University of Warsaw
Faculty of Physics

EDMR Setup

User's Manual

Abstract:

Electrically Detected Magnetic Resonance (EDMR) setup is an extension of a standard ESR setup. The main challenge was to force two measurement systems to work together. One system is Bruker ELEXSYS E580 ESR spectrometer and the second is Keithley 6221/2182A nanovoltmeter or SR830 lock-in amplifier. Two systems were synchronised with the ARM Cortex M3 microprocessor programmed in C. Whole setup is controlled by the application written in C#.

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Warsaw, 2012

Contents

1. Introduction

The EDMR setup consists of the following components:

1. Keithley 6221 current source,
2. Keithley 2182A nanovoltmeter or Stanford Research SR830 lock-in amplifier,
3. STM32 microcontroller with ARM Cortex M3 microprocessor,
4. EPR Bruker ELEXSYS E580 spectrometer,
5. Computer with the EDMR application for data collection and control.

Magnetic field during the measurement process using the Bruker ELEXSYS E580 spectrometer changes in steps. The spectrometer provides SWA (sweep advance) output in the TTL standard. Falling edge indicates a moment when magnetic field changes. A time when voltage is measured by the lock-in amplifier or the nanovoltmeter can also be accurately determined by observation of appropriate logic states of these devices. The microcontroller records SWA signals and voltage measurement signals and thus can assign the appropriate magnetic field step to specific voltage measurements that were performed during that step. Then via LAN, the microcontroller sends to a computer the magnetic field step number and corresponding numbers of voltage measurements. On a computer runs the application that receives data from the microcontroller, collects measured voltage values and presents data on a plot. Figure 1 presents the scheme of EDMR setup.

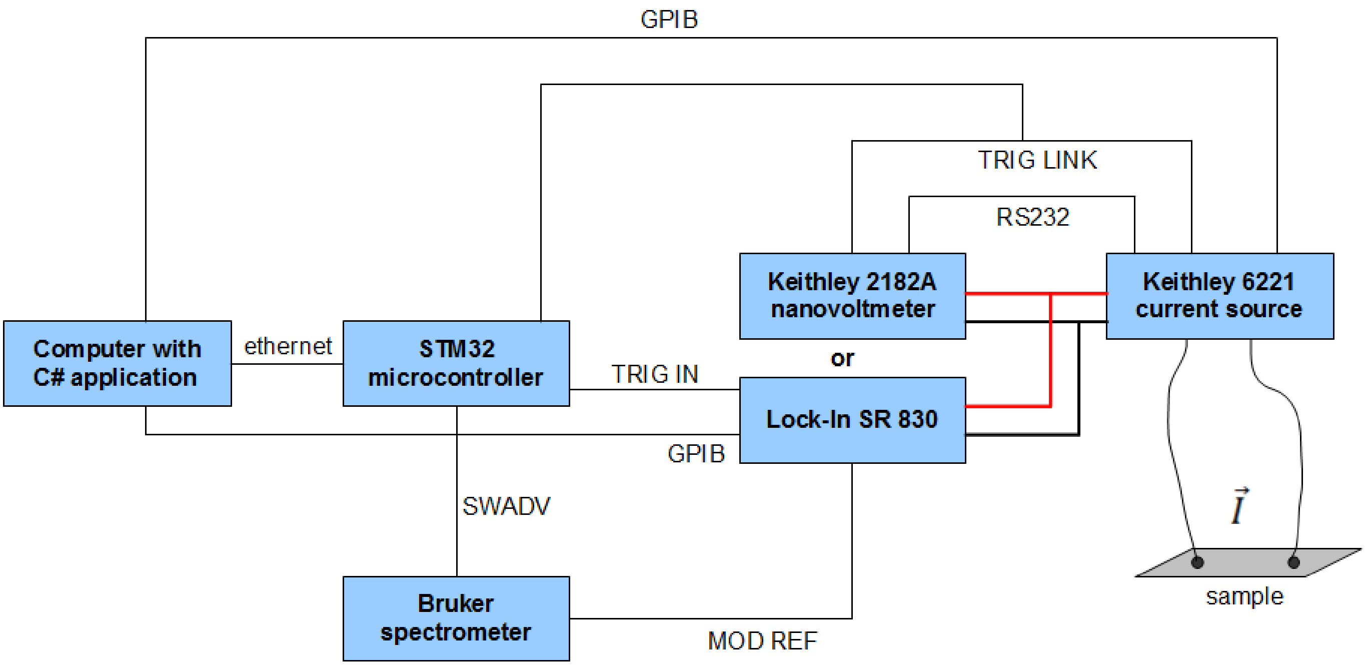


Figure 1. Scheme of the EDMR setup

2. Wiring the microcontroller

The setup can operate in two configurations. The first configuration uses the Keithley 2182 nanovoltmeter as a device for voltage measurements. The second configuration uses the SR830 lock-in amplifier. First two points are the same for both configurations:

1) Plug an ethernet cable to the microcontroller and then turn it on. The microcontroller tries to obtain an IP address using DHCP service. However, if DHCP service is not available in a network, a static IP configuration is used: IP: 192.168.0.8, mask: 255.255.255.0. Once an IP address is set, it is displayed on the microcontroller's screen.
2) Connect the SWA (sweep advance) output, which is located in the "Field controller" module of the spectrometer's console to the **PE0 (J11 26)** pin of the microcontroller. GND of the plug connects to one of the microcontroller's GNDs, for example to the pin J11 33. The pin PE0 and all other pins used in the system are presented in figure 2. Figure 3 presents the SWA output connected to the PE0 (J11 26) pin. Figure 4 shows the location of the SWA in the spectrometer's console.

NOTE: The nominal operating voltage of the microcontroller is 3.3 V. However, most ports tolerates voltage of 5 V. In case you need to use a different port of the microcontroller than described in this manual, check in the microcontroller's specification if a port tolerates voltages higher than 3.3 V.

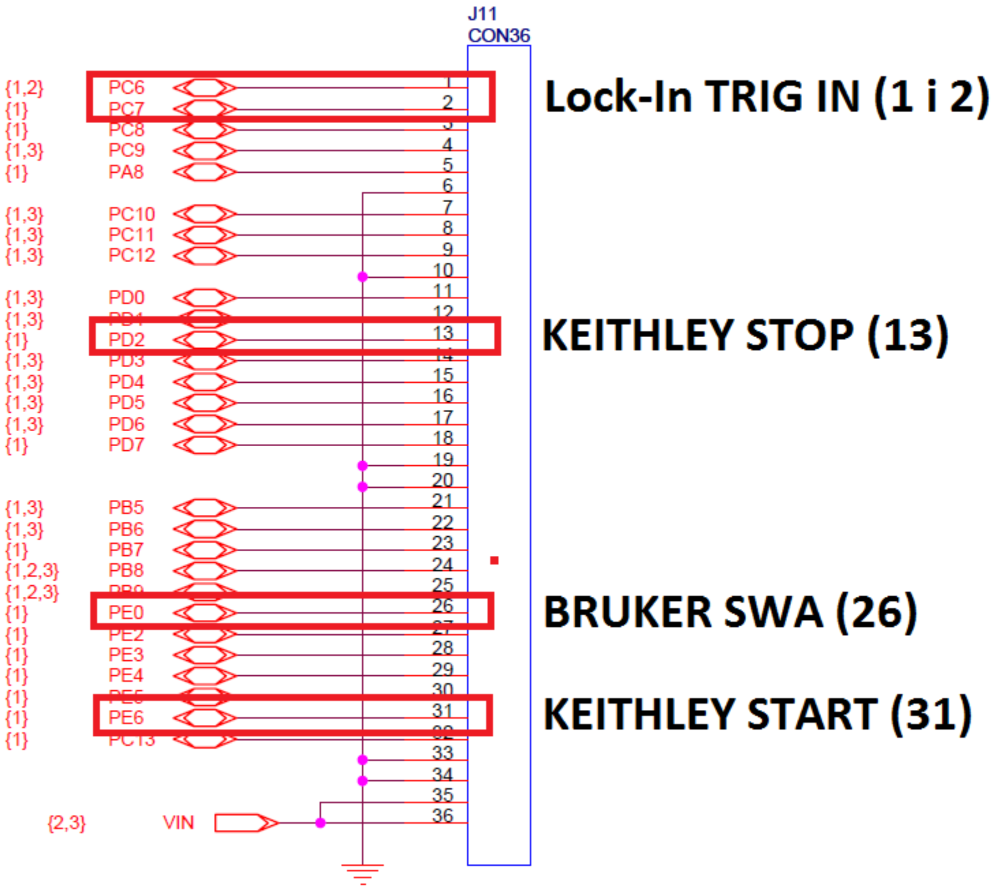


Figure 2. All ports used in the system.

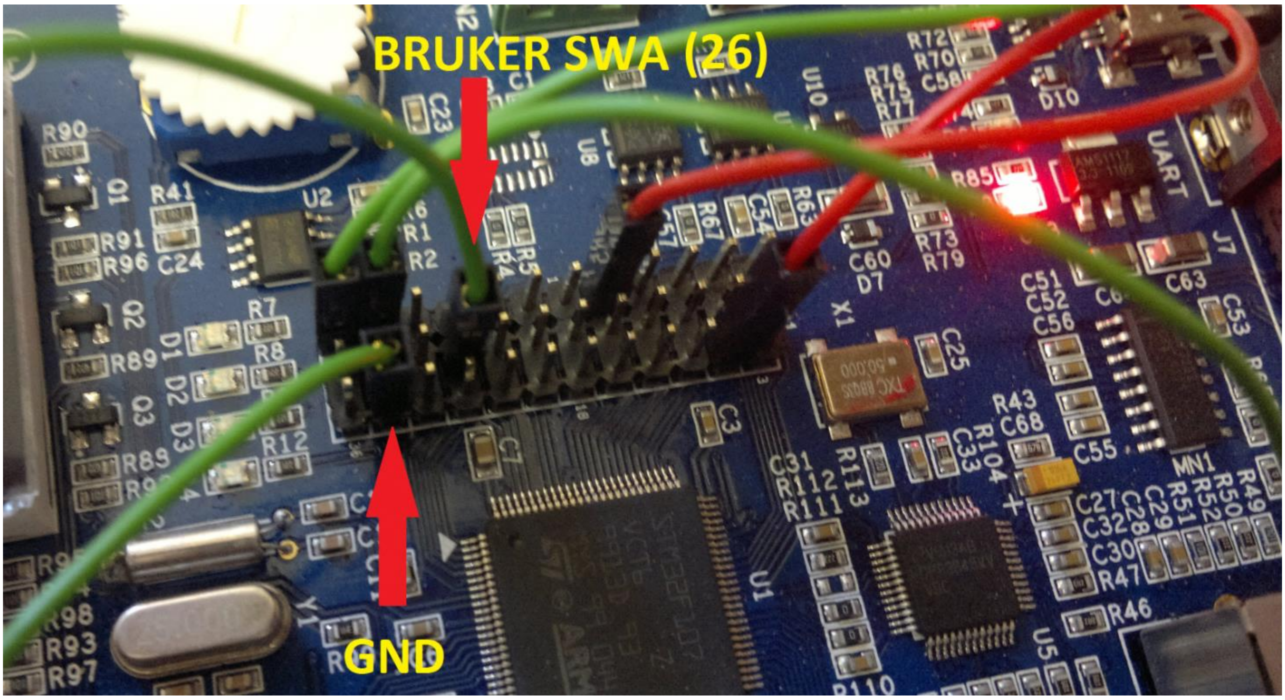


Figure 3. Wiring the SWA output to the PE0 pin of the microcontroller.

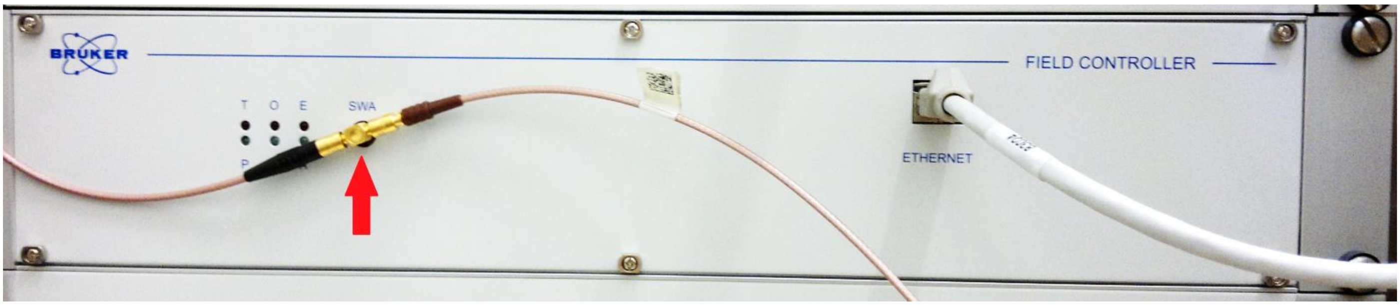


Figure 4. The SWA output on the spectrometer's console.

2.1. Configuration with Keithley 2182A nanovoltmeter

1) Figure 5 shows the triggering sequence between the Keithley 6221 current source and the Keithley 2182A nanovoltmeter. The 6221 Output Trigger (from now on the **Keithley Start**) has to be connected to the **PE6 (J11 31)** port of the microcontroller. The 2182A Output Trigger (from now on the **Keithley Stop**) has to be connected to the **PD2 (J11 13)** port of the microcontroller. Triggering signals coming from Keithley devices are available through the device visible in figure 7. By default, the Keithley Start signal is available on the line number 2, and the Keithley Stop signal is available on the line number 1.

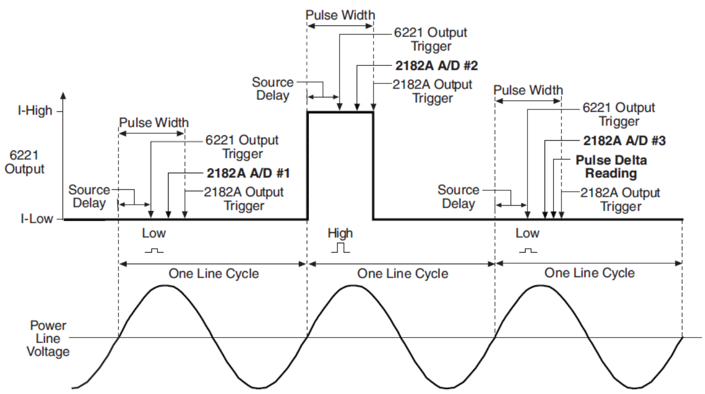


Figure 5. Triggering sequence in the pulse mode.

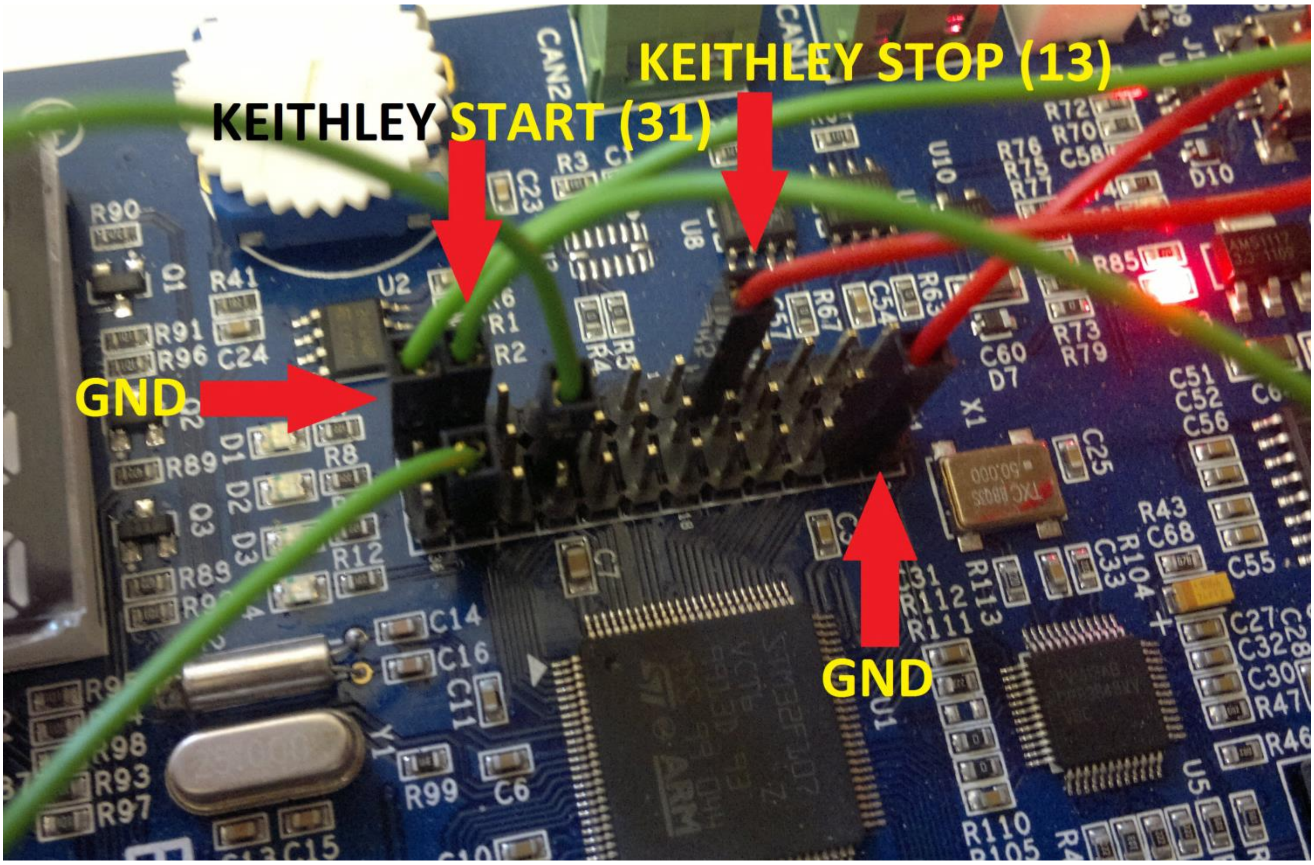


Figure 6. Wiring the KEITHLEY START and the KEITHLEY STOP outputs to the appropriate microcontroller's ports.

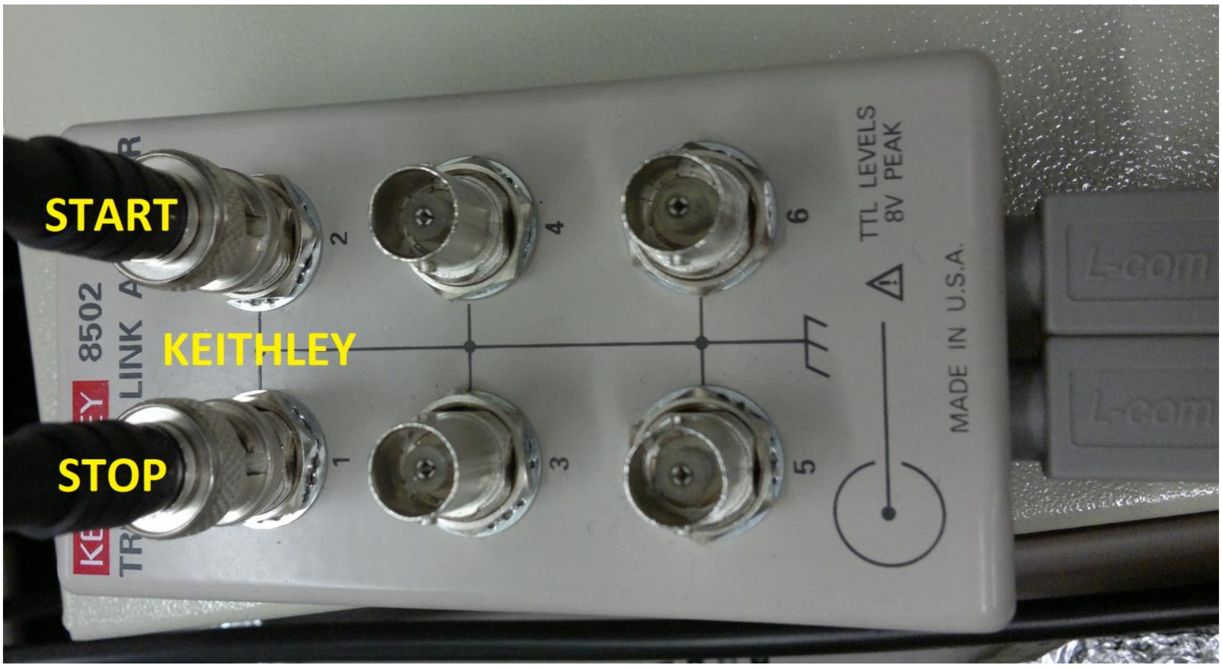


Figure 7. Location of the KEITHLEY START and the KEITHLEY STOP outputs.

2.2. Configuration with SR830 lock-in amplifier

1) Connect **PC6** and **PC7** ports (J11 1 and 2) (fig. 8) to the TRIG IN output of the SR830 lock-in amplifier, which is located on the rear panel (see fig. 9). Connect the reference signal from the spectrometer's console (fig. 10) to the REF IN, which is located on the front panel of the SR830 lock-in amplifier.

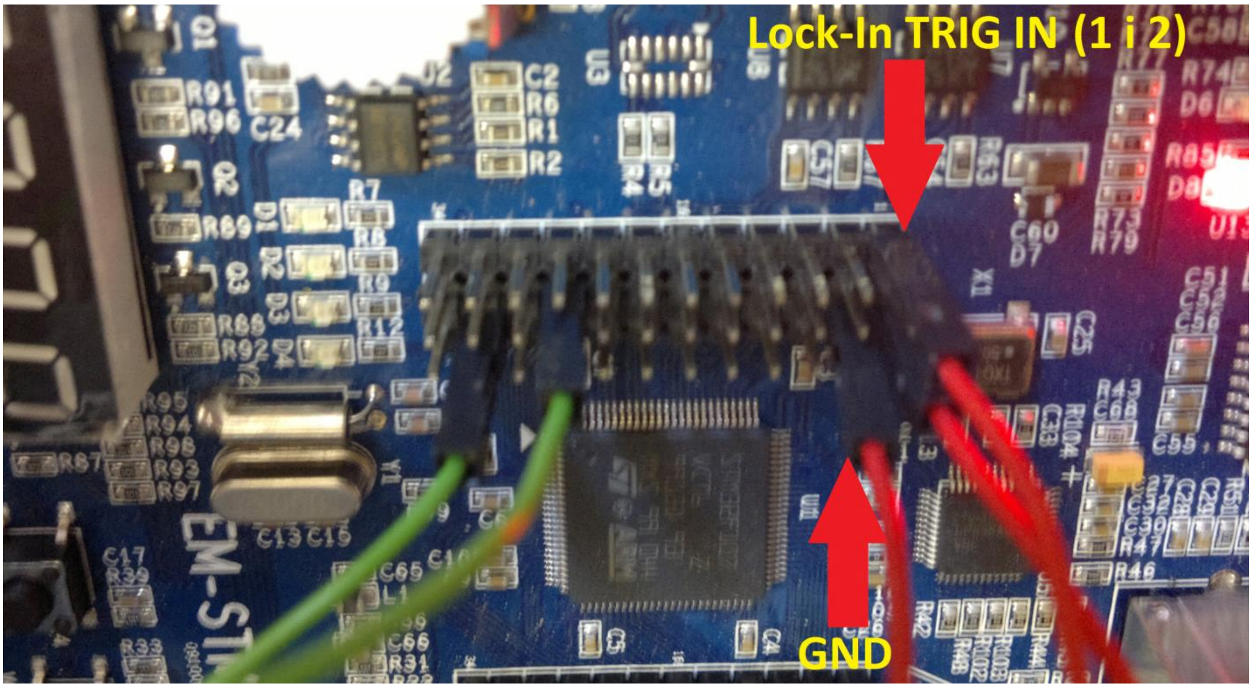


Figure 8. Wiring PC6 and PC7 to the TRIG IN input of the SR830 lock-in amplifier.

Figure 9. The TRIG IN input on the rear panel of the SR 830 lock-in amplifier.

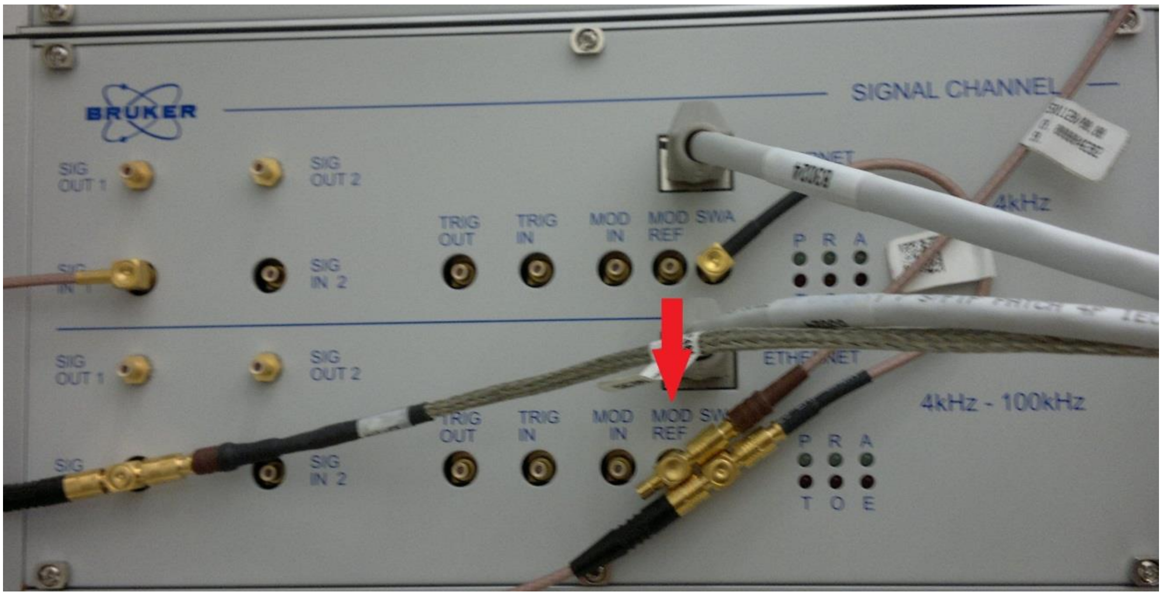


Figure 10. The MOD REF output on the spectrometer's console.

3. Using the application

For proper operation the computer on which the application is launched should meet the following requirements:

- screen resolution at least 1280 x 800,
- installed at least 4.0 version of the .NET framework,
- installed the NI4882 driver; in case the driver is not installed, an attempt to run the measurement by pressing “Measure” button (fig. 12, 16, 17) fails,
- the application was tested on Windows 7 operating system,
- installed a PDF viewer such as Adobe Acrobat Reader for opening attached manuals.

The application allows the user to perform three types of measurements: the **pulsed** and the **delta** mode using the Keithley 2182A nanovoltmeter and the **lock-in** mode using the SR830 lock-in amplifier. Measurement mode should be selected from the menu, as shown in figure 11. The program also allows the user to send any commands to measurement devices connected through GPIB using the **custom code** mode. An example of the custom code mode functionality is given at the end of the chapter.

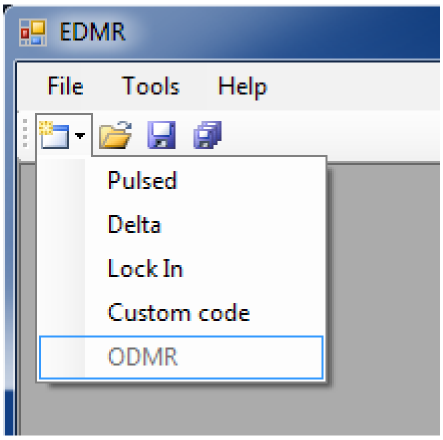


Figure 11. Measurement mode selection menu.

3.1. The pulsed mode

When the pulsed mode is selected, the window shown in figure 12 appears.

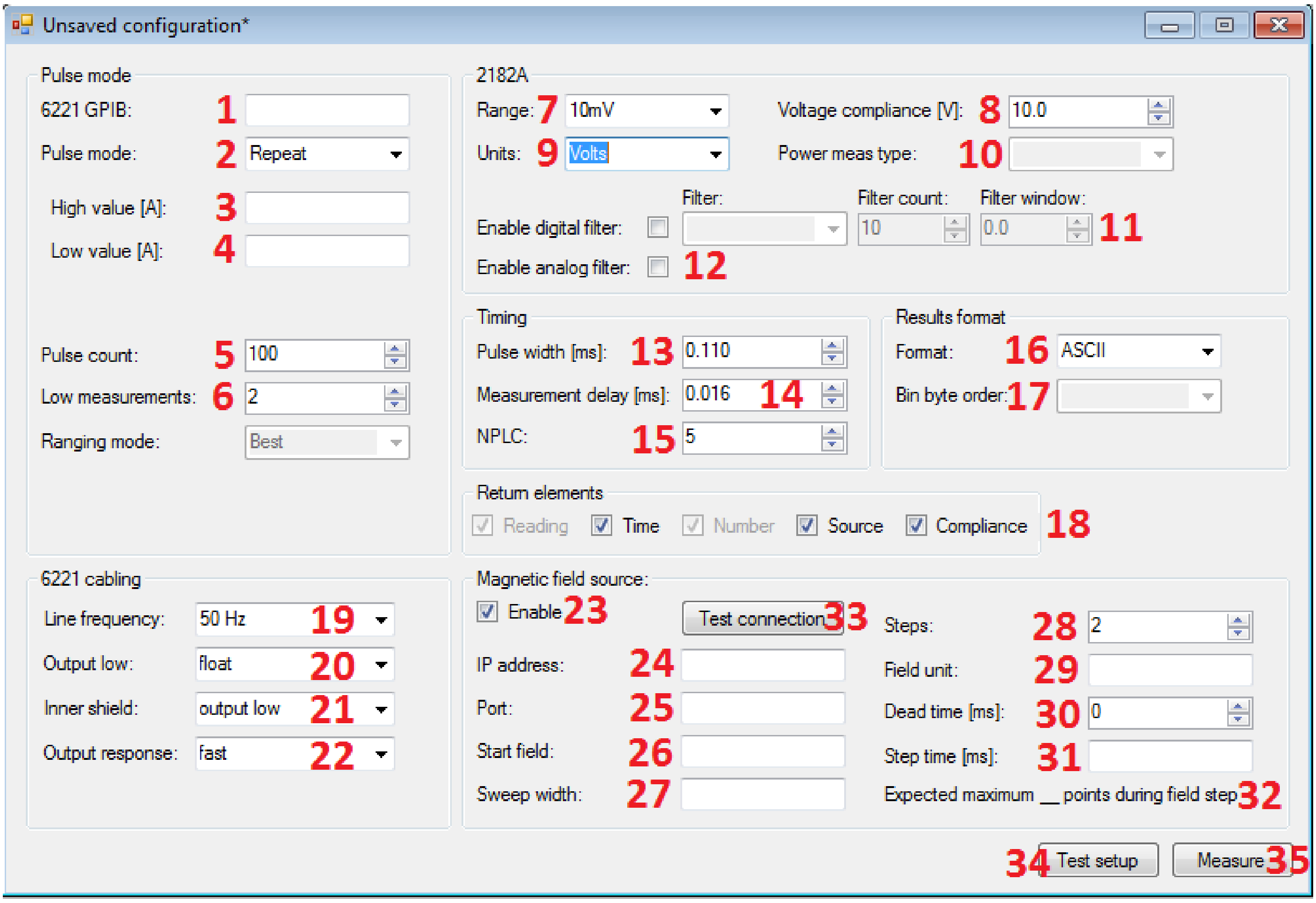


Figure 12. Configuration window in the pulsed mode.

Description of the configuration window (fig. 12) in the pulsed mode:

1) The GPIB address of the Keithley 6221.
2) Possible choices: repeat – pulses of equal current are generated, sweep – current changes, which allows measuring current-voltage characteristics.
3) High current level in the repeat mode. In the sweep mode set initial and final current level and sweep type: linear or logarithmic.
4) Low level current in the repeat mode.
5) Number of current pulses that are generated. When synchronisation with the magnetic field is enabled, it is recommended to set the maximum number of pulses – 65536.
6) Specifies how many voltage measurements are performed when current level is low. Possible choices: 1 – measurement is made before a current pulse is generated or 2 – measurement are made before and after a current pulse.
7) Measuring voltage range. Possible choices: 10 mV, 100 mV, 1 V, 10 V, 100 V.
8) Maximal allowed voltage level.
9) Choice of measurement units, possible values: Volts, Ohms, Watts, Siemens.
10) Selection of the power measurement algorithm, possible values: Average or Peak. That field is active only when Watts are selected in the previous point.
11) Digital filter settings (see 6221 and 2182A manuals for more details).
12) Analog filter enable/disable selection (see 6221 and 2182A manuals for more details).

13) Selection of the current pulse time width. Available range: 0,05 – 12 ms.
14) Voltage measurements start after the time specified in that field. Available range: 0,016 – 11,966 ms.
15) That field specifies current pulses generation frequency. The time between pulses can be calculated as follows: time=NPLC/(50 Hz or 60 Hz).
16) Format of data sent from Keithleys to a computer. Possible choices: ASCII, Single Precision Bin, Double Precision Bin.
17) Bytes order when Single or Double Precision Bin was selected in the previous point. Possible options are Swapped or Normal.
18) Kind of the data sent to a computer.
19) Current frequency in a mains power system used in a laboratory (in Poland – 50 Hz).
20) Specifies how output low (LO) of the Keithley 6221 is connected. Possible choices are float (fig. 13 A) or earth ground (fig. 13 B). **If the DUT has any low impedance path to earth ground, do not connect source-LO to earth within the 6221.** Connecting LO to earth can cause ground loops that will drive potentially large currents through leads, DUT contacts or even through the DUT itself. **If the DUT has no paths to earth, then connecting LO to earth within the 6221 will simplify connections, cabling and shielding.** LO can then be carried out to the DUT on the outer shield of the triax cable, rather than on a separate wire. (source – *help of the KI6220 application)*

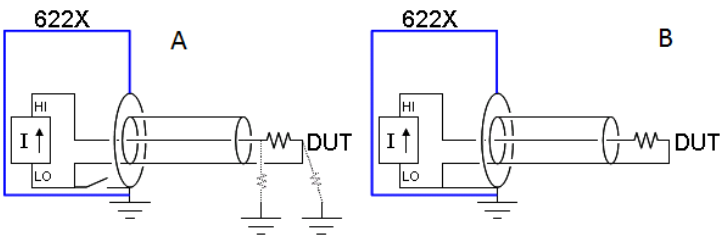


Figure 13. Output low (LO) of the 6221: A) floating, B) grounded. ***(source – help of the KI6220 application)***

21) Specifies how inner shield of a triax cable is connected. Possible choices are output low (fig. 14 A) or guard (fig. 14 B). **In the unguarded mode, cabling is simple, but cable (and voltmeter) capacitance may limit the speed of output current. The higher RDUT is, the slower the response.** If the 2182A nanovoltmeter is connected directly across the DUT (4W connections), it will add at least 500 pF, so eliminating cable capacitance will not be useful. **In the guarded mode, a driven guard eliminates cable capacitance for faster output current.** The source LO must be carried to the DUT on a separate wire. If it is acceptable to connect LO to earth within the 6221, then the outer shield of the triax cable can be used for this. (source – *help of the KI6220 application)*

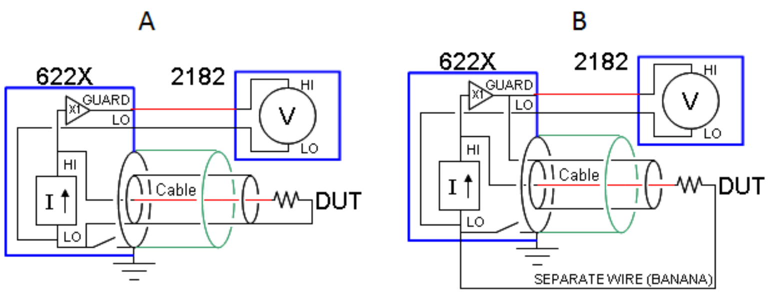


Figure 14. Inner shield connected to: A) output low (LO), B) as cable guard. ***(source – help of the KI6220 application)***

22) The 2182A nanovoltmeter has internal capacity that helps to reduce current drawing from the 2182A through the DUT. For most 2 wire measurements, 2182A capacitance does not need to be reduced. If you need the fastest response for the 2182A measuring the guard output (e.g. for pulsed measurements), the 2182A input capacitance can be minimised, to about 1/3 of its normal value. Note that on the 100 V and 10

mV measuring ranges, the 2182A rise time is limited by other factors, so the capacitance will not affect total response time. (source – *help of the KI6220 application)*

23) Magnetic field synchronisation enable/disable selection.
24) IP address of the microcontroller.
25) Microcontroller's port numer. By default set to 23.
26) Initial magnetic field set in the xepr application.
27) Magnetic field sweep width set in the xepr application.
28) Number of magnetic field steps set in the xepr application.
29) Choice of the magnetic field unit.
30) The time from the change of the magnetic field during which voltage measurements are rejected.
31) Time width of the magnetic field step (value can be taken from the xepr application). This field is not mandatory however when set, allows estimating how many voltage measurements will be performed during one magnetic field step.
32) Estimated number of voltage measurements performed during one magnetic field step. More voltage measurements, better averaging.
33) Tests connection between the microcontroller and a computer.
34) Clicking that button validates all fields and checks the availability of the Keithley 6221 and the Keithley 2182A.
35) Clicking that button displays the window for measurement process control (fig. 15).

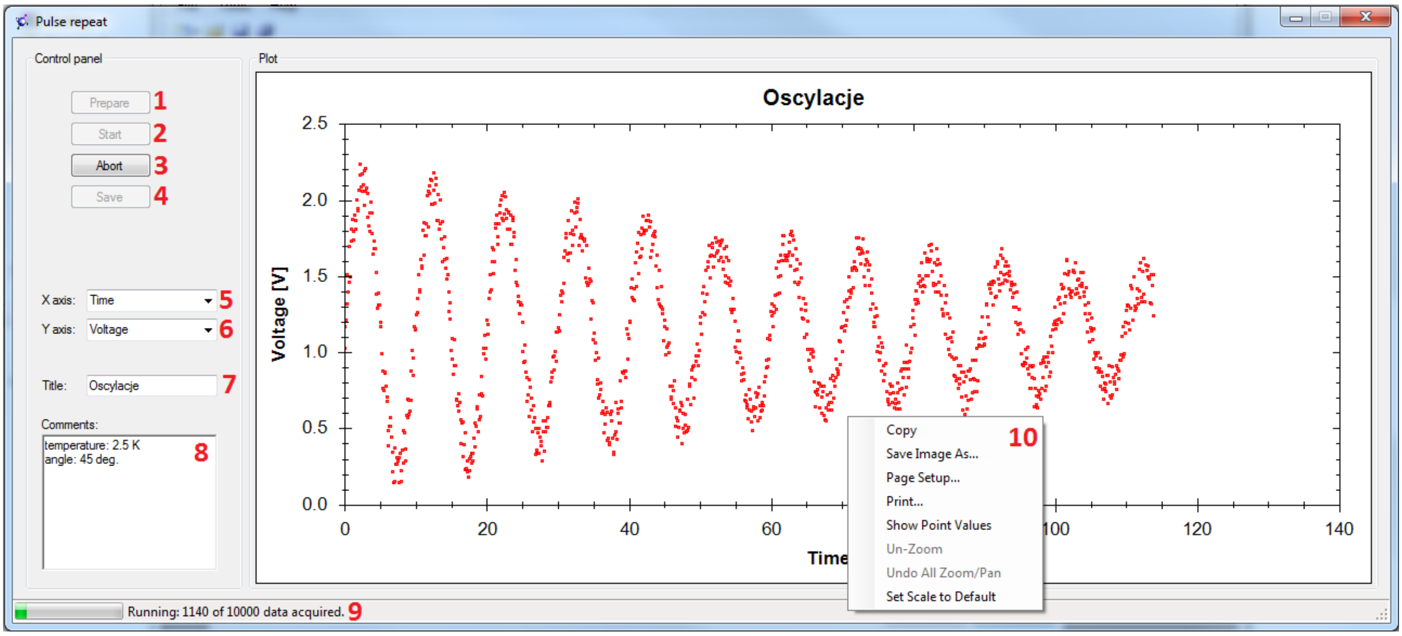


Figure 15. Measurement window.

The measurement window consists of the following elements:

1) After the prepare button is clicked, all configuration parameters selected in the previous window are sent to appropriate devices.
2) The start button initializes the measurement process. With enabled synchronisation with the magnetic field, the start button should be clicked before the measurement is started in the xepr application.
3) The abort button stops the measurement process.
4) The save button allows the user to save measured values to a file.
5) Choice of units at X axis.
6) Choice of units at Y axis.
7) The title of plot.
8) A text from the comments field will be saved to an output file.

9) Progress bar shows how many voltage measurements were made.
10) Context menu allows the user to: copy plot picture to a system clipboard, save plot picture to a file, print plot picture, display values, zooming.

3.2. The delta mode

When the delta mode is selected, the window shown in figure 16 appears.

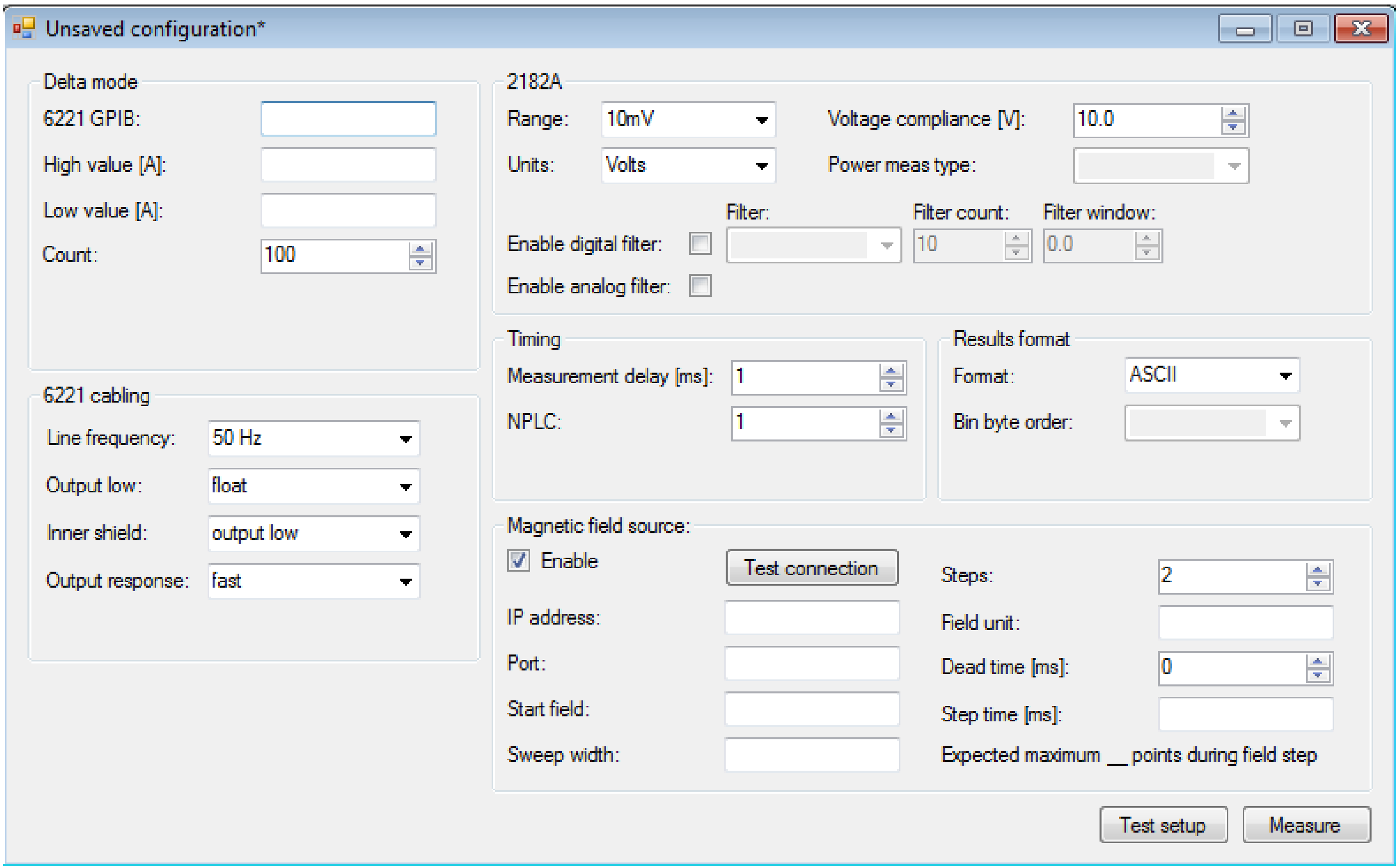
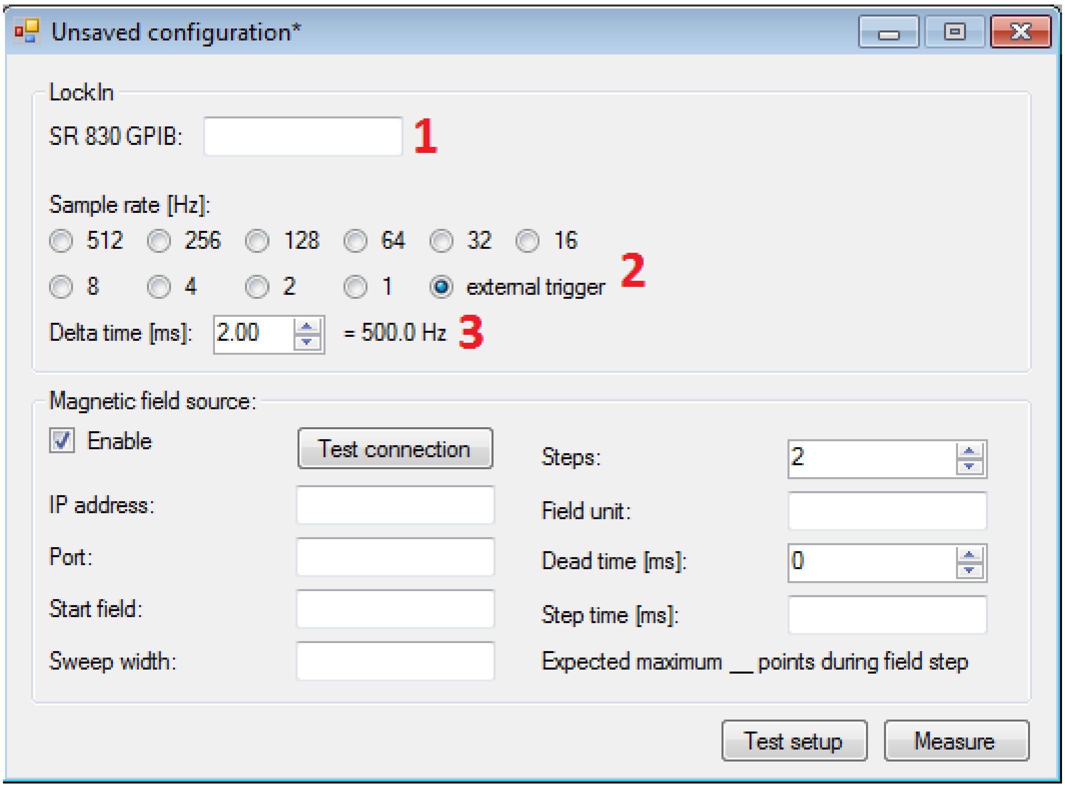


Figure 16. Configuration window in the delta mode.

Most of the fields are the same as in the pulsed mode. In the delta mode high and low current level has to be set.

3.3. The lock-in mode

When the lock-in mode is selected, the window shown in figure 17 appears.



Rysunek 17. Configuration window in the lock-in mode.

Description of the configuration window (fig. 17) in the lock-in mode:

1) The GPIB address of the SR830 lock-in amplifier.
2) Voltage measurement frequency selection. When synchronisation with the magnetic field is enabled, then external trigger option has to be selected.
3) When external trigger option is selected in the previous point, then voltage measurement frequency has to be set by specifying the time between measurements.

Description of fields from the "Magnetic field source" area is the same as in the pulsed mode (see points 23 – 32).

3.4. The custom code mode

The custom code mode allows the user to send any commands to measurement devices connected by GPIB. Figure 18 shows a window with a sample code that will be sent to a device with 12 GPIB address. The code from the figure 18 sent to the Keithley 6221 current source generates sin wave with an amplitude of 1 mA and frequency 1000 Hz.

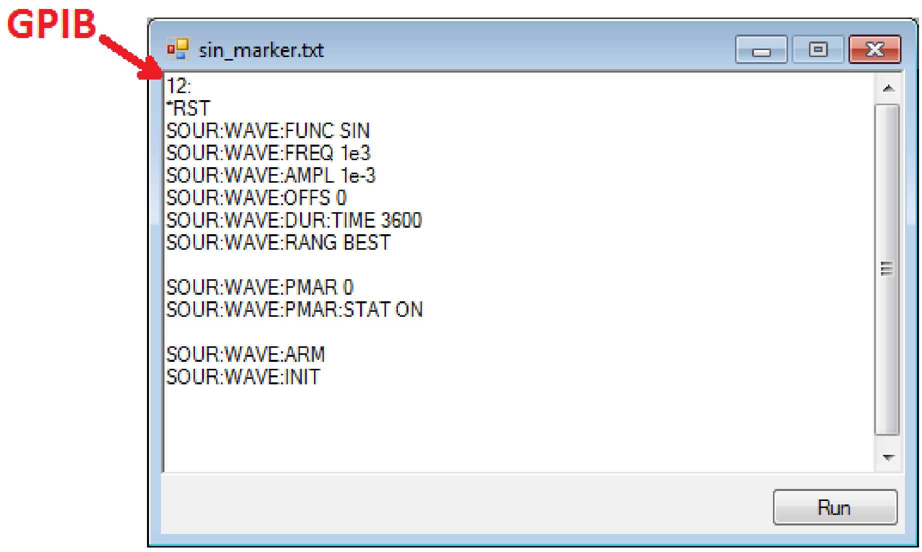


Figure 18. Custom code.

The GPIB address is set in the first line. Commands from next lines will be sent to that address. The GPIB address may also be set before a command, see and analyse the following code:

```
12:
command 1
command 2
13: command 3
command 4
14:
command 5
command 6
```

Commands 1 and 2 will be sent to the GPIB address number 12, command 3 will be sent to the address number 13, command 4 to the address number 12, commands 5 and 6 to the address number 14.

4. Final remarks and ideas for future improvements

Helpful tips for configuring measurement in the pulsed mode and the delta mode using Keithley devices are available in the help of the program KI6220. Figures 13 and 14 come from this program.

In the pulse and the delta mode sometimes measured values are arranged around zero and are alternately positive and negative. Resetting Keithleys 6221 and 2182 solves the problem.

Keithley devices are synchronised with mains frequency (in Poland – 50 Hz). It means that time *t* between pulses should equal to multiple of 20 ms when mains frequency is 50 Hz. In the delta mode for NPLC = 1 *t* should equal to 20 ms, however equal to 27,5 ms. For NPLC = 3 *t* = 60 ms but equals to 63 ms, for NPLC = 5 real time *t* = 103 ms, for NPLC = 7 real time *t* = 147 ms, for NPLC = 10 real time *t* = 207 ms. The problem occurs only in the delta mode, in the pulsed mode time between pulses is correct.

The setup can be extended/improved as follows:

- Use FAST2 mode for transferring data to a computer from the SR830 lock-in amplifier. At the moment data is read from the buffer, which has a capacity of 16383 measurements. For the frequency equal to 512 Hz buffer fills up in about 32 seconds. In the FAST2 mode buffer is not used because the data are continuously transmitted to a computer.
- In the present setup, data is sent from the microcontroller to a computer via TCP/IP. To transfer, one can try using a USB port. It may involve the need to write the appropriate driver for Windows.
- The setup can be extended with the ODMR (Optically Detected Magnetic Resonance).