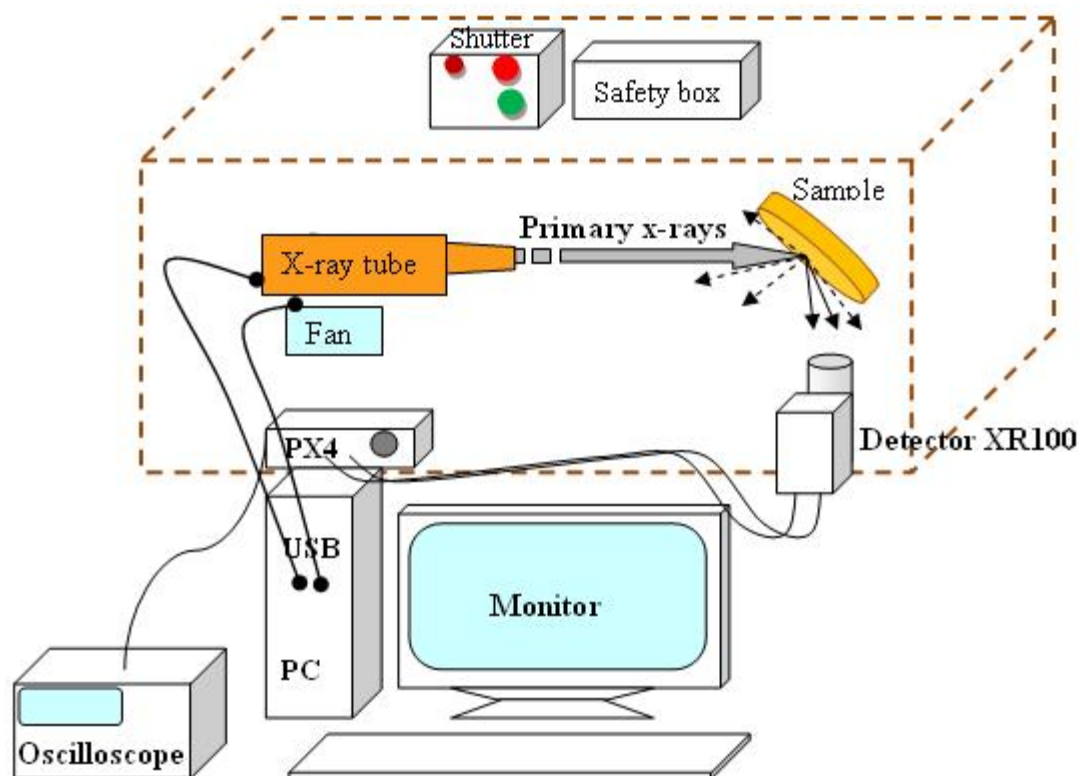


FIG.4. Schematic diagram of the apparatus

Figure 4 shows schematically the arrangement of experimental equipment used in our laboratory. Connection of the Shutter Box and Safety Box with the built in shutter and door locks are not shown for simplicity. In some experiments, a radioactive isotope is used, placed as a sample in front of the detector. The x-ray tube is not turned on in the experiments with radioactive sources.

II.1. Safety Box and Shutter

Figure 5 shows the electric circuit that includes the Safety Box, the Flashing Light Box, the X-ray tube and the safety switches of the shutter and the door.

The safety box and the shutter control box circuit is used to:

- 1) disconnect the x-ray tube power supply when both the door of the sample chamber and the shutter are open;
- 2) turn on the Flashing Light Box when the x-ray tube is operating;
- 3) identify whether the shutter is open or closed;
- 4) replace samples inside the chamber without turning off the x-ray tube.

The Safety Box and the Shutter Control Box must be turned on simultaneously. Take the key in MP 250. The Safety Box is turned on by turning the key clockwise and then pushing the red button on its top. Press the power button on the front panel of the Shutter Box. Check whether the Flashing light box has started to show that the circuit is connected and you can proceed with turning on the x-ray tube. If the light box has not started flashing, check the chamber door lock and the shutter position. If the door is unlocked while the shutter is open (the shutter handle is completely out), the circuit is disconnected.

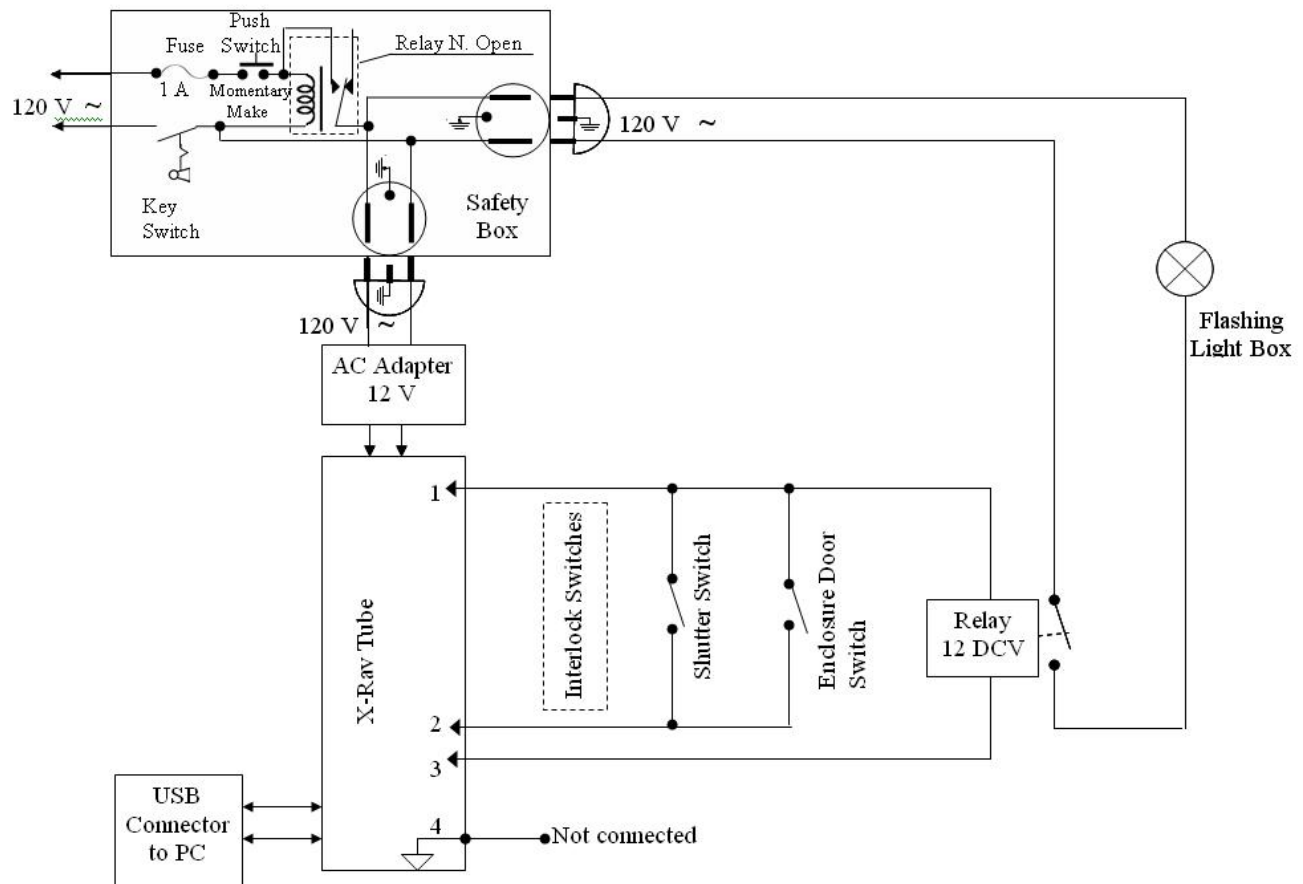


FIG.5. Safety box and shutter control box circuit.

II.2. Primary X-ray source

The Mini-X X-ray tube is shown in Fig.6a. The sizes of the miniature x-ray source are just 18 cm x 5 cm x 2.5 cm. It is turned on/off and operated with computer via USB connection. A beeper warns the user when x-rays are produced.

Some specifications

Tube current:	5 μ A min / 200 μ A max (4 W max)
Tube voltage:	40 kV max
Continuous power:	4 W max
Anode (transmission):	Ag
Be window:	127 μ m
Cooling:	Air cooled

Mini-X Control software controls voltage and current.

A heated filament of a cathode emits electrons by thermionic emission. The electrons are accelerated by voltage of tens of kiloelectronvolts applied between the cathode and the plate of the anode. When energetic electrons are made to slow down by collisions with atoms in the anode of the x-ray tube, the principal means of energy loss is that of radiation or Bremsstrahlung. The radiation spectrum is characterized by a continuum (Fig. 6b) with the maximum photon energy determined by the incident electron energy, E_e , so that $E < E_e$, where E is the emitted photon energy. Superimposed on this continuum will be strong peaks due to fluorescence of the anode material, whose atoms are ionized by direct impact of speedy electrons.

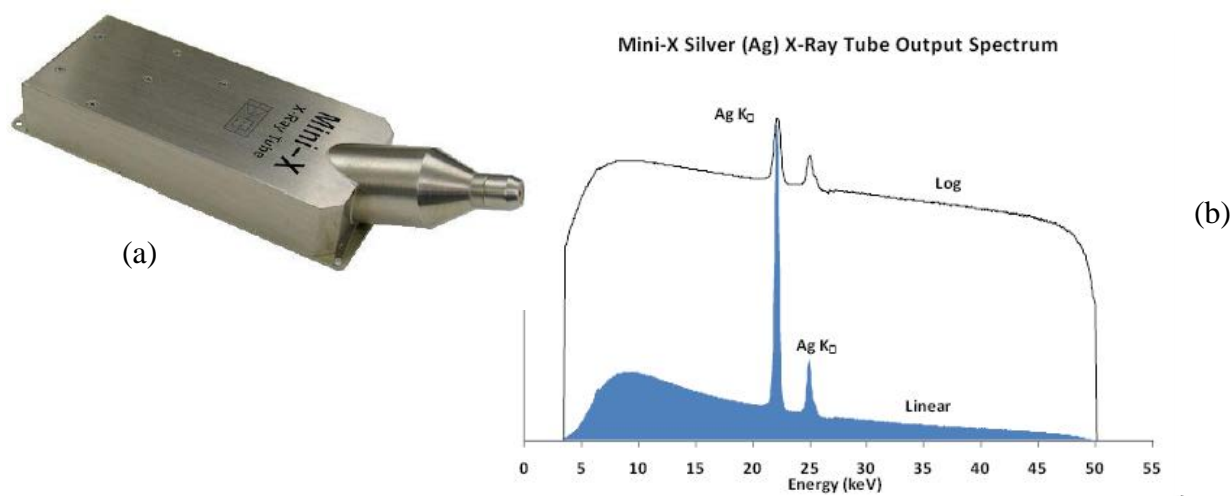


FIG.6. (a) MiniX miniature X-ray tube with built-in power supply.
 (b) X-ray spectrum produced by the x-ray source with silver anode at 50 kV.

Since the photon endpoint follows the electron energy, fluorescence can be restricted to a range of atomic numbers by choosing the accelerating voltage with value in kilovolts that exceeds the excitation energy of the electron shell, where the vacancy should be produced, in kiloelectronvolts. For example, the voltage of 25 kV can excite the K-series of atoms of palladium ($Z = 46$) with the excitation energy of the K-shell 24.4 keV, but would be useless to produce the K-series of silver ($Z = 47$) with the excitation energy of the K-shell 25.6 keV.

II.3. Sample holder

A sample holder is an optical lens mount that can rotate a target in a rotational platform to vary the angles of incident and exit x-ray beam. The arrangement of the x-ray tube, the cooling fan, the target and the detector inside the sample chamber is shown in Fig. 7. All standard samples are either produced in a shape of a thin cylinder or a circular plate stuck to a plastic ring.

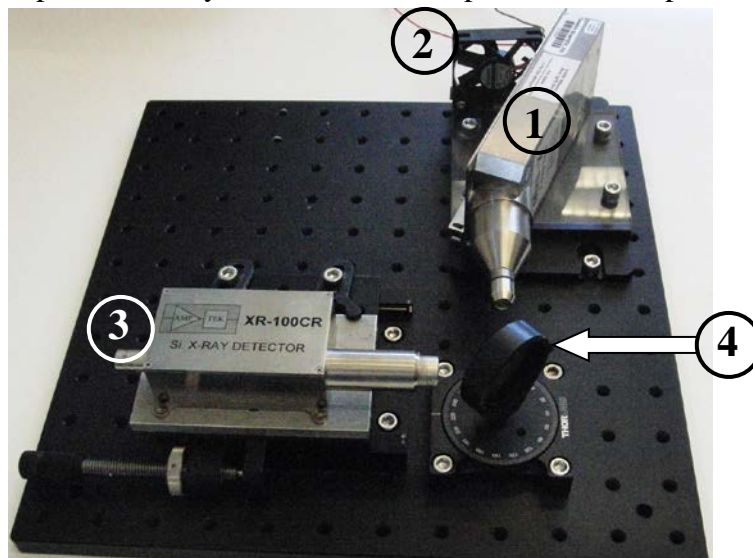


FIG.7. The arrangement of the x-ray tube (1), the cooling fan (2), the Si-detector (3) and the target in the sample holder (4) inside the sample chamber. [Wires, internal enclosure of the x-ray tube, and a shutter are not shown].

II.4. Detector and processor

The Amptek Inc. X-ray detectors XR-100CR are using Si-PIN diodes assembled as shown in Fig.8.

Some specifications of XR-100CR

- 2-stage cooler
- 13 mm² / 500 μm
- 1.0 mil (25 μm) Be
- 174 - 200 eV (according to the Manufacturer)
- P/B Ratio: 550/1 (4000/1 with external collimator)
- [The Peak to Background (P/B) Ratio is the ratio of the counts at the 5.9 keV peak to the counts at about 2 keV].

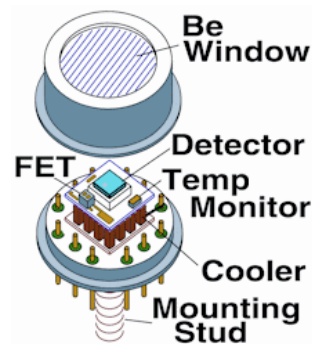


FIG.8. A unit containing the Si PIN diode and a Peltier cooler evacuated and protected with a Beryllium window

The Amptek PX4 (Fig.9) is an interface between Amptek’s XR100 series of X-ray detectors and a personal computer with data acquisition, control, and analysis software. The PX4 includes three major components: (1) a shaping amplifier, based on a digital pulse processor, (2) a multichannel analyzer - MCA, and (3) power supply.

The detector box, the PX4, a computer and possibly an oscilloscope are connected as on Fig. 10. Before starting measurements, verify that the connection is done correctly and that the PX4 is OFF. The light on the “ON/OFF” button which is located on the front panel of PX4 should be off.



FIG.9. PX4 Interface (front and back).

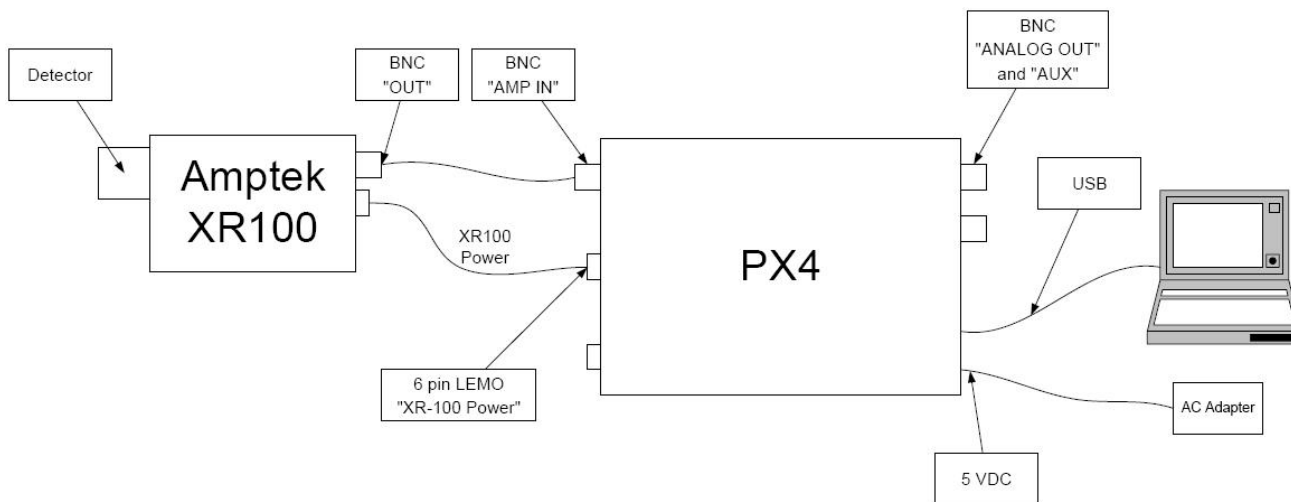


FIG.10. Spectrometer parts and connections


II.5. Software

The PX4 is provided with the ADMCA display and acquisition software application. This software controls all data acquisition parameters and can represent the output data in different formats. The Live Spectrum mode gives an image of the x-ray spectrum of the target during the preset time interval with steadily growing number of counts in each channel of the multichannel analyzer (MCA).

When the data acquisition is finished, the image of the spectrum can be saved in three formats (it is recommended to save every good spectrum immediately after the MCA stops counting): in the *.mca format; as a Bitmap Image; and as a table in the *.txt format. The *.mca file is possible to modify only on the lab computer, where the ADMCA software is installed. It gives a lot of tools for the spectrum processing. The *.bmp image can be printed out for a spectrum presentation. The *.txt file can be processed with the MATLAB or any other program for computer analysis of the spectrum and uncertainties calculations.


II.6. Set up procedure for the X-ray spectrometer (can be skipped if the default settings are applicable; verify it with the experiment supervisor)

1. Press the power button labeled “ON/OFF,” which is located on the front panel of the PX4, and hold it in for less than two seconds. You should hear a single beep.

2. Click on the ADMCA icon  in Windows Menu at the lower left corner of the PC screen.

3. In the “Starting ADMCA” box, click “Connect.” The PX4 unit should now be connected to the software. The USB symbol located at the bottom right corner of the ADMCA window should be green.

4. Next, the proper configuration must be selected for the detector. The Amptek detectors configurations are identified by the detector material, dimensions and the cooler type. Find these data for the correct detector configuration on p.10 of this Manual in section II.4.

5. Click on the acquisition setup button  of the toolbar. The dialogue box “DPP Properties” shown in Fig. 11 will appear. In the “General” window, select the configuration in the drop-down menu under the Read Amptek Detector Configuration line.

6. Once the correct configuration is selected, the indented grey area above the dropdown menu should read “Loaded Si 13mm2/500um 2-Stage Cooler configuration”. Press “Apply”.

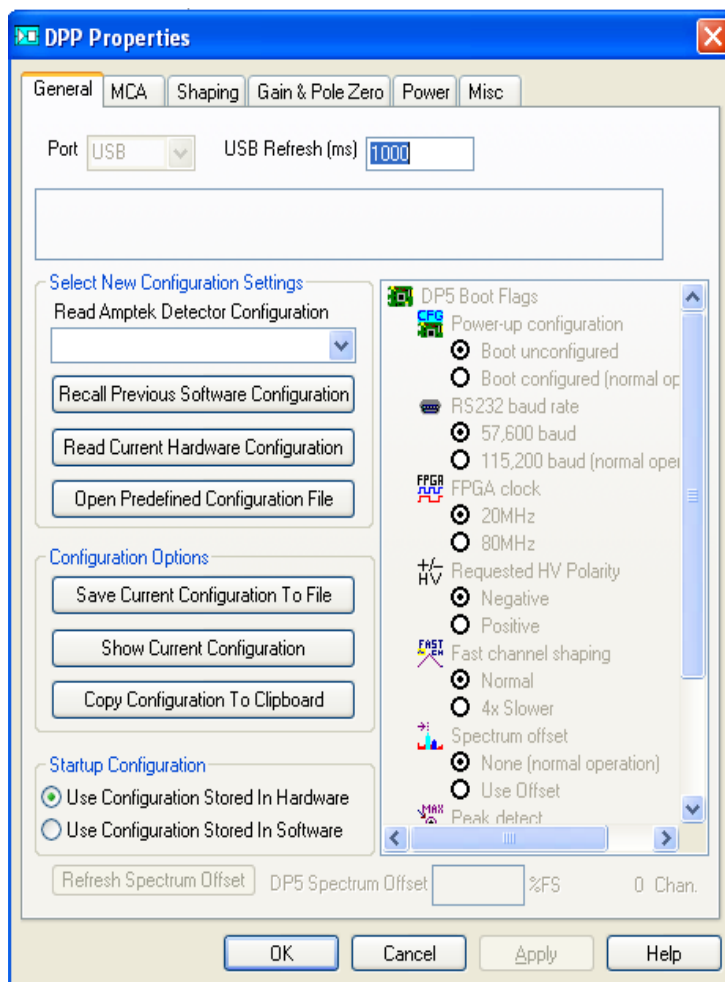


FIG.11.

7. It takes up to 5 minutes for the Amptek detector to stabilize after the configuration has been set.
8. Now, some other options can be selected in the MCA window (Fig.11). You can set the full number of channels of the MCA and the time of acquisition. Begin with 1024 channels. After you have changed settings in the MCA window, press “Apply” button.
9. Open the “Gain and Pole Zero” window. A common task is to change the energy range of a system. This is done by adjusting the gain of the amplifier. A lower gain corresponds to a higher energy range, and a higher gain to a lower energy range. For example, in some Amptek’s digital systems a gain of x100 may correspond to a 15 keV full scale and a gain of x50 to a 30 keV full scale. To adjust the energy range it is always the gain of the system that must be changed. It is not enough to change the software calibration only. If the software’s calibration is changed without the amplifier’s gain changing appropriately, the software will be displaying the wrong energy range and peaks will be incorrectly identified. We recommend to begin the data acquisition with the gain 30. Press “Apply”. Later, you may try to change the gain for the same preset time and the same total number of channels to see the difference. However, if your task is to compare a number of spectra of different samples taken under same conditions, the same gain must be chosen and set up for all the spectra.
10. Adjust some of the thresholds which prevent low-end noise and other unwanted counts from affecting the spectrum. This can be done automatically by the software. To find the correct settings start the acquisition by pressing the traffic light button at the top bar of the ADMCA screen. The green traffic light actually means same as in the street, i.e. that you can start. It is red when the data acquisition is in progress.

Follow these steps in the “Gain & Pole Zero” window:

- Be sure to REMOVE any sources from in front of the detector. Press the button “Start/Stop Acquisition” in same window.
- Click the “Tune Input Offset” button. The software will automatically set the input offset for the current Gain setting. Click on “Apply”.

11. Return to the MCA window. Click the Tune Slow Threshold button on the toolbar. Press “Apply”.
12. Open the “Shaping” window. Click the Tune Fast Threshold button on the toolbar. Steps 11 and 12 automatically set the slow threshold (LLD) and the fast threshold. Press “Apply”. Stop data acquisition. It is necessary to readjust the thresholds whenever the gain is changed.
13. Place the radioactive isotope ^{210}Pb in the sample holder in front of the detector as close to the detector as possible. Start the data acquisition. Open the window “Misc” of the DPP Properties dialog box. Press the button Oscilloscope. The growing signal shows that the data acquisition is going on. Select the “OK” button to exit the DPP Properties dialogue box.

Click "Show Current Configuration" to view the current detector and analyzer settings. Click OK to exit the box if the settings are same as in Fig.12. If they are not, or if you find problems with spectrum data later, go to APPENDIX C to correct the settings.

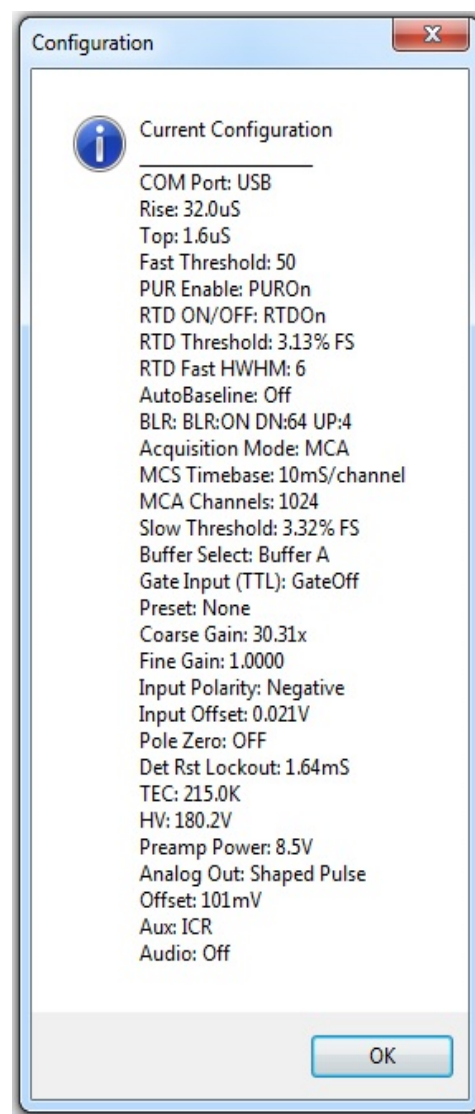


FIG.12. Correct spectrometer configuration and

III. Compulsory exercises

Two compulsory exercises should be submitted for Progress Check.

Exercise 1. Calibration of the multichannel analyzer with a radioactive sources.

In this exercise, the x-ray tube is not used. Instead, a sample with a radioactive isotope is placed directly in front of the entrance window of the detector and its radiation is examined with the spectrometer. The Exercise will make you skilled working with the multichannel analyzer (MCA) and processing the spectrum with the ADMCA software. Energy for all peaks of the radioactive isotope (radioisotope) is well known. The goal of the experiment is to obtain the positions of the peaks in the spectrum of the radioisotope and calibrate the horizontal scale of the multichannel analyzer (MCA) in energy units (eV).

To accurately calibrate a spectrum, there must be at least two known peaks in the spectrum. You are recommended to use ^{210}Pb radioisotope. However, it is preferable to use two isotopes at low and high energy limits of the spectrometer between 4 keV and 40 keV.

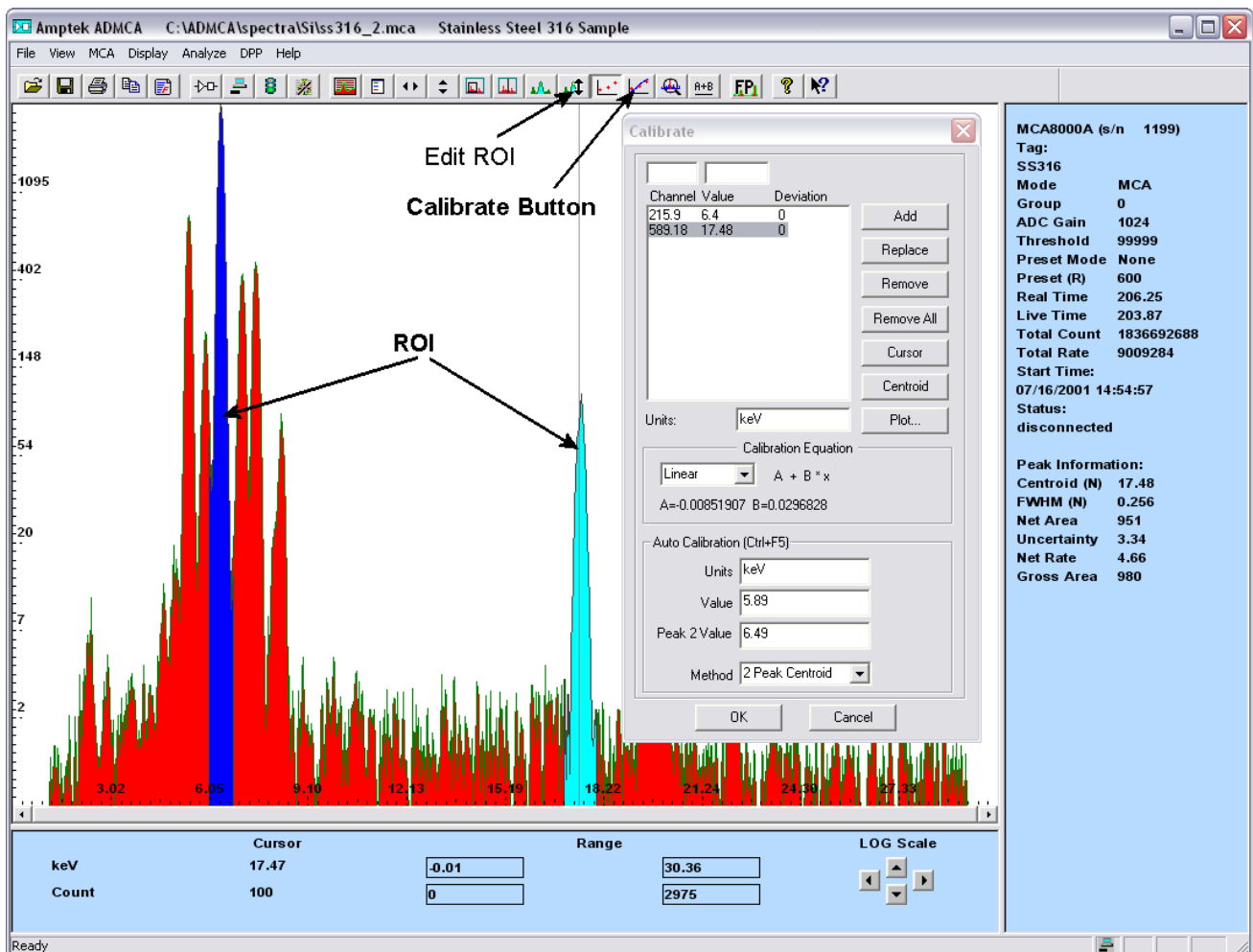


FIG.13. Calibration procedure for two chosen peaks of the spectrum with ADMCA software.

To calibrate the scale in the Amptek ADMCA software, see Fig.13 and follow these steps:

1. Chose the two peaks that will be used for the calibration and make note of their energies.
2. Mark an ROI (region of interest) around each peak. This can be done by clicking the cursor at the left base of the first peak and then holding down the “U” key on the keyboard until the

whole peak is highlighted, or by using the *Edit ROI* button on the toolbar and then clicking and dragging across the peak until it is highlighted. For more information on marking ROIs, see the Help option under Analyzing Data with the ADMCA Software → Regions of Interest and How to Define Them.

3. Now click on the toolbar button *Calibrate* to open the calibration dialog box.
4. Move the dialog box such that both peaks are visible and then click the cursor into the first peak. The peak should be highlighted and the Peak Information section on the right-hand information panel should be filled in. If the peak is highlighted and there is no peak information, then the ROI is not marked correctly. Try adjusting the start and end points of the ROI.
5. Click the *Centroid* button on the dialog box. This will enter the center position of the peak into the Channel box. Then enter the energy of that peak into the Value box, e.g. 6.4 (6.4 keV is the energy of the Fe K_{α} peak), and click Add.
6. Now click the cursor into the second peak (it will highlight). Click on Centroid, enter the second energy, e.g. 17.48, and click Add. There should now be information about the two characteristic peaks in the box.
7. In the Units box type in the energy units, e.g. keV.
8. Click OK. The scale should now be in energy. If it is not, press F7, this toggles between calibrated units and channels. If the same energy range is to be used (i.e. the gain has not changed), ADMCA can be set to load a calibration automatically on startup. Save a spectrum that has been calibrated by following the steps above. Click on the View menu and select Preferences. On the General tab in Spectrum Template enter in the path and file name of the file that was just saved. Check Load Calibration on Startup and click OK. ADMCA will now automatically load that calibration every time it opens.

In addition, a calibration can be applied from one file to another. For this procedure see the Help file (F1) under Analyzing Data with the ADMCA Software -> Calibrating a Spectrum / Applying a Calibration.

QUESTION 1.

For calibration with ^{210}Pb isotope, explain the difference in the values of the energy of the Pb L-series peaks as given in APPENDIX A and the actual highest peaks' energy of about 10.8 keV and about 13.1 keV. Basing on your reasoning, find the more precise energy of the registered peaks.

Exercise 2. Calibration of the multichannel analyzer with a set of pure metals.

This exercise is done with the x-ray tube whose primary x-rays induce x-ray fluorescence in a sample produced of a pure metal. As it was stated in the previous exercise, the software must always be calibrated in order to change the channel scale into an energy scale. There is a box in the lab room with a set of samples for calibration. To accurately calibrate a spectrum in a wide range of energies, as many known peaks as possible should be used.

Before starting the data acquisition, you must choose the samples for the most effective calibration procedure. The spectrometer allows registering characteristic peaks in the range between 2 keV and 50 keV. Using the table in APPENDIX A choose an element with the energy of its K_{α} peak close to the lower limit. We can recommend vanadium ($Z=23$, symbol of element V) with its K_{α} energy of about 5 keV. To choose the sample with the peak close to the upper limit of the MCA, you should check x-ray excitation energies in the table of APPENDIX B. The 40-keV primary x-rays can ionize the energy levels with energies under 40 keV and consequently induce x-ray fluorescence that follows the electron transition to this level.

QUESTION 2.


Explain the difference in the values of excitation energy of the K-level (K electron shell) and the K_{α} peak and write the explanation into the notebook.

Each sample, selected for calibration, will be represented by at least two K-series peaks and/or two L-series peaks (see APPENDIX A). To improve the quality of calibration, it is recommended to select three to four more chemical elements with their characteristic peaks evenly distributed between extreme characteristic peaks of the two primarily selected elements.

Examine the sample you will take for the first data acquisition: its surface must be smooth, clean and flat. Place the sample into the sample holder and give the plane of the sample orientation of 45° of the angle of incidence of the primary x-rays from the x-ray tube. This position also matches the 45° of the exit angle for the fluorescence beam from the sample to the detector (Fig.7).

Turn on procedure for the X-ray tube

With the shutter inside the sample chamber is closed in its lower position and the door of the sample chamber is securely locked, start with turning on the Safety Box and the Shutter Box as explained in section II.1 on page 7.

- Plug in the USB connector from the x-ray tube into the computer.
- Plug in the USB connector from the fan into the computer.
- Click on the icon  of Mini-X in Windows Menu at the lower left corner of the desktop and press “Start Amptek Mini-X” at the top of a small window (see Fig.14).

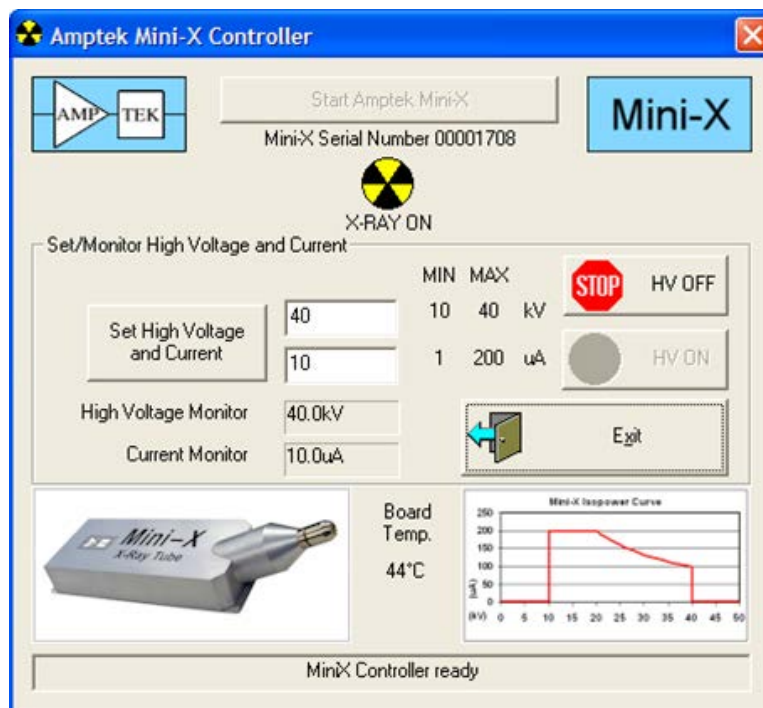


FIG.14. Turning on the x-ray tube.

- Set voltage in the range of 10 – 40 kV, and current in the range 5 – 200 μ A (see the current vs. voltage diagram on the display) with the computer keyboard and press “Enter”. You may vary the combination of voltage and current, but the power should be strongly < 4 W! If the high voltage is 38-40 kV (recommended), the current should be $\leq 90 \mu$ A.

- Use a computer mouse to click on the “HV ON” and press “Yes” in the opened window.
- The AMPTEK window starts displaying “X-RAY ON”.
- With the shutter still closed, open the door of the sample chamber and mount the sample in the sample holder. The setup must look like in Fig. 15 (a).
- Close the door.
- Open the shutter by pulling the shutter handle completely out and fix it with a key rod. Now, the machine is ready for data acquisition, and the setup looks like in Fig. 15 (b).

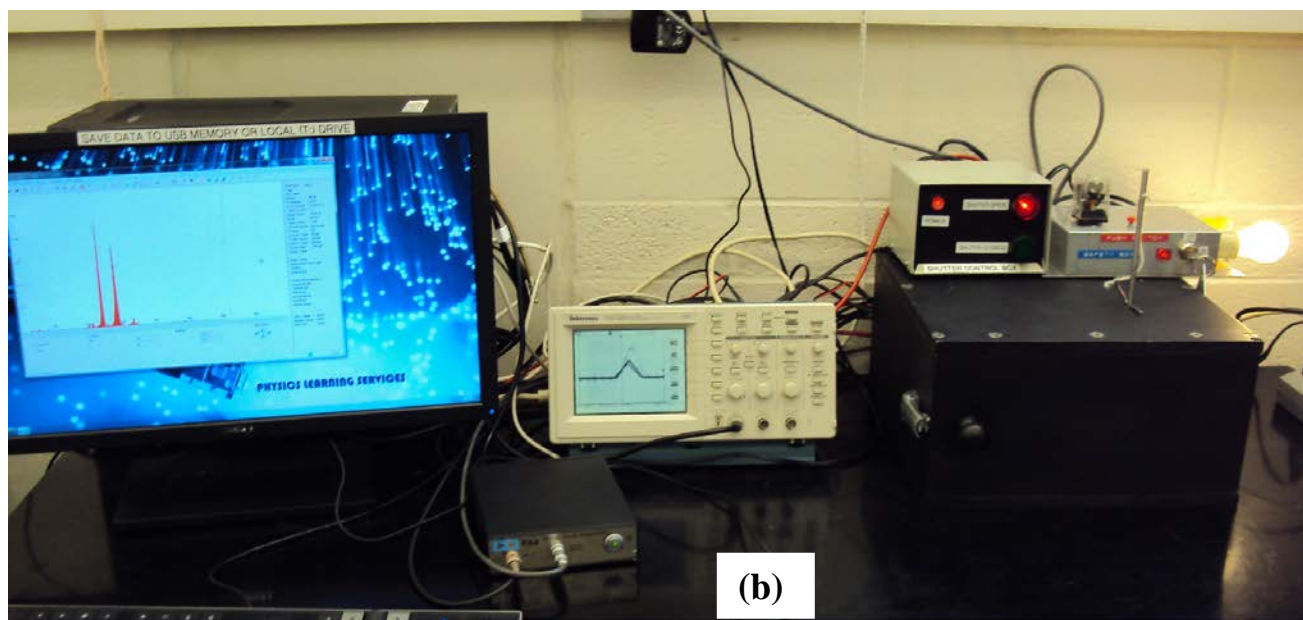
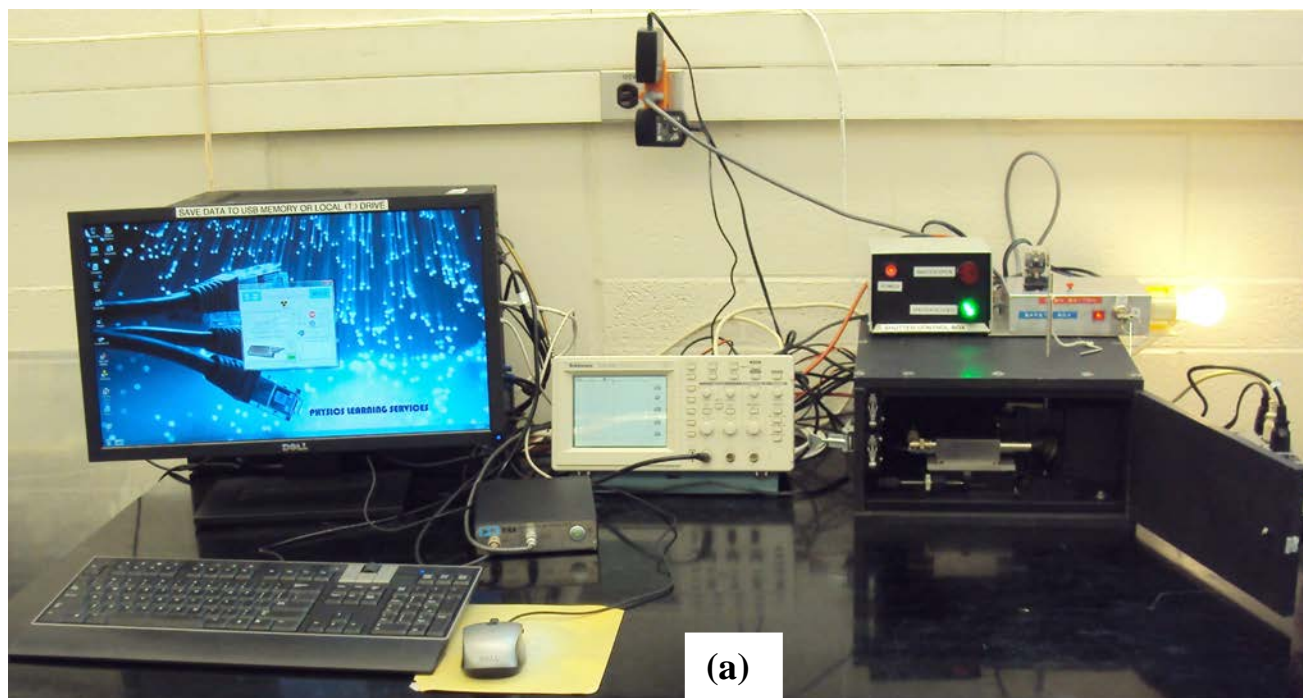


FIG.15. (a) – shutter closed, door opened for replacing a sample.

(b) – shutter opened, door closed; data acquisition has started; the oscilloscope shows strong signal of a perfect shape.

QUESTION 3.

Using the table of APPENDIX B, determine the last element that can be identified with its K-series characteristic x-ray spectrum if the x-ray tube voltage is set up to 40 kV.

After the spectrum of the first sample is taken and processed by the software, close the shutter by pulling the shutter's handle completely in. Open the door of the sample chamber, change the sample, lock the chamber door and open the shutter. The apparatus is ready for the next spectrum acquisition.

For each sample, find the position of the K_{α} and K_{β} or L_{α} and L_{β} peaks, as in the Exercise I. Using any software, apply the linear fit to the entire number of peaks from the Exercises I and II, find the slope with its error and print out the calibration line for your further experiments. Take into account that the calibration is done for specific gain, time of acquisition and the total number of channels of the MCA. As soon as you decide to change any of these settings, you will have to calibrate the analyzer for the new conditions.

QUESTION 4.

Basing on the obtained calibration, explain why you are using the average value for the energy of $K_{\alpha 1}$ and $K_{\alpha 2}$ peaks, the average value for a number of K_{β} peaks, instead of using the precise value of energy of each characteristic line according to Fig. 3 of Introduction (page 6).

If you have studied all spectra you planned, press "HV OFF". The tube is turned off. Turn off the Shutter Box and the Safety Box in the reverse order to that for the turning on procedure.

Troubleshooting

1. You may suddenly notice that there is no more counts acquiring by the spectrometer, but the beep signal still identifies that the x-ray source is on and the set time has not elapsed. More probably, this is a result of wrong settings for the x-ray source high voltage and current. The x-ray tube is overheated.
 - Turn off the x-ray tube by pressing the virtual button HV OFF (see Fig. 14).
 - Open the chamber and the shutter to cool down the x-ray tube.
 - Wait for about 5 minutes.
 - Close the shutter, close the chamber and repeat the turning-on procedure for the x-ray tube as explained above on p.15.
 - Set up a combination of the high voltage and current across the x-ray tube that result in power $P < 4W$, e.g. 40 keV and 90 μA .
 - For more help, read the Mini-X Manual in T-folder.
2. If the oscilloscope signal is not perfect (see Fig. 15b) or the spectrum looks unexpectedly poor, this is a result of software malfunctioning. Don't touch the chamber and x-ray source.
 - Turn off the PX4.
 - Turn off the computer.
 - Turn on the PX4.
 - Reboot the computer to re-load drivers.

APPENDIX A

Energies (keV) of the strongest characteristic X-ray lines

Atomic number and element	KM _{III}	KM _{II}	KL _{III}	KL _{II}	L _I N _{III}	L _I M _{III}	L _I M _{II}	L _{II} N _{IV}	L _{II} M _{IV}	L _{III} N _V	L _{III} M _V	L _{III} M _{IV}	L _{III} M _I
	K β_1	K β_3	K α_1	K α_2	L γ_3	L β_3	L β_4	L γ_1	L β_1	L β_2	L α_1	L α_2	L $_I$
Intensity	~20	~10	100	50–53	~5	50–35	20	~5	~50	~5	~90	10	20–5
11 Na	1.071		1.041										
12 Mg	1.302		1.253										
13 Al	1.557		1.487	1.486									
14 Si	1.836		1.740	1.739									
15 P	2.139		2.014	2.013									
16 S	2.464		2.308	2.307									
17 Cl	2.816		2.622	2.620									
18 Ar	3.190		2.958	2.956									
19 K	3.590		3.314	3.311									
20 Ca	4.013		3.692	3.688									
21 Sc	4.461		4.090	4.086					0.400		0.395		0.348
22 Ti	4.932		4.511	4.505					0.458		0.452		0.395
23 V	5.427		4.952	4.944		0.585			0.519		0.511		0.446
24 Cr	5.947		5.415	5.405		0.654			0.583		0.573		0.500
25 Mn	6.490		5.899	5.888		0.721			0.649		0.637		0.556
26 Fe	7.058		6.404	6.391		0.792			0.719		0.705		0.615
27 Co	7.649		6.930	6.915		0.870			0.791		0.776		0.678
28 Ni	8.265		7.478	7.461		0.941			0.869		0.852		0.743
29 Cu	8.905	8.903	8.048	8.028		1.023	1.019		0.950		0.930		0.811
30 Zn	9.572	9.567	8.639	8.616		1.107	1.102		1.035		1.012		0.884
31 Ga	10.271	10.261	9.252	9.231		1.197	1.191		1.125		1.098		0.957
32 Ge	10.983	10.978	9.887	9.856		1.294	1.289		1.218		1.188		1.036
33 As	11.727	11.721	10.544	10.509		1.386	1.380		1.316		1.282		1.120
34 Se	12.496	12.489	11.222	11.181		1.492	1.485		1.419		1.379		1.204
35 Br	13.292	13.285	11.924	11.878		1.600	1.593		1.523		1.480		1.294
36 Kr	14.113	14.105	12.650	12.598		1.706	1.698		1.637		1.586		1.386
37 Rb	14.962	14.952	13.396	13.336	2.051 ²	1.827	1.817		1.752		1.694	1.692	1.482
38 Sr	15.836	15.826	14.166	14.098	2.197 ²	1.947	1.937		1.872		1.806	1.804	1.582
39 Y	16.737	16.725	14.958	14.882	2.347 ²	2.072	2.060		1.996		1.923	1.920	1.685
40 Zr	17.662	17.649	15.770	15.692	2.503 ²	2.200	2.187	2.292	2.118	2.215	2.043	2.040	1.792
41 Nb	18.623	18.606	16.615	16.521	2.660 ²	2.336	2.319	2.449	2.257	2.357	2.166	2.163	1.902
42 Mo	19.608	19.590	17.479	17.374	2.825 ²	2.473	2.455	2.611	2.396	2.508	2.295	2.291	2.016
43 Tc	20.619	20.599	18.367	18.251	3.001 ²	2.618	2.598	2.778	2.537	2.664	2.424	2.421	2.131
44 Ru	21.656	21.637	19.279	19.150	3.179 ²	2.763	2.744	2.952	2.683	2.825	2.556	2.554	2.253
45 Rh	22.723	22.698	20.216	20.073	3.365 ²	2.915	2.890	3.132	2.835	2.992	2.698	2.692	2.377
46 Pd	23.819	23.792	21.178	21.021	3.557	3.073	3.046	3.318	2.990	3.163	2.838	2.833	2.503
47 Ag	24.943	24.912	22.163	21.991	3.754	3.234	3.203	3.511	3.151	3.342	2.985	2.979	2.634

	$K\beta_1$	$K\beta_3$	$K\alpha_1$	$K\alpha_2$	$L\gamma_3$	$L\beta_3$	$L\beta_4$	$L\gamma_1$	$L\beta_1$	$L\beta_2$	$L\alpha_1$	$L\alpha_2$	L_1
<i>Intensity</i>	~20	~10	100	53–65	~5	35–20	20	5–25	100	5–20	~90	10	20–5
48 Cd	26.095	26.061	23.173	22.985	3.960	3.402	3.368	3.710	3.319	3.525	3.134	3.131	2.767
49 In	27.275	27.237	24.209	24.002	4.169	3.572	3.534	3.915	3.487	3.712	3.288	3.280	2.905
50 Sn	28.491	28.439	25.272	25.044	4.377	3.750	3.703	4.127	3.661	3.903	3.442	3.433	3.045
51 Sb	29.725	29.677	26.359	26.110	4.609	3.932	3.884	4.345	3.843	4.101	3.604	3.594	3.189
52 Te	30.995	30.944	27.472	27.201	4.837	4.120	4.069	4.568	4.030	4.302	3.770	3.759	3.336
53 I	32.295	32.239	28.612	28.317	5.072	4.313	4.257	4.799	4.221	4.509	3.938	3.926	3.485
54 Xe	33.625	33.562	29.779	29.459	5.319	4.516	4.453	5.035	4.415	4.720	4.110	4.095	3.625
55 Cs	34.985	34.918	30.973	30.625	5.567	4.719	4.652	5.278	4.619	4.936	4.289	4.271	3.795
56 Ba	36.378	36.303	32.194	31.817	5.820	4.928	4.853	5.529	4.827	5.158	4.470	4.450	3.954
57 La	37.802	37.721	33.442	33.034	6.080	5.143	5.062	5.786	5.037	5.385	4.651	4.629	4.122
58 Ce	39.258	39.170	34.720	34.279	6.349	5.364	5.276	6.051	5.261	5.617	4.839	4.820	4.289
59 Pr	40.748	40.653	36.026	35.550	6.622	5.592	5.497	6.321	5.485	5.853	5.034	5.009	4.455
60 Nd	42.272	42.166	37.361	36.847	6.902	5.829	5.723	6.597	5.722	6.091	5.231	5.208	4.633
61 Pm	43.825	43.713	38.725	38.171	7.193	6.071	5.959	6.880	5.962	6.334	5.433	5.408	4.785
62 Sm	45.413	45.289	40.118	39.523	7.490	6.319	6.195	7.169	6.205	6.582	5.635	5.610	4.995
63 Eu	47.036	46.902	41.542	40.902	7.794	6.574	6.440	7.467	6.455	6.835	5.843	5.815	5.177
64 Gd	48.696	48.554	42.996	42.309	8.100	6.832	6.690	7.772	6.713	7.034	6.058	6.026	5.362
65 Tb	50.382	50.228	44.481	43.744	8.423	7.096	6.942	8.086	6.976	7.358	6.273	6.239	5.547
66 Dy	52.119	51.956	45.999	45.208	8.743	7.371	7.208	8.409	7.249	7.627	6.496	6.458	5.743
67 Ho	53.878	53.707	47.547	46.699	9.080	7.650	7.479	8.740	7.529	7.901	6.719	6.681	5.944
68 Er	55.681	55.491	49.128	48.221	9.425	7.942	7.752	9.078	7.813	8.180	6.951	6.906	6.153
69 Tm	57.513	57.303	50.742	49.773	9.782	8.236	8.026	9.426	8.103	8.465	7.181	7.134	6.342
70 Yb	59.374	59.157	52.389	51.354	10.148	8.531	8.314	9.781	8.402	8.755	7.415	7.367	6.546
71 Lu	61.286	61.049	54.070	52.965	10.518	8.844	8.607	10.144	8.709	9.049	7.655	7.604	6.753
72 Hf	63.236	62.979	55.790	54.611	10.890	9.153	8.896	10.517	9.016	9.348	7.891	7.837	6.960
73 Ta	65.221	64.946	57.533	56.277	11.278	9.488	9.213	10.894	9.345	9.649	8.147	8.089	7.173
74 W	67.244	66.951	59.318	57.982	11.675	9.819	9.526	11.284	9.671	9.959	8.396	8.335	7.388
75 Re	69.309	68.994	61.140	59.718	12.082	10.161	9.846	11.682	10.006	10.273	8.651	8.584	7.604
76 Os	71.416	71.077	63.001	61.487	12.503	10.515	10.176	12.092	10.349	10.592	8.905	8.835	7.822
77 Ir	73.560	73.203	64.896	63.287	12.925	10.865	10.508	12.514	10.705	10.919	9.175	9.096	8.046
78 Pt	75.751	75.364	66.832	65.123	13.363	11.231	10.844	12.944	11.073	11.251	9.439	9.364	8.271
79 Au	77.985	77.580	68.804	66.990	13.806	11.609	11.204	13.383	11.432	11.585	9.705	9.618	8.494
80 Hg	80.261	79.822	70.819	68.894	14.258	11.987	11.548	13.834	11.823	11.927	9.999	9.898	8.722
81 Tl	82.575	82.384	72.872	70.832	14.736	13.387	12.196	14.293	12.217	12.272	10.271	10.117	8.953
82 Pb	84.936	84.450	74.969	72.804	15.222	12.791	12.305	14.769	12.618	12.625	10.555	10.453	9.185
83 Bi	87.354	86.831	77.118	74.815	15.717	13.205	12.682	15.261	13.031	12.981	10.836	10.728	9.421
84 Po	89.801	89.250	79.301	76.863	16.230	13.628	13.077	15.756	13.452	13.342	11.131	11.014	9.664
85 At	92.302	91.722	81.523	78.943	16.748	14.067	13.487	16.262	13.882	13.708	11.427	11.302	9.858
86 Rn	94.866	94.246	83.793	81.065	17.262	14.511	13.891	16.777	14.323	14.079	11.727	11.595	10.085
87 Fr	97.477	96.807	86.114	83.231	17.805	14.976	14.306	17.307	14.775	14.456	12.031	11.892	10.340
88 Ra	100.130	99.432	88.476	85.434	18.352	15.443	14.745	17.848	15.238	14.839	12.340	12.196	10.622
89 Ac	102.846	102.101	90.884	87.675	18.922	15.931	15.186	18.402	15.711	15.227	12.652	12.502	10.835
90 Th	105.611	104.831	93.358	89.952	19.498	16.419	15.639	18.993	16.215	15.622	12.970	12.809	11.119
91 Pa	108.435	107.606	95.883	92.287	20.095	16.924	16.095	19.581	16.715	16.022	13.300	13.119	11.366
92 U	111.303	110.424	98.440	94.659	20.712	17.454	16.575	20.167	17.219	16.429	13.614	13.438	11.619
93 Np	114.243	113.312	101.068	97.077	21.340	17.992	17.061	20.785	17.751	16.840	13.944	13.760	11.890

	$K\beta_1$	$K\beta_3$	$K\alpha_1$	$K\alpha_2$	$L\gamma_3$	$L\beta_3$	$L\beta_4$	$L\gamma_1$	$L\beta_1$	$L\beta_2$	$L\alpha_1$	$L\alpha_2$	L_1
<i>Intensity</i>	~20	~10	100	53–65	~5	35–20	20	5–25	100	5–20	~90	10	20–5
94 Pu	117.261	116.277	103.761	99.552	21.982	18.540	17.556	21.417	18.293	17.256	14.279	14.084	12.124
95 Am	120.360	119.317	106.523	102.083	22.637	19.106	18.063	22.065	18.852	17.676	14.617	14.412	12.384
96 Cm	123.423	122.325	109.290	104.441	23.306	19.663	18.565		19.552		14.959	14.703	
97 Bk	126.663	125.443	112.138	107.205	24.040	20.348	19.128		20.019		15.320	15.086	
98 Cf	130.851	129.601	116.030	110.710	24.831	21.001	19.751		20.763		15.677	15.443	
99 Es	134.238	132.916	119.080	113.470	25.579	21.648	20.326		21.390		16.036	15.780	
100 Fm	137.693	136.347	122.190	116.280	26.334	22.303	20.957		22.044		16.402	16.134	
101Md	141.234	139.761	125.390	119.170	27.120	22.984	21.511		22.707		16.768	16.487	
102 No	144.852	143.295	128.660	122.100	27.932	23.692	22.135		23.403		17.139	16.843	
103 Lw	148.670	146.920	132.020	125.100	28.760	24.530	22.780		24.130		17.500	17.210	

Approximate K and L line intensities are given at the head of the columns in the table, relative to the line in the series which is normally the strongest. The values given are based on the compilation of experimental relative intensities of Salem, Panossian and Krause and the intensities calculated by Scofield (Salem *et al.*, pp. 121–37).

Unresolved lines:

1— $KN_{II,III}$ ($K\beta_2$); 2— $L_1N_{II,III}$ ($L\gamma_{2,3}$)

Depending on the resolving power of the dispersing system used (e.g. crystal spectrometer, solid state energy dispersive detector) line pairs shown separately in the table may not be resolved and the effective energy of the doublet will be close to the mean value weighted by the relative intensity of the components.

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Data were taken from *Tables of Physical & Chemical Constants (16th edition 1995)*. 4.2.1 X-ray absorption edges, characteristic X-ray lines and fluorescence yields. Kaye & Laby Online. Version 1.0 (2005) www.kayelaby.npl.co.uk

APPENDIX B

X-ray Excitation Energies of the Elements

H	1	He	2
	0.014		0.025
Li	3	Be	4
	0.055		0.112
Na	11	Mg	12
	1.072		1.305
			0.063
			0.050
			0.049
K	19	Ca	20
	3.607		4.038
			0.341
			0.297
			0.295
Rb	37	Sr	38
	15.200		16.105
			2.065
			1.863
			1.805
Cs	55	Ba	56
	35.985		37.441
			5.713
			5.359
			5.012
Fr	87	Ra	88
			Ac
			89
Symbol	Z	Cu	29
<i>K</i>	→		8.979
<i>L_I</i>	→		1.100
<i>L_{II}</i>	→		0.952
<i>L_{III}</i>	→		0.932
B	5	C	6
	0.188		0.284
			0.402
			0.537
			0.686
			0.867
Al	13	Si	14
	1.560		1.839
			0.087
			0.073
			0.072
Ga	31	Ge	32
	10.367		11.104
			1.302
			1.142
			1.115
As	33	Se	34
	11.868		12.658
			1.653
			1.477
			1.436
Br	35	Kr	36
	13.474		14.322
			1.782
			1.596
			1.550
In	49	Sn	50
	27.940		29.200
			4.465
			3.938
			3.730
Tl	81	Pb	82
	85.528		88.006
			15.860
			14.698
			13.035
Bi	83	Po	84
	90.527		90.527
			16.385
			15.708
			13.418
Sb	51	Te	52
	30.491		31.813
			4.939
			4.612
			4.341
I	53	Xe	54
	33.169		34.582
			5.188
			4.852
			4.781
At	85	Rn	86
	4.557		98.417
			18.055
			17.334
			14.612

Based on tables of McMaster *et al.*

Energies are in KeV

Ce	58	Pr	59	Nd	60	Pm	61	Sm	62	Eu	63	Gd	64	Tb	65	Dy	66	Ho	67	Er	68	Tm	69	Yb	70	Lu	71
	40.444		41.991		43.569		45.184		46.835		48.520		50.240		51.996		53.789		55.618		57.486		59.390		61.332		63.314
			6.835		7.126		7.428		7.737		8.052		8.376		8.708		9.047		9.395		9.752		10.116		10.488		10.870
			6.165		6.441		6.722		7.013		7.312		7.618		7.931		8.252		8.581		8.919		9.265		9.618		9.978
			5.724		5.965		6.208		6.460		6.717		6.977		7.243		7.515		7.790		8.071		8.358		8.648		8.943
Th	90	Pa	91	U	92	Np	93	Pu	94	Am	95	Cm	96	Bk	97	Cf	98	Es	99	Fm	100	Md	101	No	102	Lr	103
	109.649		115.603		121.760		127.095		132.912		138.925		145.042		151.269		157.602		164.047		170.596		177.253		184.019		190.896
			20.470		21.756		23.095		24.488		25.937		27.442		28.995		30.599		32.254		33.961		35.711		37.506		39.346
			19.692		20.947		22.263		23.628		25.044		26.511		28.029		29.599		31.221		32.896		34.626		36.411		38.251
			16.300		17.167		18.053		18.953		19.867		20.795		21.738		22.695		23.667		24.654		25.656		26.673		27.705

Compilation of X-Ray Cross Sections

W. H. McMaster, N. Kerr Del Grande, J. H. Mallett, and J. H. Hubbell

Lawrence Livermore National Laboratory Report

UCRL-50174 Section II Revision I (1969)

APPENDIX C. Spectrometer settings and data acquisition guide.


Written by Yaroslav Babich, EngSci student in SURF June 14, 2012.

I. Geometry considerations.

The samples must be placed in a consistent manner, as geometry has a fairly significant effect on the peak amplitudes. The vertical plane and the horizontal angle of the sample should be fixed. The horizontal angle is varied by rotating the sample holder. Set it so that it forms a 45 degree angle with both the X-ray tube and the detector, in order to minimize Rayleigh and Bragg scattering.

Low-energy X-rays (those below 4keV) get severely attenuated in air. As such, if you want to see low-energy peaks, such as Si K_{α} , Sn L_{α} , etc. you must try to minimize the detector-sample distance.

II. Checking initial (saved) settings.

Before taking spectra, a quick check of the apparatus settings will save your time. On the PC desktop click on an icon of the ADMCA software . In a selection window click on the “Connect to MCA” option.

In the opened window, find a function DPP in the upper row of options, and in the drop-down menu select “Show Current Configuration”. You will see the configuration of the detector with current setting as in Fig.C1.

If the real values are different from those in Fig.C1, start re-setting the detector manually.

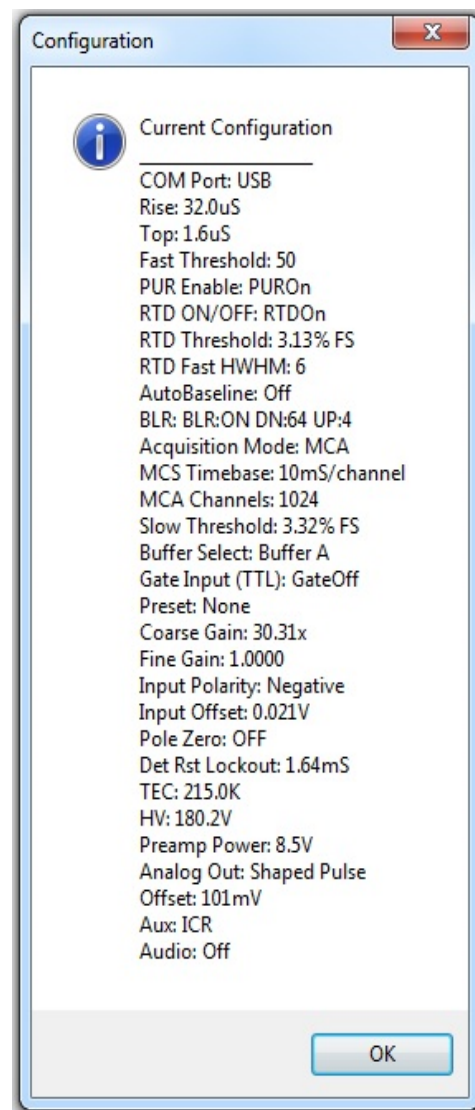



FIG.C1.

III. Detector settings adjustment.

The settings for the detector can be accessed and changed via the Amptek ADMCA software, either by pressing F9, going into the MCA menu and selecting “Acquisition Setup”, or by clicking  in the toolbar. The following window should pop up:

1 – General. Selecting a detector.

The “General” window is shown in Fig. C2.

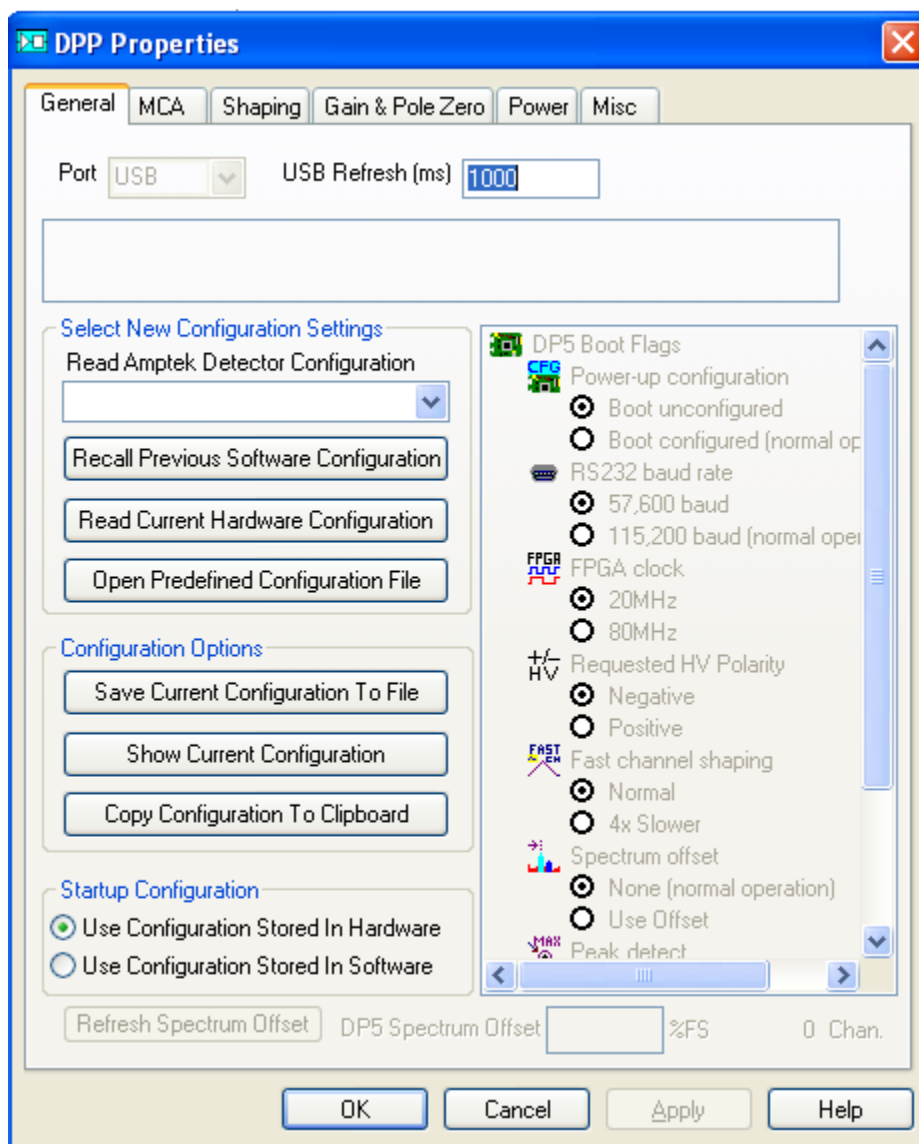


FIG.C2.

In the drop-down menu under “Read Amptek Detector Configuration”, select the Si 13mm²/500μm detector settings. Reload these every time you (re-)open the software, or when you want to revert to the default settings after changing something – these are the working and optimized settings for the detector. Click Apply, click OK.

2 – MCA settings (MCA).

Switching from General to MCA window, you will find three main settings: the “MCA Channels”, the “Preset Time (sec)” and “Slow Threshold” (Fig. C3).

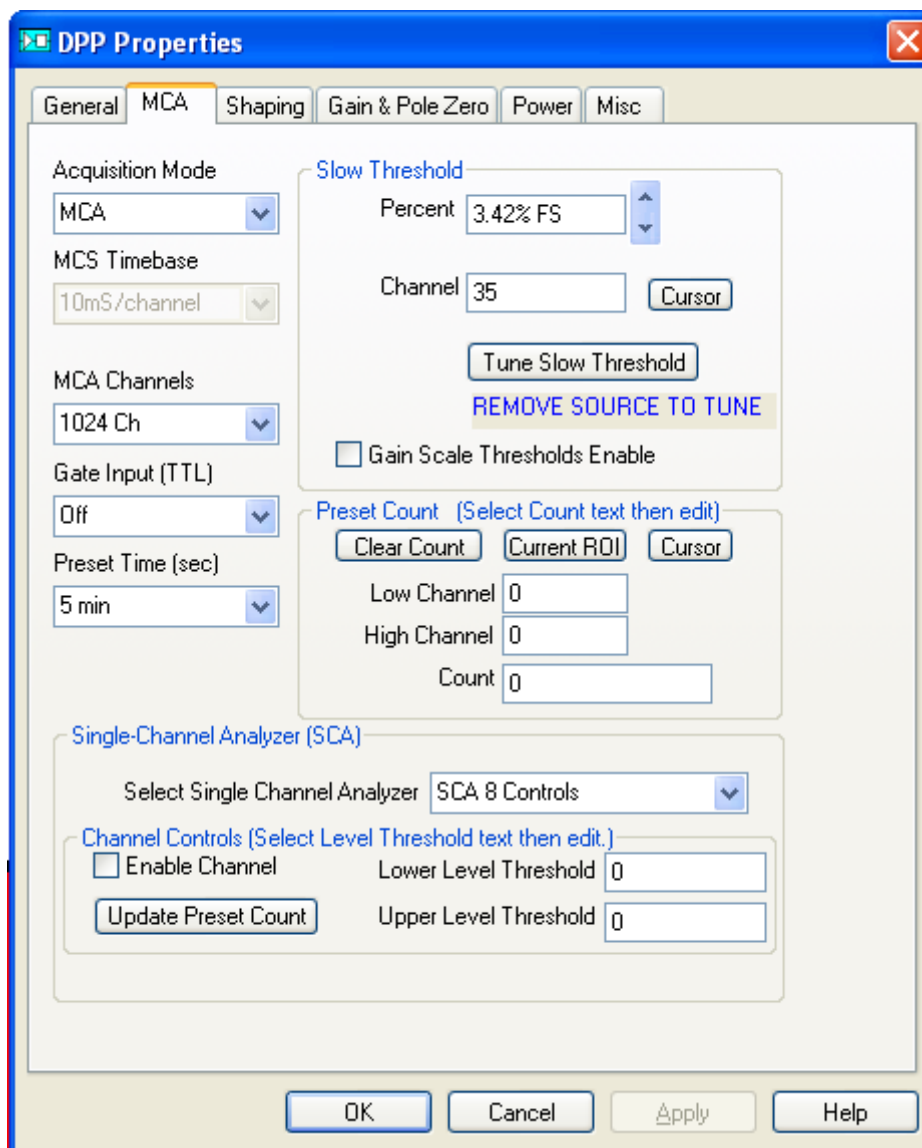


FIG.C3.

MCA Channels

The number of channels does not affect the resolution or energy range of the spectrum; it only makes the spectrum more or less refined. If the peaks being taken are not smooth, then you can decrease the number of channels (or increase the acquisition time). If the FWHM (Full Width at Half Maximum) of the spectrum is less than 5 channels, then increase the number of channels.


Preset Time

Fairly obvious, you can preset the acquisition time here. The acquisition should be run long enough for all the peaks being considered to have a small error, and therefore a large area. An area of a few hundred counts is usually sufficient.

Slow Threshold

The slow threshold is the channel (or percentage of the number of channels) below which no counts are taken. Electrical noise manifests as low-amplitude, low-energy pulses, and is consequently recorded as counts in the low-energy channels. The slow threshold should ideally be set such that it is just above this noise.

The slow threshold can be set automatically or manually. To set it automatically, remove any radiation sources from in front of the detector, start an acquisition, and click “*Tune Slow*

Threshold”. It is also tuned when the  button is pressed on the toolbar (again, an acquisition must be running).

To set the slow threshold manually, start a source-less acquisition, click on the first channel, and press F8. This sets the slow threshold to the first channel. You should see the noise peak start growing immediately. Click a few channels above the highest-energy noise, and press F8 again. You should go through the exercise of manually tuning the slow threshold in order to be able to recognize and remove the noise peak if you see it in the future.

The slow threshold (and fast threshold and input offset) should be retuned every time you change the gain, the peaking time, or the high voltage bias.

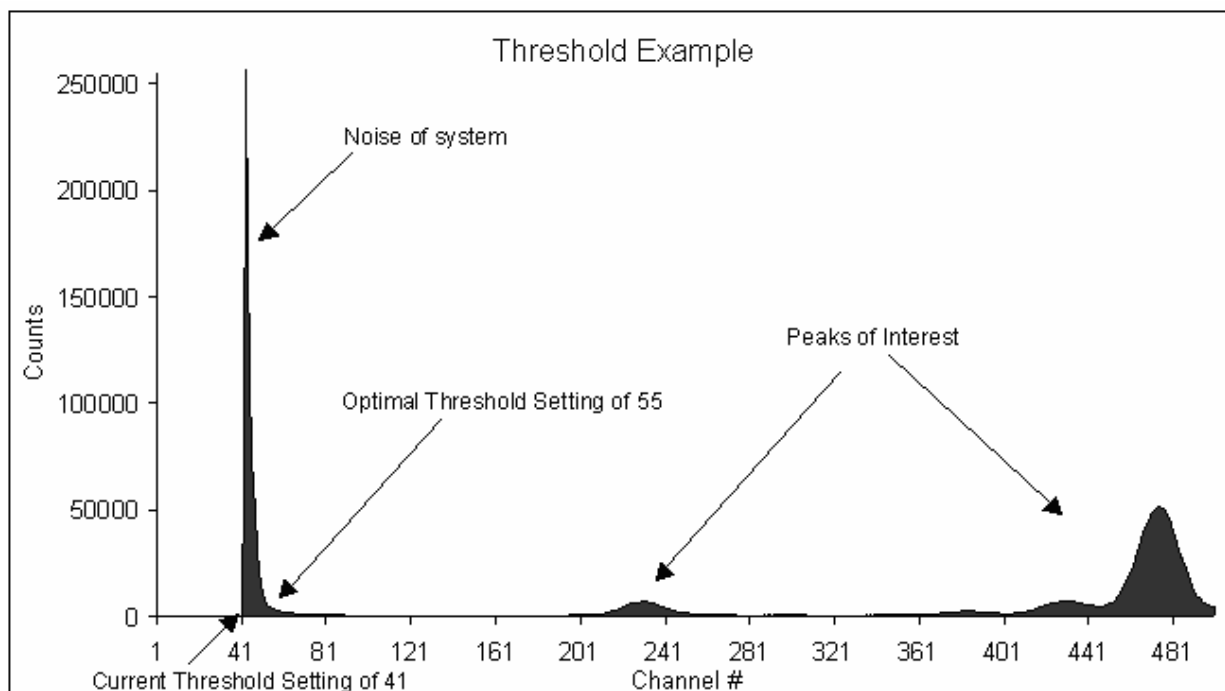


FIG.C4.

3 – Shaping.

The main settings here are the “*Peaking Time*”, “*Pile-Up Rejection*”, “*Fast Threshold*” and the “*Rise Time Discrimination*”. See Fig. C5.

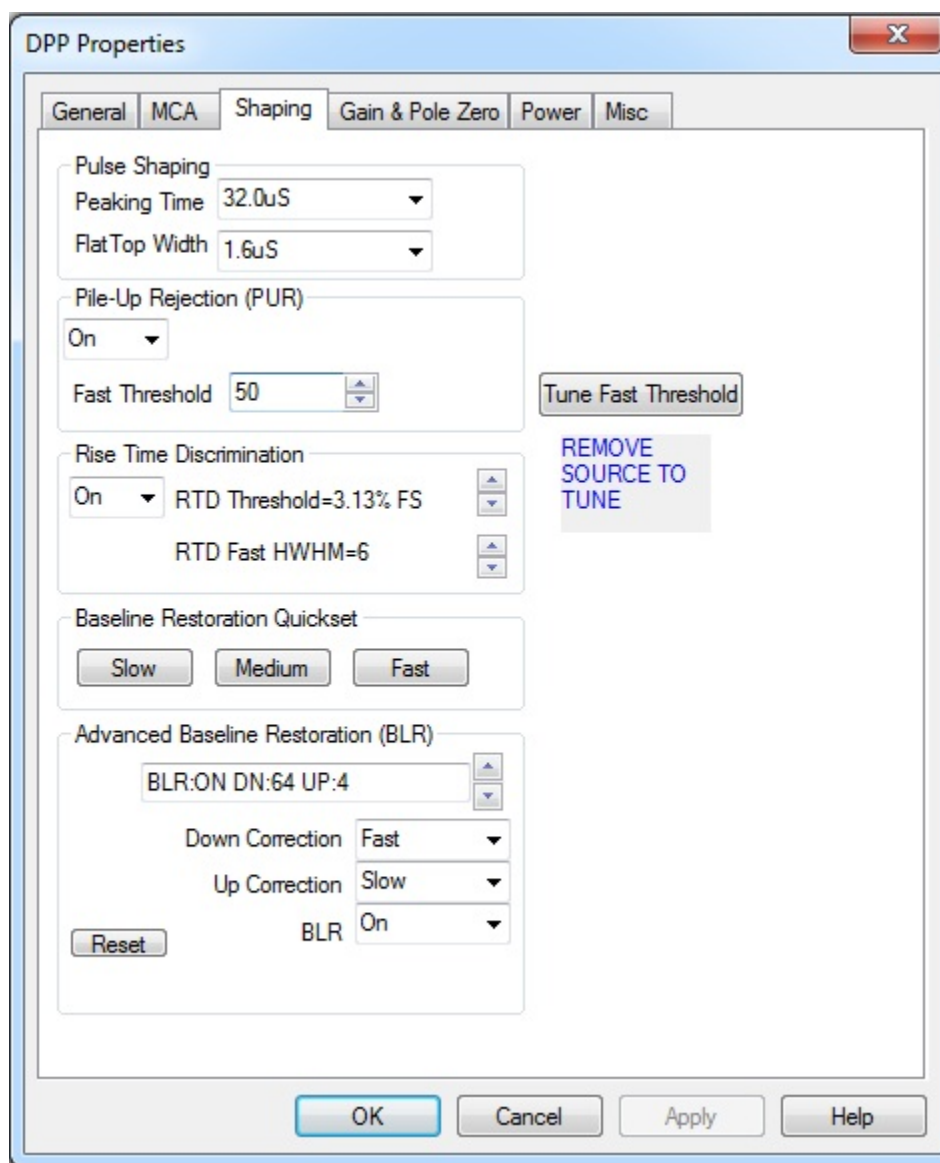


FIG. C5

Peaking Time

Most likely, you will never change this. The peaking time is directly proportional to the width of the pulse that is recorded by the detector. You want the pulses to overlap as rarely as possible, in order to not have to deal with pile-ups. In this respect, smaller peaking times are better. However, peaking times below 12.8 μ s begin to decrease the resolution of the spectrum, so only use lower peaking times if the dead time of the spectrum (percentage of discarded counts) is above ~20%.

There are other ways to deal with a large dead time: a) decrease the current, b) move the sample and X-ray tube further apart, or c) move the detector and sample further apart. Note that the step c) will probably cause you to lose low-energy peaks as they are attenuated more in air than are high-energy peaks. Only if these options are exhausted or unavailable should you decrease the peaking time. Also, before doing any of this, make sure your fast threshold is not too low, as this could result in a spurious input count

Pile-Up Rejection (PUR)

Understanding this requires some knowledge on the inner workings of the detector. It has two parallel processing channels: the fast channel and the slow channel. The fast channel essentially sees every pulse that is separated by at least $0.4 \mu\text{s}$ but cannot accurately measure their amplitude (energy). The slow channel can only resolve pulses that are separated by at least the peaking time, which is larger than the $0.4 \mu\text{s}$ of the fast channel. If two pulses in the slow channel are separated by, for example, $0.6 \mu\text{s}$, the fast channel sees two pulses, while the slow channel will only see one pulse with an amplitude equal to the sum of the two pulses. With PUR turned off, this sum-pulse is recorded in a high-energy channel. With PUR turned on, the pulse is rejected. The two fast channel counts are still counted as input counts, but total counts (the number of recorded pulses) are unchanged.

In practice, PUR should always be turned on. The only reason to turn it off would be if you were interested in seeing the pile-up peaks, in which case you need an input count rate at least in the thousands, and a strong peak in the left half of the spectrum.

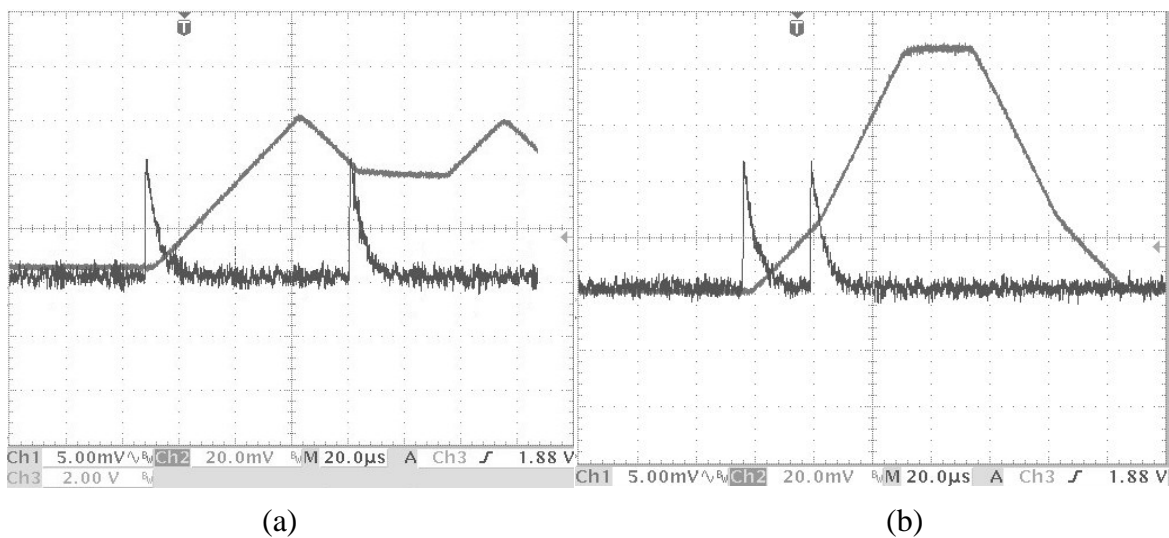



FIG.C6.

Fast Threshold

The fast channel (see PUR) also measures the amplitude, but not as accurately as the slow channel. However, to get an accurate input count, it is still necessary to distinguish electrical noise from actual counts, thus a fast threshold is implemented which is identical to the slow threshold in the slow channel. Fast channel pulses that map to a channel below the fast threshold are rejected – counted as neither input counts nor total counts, even if a pulse occurred in the slow channel. Thus, a fast channel that is too high would cause legitimate pulses in the slow channel to be rejected, while a fast channel that is too low would cause an inaccurate measured input count, as the fast channel would trigger on electrical noise.

The fast threshold can be set automatically or manually. To automatically set it, you can click the “*Tune Fast Threshold*” button in the Shaping tab of the Acquisition set-up, or it is automatically tuned when you click  in the toolbar.

To manually set the fast threshold, remove any source from in front of the detector, start an acquisition, and observe the input counts. You want the input counts to increase by 2-5 counts per second. If they are increasing faster, increase the fast threshold. If they are increasing slower or not at all, decrease the fast threshold.

The fast threshold (and slow threshold and input offset) should be retuned every time you change the gain, the peaking time, or the high voltage bias.

Rise Time Discrimination (RTD)

In this 500 μm detector, the back 100 μm of Si is fully depleted. However, to discard a slower-rising lower-amplitude pulse, the rise-time discriminator is used. RTD discards such pulses that have a half-width at half-maximum greater than the specified “RTD Fast HWHM”. Via trial and error, the best settings for HWHM for various peaking times and gains are between 6 and 10. Try 6 first.

The RTD Threshold is the channel (percentage of total channels) above which RTD begins to get activated.

RTD should be turned on at all times, especially if there are high-energy (above 10 keV) peaks. If the dead time is very large, then RTD can be turned off to speed up data collection, at the price of some inaccuracy (in addition to skewing the spectrum slightly towards low energies, it causes the assumed value of the detector efficiency to be incorrect).

4 – Gain and Pole Zero.

The main settings here are “Gain” and “Input Offset” (Fig.C7). The rest of the settings should not be changed.

Gain

Pulse amplitudes are multiplied by the gain before being mapped to different channels. In essence, a pulse at a lower gain will map to a lower channel than the same pulse would at a higher gain.

There is a different channel-energy calibration for each different gain value. They can be found by making a linear fit of at least two known peaks of a spectrum taken at the gain value in question (recommended), or consulting the table below (accuracy dubious, may depend on other settings):

Gain	Comparison of calibration at 1024 channels for different gains (may differ from yours in specific numbers): $\text{channel number} = \text{const}_1 \times E + \text{const}_2$ (± 2 channels)
91.84	$96.088 \times E + 12.314$
76.68	$80.323 \times E + 9.8454$
60.30	$62.996 \times E + 7.3636$
49.92	$52.215 \times E + 5.557$
41.58	$43.465 \times E + 4.6135$
34.72	$36.359 \times E + 3.1665$
30.30	$31.691 \times E + 2.8848$
25.09	$26.227 \times E + 2.4313$
20.90	$21.833 \times E + 1.92$
17.46	$18.24 \times E + 1.6674$
14.27	$15.014 \times E + 1.2661$
11.82	$12.41 \times E + 1.1738$
9.84	$10.327 \times E + 0.9639$

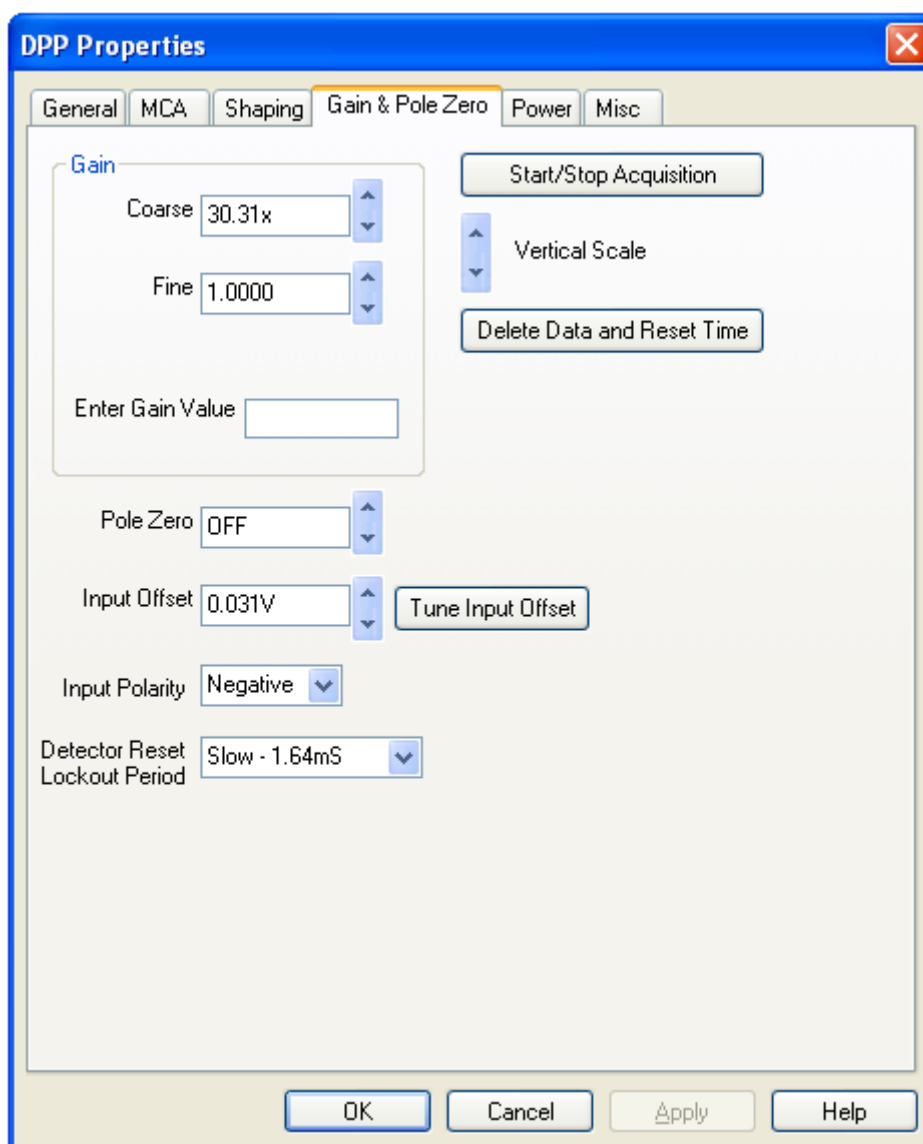


FIG.C7.

The gain should be selected such that the highest-energy peak that you expect to see appears in the spectrum. To see any peak that can be excited by a 40-kV X-ray tube, begin with 30.30x gain.

Input Offset

The input offset (and slow and fast thresholds) should be tuned whenever the gain is changed, with no source in front of the detector.

5 – Power.

Relevant settings here are the “*Cooler Temperature*” and the “*High Voltage Bias*”.

Cooler Temperature

The colder, the better. As it happens, this detector has a two-stage Peltier cooling system, so it can cool itself to 75-80 K below room temperature (i.e. to 213-218 K). As long as the temperature is relatively constant (fluctuations by a few degrees are okay), the spectra will be consistent. The recommended setting is 215 K.

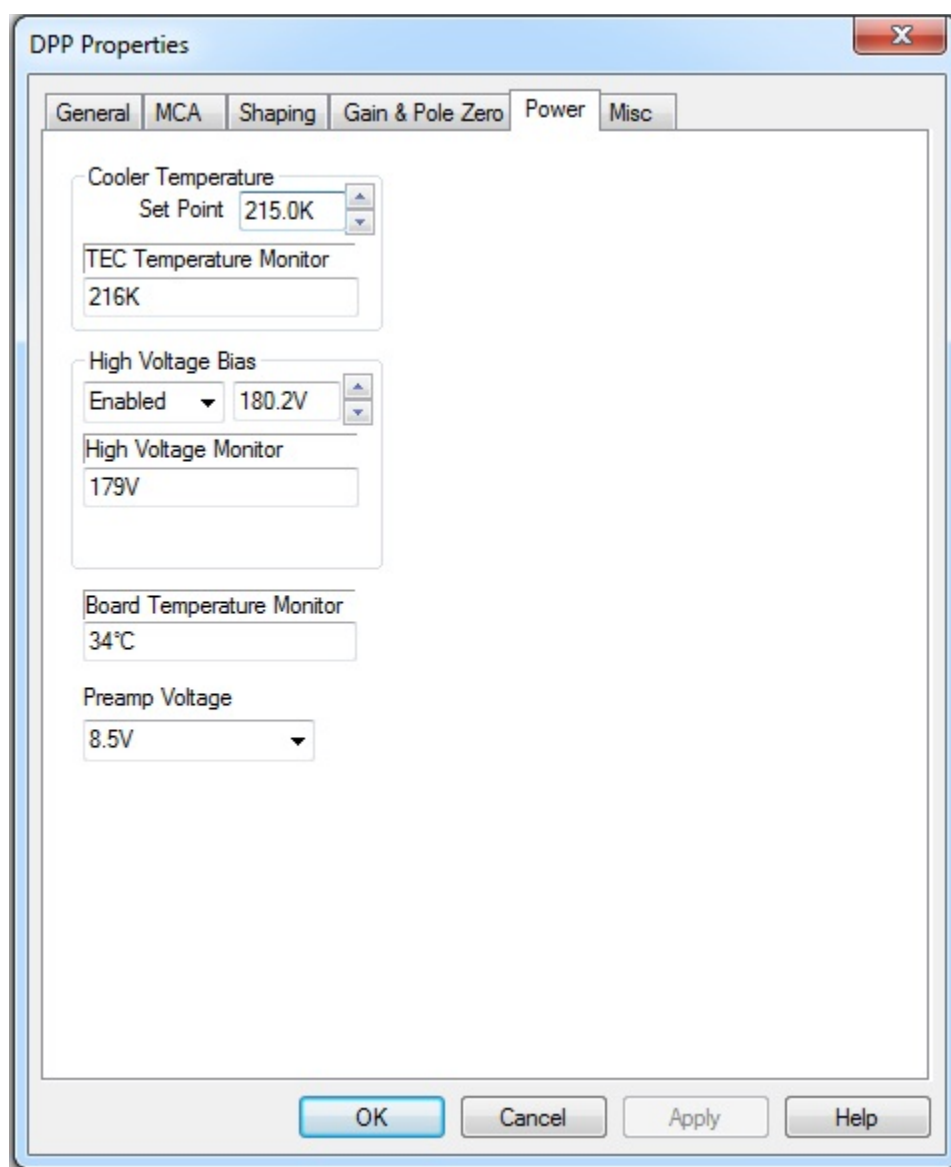


FIG. C8.

High Voltage Bias (HV Bias)

It is not recommended that you change the HV bias from 180 V. If it is decreased, you may lose the pulse and be unable to restore it without turning off the detector, waiting a few minutes, turning it back on and tuning fast and slow thresholds if necessary. If it is increased, you will have decreased resolution, and when decreasing it back later you may again lose the pulse and have to troubleshoot again.

The disappearing pulse issue has never occurred when the detector is turned on and the HV bias is immediately set to 180 V, so that is the recommended course of action.

6 – Misc.

There isn't much of interest in the miscellaneous settings, except just one: the virtual oscilloscope can show whether the signal is being registered by the detector. This can be useful during troubleshooting when the source of absence of a signal is to be determined.