



Characterisation of an electron source to ionize gas molecules through electron ionization

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September 9, 2014

DESY - Hamburg

Abstract

The electron ionization is a method to produce ions through the interaction between energetic electrons and gas molecules.

The purposes of this project are to set up all necessary tools and to identify the best operation parameters for the electron source in order to achieve optimal electron ionization. A time-of-flight mass spectrometer is used to observe the ionization of molecules hit with the electron beam.

The characterisation of the electron source might be useful for testing the time-of-flight spectrometer and all other devices, that are necessary for the next experiments in FLASH at DESY.

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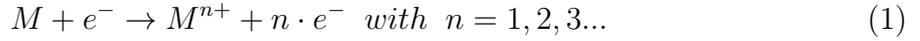
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1 Introduction

Electron ionization (or electron impact ionization) is a useful method to produce ions through the interaction between energetic electrons and gas molecules. The electron ionization process is described by the following formula:



where M is a gas molecule (in our case, argon gas) to analyze, e^- is an electron which interacts with the gas molecule M and M^{n+} is the resulting ion.

The purposes of the project, described in this report, are to set up all necessary tools for electron ionization and to identify the best operation parameters for the electron source in order to achieve optimal electron ionization. The characterisation of the electron source might be also useful for testing the time-of-flight spectrometer and all other devices for the next experiments at FLASH or other facilities.

2 Apparatus and experimental details

The figure 1 shows the experimental setup that we used to characterize the electron source:

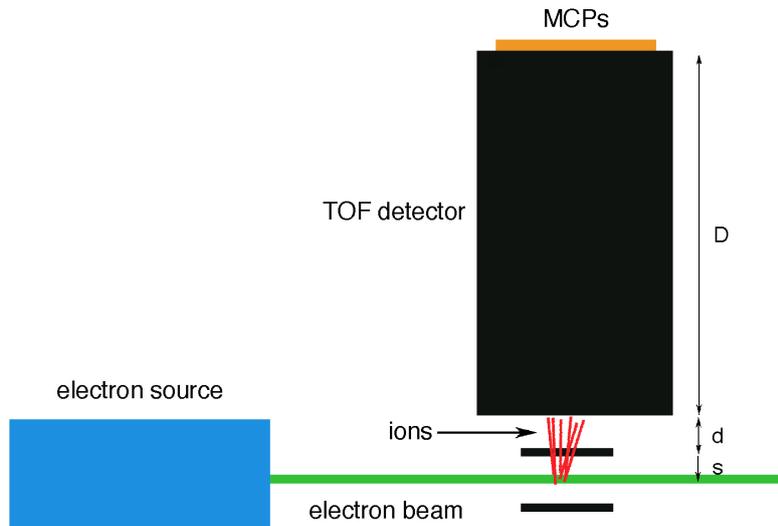


Figure 1: Scheme of experimental setup inside the vacuum chamber. The TOF mass spectrometer is composed by three regions: ionization region (s), acceleration region (d) and field-free drift region (D).

An electron beam is used to ionize the gas molecules inside the vacuum chamber and then the ions are analyzed through a time-of-flight (TOF) mass spectrometer to determinate the mass-to-charge ratio of each ion, formed by electron ionization.

In the following paragraphs, we are going to discuss the vacuum chamber, the electron source and the TOF mass spectrometer in detail.

2.1 Vacuum chamber

In the vacuum chamber the air is removed by two turbo-molecular pumps, which are set on the bottom and on a side of chamber backed by a roughing pump system providing a pre-vacuum of 10^{-1} to 10^{-2} *hPa* for the turbopumps.. Moreover a cooling system with water decreases the temperature of pumps (*figure 2*).

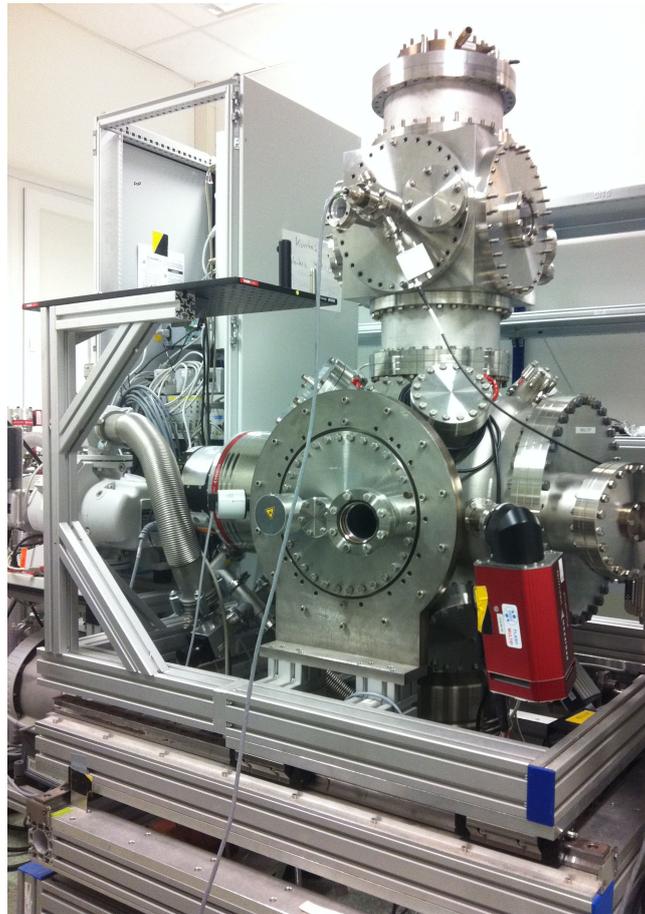


Figure 2: This picture shows the vacuum chamber. On the left side it is possible to look at the lateral vacuum pump, whereas the pump on the bottom of the chamber is hidden by the support of the chamber. On the upper part of the chamber we set the electron source (it's visible only the voltage divider) and on the top there are the TOF spectrometer and the MCP detector.

The maximum rotational speed of the pump is 525 Hz and the order of magnitude of the pressure inside the vacuum chamber is about $10^{-7} - 10^{-8} \text{ hPa}$. The vacuum chamber is made of stainless steel. The *figure 3* shows the top of vacuum chamber without a flange. It is possible to observe two kinds of flange (covered or window flange) and there are a lot of possibilities to insert tools inside the chamber. We chose the central opening to insert the electron gun.

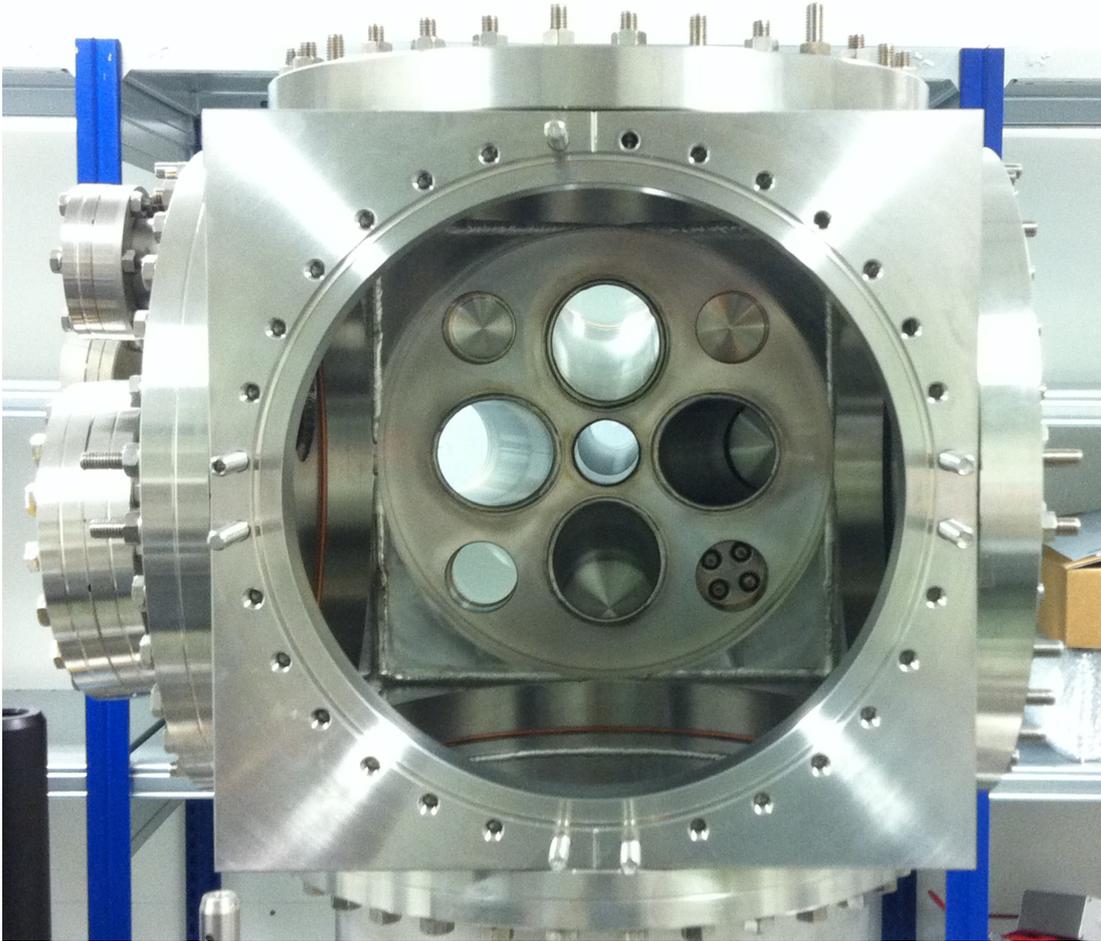


Figure 3: Vacuum chamber without a flange.

2.2 Electron source

An electron gun (called also electron emitter or electron source) produces a narrow and collimated electron beam with a precise kinetic energy. It is used usually in scanning applications (i.e. scanning electron microscope), in Auger measurements, in electron pulse experiments and in a broad range of devices.

The electron gun is a source for electrons up to 5 keV . It consists of three main parts (*figure 7*):

- beam generation (filament, wehnelt cylinder and anode);
- focusing (einzel lens);
- deflection (two pairs of deflection plates).

The length of electron gun is 21.0 cm and the energy range is $0 - 5\text{ keV}$ with energy width about of 0.6 eV . From the information on the data sheet, the spot size of electron gun can be adjusted down to $50\text{ }\mu\text{m}$ and the pressure requirements for stable operation have to be smaller than 10^{-6} hPa .

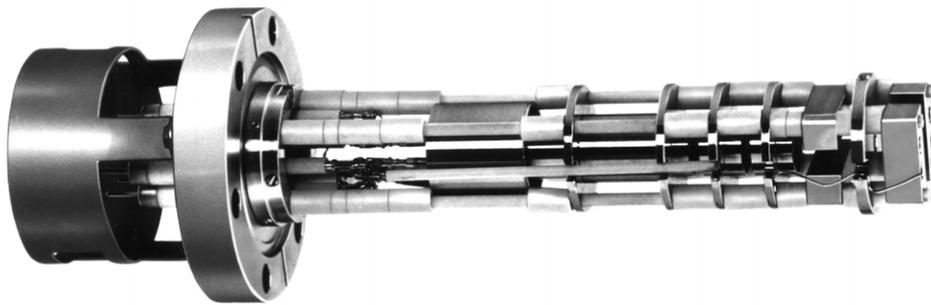


Figure 4: Design of the electron source (Specs EQ 22/35) without the covering [4].

By heating the tungsten filament, which is on negative potential, electrons are emitted through thermionic effect and accelerated towards the anode. A large voltage between cathode and anode is used to accelerate electrons up to the desired kinetic energy. Functions of wehnelt cylinder, which is on negative potential in comparison to the filament potential, are:

- to influence the amount of emitted electrons;
- to focus the electron beam, in order to increase or decrease the diameter of the beam.

Moreover, an einzel lens is used to focus the electron beam further. An einzel lens focuses charged particles (in this case, electrons) without changing the total energy of the beam. Basically it consists in three cylindrical tubes in series along an axis. The middle tube is on a positive potential and the two external tubes are on ground potential, in order to the electrostatic field inside the einzel lens is symmetrical. The direction and velocity of outer electrons are altered by electrostatic field, so they arrive in the focus slightly later than electrons with a straight path. For this reason, an einzel lens focuses

the incoming electron beam (*figure 5*).

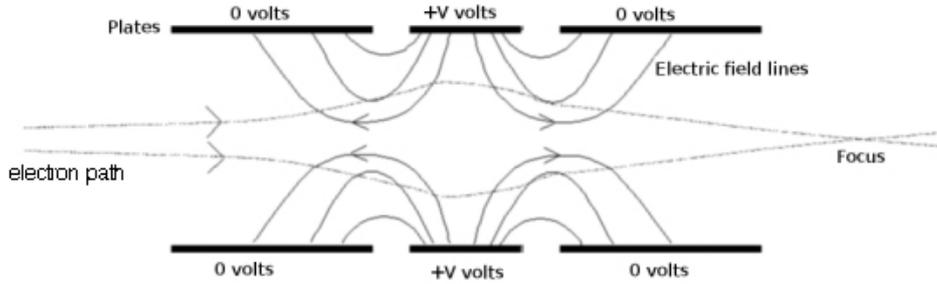


Figure 5: Outline of an einzel lens (2-dimensions and 3-dimensions) [6].

At the end of the electron gun, there are two pairs of deflection plates which deflect the electron beam on the sample, through the vertical electric field inside each pair of plates. All parts of the electron gun are separated by ceramic sleeves, which serve as distance holder and as electric insulation too.

The electron gun is combined with a pulse generator in order to use it as a pulsed electron source. Electron pulses are produced by applying a quick voltage pulse to the wehnelt cylinder, so electrons are emitted by the gun only for the duration of the pulse. Since there are internal capacitors inside the pulse generator, the energy of electron beam must not exceed 4 keV [5].

Moreover the minimum of energy should be about 100 eV , because under this value the kinetic energy of electrons coming from filament is too low and the duration of pulse is so short for allowing the emission of the electron beam.

As mentioned previously, one of our purposes in this project is to characterize the electron source in order to ionize the gas molecules (in our case, argon gas) satisfactory.

2.3 Time-of-flight mass spectrometer

Time-of-flight (TOF) mass spectrometry is a method of mass spectrometry in which the mass-to-charge ratio of ions is determined through a time measurement.

Using a TOF mass spectrometer has some advantages, such as a complete spectrum can be obtained in few microseconds, so one can study samples that change rapidly. The main disadvantage of TOF spectrometer is the intensity of ion current which is much lower than other types of spectrometer, for example the deflection mass spectrometer. In fact a TOF mass spectrometer has to work in a pulsed fashion, so the total amount of ions that can be detected is reduced.

The TOF mass spectrometer, used in the data acquisition, consists of three main regions (*figure 6*):

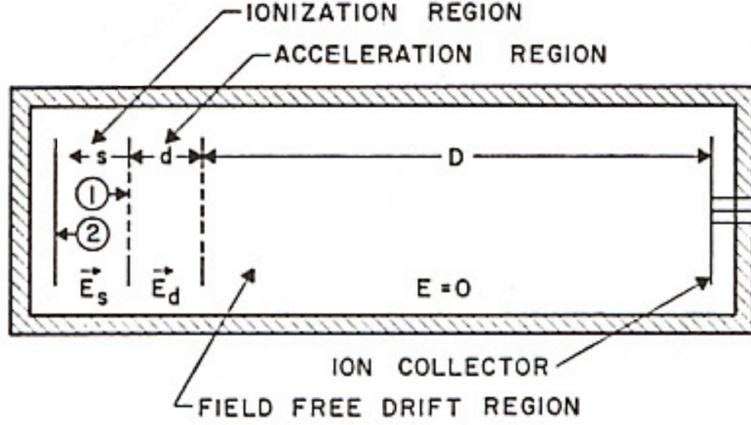


Figure 6: Basic geometry of a TOF mass spectrometer [1].

- **Ionization region**, in which the sample is hit by the electron beam. The ions, formed in the ionization region, will be accelerated out of the sample towards the collector by a constant electric field (E_s). In the spectrometer, used in the data acquisition, the accelerating field is pulsed on at the end of ion formation in order to improve the energy resolution, as it will be discussed later. The pulse of electric field E_s lasts until all ions left this region.
- **Acceleration region**, in which there is another electric field E_d to accelerate the ions along the distance d . Therefore the energy of each ion will increase to a value U at the end of this region:

$$U = U_0 + qsE_s + qdE_d \quad (2)$$

where U_0 is the initial energy, s is the distance travelled by ion in the ionization region and q is the charge of ion. The energy U is independent from the mass of ion (m), but it is dependent on the initial energy U_0 .

- **Field-free drift region**, in which there are not electric and magnetic fields, so each ion drifts along the distance D , depending on the velocity that is got in the first two accelerating regions.

When an ion arrives into the field-free drift region, it will have the kinetic energy U :

$$U = \frac{1}{2}mv^2 \rightarrow U = U_0 + qsE_s + qdE_d = \frac{1}{2}mv^2 \quad (3)$$



Figure 7: In this photo it is possible to identify the three main regions of TOF mass spectrometer.

The velocity (v) of each ion is equal to:

$$v = \frac{D}{t} \quad (4)$$

where D is the length of field-free drift region and t is the time-of-flight (TOF) of the ion in this region.

Thus recasting the equation (3) and the equation (4) and considering negligible the initial energy U_0 , it can be shown that the time-of-flight (TOF) of the ion is proportional to the square root of mass-to-charge ratio:

$$TOF \propto \sqrt{\frac{m}{q}} \quad (5)$$

If all ions were formed in a plane parallel to the collector and with zero initial velocity, the time-of-flight would be the same for all ions which had the same mass-to-charge ratio ($\frac{m}{q}$). The resolution of a TOF mass spectrometer depends on its ability to reduce the

energy spread and the spatial distribution of the initial ion cloud. [1]

In the 1955 W.C.Wiley and I.H.McLaren developed a clever method to improve the energy spread compensation, introducing a time-lag between the formation of ions and the application of the first acceleration pulse (in this case, E_s). During the time-lag, ions move to a new position because of their initial velocities, so the arrival time spread due to initial kinetic energy differences is eliminated under proper experimental conditions. The TOF mass spectrometer, used for the data acquisition, is based exactly on time-lag principle.

When the ions arrive at the end of the field-free drift region, they are collected by a micro-channel plate (MCP) detector and converted in an electrical signal (*figure 8*).

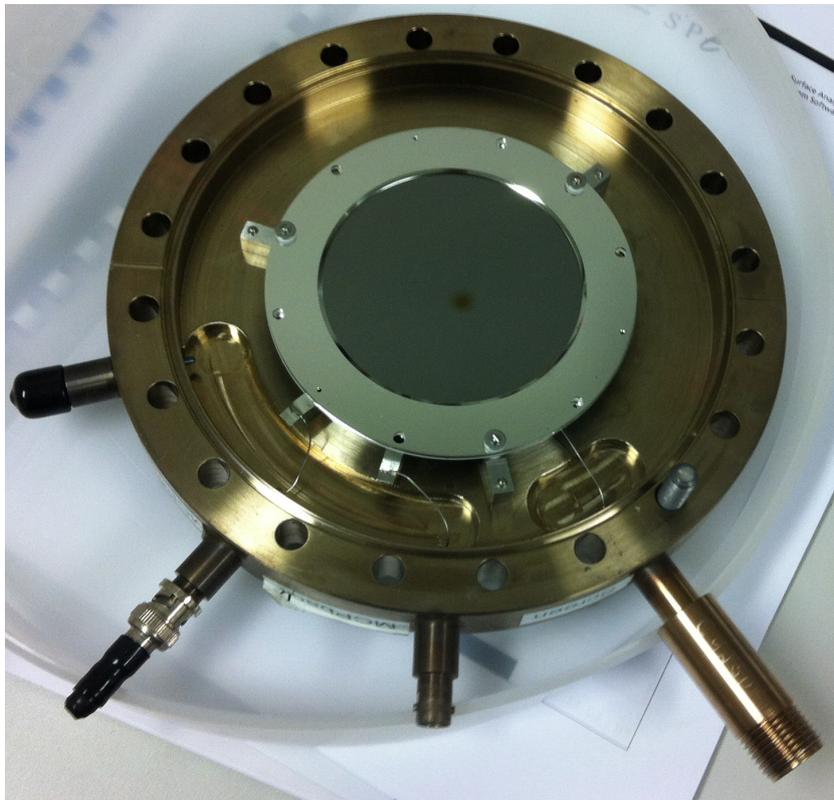


Figure 8: Micro-channel plate detector.

A micro-channel plate detector is composed by many electron multipliers, oriented parallel to each other and tilted to MCP input surface of about 10 degrees. Each channel can be considered as a continue structure of dynodes in order to multiply the incoming charged particle in a lot of outgoing electrons (*figure 9*). The order of magnitude of gain is typically equal to 10^6 .

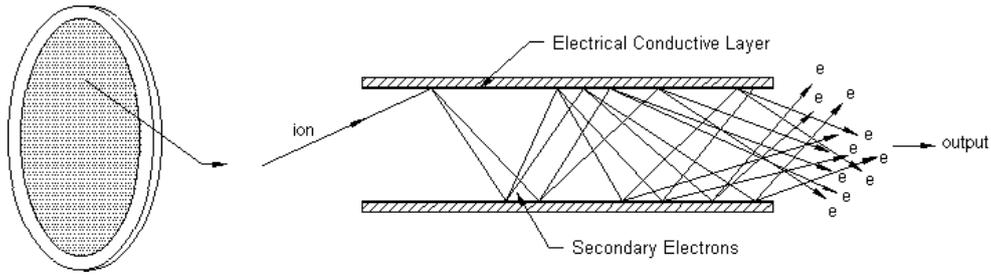


Figure 9: A typical micro-channel of an MCP detector.

3 Data analysis

As mentioned previously, the electron gun is combined with a pulse generator in order to use it as a pulsed electron source. The pulse generator provides electron pulses of 1.6 ns and a repetition rate of up to 50 kHz . Since the width of pulses should be a few microseconds and the repetition rate should be of few kilohertz to acquire data correctly, it is possible to change these parameters with an external pulse signal (*figure 10*). When the pulse generator produces the electrical signal, an electron beam is emitted by the gun for the entire duration of the pulse.

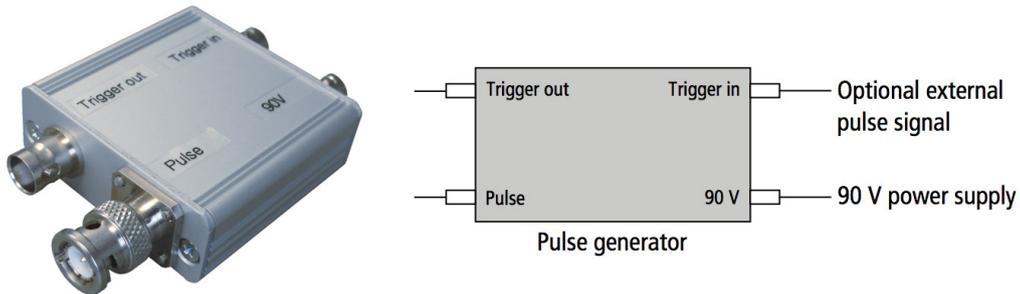


Figure 10: Pulse generator connections [5].

For data acquisition we have used an oscilloscope (LeCroy) with sample rate equal to 2 GS/s . When electrons are emitted by the gun to ionize the molecules inside the vacuum chamber, the oscilloscope can measure and save the waveform coming from the MCP detector. The length of the saved waveform is chosen in a way that also the heaviest ion present is recorded. The type of this file is *.trc*. Typical waveforms are shown in the *figure 11*.

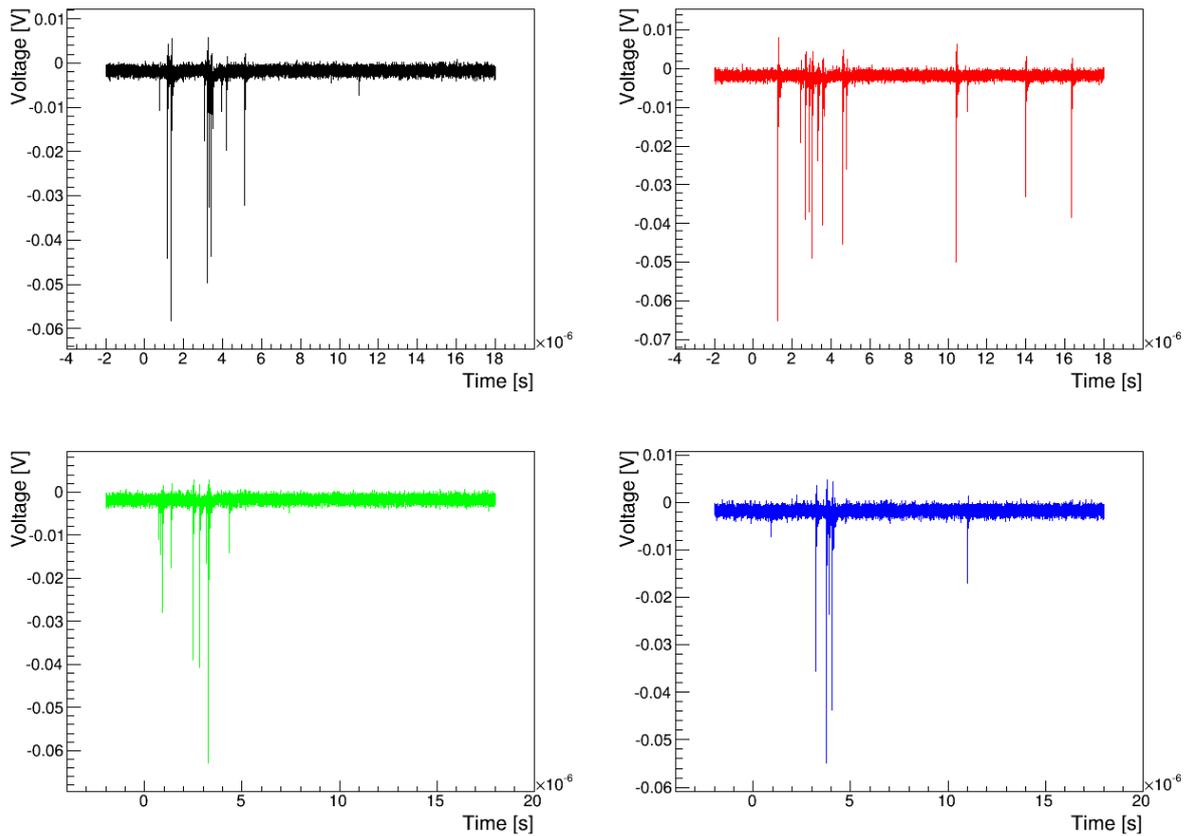


Figure 11: In this figure there are some typical waveforms acquired by LeCroy oscilloscope. One can observe that waveforms are different between individual shots.

Many waveforms are required to acquire important information about the time-of-flight of ions.

I have written a script in MATLAB to read and analyze a lot of waveforms, acquired with the same settings. The script has two main functions:

- to read each waveform, saved in the file, and storing only events which exceed arbitrary voltage threshold;
- to plot in a histogram the time-of-flight of all events, which are stored previously.

The first part of the script clears all variables and commands, used in the MATLAB software previously. Of course arbitrary threshold may be modified. Moreover the value of selected threshold will appear on the screen during running of the script.

```
1 %Clear all variables
2 clear;
```

```

3 clc;
4
5 %Set the threshold to select the interesting events only
6 threshold = -0.008;
7 disp( strcat('Threshold: ', num2str(threshold), ' V') )

```

After setting some important variables, as for example the number of histogram's bins (*nbins*), the function *ReadLeCroyBinaryWaveform* is called for each file inside the selected folder. The code of this function can be viewed on the Internet: [9]
<http://www.mathworks.com/matlabcentral/fileexchange/2114-readlecroybinary-waveform-m/content/ReadLeCroyBinaryWaveform.m>.

The return value of this function is a record containing four elements:

- *ans.info*: some information about waveform, for example oscilloscope ID, sampling time and other settings;
- *ans.desc*: in our case, this element is empty;
- *ans.y*: voltage values sampled by the oscilloscope;
- *ans.x*: array of time values corresponding to *ans.y*. It is important to underline that the time "0" marks the trigger event (in our case, the leading edge of pulse).

The second *for loop* analyzes the sampled values and stores only those that exceed the threshold. The time-of-flight measurements of collected ions are stored in the variable *data()*.

```

1 %Set some variables for the loops and set the number of bins
2 n = 1;
3 data = [];
4 d=dir('D://FileROOT/file_data/test-trc/'); % directory of the ...
   folder in which there are all files .trc
5 nbins=1000; % nbins = number of bins -- if nbins = 10000, then the ...
   width of each bin is equal to 2 ns.
6
7 %Two for loop to read all files (.trc) in the selected folder and ...
   fill the vector data() with events that exceed the threshold
8 for i = 1:(size(d)-2) % processing each file .trc in the folder
9     ReadLeCroyBinaryWaveform( ...
       strcat('D://FileROOT/file_data/test-trc/', d(i+2).name) );
10
11     for k = 1:20001 % length of ans.y (and of course ans.x) is ...
       equal to 20000
12         if (ans.y(k) < threshold)
13             data(n)=ans.x(k); % 'time' (x-axis) concerning ...
               the event that exceed the selected threshold
14             n=n+1;
15         end
16     end

```

```

17
18     %Clear the ans.x and ans.y, corresponding to the previous event
19     clear ans.x
20     clear ans.y
21 end

```

The following code is necessary to plot all values, stored in variable `data()`, in a histogram. The number of bins in the histogram is selected previously in the variable `nbins` (in our case `nbins = 1000`, so the width of each bin is equal to 20 ns). Afterwards the title of histogram and the label of axes are set.

```

1 %Plot the histogram: the number of bins is set in the variable 'nbins'
2 f = figure('Visible', 'on'); % on = the canvas is visible or off = ...
   the canvas is not visible
3 hist(data,nbins)
4 title('Spectrum')
5 xlabel({'time [s]'})
6 ylabel({'counts'})

```

In the *figure 12* you can see an example of histogram. Data of histogram in *figure 12* and in the following figures of this paragraph were acquired with the same oscilloscope used for this project, but the experimental setup was completely different.

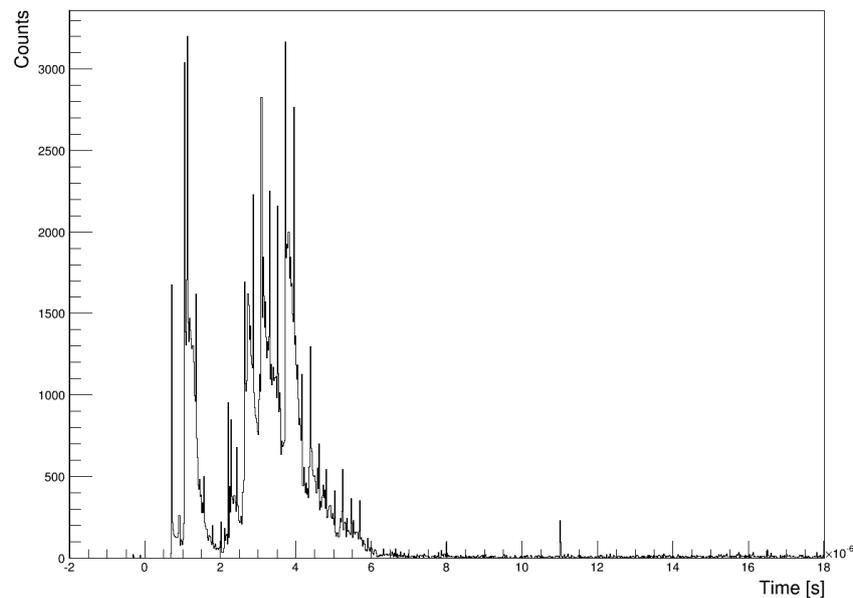


Figure 12: Example of histogram. The value of threshold to get this histogram is equal to -0.008 V and the width of each bin is equal to 20 ns . Data of histogram don't concern the experimental setup described previously, but they are used to check the MATLAB script.

Moreover the following code may be useful:

- to fit the histogram with a Gaussian function in specified range X_{range} (from *line 1* to *line 4*);
- to search the peaks in the histogram automatically, after setting conveniently the variables *minpeakheight* and *minpeakdistance* corresponding to the minimum peak height and the minimum separation among near peaks (from *line 5* to *line 12*);
- to save the value of the bin and corresponding count in two columns of a file *.txt* (from *line 13* to *line 20*);
- to save the histogram as a picture *.png* (from *line 21* to *line 24*).

```
1 %Code to fit with a Gaussian function in a specific range of x-axis
2 Xrange = (data ≥ 3.7E-6) & (data ≤ 3.95E-6); % Specify the range of ...
   x-axis
3 histfit(data(Xrange))
4
5 %Code to search peaks: set the MINPEAKHEIGHT and MINPEAKDISTANCE ...
   conveniently.
6 [histo_height, histo_bin] = hist(data, nbins);
7 [peak_counts, peak_timebin] = findpeaks(histo_height, ...
   'MINPEAKHEIGHT', 200, 'MINPEAKDISTANCE', 10);
8 hold on
9 plot(histo_bin(peak_timebin), ...
   peak_counts+20, 'r.', 'markerfacecolor', [1 0 0])
10 hold off
11 percentage = (peak_timebin * 100)/sum(peak_timebin);
12
13 %Code to write a text file (.txt)
14 [histo_height, histo_bin] = hist(data, nbins);
15 A = [histo_bin; histo_height];
16 directory='D://FileMATLAB/histo_fileTXT/histo_data.txt'; % ...
   directory of the folder in which to save data of histogram
17 fileID = fopen(directory, 'w');
18 fprintf(fileID, '%d %d \r\n', A);
19 fclose(fileID);
20
21 %Code to save the histogram as a picture
22 axis([-2E-6 18E-6 0 6000]);
23 directoryFigure='D://FileMATLAB/histo_fileTXT/figura20.png'; % ...
   directory of the folder in which to save the picture of histogram
24 saveas(f, directoryFigure);
```

The entire code of MATLAB script is included in the appendix.

One of the most important questions in data analysis is to choose opportune threshold. In fact some peaks could be hidden by background noise if the threshold is too high

(figure 13 on the top), whereas same or other peaks don't appear in the histogram if the threshold is too low (figure 13 on the bottom). Therefore it is important to select opportune threshold in order to look for a compromise (figure 14).

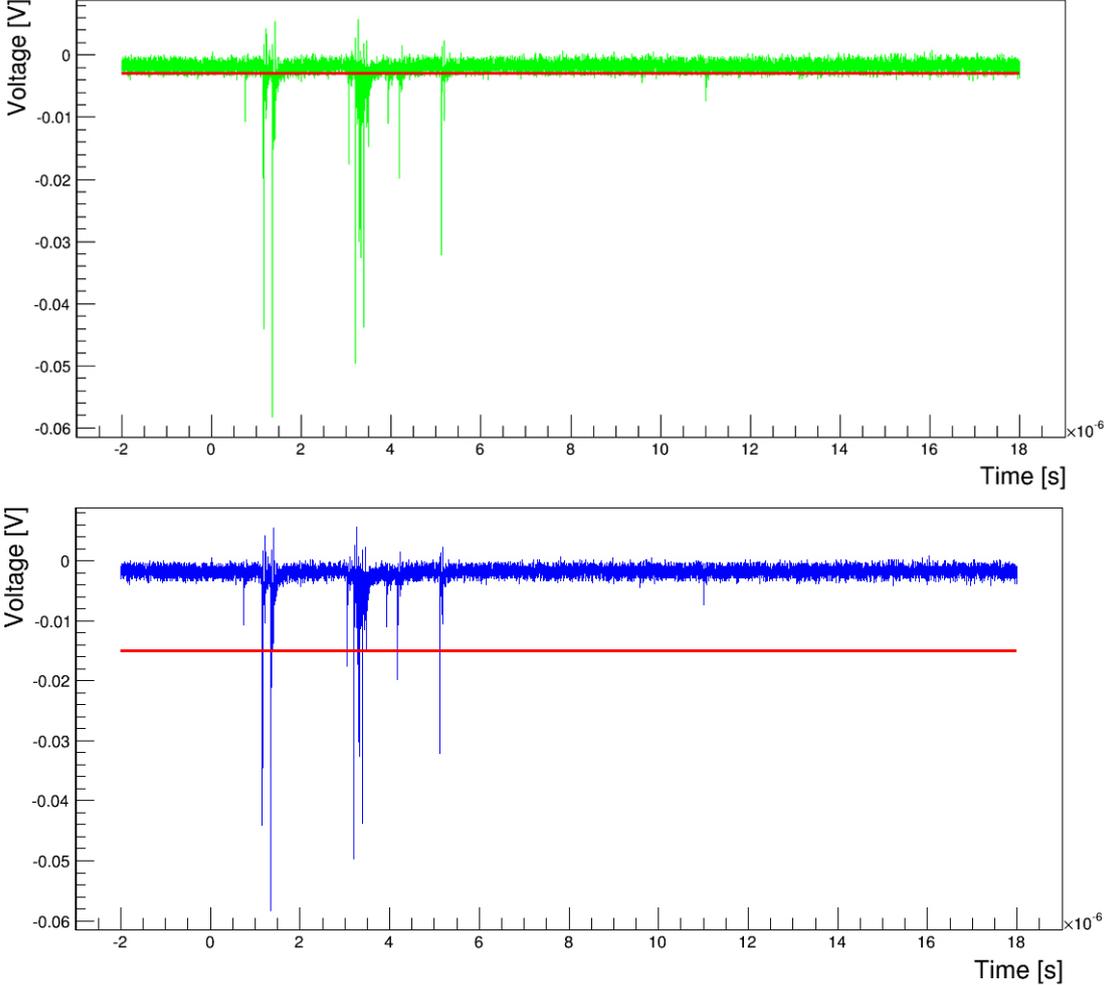


Figure 13: Two examples of waveform with different thresholds. On the top the threshold is equal to -0.003 V and is too high, whereas on the bottom the threshold is equal to -0.015 V and is too low.

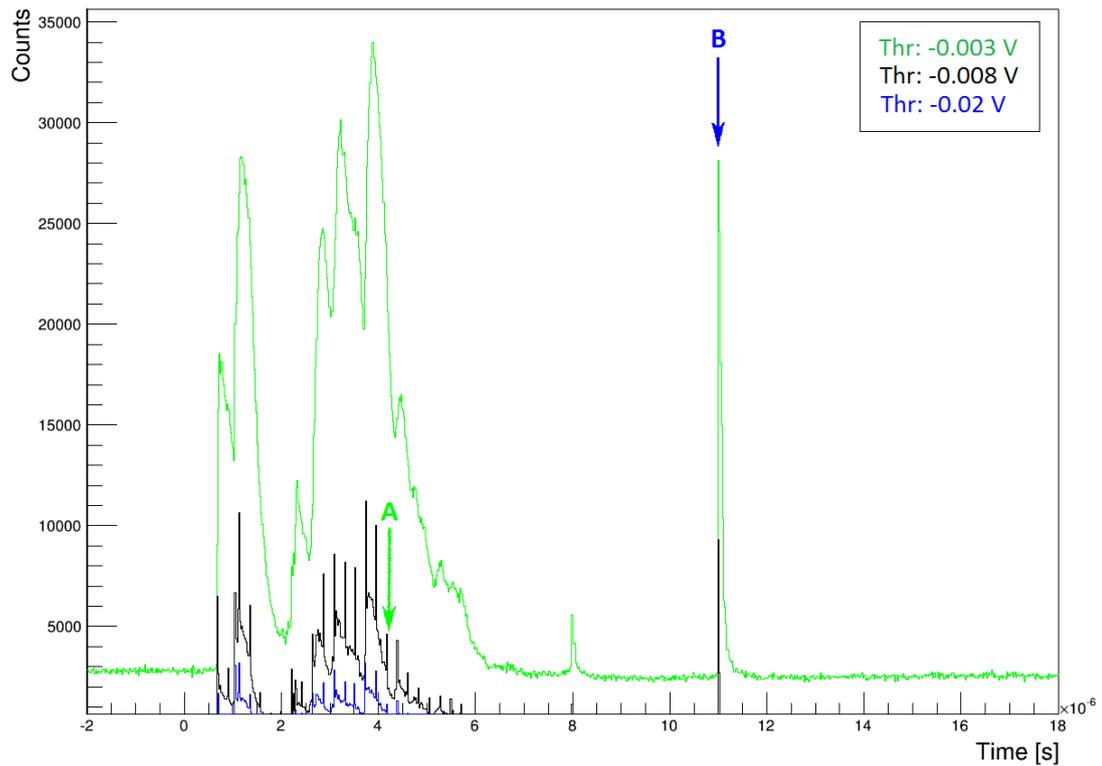


Figure 14: The threshold is equal to -0.003 V (it's too high) to get the green histogram and the threshold is equal to -0.015 V (it's too low) to get the blue histogram. The histogram in black is a compromise for available data, in fact the relative threshold is equal to -0.008 V .

In the *figure* 14 you can see that some peaks (one of these is indicated by the green arrow [A]) are hidden by background noise, whereas other peaks (one of these is indicated by the blue arrow [B]) aren't visible because the threshold is too low.

4 Results and discussion

The most important purpose of this project is to characterize the electron source in order to identify the best parameters for electron ionization inside the vacuum chamber. Other purposes of this project may be:

- to verify the correctness of data measurements, for example to prove the repeatability of data measurements and to check that the time-of-flight of ions is really proportional to the square root of mass-to-charge ratio ($TOF \propto \sqrt{\frac{m}{q}}$);
- to measure some ionization energies of argon gas and to compare them with other and independent values, for example with the values of National Institute for Standards and Technologies (NIST) [10]. It is important to underline that the energy range of electron beam is about 100 – 4000 eV (more information in the paragraph 2.2), so only a few ionization energies will be possible to measure;
- to measure the energy resolution of time-of-flight mass spectrometer on varying of some parameters, for example wehnelt voltage, focus voltage, pressure inside vacuum chamber, etc.

Before starting the data acquisition, we checked the correct operation of the electron source, so the laboratory measurements were compared with the test report of our electron gun, which was done by a SPECS engineer on 15 January 2014.

In this test report, the wehnelt voltage was adjusted for an emission current of about 1 μA , moreover the focus voltage is calculated as a percentage of electron energy (in this case 74%). In the following table and in the *figure 15* you can see some measurements of the test report.

	Electron energy [eV]	Focus voltage [V]	Filament current [A]	Emission current [μA]	Total sample current [μA]	Wehnelt voltage [V]
#1	60	46.8 [78 %]	2.200	1.00	-0.102	5.4
#2	200	148 [74 %]	2.200	1.01	-0.323	9.7
#3	600	444 [74 %]	2.200	1.08	-0.684	21.6
#4	1000	740 [74 %]	2.200	1.00	-0.753	33.5
#5	2000	1480 [74 %]	2.200	1.04	-0.853	63.0
#6	3000	2220 [74 %]	2.200	1.04	-0.886	92.5
#7	4000	2960 [74 %]	2.200	1.03	-0.989	122.0
#8	5000	3700 [74 %]	2.200	0.98	-0.855	151.6

During the data acquisition in the laboratory, the pressure inside the vacuum chamber is always equal to $(6.07 \pm 0.05) * 10^{-8}$ hPa. The laboratory measurements of emission current with the same parameters of test report are not equal to the values in the test report. This mismatch is also present when the focus voltage and wehnelt voltage are changed in whole range. In fact, when the energy of electron beam is set below about 1.5 keV, the emission current is equal to 0.00 μA , but the data measurements in test

report are completely different. Instead if the energy of electron beam is set above more or less 1.5 keV , there are a lot of fluctuations of emission current: in fact this value changes in the entire range ($0 - 250 \mu\text{A}$) rapidly, without ever becoming stable. There are also a lot of fluctuations of electron energy.

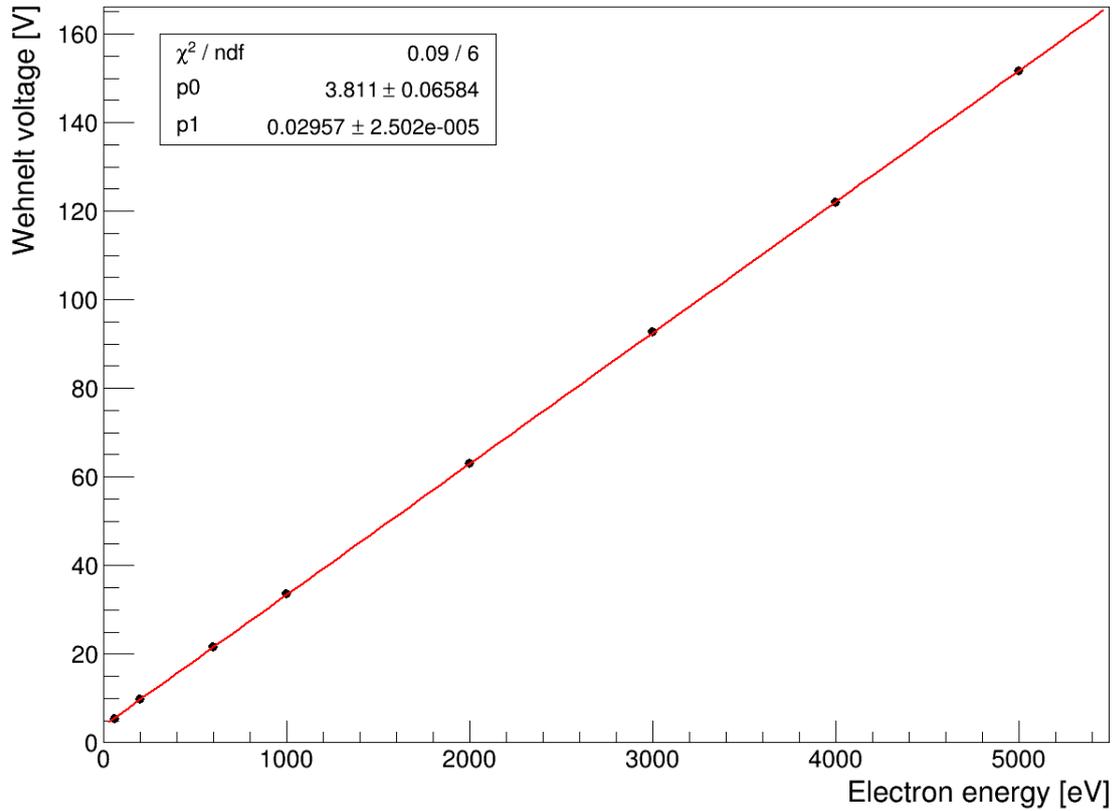


Figure 15: This graph shows the linear dependence between wehnelt voltage and energy of electron beam when the emission current is constant and equal to $1 \mu\text{A}$.

These problems may be due to malfunctioning of electron source and/or its power supply. For these reasons, it was not possible to acquire data during the summer student program at DESY. The electron source is going to be sent back for repair, so it will not be possible to obtain results because of time constraints.

5 Conclusions

The apparatus for data measurements is assembled, so all devices are adjusted and all electronic connections are plugged. The pressure inside the vacuum chamber reaches the desired value ($< 10^{-6} \text{ hPa}$).

A MATLAB script was written to analyse the waveforms coming from the time-of-flight mass spectrometer, in fact it is important to select the signals of ions from noise.

During the summer student program at DESY, it was not possible to acquire data measurements and to identify the best parameters for electron ionization because the electron source does not operate correctly, so it absolutely needs to be repaired.

6 Appendix

In this appendix there are all code lines of MATLAB script.

```
1 %Clear all variables
2 clear;
3 clc;
4
5 %Set the threshold to select the interesting events only
6 threshold = -0.008;
7 disp( strcat('Threshold: ', num2str(threshold), ' V') )
8
9 %Set some variables for the loops and set the number of bins
10 n = 1;
11 data = [];
12 d=dir('D://FileROOT/file_data/test-trc/'); % directory of the ...
    folder in which there are all files .trc
13 nbins=1000; % nbins = number of bins -- if nbins = 10000, then the ...
    width of each bin is equal to 2 ns.
14
15 %Two for loop to read all files (.trc) in the selected folder and ...
    fill the vector data() with events that exceed the threshold
16 for i = 1:(size(d)-2) % processing each file .trc in the folder
17     ReadLeCroyBinaryWaveform( ...
        strcat('D://FileROOT/file_data/test-trc/', d(i+2).name) );
18
19     for k = 1:20001 % lenght of ans.y (and of course ans.x) is ...
        equal to 20000
20         if (ans.y(k) < threshold)
21             data(n)=ans.x(k); % 'time' (x-axis) concerning ...
                the event that exceed the selected threshold
22             n=n+1;
23         end
24     end
25
26     %Clear the ans.x and ans.y, corresponding to the previous event
27     clear ans.x
28     clear ans.y
29 end
30
31 %Plot the histogram: the number of bins is set in the variable 'nbins'
32 f = figure('Visible', 'on'); % on = the canvas is visible or off = ...
    the canvas is not visible
33 hist(data,nbins)
34 title('Spectrum')
35 xlabel({'time [s]'})
36 ylabel({'counts'})
37
38 %Code to fit with a Gaussian function in a specific range of x-axis
39 Xrange = (data ≥ 3.7E-6) & (data ≤ 3.95E-6); % Specify the range of ...
    x-axis
```

```

40 histfit(data(Xrange))
41
42 %Code to search peaks: set the MINPEAKHEIGHT and MINPEAKDISTANCE ...
    conveniently.
43 [histo_height,histo_bin] = hist(data,nbins);
44 [peak_counts,peak_timebin] = findpeaks(histo_height, ...
    'MINPEAKHEIGHT', 200, 'MINPEAKDISTANCE', 10);
45 hold on
46 plot(histo_bin(peak_timebin), ...
    peak_counts+20,'r.','markerfacecolor',[1 0 0])
47 hold off
48 percentage = (peak_timebin * 100)/sum(peak_timebin);
49
50 %Code to write a text file (.txt)
51 [histo_height,histo_bin] = hist(data,nbins);
52 A = [histo_bin; histo_height];
53 directory='D://FileMATLAB/histo_fileTXT/histo_data.txt'; % ...
    directory of the folder in which to save data of histogram
54 fileID = fopen(directory,'w');
55 fprintf(fileID,'%d %d \r\n',A);
56 fclose(fileID);
57
58 %Code to save the histogram as a picture
59 axis([-2E-6 18E-6 0 6000]);
60 directoryFigure='D://FileMATLAB/histo_fileTXT/figura20.png'; % ...
    directory of the folder in which to save the picture of histogram
61 saveas(f,directoryFigure);

```

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