ORIGINAL INSTRUCTION

ELECTRON PARAMAGNETIC
RESONANCE SPECTROMETER

ADN23.00.00.000-06 OM

# SPINSCAN X

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This operation manual ADN23.00.00.000-06 OM for the Electron Paramagnetic Resonance Spectrometer (EPR) SPINSCAN X (hereafter referred to as EPR Spectrometer or Spectrometer) contains information on the design, operation preparation, operation procedures, maintenance and safety measures, transportation and storage.

Only personnel who have fully studied this manual and received the appropriate training in the manufacturer's program is allowed to work with the Spectrometer.

# TABLE OF CONTENTS

# WARNING SYMBOLS

Three types of warning symbols are used in this manual: in order of importance is **Warning, Caution** and **Notice**. Their description is given below.

| | |
|---|---|
| ⚠ WARNING | **INDICATES CONDITIONS OR SITUATIONS WHICH, IF NOT AVOIDED, COULD CAUSE SERIOUS PERSONAL INJURY AND/OR CATASTROPHIC DAMAGE TO THE EQUIPMENT OR DATA.** |
| ⚠ CAUTION | **Informs of conditions or situations important for equipment proper operation.** |
| NOTICE | *Notice of advisory nature which requires paying attention to specific facts or conditions.* |

# GENERAL

## SECTION 1

# 1 GENERAL

## 1.1 GENERAL DESCRIPTION

The SPINSCAN X Electron Paramagnetic Resonance[1] (hereafter, EPR) spectrometer (Fig.2.1) is intended for recording spectra and measuring the spectral parameters of EPR signals. Electron paramagnetic resonance spectrometer X range (frequency range – 9,3-9,5 GHz, magnetic field sweep from 0 to 750 mT) SPINSCAN X (Figure 1.1) is designed for registration of EPR spectra of paramagnetic substances[2] in liquid or solid phase, as well as for measuring the spectra parameters (intensity, line width, etc.) The EPR spectrometer may be used for solving a wide range of applied problems in various fields of science and technology (physics, chemistry, biophysics, geology, medicine, etc.).

Spectrometer operation control, registration and measurement of sample spectrum parameters are performed from the operator's work station based on a personal computer with specialized software.

The main functional assemblies of SPINSCAN X are an EPR signal sensor unit with an electromagnet and operating resonator, magnetic field control unit, processing and setting unit, power supply unit.


**EPR** is the effect of absorbing resonant electromagnetic radiation by a paramagnetic substance in a constant magnetic field. Electron paramagnetic resonance is determined by quantum transitions between magnetic sublevels of paramagnetic atoms and ions (Zeeman effect). Electron paramagnetic resonance spectra are observed mainly in the microwave range.

[1] **Paramagnetic substance** is a substance that is magnetized in an external magnetic field in the direction of an external magnetic field and has a positive magnetic susceptibility, the value of which is much lower than 1.

Figure 1.1 – SPINSCAN X

The spectrometer provides an opportunity to research samples in cuvettes, ampoules, capillaries and tubes placed in the resonator. The working area of the resonator is a cylinder with a diameter of 8 mm and a height of 15 mm.

The spectrometer is classified by the following type:

- *Protection against electric shock*: Class I – Type B applied parts;
- *Ingress protection rating:* IP20;
- *The degree of protection provided by the ESR spectrometer case against external mechanical influences: IK06.*

The spectrometer should not be used in places where there is a risk of explosion or fire.

The spectrometer is electrically powered from a single-phase general-purpose AC power supply with a frequency (50/60) Hz and a nominal voltage of 110-240 V.

## 1.2 OPERATION PRINCIPLES

The paramagnetic sample is positioned inside a measuring resonator located between the poles of the electromagnet. Microwave power with a frequency equal to the resonator frequency is supplied to the resonator with the sample. The magnetic field generated by the electromagnet changes over time according to a linear law and is simultaneously modulated at high frequency. When magnetic field induction reaches a value that corresponds to the condition of appearance of quantum transitions between the Zeeman levels of paramagnetic particles, a resonant absorption of microwave oscillation energy occurs. It leads to a proportional change in the microwave signal reflected from the resonator. Microwave signal is converted to an EPR signal at magnetic field modulation frequency. The amplified Fourier-transformed signal is measured as an absorption derivative of the magnetic field induction function.

## 1.3 OPERATOR'S WORK STATION (AWS)

EPR spectrometer has Ethernet interface and is connected to the AWS via an Ethernet cable.

The spectrometer software allows to run the spectrometer r and it has the following functionalities:

- Parameters setting central value of polarizing magnetic field induction;
- the width and time of the magnetic field sweep;
- magnetic field modulation amplitude;
- attenuation of microwave power / microwave power;

  - time constant;
  - number of smoothing points;
  - number of sweep points;
  - number of scans;
  - delay before scans;
  - left side offset.
- Resonator and microwave bridge connection control;
- Measurement of EPR spectra;
- Processing of EPR spectra:
  - spectrum parameters determination;
  - zoom;
  - baseline correction;
  - signal detection phase adjustment;
  - spectrum line parameters determination (report with g-factor calculation);
  - integration and differentiation;
  - peaks identification and analytical curve fitting;
  - subtraction (addition) of spectra (Linear algebra);
  - multiple harmonics;
  - extraction.
- Export / Import of data;
- Data archiving;
- Printing;
- Automatic self-diagnostics of parameters and diagnostics by remote access via the Ethernet interface.

* *Completeness of the ARM - in agreement with the customer. More detailed information is given in Section 10.*

## 1.4 PRODUCT IDENTIFICATION

There are plates on the spectrometer shell containing the following inscriptions (when delivered for export - in accordance with the contract terms):

- Trademark and manufacturer;
- Model;
- Voltage;

- Freqency;
- Current;
- Classification;
- Serial No.;
- Date of manufacture (month, year);
- Place of manufacture;

Besides, there are signs on the housing::

**- «Warning, Danger», «Refer to operation manual»**

**- «Caution! Electric voltage»**

Packaging for the transport of the spectrometer is marked with basic and additional inscriptions according to the design documentation, and with marking signs such as **“Top”**, **“Fragile”**, **“Handle with care”**, **“Keep dry”**, **“Temperature limitations”**.

# SAFETY

## SECTION 2

# 2 SAFETY

## 2.1 GENERAL INFORMATION

**WARNING**

**IF SAFETY PRECAUTIONS ARE NOT FOLLOWED, ANY ELECTRICAL EQUIPMENT IS A HAZARD TO PERSONNEL.**

Although the spectrometer is designed to be safe from injury, electric shock and micro-wave radiation, personnel must be familiar with the current safety standards, regulations and instructions.

**WARNING**

**ALL PERSONS INVOLVED IN OPERATION SHOULD READ THE SAFETY INSTRUCTIONS IN THIS MANUAL.**

**BEFORE PUTTING THE SPECTROMETER INTO OPERATION, THE CONTENTS OF THIS DOCUMENT AND THE INSTRUCTIONS (AND OTHER TECHNICAL DOCUMENTS) FOR ADDITIONAL COMPATIBLE EQUIPMENT MUST BE CAREFULLY STUDIED.**

## 2.2 RESPONSIBILITIES

Personnel operating the spectrometer should take appropriate precautions to protect against microwave radiation, which may affect those present due to their carelessness, negligence, or ignorance.

**WARNING**

**THE SPECTROMETER DESCRIBED HEREIN IS SOLD WITH THE CONDITION THAT NEITHER THE MANUFACTURER (ADANI), NOR THEIR AGENTS OR REPRESENTATIVES BEAR RESPONSIBILITY FOR ANY HARM TO HEALTH OR ANY OTHER DAMAGE WHICH CAN RESULT FROM EXCESSIVE MICRO-WAVE RADIATION THROUGH THE BUYER'S FAULT.**

**ADANI DOES NOT ACCEPT ANY RESPONSIBILITY FOR THE SPECTROMETER THAT HAS NOT BEEN SERVICED AND MAINTAINED IN ACCORDANCE WITH THE MANUFACTURE INSTRUCTIONS, OR WHICH HAS BEEN MODIFIED OR TEMPERED WITH IN ANY WAY.**

**DO NOT USE THE SPECTROMETER WITH THE OPEN PANELS.**

## 2.3 ELECTRICAL SAFETY

In terms of electrical safety, the spectrometer complies with IEC 61010-1-2014, according to the type of protection against electric shock, the spectrometer belongs to Class I.

The housing of the spectrometer ensures protection against contact with dangerous parts and against penetration of solid objects (degree of protection is IP20).

The spectrometer ensures an electrical connection of all metal non-current-carrying parts that may be under voltage, with grounding elements.

The value of the total electrical impedance between the protective grounding terminal and any available metal part having protective grounding does not exceed 0,1 Ohm.

The design of the spectrometer in terms of insulation of conductive parts, the size of gaps and leakage paths meets the requirements of the installation category (overvoltage category) II at pollution degree II according to IEC 61010-1-2014.

The ground-leakage current in normal condition does not exceed 3,5 mA.

## 2.4 OPERATING CONDITIONS

The device is intended for use in rooms at ambient temperature from 10°C to 35°C.

## 2.5 PHYSICAL FACTORS PARAMETERS

The electrostatic field intensity on the spectrometer surface corresponds to local regulations and does not exceed 20 kV/m.

The electrostatic field intensity of a 50 Hz industrial frequency current generated by the spectrometer complies with the local regulations and does not exceed 5,0 kV/m.

The electrostatic field intensity generated by the spectrometer complies with the local regulations and does not exceed 50,0 V/m.

The noise level of the EPR spectrometer does not exceed 70 dBA.

If it is necessary to measure or calculate the sound pressure level, the responsible authority should measure the sound level both in the operator's area during normal use and at a distance of 1 m from the spectrometer housing.

## 2.6 MECHANICAL SAFETY

When using the spectrometer, there may be a risk of injury when carrying it.

In order to prevent injuries from protruding parts of the device, all edges, protrusions, corners, holes, handles are designed smooth and rounded.

**WARNING** **SPINSCAN X SHOULD BE CARRIED BY TWO PERSONS.**

## 2.7 EMERGENCY ACTIONS

In case of an emergency, such as:

- damage to the spectrometer microwave radiation protection;
- short circuit or breakage in the electric circuits of the spectrometer,
- electric current passing through the human body;
- mechanical damage to the spectrometer elements;

Disconnect the spectrometer from the electric mains by pressing the emergency stop button and provide first aid to the injured (if necessary). Then switch off the computer and contact the spectrometer warranty or post-warranty service company.

# INTRODUCTION

## SECTION 3

# 3 INTRODUCTION

## 3.1 DESCRIPTION

The spectrometer SPINSCAN X consists of a base where all main components of the device are mounted on. For more efficient air cooling of the spectrometer there is a grid and fans on the rear and side panels (Fig. 3.1).

The top cover of SPINSCAN X has a recess in which the holder for the cuvette (capillary, ampoule, etc.) is located. On the front facade of the spectrometer there is a window for supplying the radiation to the sample in the measuring resonator, protected with a decorative screw. Before installing the radiation source, the decorative screw must be unscrewed (Fig. 3.2).

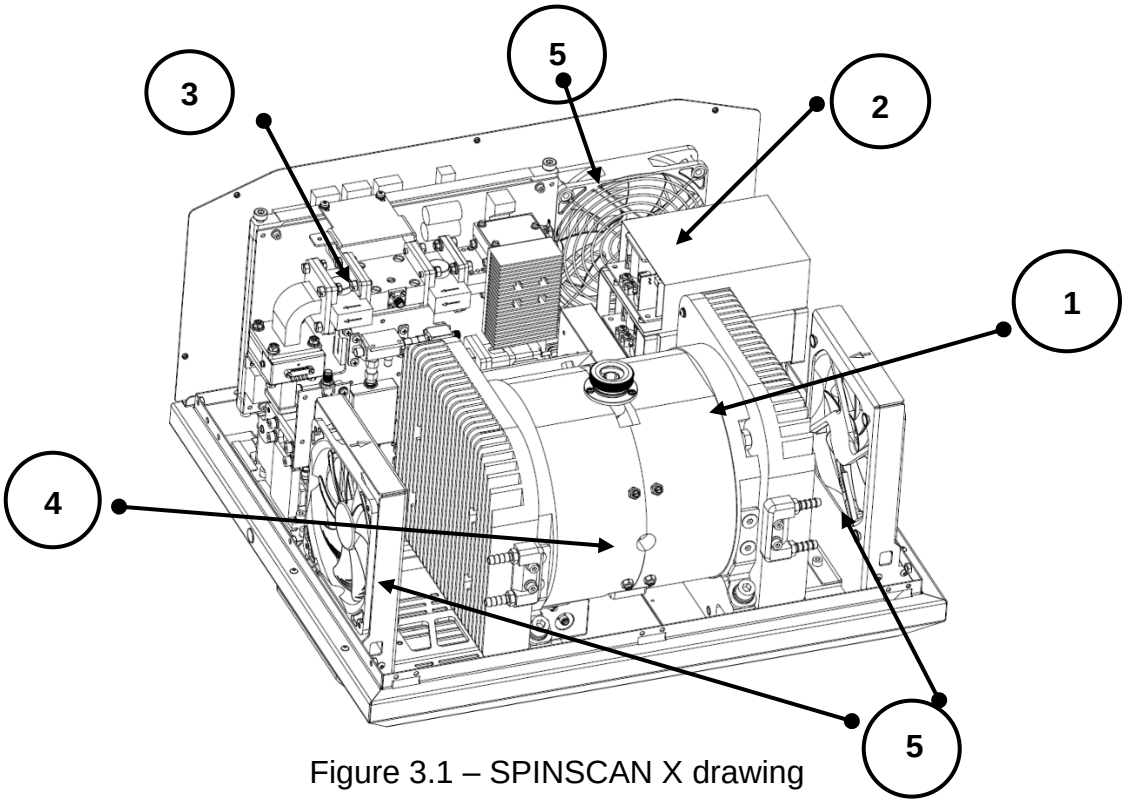



Figure 3.1 – SPINSCAN X drawing

1 - Electromagnet

2- Power Supply

3 - Microwave Bridge

4 - Window for irradiation

5 - Fans

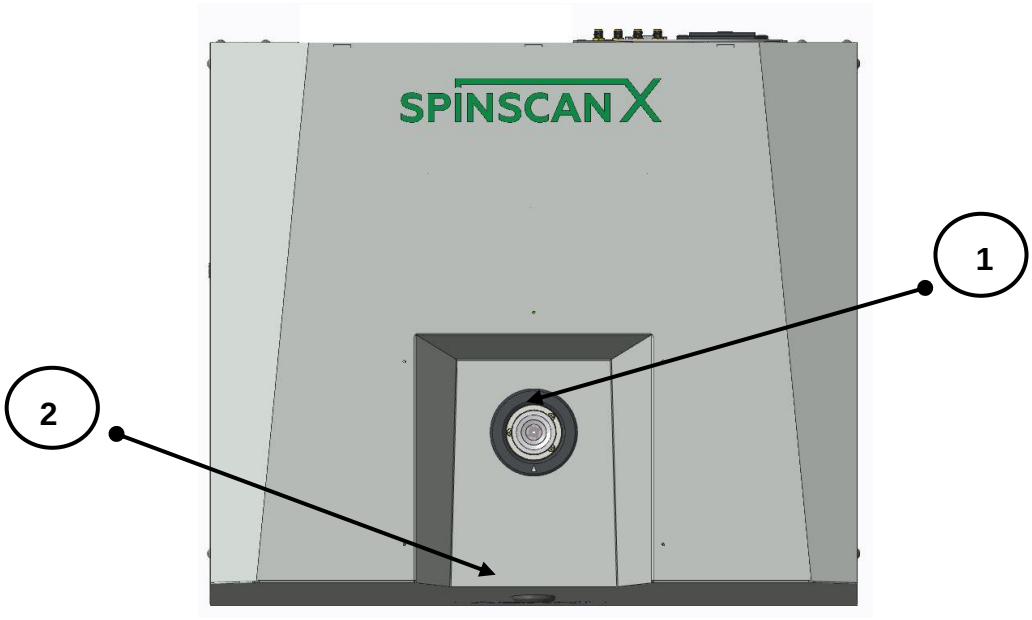



Figure 3.2 – EPR Spectrometer top view

1 – Section for sample holder

2 – Protective screw for window for light access

The Figure 3.3 shows the rare panel of the spectrometer, where the fan and the connectors panel are located (Fig. 3. 4).

To access the internal components of the spectrometer, unscrew the screws on the rear panel of the spectrometer, insert the screwdriver slightly into one of the holes, and gently pull the panel towards you.

Figure 3.3 – EPR Spectrometer, Rear View

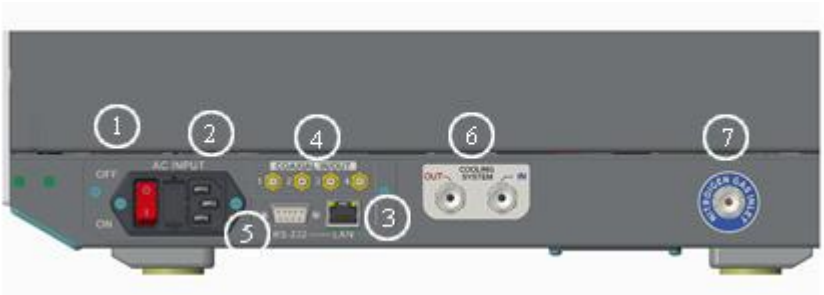



Figure 3.4 – SPINSCAN Rear Connection Panel

1 – I/O power switch; 2 – power cable connector; 3 – Ethernet port; 4 –SMA1 and SMA2 ports for external peripheral devices (external synchronization input and external analog signal input); 5 – RS-232 serial port for external devices connection/

To remove the cover of spectrometer:

- remove the back panel of the spectrometer (see the description above);

- remove the screws on the side panels and then carefully lift the cover up (Fig. 3.5).

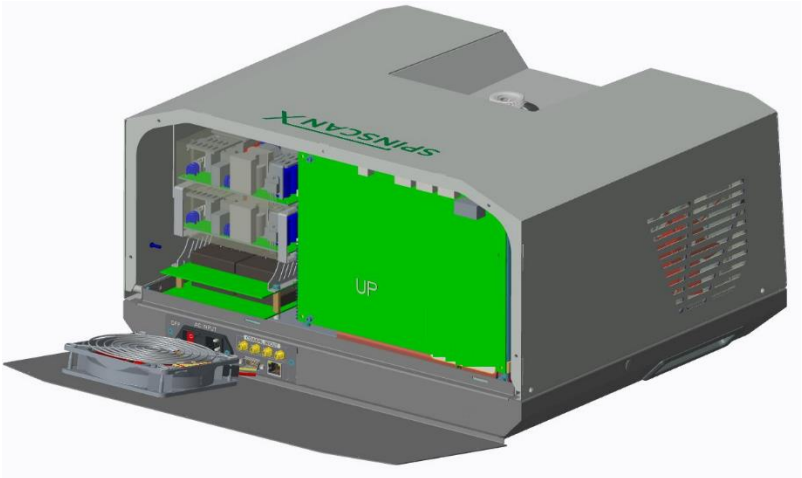



Figure 3.5

## 3.2 BASIC COMPONENTS OF EPR SPECTROMETER SPINSCAN

The spectrometer consists of four units:

- EPR signal sensor unit;
- Magnetic field control unit;
- Processing and tuning unit;
- Power supply unit.

**CAUTION** **In case of the component failure, the user should contact the manufacturer for replacement of non-working components and assemblies.**

EPR signal sensor unit is designed to generate polarizing magnetic field and microwave power, to create conditions for interaction between microwave electromagnetic field and sample and to detect EPR signal at the frequency of magnetic field modulation.

- *EPR signal sensor unit:*
  - Electromagnet;
  - Measuring resonator;
  - Microwave bridge;
  - Microwave preamplifier;
  - Microwave power meter;

– Microwave frequency meter;
– Hall sensor;

*- magnetic field control unit:*

– Current source of a Hall sensor;
– Magnetic field establishing device;
– Commutator and integrator;

*- processing and setting unit:*

– Signal processing controller at magnetic field modulation frequency;
– Microwave bridge control device;
– Microwave bridge tuning device;
– Magnetic field frequency and modulation amplitude setting device;
– Interface.

## 3.2.1 EPR SIGNAL SENSOR UNIT

The EPR signal sensor unit consists of a small electromagnet, a microwave bridge, a resonator with a magnetic field modulation device and a Hall sensor.

The functional scheme of the EPR signal sensor unit is shown in Figure 3.6.

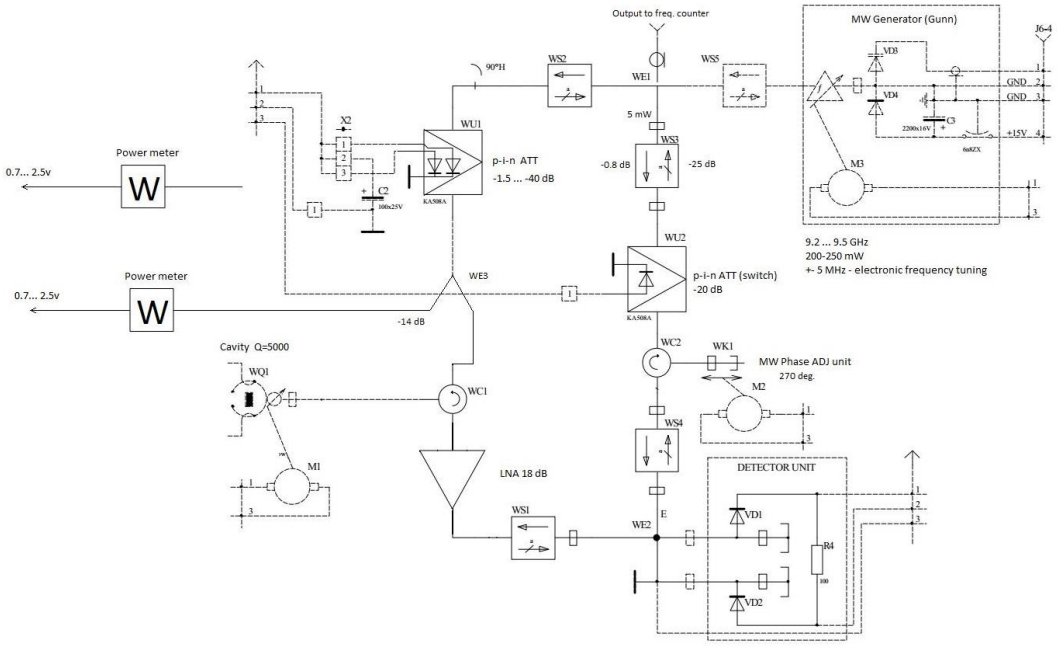



Figure 3.6 – EPR Signal Sensor Unit Functional Scheme

### 3.2.1.1 Electromagnet

The electromagnet is used to create a polarizing magnetic field and its time-based sweep.

The core of the electromagnet is designed as a body of rotation formed by two cylindrical cups connected to each other.

The core cups have several poles, used to feed the microwave bridge to the resonator and to position the electric drive elements for proper connection with resonator, and there is also a window for providing exposure to samples of optical or other ionizing radiation.

Core cups contain inner poles with conical pole tips.

The core of the electromagnet and the pole tips are made of Armco type steel. The electromagnet has a pair of coils (Figure 3.6). The main coils serve for creation of a polarizing magnetic field.

Temperature of electromagnet during operation does not exceed 36 ... 40ºC at ambient temperature 20ºC.

The electromagnet has the following main parameters:

| | |
|---|---|
| Maximum magnetic field induction, T | 0,7 |
| Magnetic field nonuniformity in the volume of Ø 1 x 10 mm, μT, maximum | 6 |
| Maximum power consumption, VA | 150 |
| Diameter of the pole tips, mm | 60 |
| Pole gap, mm | 14 |
| Weight, kg | 25 |

4

A modulation transformer with a ratio of 30 and a commutate resonance frequency in the primary side is fixed to the electromagnet. It serves to match the output resistance of the magnetic field modulation amplifier with the low active resistance of the magnetic field modulation circuit in the resonator WQ1.

### 3.2.1.2 Microwave bridge

The microwave bridge is designed according to the homodyne scheme (with the microwave detector shift channel), which ensures high sensitivity of a spectrometer in all range of power of microwave radiation, led to a resonator. Microwave bridge contains two channels - measuring channel and the microwave detector shift channel. The measuring channel is used to set the microwave power required for measurements in the resonator. The Microwave detector shift channel is used to set the optimal working conditions for microwave detectors.

Both channels consist of identical elements: electronically controlled attenuators WU1 and WU2, isolators WS2, WS1 and WS3, WS4, three-arm circulators WC1 and WC2. The second arm of the circulator WC1 of the measuring channel is connected to the resonator WQ1. The second arm of the circulator WC2 of the shift channel is connected to the controlled short-circuit reflector WK1 (Figure 3.7).

The microwave power from the generator G1 is supplied to the measuring channel and microwave detector shift channel through the isolator WS5 and waveguide directional coupler WE1. The signal reflected from the resonator, due to the absorption the microwave power sample, is fed to the T-shaped waveguide bridge WE2 through the third arm of coaxial circulator WC1, microwave preamplifier LNA and coaxial isolator WS1. The microwave signal of the microwave detector shift channel, the phase of which can change with the movement of the reflector short-circuiter WK1, is also fed to the bridge WE2 (Figure 3.7).

The microwave detector section is balanced and is based on two microwave detector diodes VD1 and VD2 with identical parameters.

To provide control and tuning, resonator frequency and coupling control units, microwave detectors shift channel phases tuning unit are equipped with the M1, M2 and M3 electric drivers.

The microwave generator G1 is based on the Gunn diode and has an output power of at least 180 mW. The range of mechanical frequency tuning is 9.3-9.5 GHz. The band of electronic frequency tuning is 8 MHz. To measure the generator frequency, some power is shunted off with WE1 directional coupler via coax connection on the CU3 frequency divider.

For valves, compact standard flange insulators are used. T-shaped flange insulators are used as circulators. Isolators and circulators provide the discrimination min 25 dB.

Microwave power waveguide attenuators WU1 and WE2 are based on a resonant iris controlled by a semiconductor p-i-n diode. Maximum attenuation of microwave power in the measuring channel is 40 dB.

Isolators and circulators provide isolation of minimum 25 dB.

### 3.2.1.3 Measuring resonator

The measuring resonator has a rectangular shape and belongs to $H_{102}$ type.

The resonator casing has a through hole (11,0 mm), in which a quartz tube is inserted to protect the resonator from contamination; a hole for inserting the coupling adjustment rod; a hole for irradiation of the sample recesses for installation of the Hall sensor and temperature sensor.

| | |
|---|---|
| Frequency range with quartz tube, GHz | 9,3-9,5 |
| Frequency range of an empty resonator, GHz | 9,5-9,7 |
| Quality of the unloaded resonator | 5000 |
| Vibration type | $H_{102}$ |
| Maximum test tube diameter with sample, mm | 6 |
| Resonance cavity dimensions, $mm^3$ | 23x10x42 |
| Housing dimensions (without frequency and coupling connection elements):<br>L x H x W, mm | 79 x 68x 12 |
| Permissible temperature of resonator housing, ℃ max | 40 |

The connection of the microwave input waveguide with the resonant cavity is carried out by changing the size of the coupling hole by moving along the hole of the cylindrical metal element attached to the end face of the Teflon rod. The coupling connection unit is equipped with electric drive elements. To modulate the magnetic field, a modulation circuit is placed inside the resonance cavity, which is formed by two rods, one of which has a circular cross-section (diameter 1.5 mm), and the other is flat (0.5 x 3.5 mm) and is located on the side of the hole for irradiating the sample. One end of each of the rods is in electrical contact with a narrow wall of the resonant cavity, and the other is insulated, removed through the resonator body and connected to a modulation current transformer.

### 3.2.1.4 Microwave preamplifier

Coax microwave preamplifier LNA has a gain of 16 dB and a noise factor of 0.9 dB in the operating frequency range of 9.3-9.7 GHz. The microwave control unit is equipped with a protection circuit for the maximum permissible input power at the level of 0.15 mW using a power meter CU1 and microwave detectors VD1, VD2 (Fig. 3.7).

### 3.2.1.5 Microwave power meter

Measurement of the microwave power supplied to the measuring resonator is performed using a directional coupler CE1 of 15 dB and a coaxial power meter CU1 (Fig. 3.7). The power meter allows you to control and stabilize the microwave power supplied to the measuring resonator in the power range 100 μW - 10 μW with an accuracy of 0.5 dB in the attenuation range 0 - 40 dB.

### 3.2.1.6 Microwave frequency meter

The microwave frequency meter is constructed using a microwave frequency divider CU3 with a division coefficient of 256, the output signal of which is fed to a low frequency meter with a bandwidth of up to 50 MHz (Fig.3.7).

### 3.2.1.7 Hall sensor

The Hall sensor is used to set and stabilize the polarizing magnetic field.

The Hall sensor board is in a plastic housing. The Hall sensor BB1 is inserted into the housing hole of the resonator located in the pole gap of the electromagnet.

## 3.2.2 MAGNETIC FIELD CONTROL UNIT

The unit controls the source of electromagnet current and sets the induction of polarizing magnetic field. Stabilization of the magnetic field value is performed using the Hall sensor as a magnetic field meter.

The unit includes the following devices: magnetic field tuning device, Hall sensor current source, commutator and integrator.

### 3.2.2.1 Hall sensor current source

The Hall sensor current source generates the current and the reference signal for DACs of the magnetic field tuning device. Current and reference signals have a sinusoidal shape with a frequency of 1 kHz. The current is set at 50 mA.

### 3.2.2.2 Magnetic field setting device

The magnetic field tuning device is used to generate a control signal at 1 kHz, the amplitude of which is proportional to the set value of the magnetic field induction.

The main elements of the device are three 16-bit DACs, which provides the magnetic field setting in the range of 0-650 mT with a minimum pitch of a field setting of 1 μT.

### 3.2.2.3 Commutator and integrator

The commutator provides a 4 kHz AC switch for the Hall sensor current inputs and outputs. The action of the commutator allows to compensate effect of non-equivalence of the Hall sensor (its temperature coefficient), and magnetoresistive effect.

The integrator provides a time constant of electromagnet current control circuit to achieve the instability of the magnetic field at 5 μT/20 minutes.

## 3.2.3 PROCESSING AND TUNING UNIT

This unit includes: the signal processing controller at the frequency of the magnetic field modulation, microwave bridge control device, microwave bridge tuning device, frequency and magnetic field and amplitude modulation tuning device, the spectrometer parameters controller and the interface.

### 3.2.3.1 Signal processing controller at the frequency of magnetic field modulation

The processing controller allows to measure first and second derivatives of EPR signals in the magnetic field function in the range of 0°-360° detection phases at magnetic field modulation frequency. The controller is based on an 18-bit ADC, working with averaging over $F_m$ × 128 μs points, which achieves a 24-bit conversion.

$F_m$ – magnetic field modulation frequency.

### 3.2.3.2 Microwave bridge control device

The device includes AFC generator circuit according to the resonator frequency with an accuracy of min 3x10-6 and a holding range of 8 MHz; a microwave power attenuation setting device based on a 12-bit DAC with a range of 0-40 dB and an accuracy of 0.4 dB.

### 3.2.3.3 Microwave bridge assembly tuning device

The automatic tuning device includes three control drivers for the stepper motors mechanically controlling the microwave generator frequency, resonator connection and microwave detectors shift channel signal phase.

# TUNING PROCEDURES

## SECTION 4

### 3.2.3.4 Frequency and magnetic field modulation amplitude tuning device

The modulation frequency tuning is performed using DDS digital frequency synthesizer in the range of 10-500 kHz, and the modulation amplitude tuning is based on 12-bit DAC in the range of 5 µT - 1 mT.

### 3.2.3.5 Interface

The interface includes a 32-bit processor with deployed peripherals for controlling the spectrometer and high-speed Ethernet-COM converter for connection via Ethernet network with externally controlled computer.

# 4 TUNING PROCEDURES

The user (buyer) receives the spectrometer packed in shipping containers.

**CAUTION**

**Shipping containers must be in the position corresponding to the signs (markings).**

After receiving the equipment from the transport company, the buyer (user) must check the appearance and integrity of the packaging. If damage is detected, do not unpack the spectrometer until a damage report has been drawn up or a corresponding note has been made on the delivery note.

After the equipment was unpacked, the user should visually inspect the contents of the packaging and check the completeness for compliance with the packing list, as well as for mechanical damage. Claims for lack of completeness or mechanical damage to the spectrometer will be accepted only if the package is not damaged.

## 4.1 UNPACKING

Unpacking and installation are carried out as follows:

1. Open the shipping containers, carefully remove the accessories and place them according to the pre-installation requirements;

2. Remove the upper and middle part of the packing.

3. Carefully remove the instrument from the box, holding it on both sides and place it on a table. There are hand grooves on both sides of the device for easy transportation and holding.

**WARNING**

**The weight of the spectrometer is more than 40 kg, so the SPINSCAN X should be taken out of the box and moved by 2 people.**

## 4.2 SWITCHING ON

**CAUTION**

**Before operation, check the air temperature in the room where the spectrometer is installed. If temperature is below 10 °C, warm up the room to the air temperature plus (25 ±10) °C, holding the equipment at this temperature for at least two hours. Then switch on the spectrometer and warm it up for at least 90 minutes.**

To connect, follow the instructions:

1. Insert the Ethernet cables into the connectors of the spectrometer and the computer and press until it clicks;

2. Connect the Ethernet cables from the spectrometer and computer to the router as shown in the Figure 4.1, depending on whether you want to access the local network or not;

3. Carry out the Software Installation (paragraph 4.3);

4. Check Ethernet connection on the computer;

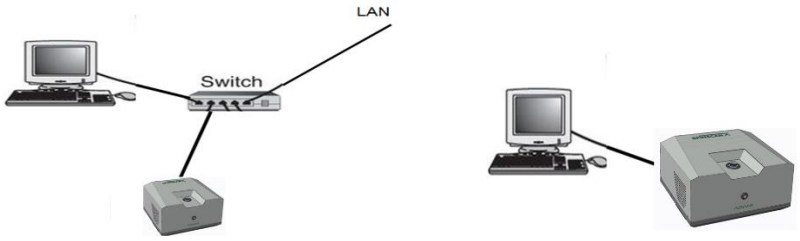
5. If the operator workstation (computer) is off, switch on the computer power;

6. Switch on the spectrometer by pressing I the power supply button on the rear panel. After that, the IP address of the equipment in the network will be recognized;

7. If necessary, use an uninterruptible power supply (UPS);

**NOTICE** *Your PC should have an 8P8C connector (for Ethernet cable)*

8. Before shipping the equipment, the manufacturer configures the router and spectrometer parameters with dynamic IP addresses. If you connect the router to the local network - contact your system administrator to inform about the new device in the local network and to install it correctly;




a) b)

Figure 4.1 (a, b)

9. Set the Ethernet connection parameters on your computer.

**Ethernet connection on the computer:**

- Press **Start –> Control panel** in the list of parameters, select– > **My Network Places**, located on the desktop – > Connection – > press OK;
- Press **Properties;**
- Right-click on a position **Local Area Connection Properties** (Status Local Area Connection);
- Press **Properties**;
- **Local Area Connection Properties** window will appear;

- Select **Internet Protocol Version 4** (TCP / IP) (Internet Protocol), and press **Properties**;
- Select **Use the following IP Address** and press **Obtain an IP address automatically**;
- Press **OK**;
- If any problem occurs (e.g. IP address conflict in the local network), contact your system administrator or ADANI Service Center to resolve the problem.

The spectrometer may be connected directly to a computer without using a router. This connection type requires the correct tuning of static IP addresses for computer and spectrometer. In this case, contact the manufacturer with this request.

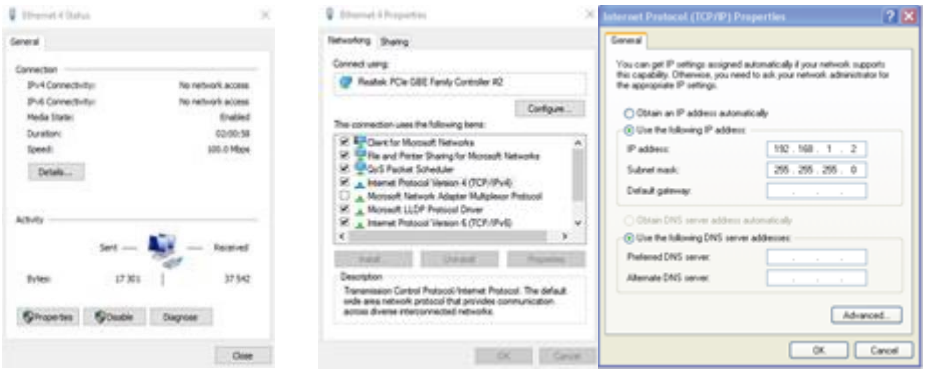


Figure 4.2

## 4.3 SOFTWARE INSTALLATION

The e-Spinoza installation package is supplied with the device on a USB flash drive.

1) In the case when the spectrometer is supplied with a computer, the installation of the software is carried out at the manufacturer of the equipment before delivery. To start the software, it is necessary to find the e-Spinoza icon on your computer's desktop and double-click on it.

2) If you use your own computer to control the spectrometer, install the software as follows:

- Open **e-Spinoza_1.0.0.X.exe** file from the Installation USB flash driver (where X is an integer representing the version number of the program);
- Click **OK** in the dialog box for subsequent installation;
- Follow the interactive Software installation instructions;
- Wait until the installation is completed.

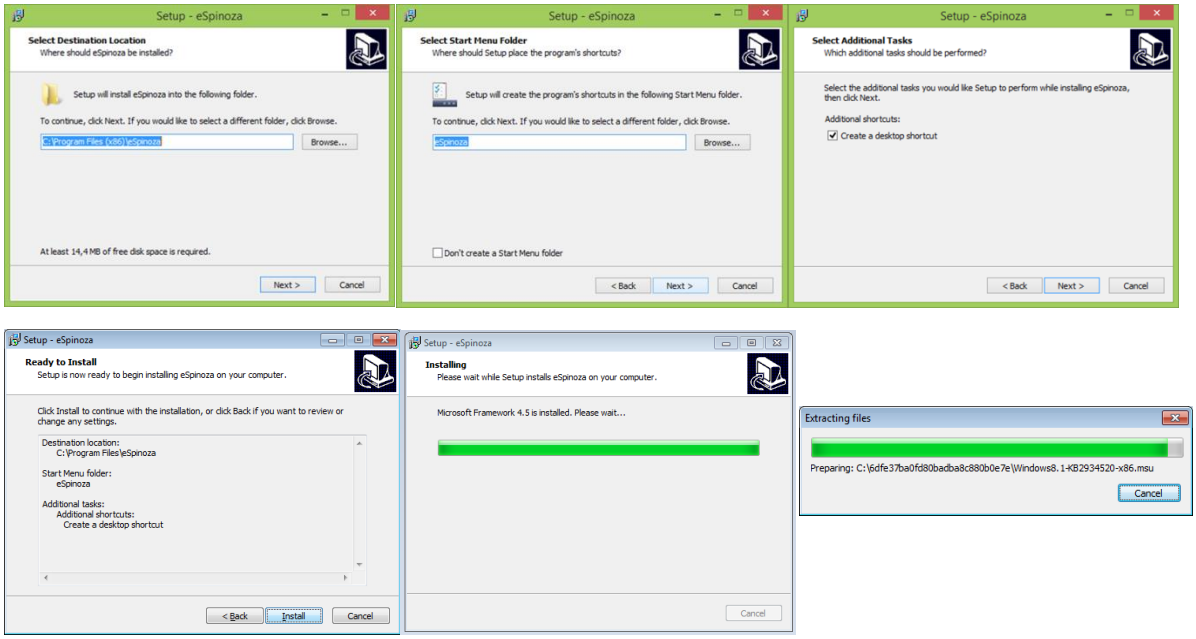
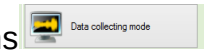


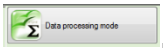

Figure 4.3

When extraction of files is completed, the Microsoft.NET Framework 4.5 window will appear. It is necessary to agree to the license agreement to continue the installation. After that, the e-Spinoza installation will be finished.

# e-SPINOZA
# SOFTWARE MANUAL
## SECTION 5

# 5 SOFTWARE INTERFACE DESCRIPTION

The program interface provides operation in two modes: data collecting mode and data processing mode. Switching between modes is performed using the buttons 

and 

, located in the upper right corner of the window or using the icon  on the toolbar.

*Data collection mode* is intended to control the spectrometer and contains the menu bar, tool bar, stack bar, external devices bar, tuning menu, selection of experiment type, indicator menu, status bar (Figure 5.1).


Figure 5.1 – Main window - *Data collection mode*

Data processing mode is intended to process measurement results and contains the menu bar, tool bar, stack bar (windows). On the right side there is a drop-down menu with mathematical functions for data processing (Fig 5.2).

The graphic window in the central part of both modes displays the current/modified spectrum.

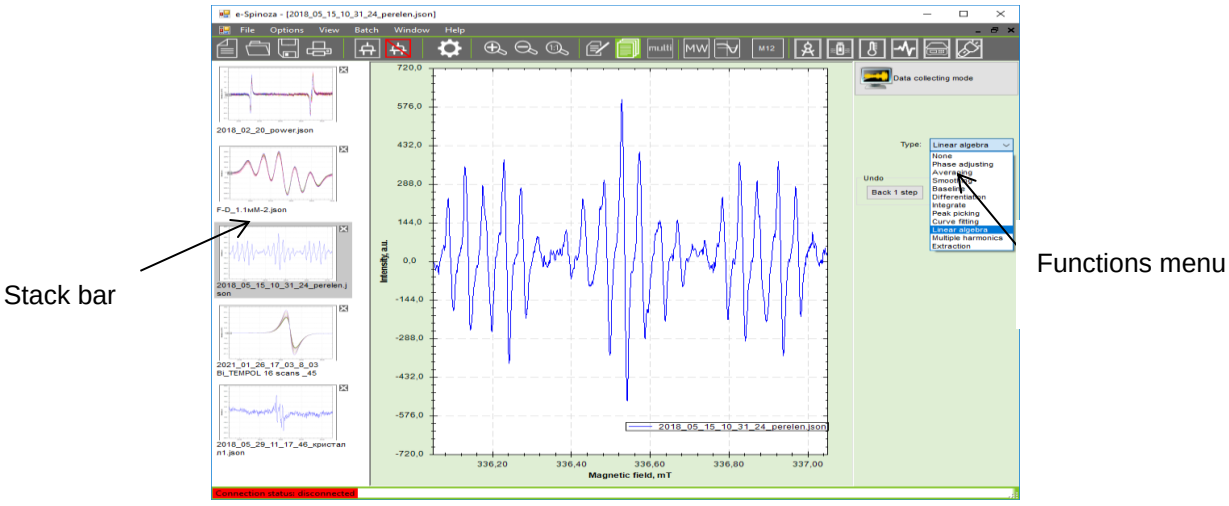



Figure 5.2 – Main window - *Data processing mode*

## 5.1 MENU BAR

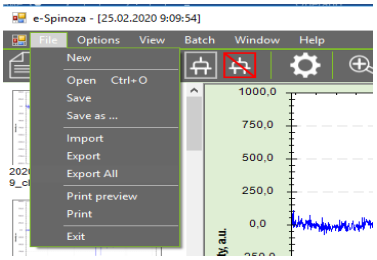



Figure 5.3 – File Menu Options

Tabs:

**New** – creates a new file with preset default parameters for scan and empty graph window;

**Open** – loads from the chosen folder an existing *.json file;

**Save** – saves the current file by date and time (Year_Month_Day_Hour_Minutes). This path may be changed: **View– > Tunings– > Interface – > Line Default location for auto-saved files**;

**Save As** – saves the current file under a new name;

**Import**

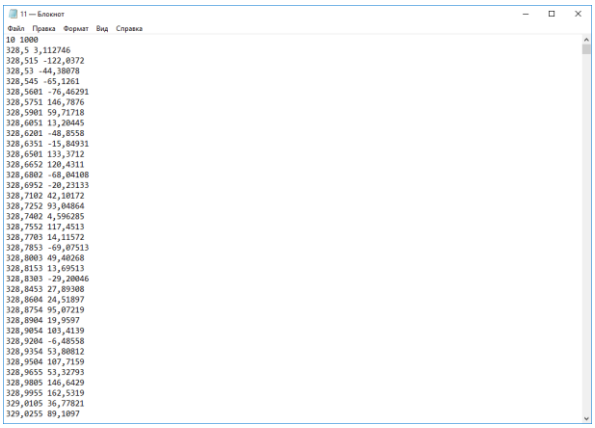
– opens the data file in the ***.spec** format (EPR Commander format of software data for the CMS 8400 spectrometer model);

**Export**

**Export All**

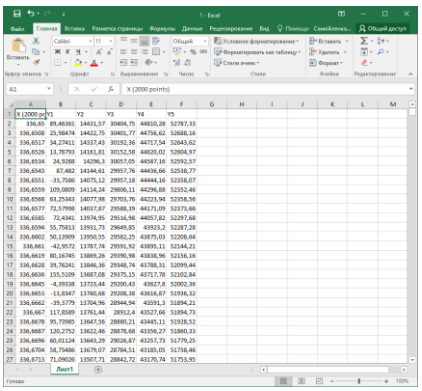
– converts the data file to:

**ASCII codes (*.txt)** – text format with measuring unit of the magnetic field in mT and Gauss (represented as 2 columns: magnetic field strength and average intensity)




**Excel file (*.xlsx)**

The exported to Excel file contains minimum 3 data columns. The first column is the magnetic field strength (expressed in mT units), the second column is the average signal intensity (a.u), the subsequent columns are the EPR signal intensity after each scan;



**PNG picture (*.png)** – generates a graphic window output as an image;

**PDF files (*.pdf)** – generates a pdf file with an image of the measured spectrum and a short report

– Converts all open data files to

**ASCII codes (*.txt)**

**Excel file (*.xlsx)**

**PNG picture (*.png)**

**Print preview** – opens preview window of the current file with spectrum(a) and the scanning parameters report before printing;

**Print** – prints the page with current spectrum(a) with the scanning parameter report;

**Exit** – closes the program.

### 5.1.1 OPTIONS MENU

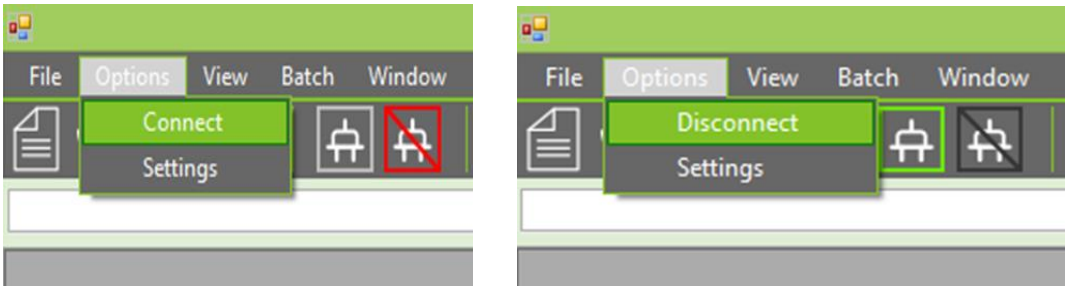



Figure 5.4 – Options menu

**Connect/Disconnect** – Connects/disconnects the Ethernet connection between the spectrometer and the computer. By pressing the '**Connect**' Button, the motor initialization is started, and the default measurement parameters are installed in the spectrometer;

**Settings** – opens the Settings window, where unique parameters specific to this spectrometer are stored. Also, there is an icon on the toolbar for quick access to the Settings window.

**Connection Tab**

In **Connection** Tab (Fig. 5.5) displays device address, IP address and port number of the spectrometer that is set by the manufacturer.

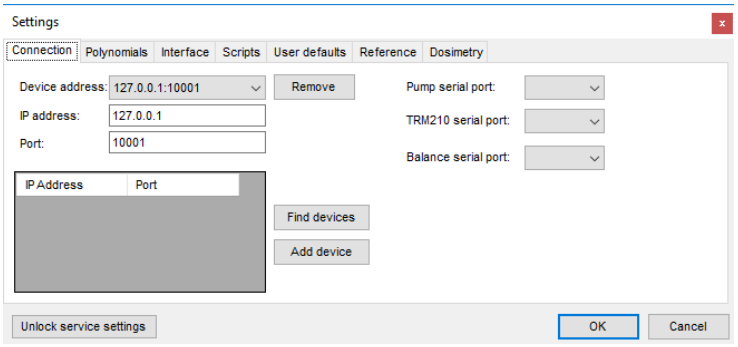



Figure 5.5 –Connection Tab Settings window

To setup the connection between the spectrometer and the computer, switch on the spectrometer, check the IP address on the **Connection** tab. If IP address is absent, press the **Find devices** button and select IP address from the found options. Then, press **Add device** and **OK.** At the end of this procedure, the connection status in the lower left corner will be green, the icon  on the **Connect** toolbar will be the same color.

If any problems with the connection are detected, contact your system administrator or the ADANI Service Center.

### Polynomials Tab

The calibration parameters of the spectrometer are stored in the **Polynomials** tab (Fig.5.6). These parameters are unique and are installed by the manufacturer during manufacturing. The ID unique number of the spectrometer is displayed in the same tab.

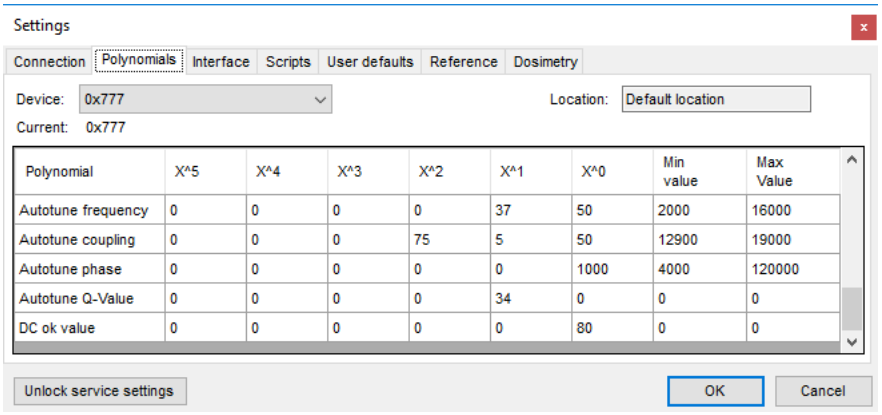
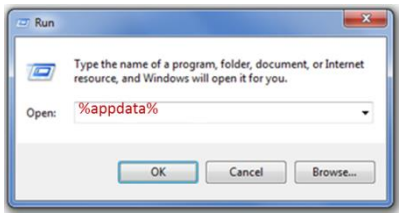



Figure 5.6 – Settings window Polynomials Tab (example)

The calibration parameters are also stored in the file **espol.stgj** file, which may be found on the USB flash drive supplied with the spectrometer. To avoid losing unique data, make a backup copy of **espol.stgj** on computer. If it is necessary to restore the polynomial parameters (for example, if all values in the polynomial tab are zero), perform the following:

1. Open the **“Run”** by pressing **Win+R** buttons


2. Paste or type ***%appdata%*** in the dialog box




3. Press **Ok.** The folder **Application Data** will open;
4. Find the folder **e-Spinoza** in the folder ADANI**;**
5. Open **e-Spinoza** folder**;**
6. Copy file **espol_MAC address.stgj (**for example espol_61_D7_E8_98_01_EE.stgj) from the installation USB-flash driver (or backup copy) and replace the selected file with the one copied in the **e-Spinoza** folder.

**CAUTION** **Do not change the calibration parameters in the Polynomials tab. If necessary, contact the manufacturer's service center.**

### Interface Tab

The autosave path for autosaving experiment files is set by default in the system folder **Documents \e-Spinoza.** It is possible to set the path to the autosave folder by selecting the **Interface** tab and defining the convenient storage location.

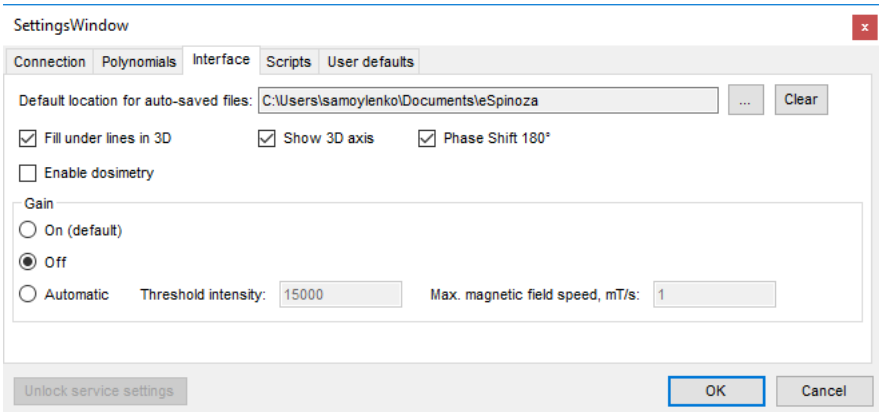



Figure 5.7 – Settings Window of the Interface Tab

For better visualization of the results of 3D experiments for the spectra imaging it is possible to apply coloring of the areas under/over the spectrum line. For this purpose, it is necessary to check the **Fill under lines in 3D** window.

If the Show **3D axis box** is checked, the values of the measured quantity along the 3rd axis will appear near each line.

The manufacturer keeps the right to modify and add new functions for spectrometer. Additional software for the analyzer is supplied separately on request.

If the operator needs to display the spectrum in the opposite phase during signal registration, the **Phase shift 180** function should be activated at the end of the measurement, press **Ok** and repeat the experiment. Then the phase of the measured spectra will be displayed automatically shifted by 180°.

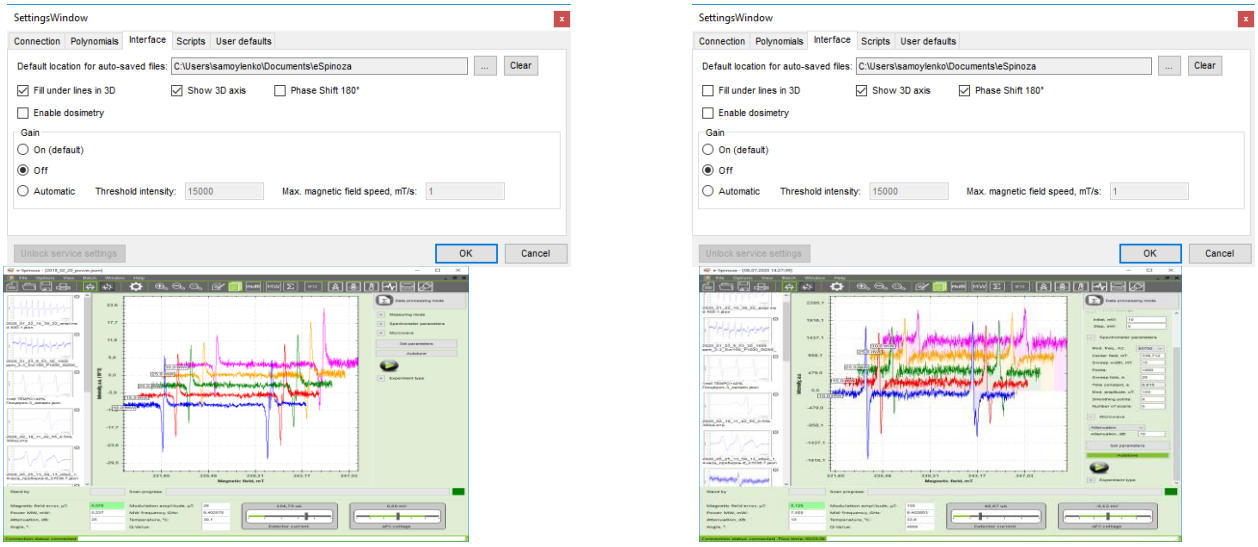
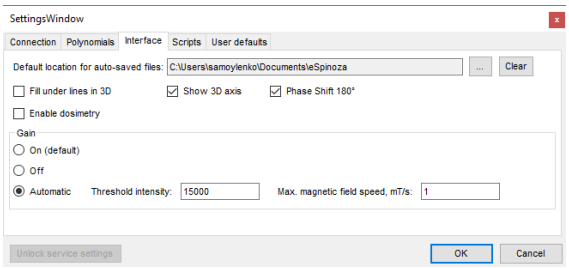



Figure 5.8

This operation can be performed manually after completing spectrum registration by going to the mathematical processing interface (Section 7.1).

Thanks to a wide range of measurements, the SPINSCAN X spectrometer implements auto-scaling of the signal. It allowes to measure samples with different line intensities in spectrum. However, in some individual cases, there is a function to switch the amplifier to reduce attenuation 10 times gain mode, when there is a distorted line (cutting along the vertical axis), and a decrease in power and modulation parameters does not lead to intensity compensation. That is why do you need to check **Automatic** in the box and continue the measurement.



Value of **Threshold intensity**, at which the switching of the gain, and **Max. Magnetic field speed** are set by the manufacturer. In case of problems, contact the manufacturer.

### Scripts Tab

If it is necessary to operate with different types of experiments it is possible to add the corresponding buttons which run the script files thus automating the desired processes.

In a case you want to use the spectrometer as Alanine analyzer you have to activate the box **Enable dosimetry** ( special software package for alanine dosimetry on request). After that in the window **Settings** new tabs with set default parameters for alanine experiments appear (Fig.5.9). The description of protocol for alanine dosimetry see in Annex A.

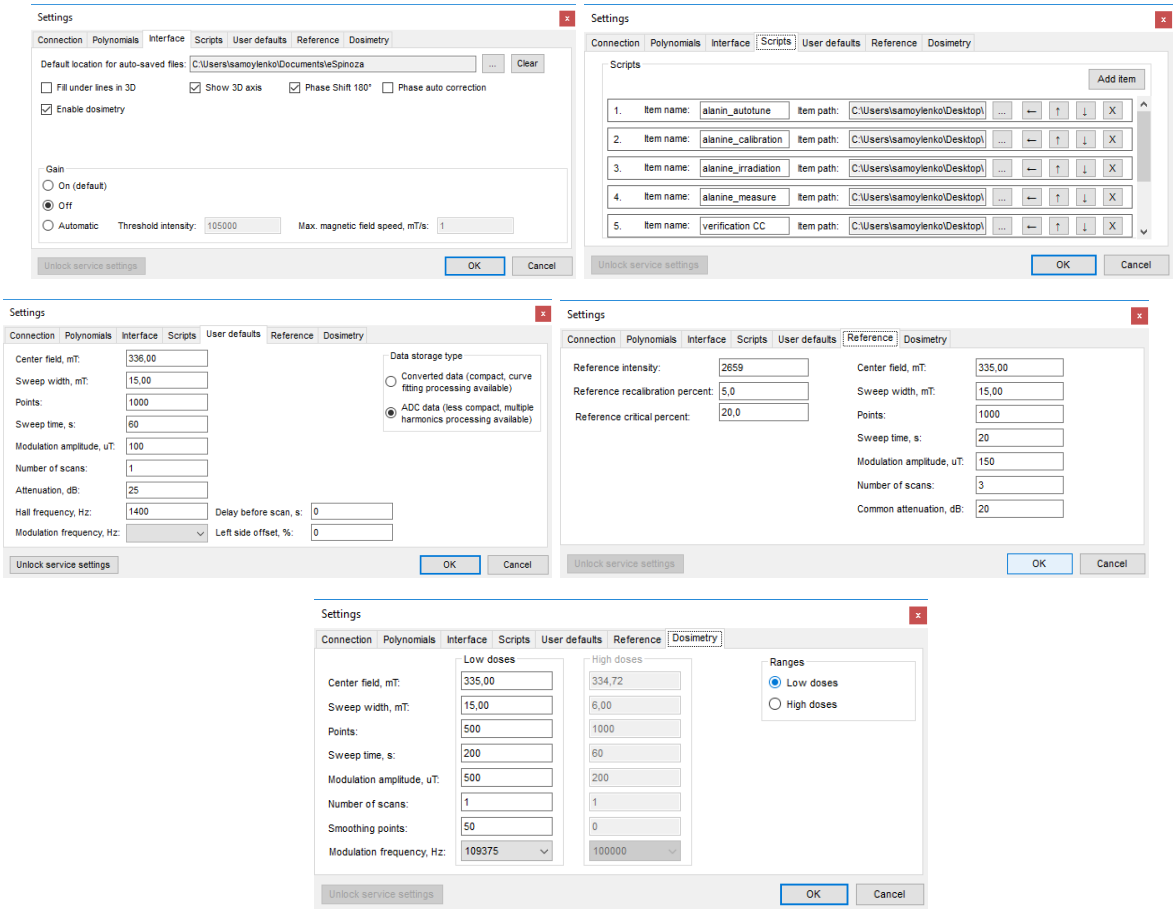



Figure 5.9 – Interface Tab (example) for Alanine Dosimetry experiment

### Autotune

Spectrometer control parameters for automatic tuning the microwave bridge and the resonator are installed by the manufacturer and stored in the polynomial for this device.

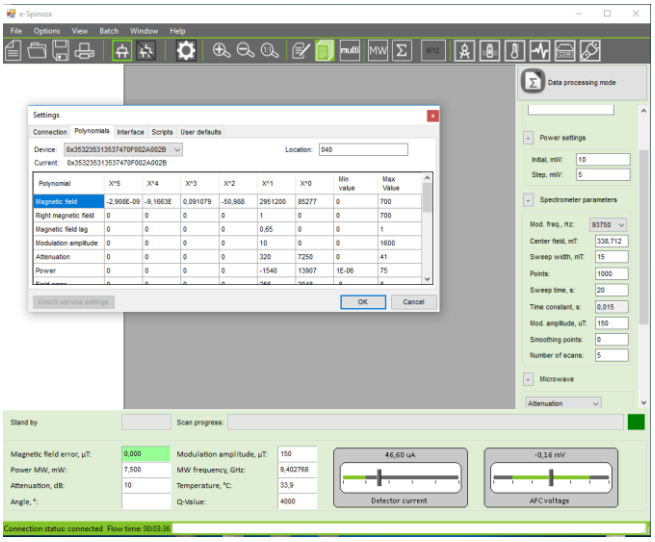


Figure 5.10

## 5.1.2 VIEW MENU

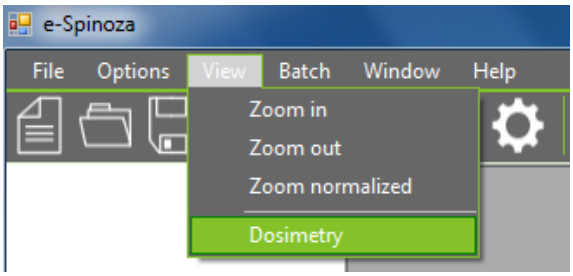



Figure 5.11 – View menu options

**Tabs:**

**Zoom in** – horizontally and vertically stretching of the spectrum in the window;

**Zoom out** – returns the zoomed spectrum to its actual size;

**Zoom normalized** – returns the zoom to its original size;

**Dosimetry** – opens the dosimetry database (works only in spectrometer mode as an alanine analyzer).

## 5.1.3 BATCH MENU

The Script File Editor (Batch Editor) is in the Batch menu, where it is possible to create script files and modify them.

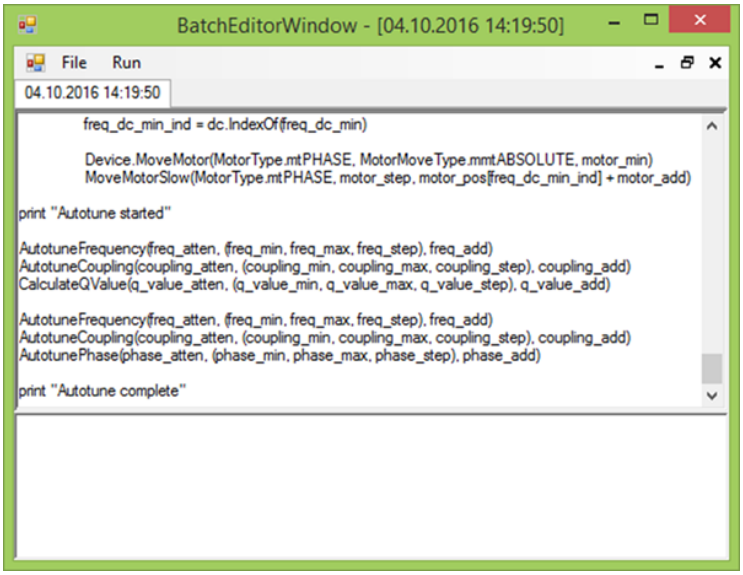



Figure 5.12 – Batch menu

> **CAUTION** **Script files are very useful. If you do not want to repeat the same operations several times manually, you can create a script file, run it and wait for it to be done.**

## 5.1.4 WINDOW MENU

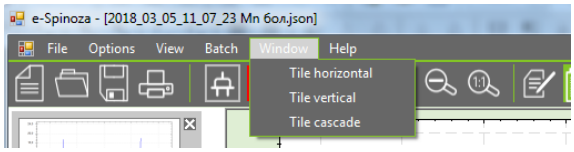



Figure 5.13 – Window menu

This tab allows to view several files with spectra as sub-windows in the area of the main graphic panel. These sub-windows can be placed horizontally, vertically or as a cascade.

Select one of three variants:

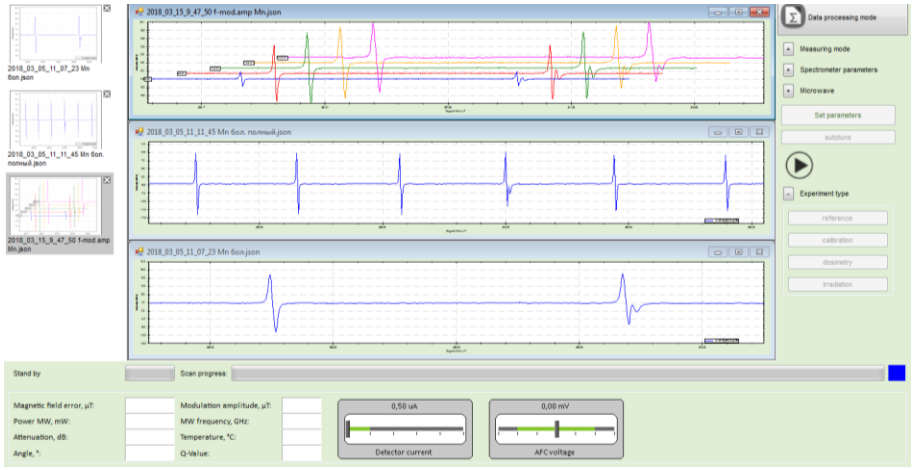


Figure 5.14 – Horizontal Placement of Sub-Windows

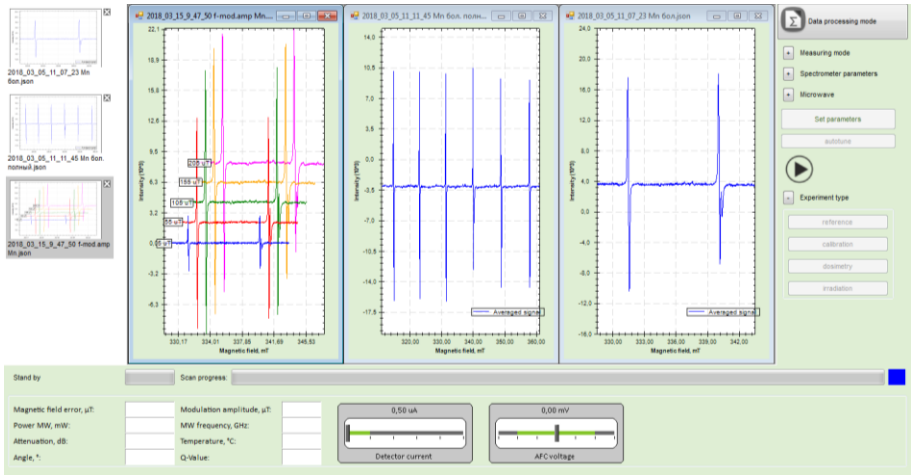


Figure 5.15 – Vertical Placement of Sub-Windows

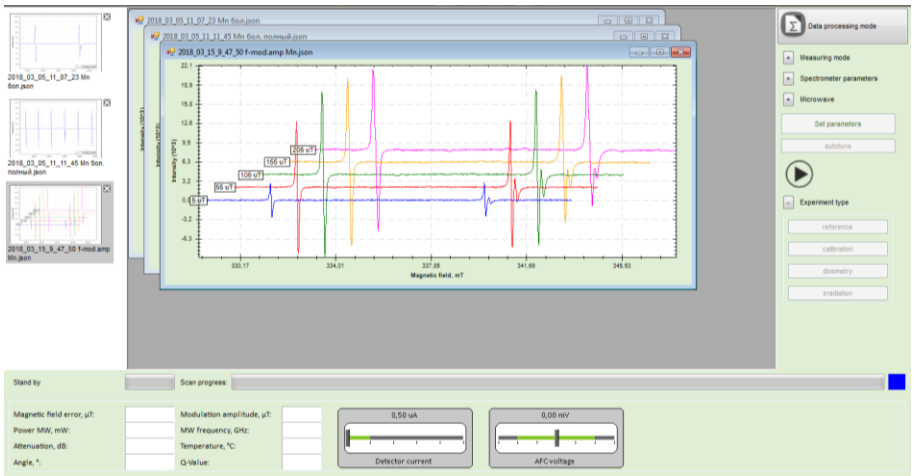


Figure 5.16 – Cascade Placement of Sub-Windows

**Tile horizontal** – all open graphic windows are displayed horizontally one by one;

**Tile vertical** – all open graphic windows are displayed vertically one by one;

**Tile cascade** – all open graphic windows are overlapped so that only file names are visible.

## 5.1.5 TOOLS BAR



- creates a new file with preset default parameters for scan and empty graph window;


- opens an existing *.json file from the chosen folder;

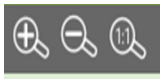

- saves the current file to the folder;


- prints the page with current spectrum and spectrum parameters report;


- establishes an Ethernet connection between the spectrometer and the computer;


- interrupt the Ethernet connection between the spectrometer and the computer;


- opens settings menu;


- zooms in and out or returns to actual size


- opens the window with a spectrum parameters report (it is possible to move the window manually to the main window area);

- activate/deactivate the side panel;


- activates the multi-plot mode;

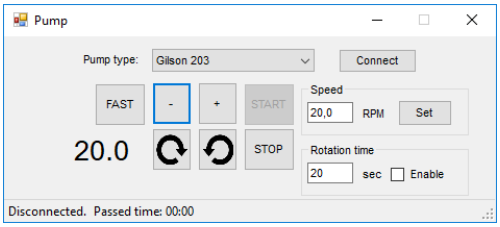

- opens the microwave bridge tuning window;


- switches the window to the data collecting mode;

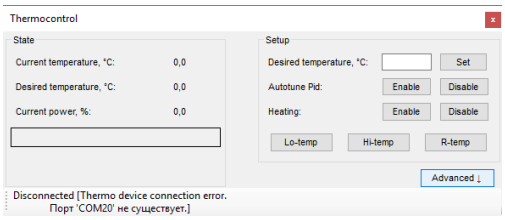

- spectrum work-up using markers;

- activates the goniometer control window;

- activates the peristaltic pump control window;

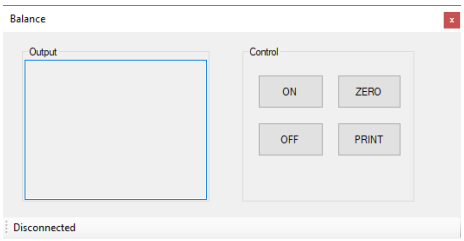



- activates the temperature control window;

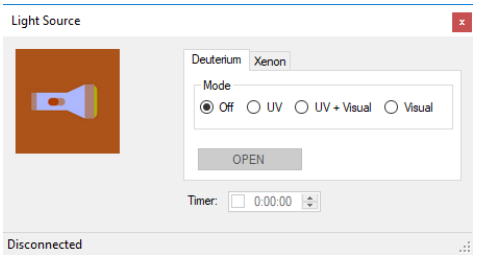



- activates the oscilloscope control window;

- activates the analytical balance control window;



- activates the light source control window.



### 5.1.5.1 MICROWAVE BRIDGE TUNING WINDOW

The microwave path tuning window can be open from the toolbar by pressing the 

button.

As mentioned in paragraph 3.2.1.2, in the microwave bridge there are 3 stepper motors that provide linear movement of the control elements to set the frequency of the microwave generator, connection of the measuring resonator with the fill line, the signal phase of the microwave detectors shift channel. In the **MW tuning monitor** window, you can find the control buttons for each of these motors. The microwave bridge tuning can be performed in automatic, semi-automatic and manual modes.

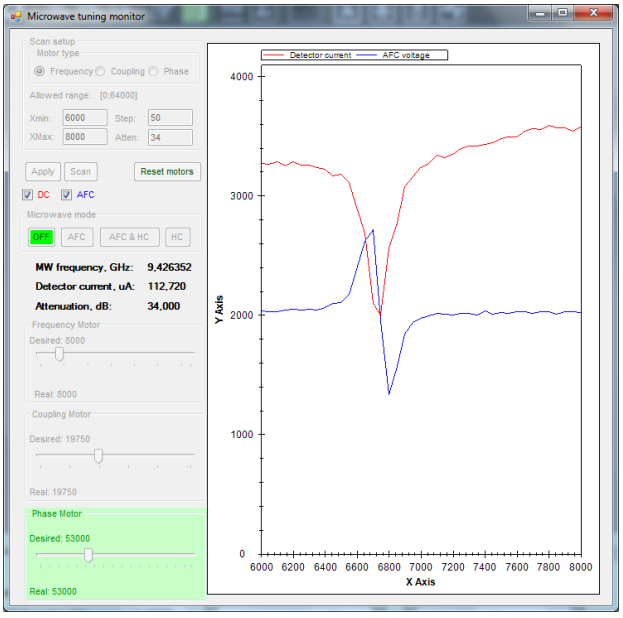



Figure 5.17 – Microwave bridge setup window

**Scan setup:**

**Motor type (frequency, coupling, phase)** – a selected motor type provides of frequency, coupling and signal phase adjustment, which takes place in automatic, semi-automatic and manual modes. The graphical window on the right displays the tuning process for selected motor;

**Xmax** and **Xmin** – minimum and maximum values for the motion range of the selected motor type (in steps) specific to the given spectrometer;

| NOTICE | *The parameters of the spectrometer for automatic connection are set by the manufacturer. You can use them as the reference for manual tuning as well.* |
|---|---|

**Step** – number of motor steps from Xmin to Xmax;

**Apply** – applies the input parameters;

**Scan** – starts scanning in the set range X min – X max for selected motor type.

**Microwave mode:**

**Microwave frequency** – display the actual resonance frequency;

**OFF** – AFC switch off button. The initial step before any tuning;

**AFC** (auto frequency control) – Switch on the automatic frequency control mode ;

**AFC&HC** (auto frequency control&homodyne channel) – Switch on both the homodyne channel and the AFC;**HC** – Switch on the homodyne channel;

**Frequency motor scroll bar** – a cursor displaying the current linear position of the microwave generator frequency control plunger. In manual frequency control mode, the cursor is activated to perform rough and fine tuning of the generator frequency;

**Coupling motor scroll bar** – a cursor displaying the current linear position of the coupling control plunger. In manual coupling control mode, the cursor is activated to perform a rough and fine coupling tuning;

**Phase motor scroll bar** – a cursor displaying the current linear position of the phase control plunger. In manual phase control mode, the cursor is activated to perform rough and fine phase tuning;

| NOTICE | *The movement of the cursor along the scroll bar sets the position of the control plunger. 100 steps can be performed by left clicking on the axis on the right or left of the cursor. Fine tuning may be performed by moving the mouse scroll forward and back.* |
|---|---|

***Semi-automatic and manual modes***

Semi-automatic tuning mode allows to set the linear position of each motor, where resonant conditions are achieved, and then to perform manual fine tuning. For this purpose, the scanning modes must be set sequentially (frequency, coupling, phase shift) and for each motor type, the scanning must be performed to determine the position values required for microwave bridge fine tuning.

The scanning range and step size of the motor are expressed in arbitrary units. The limit values for X min and X max range for every motor are determined by the manufacturer and saved in the **Polynomials** tabs (column corresponds to X min  X max).

When you open the **MW bridge monitor,** the frequency tuning mode appears by default. To access the next mode, select the motor type in the drop-down menu and repeat the scan sequentially.

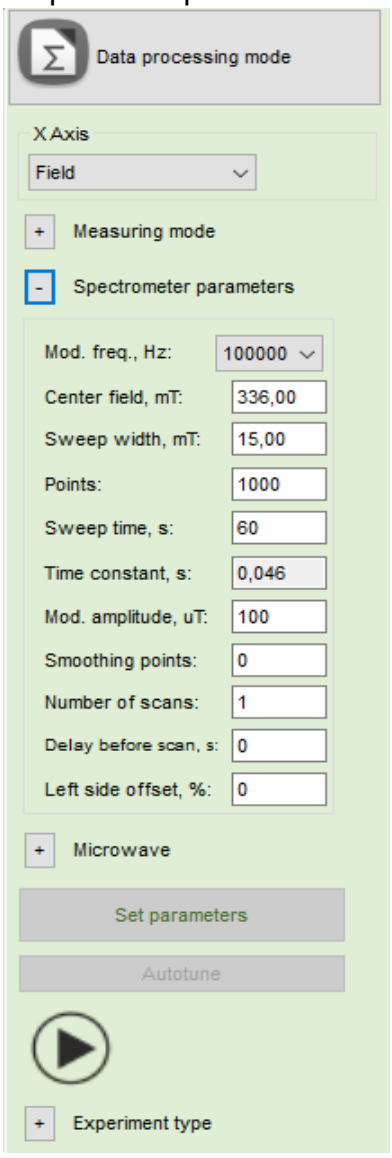
The graphics window will display the dependence of microwave power on the detectors on the linear position in the steps of the corresponding control unit after the **Scan** command is pressed during the tuning process. When the scan is complete, the approximate value for the selected motor position will correspond to the X scale resonance band position.

After that, the motor position must be precisely tuned. Use the values obtained from the auto scan and complete the tuning manually by tuning the motor position and moving the cursor until successful connection.

The sequence of actions for the complete MW bridge tuning cycle will be described in the section below.

## 5.1.6 SPECTROMETER SETTINGS PANEL

In **Data collection mode**, you can select and/or set the experiment parameters in the panel on the right side of the main screen:




- Spectrum display mode depending on the selected value of the X-axis: Field, g-factor, Frequency, time;

- Measuring mode: Field, Field-Delay, Field-Mod.ampl, Field-Temperature, Field-Power;

- Spectrometer parameters for current experiment (spectrum);

- Microwave generator parameters: power or attenuation value;

- The button for automatic tuning of the microwave bridge. Microwave bridge tuning can be optimized by the User;

- The control of different types of experiments by means of scripts. Connection and change of different types of experiments is performed via the Interface panel (see **Settings menu**).

## 5.1.7 INDICATOR MENU

In the indicator menu of Data processing mode (Figure 5.19), you can see the current status of the scanning, magnetic field tuning error, microwave power applied to the resonator, microwave power attenuation, microwave generator frequency, magnetic field modulation amplitude, resonator and magnet temperatures, resonator Q-factor.

The **Detector current** indicator shows the current value of the microwave detector current in proportion to the microwave power supplied to the detectors. The AFC voltage indicator shows the control signal level of the microwave generator frequency in the microwave generator AFC device by the frequency of the measuring resonator. AFC device keeps the frequency of the microwave generator equal to the frequency of the resonator.

| NOTICE | *Scroll up the scroll bar to see all tuning panel parameters and command buttons if the size of the monitor does not allow to display the whole window.* |
|---|---|

| | |
|---|---|
| NOTICE | *Pay attention to the indicators after you have finished the connection between microwave bridge with the resonator. The detector current indicator should be in the area between two divisions 30 μA - 60 μA. Before scanning, make sure that the cursor of the detector current indicator is in the green zone.* |
| NOTICE | *The resonator Q-factor is displayed only after the coupling of the microwave bridge has been completed. In case of manual tuning of the microwave bridge this indicator is unfunctional.* |

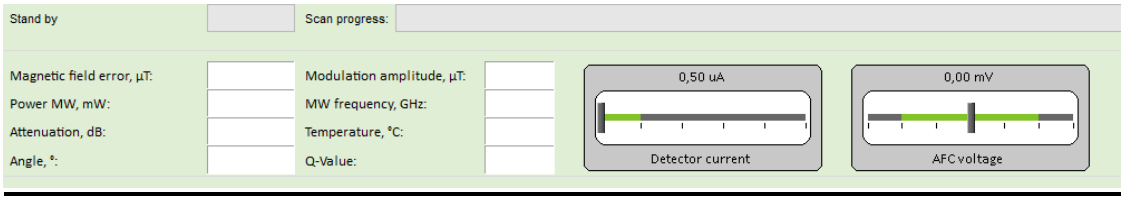



Figure 5.18 – Indicator panel

## 5.2 BATCH MENU

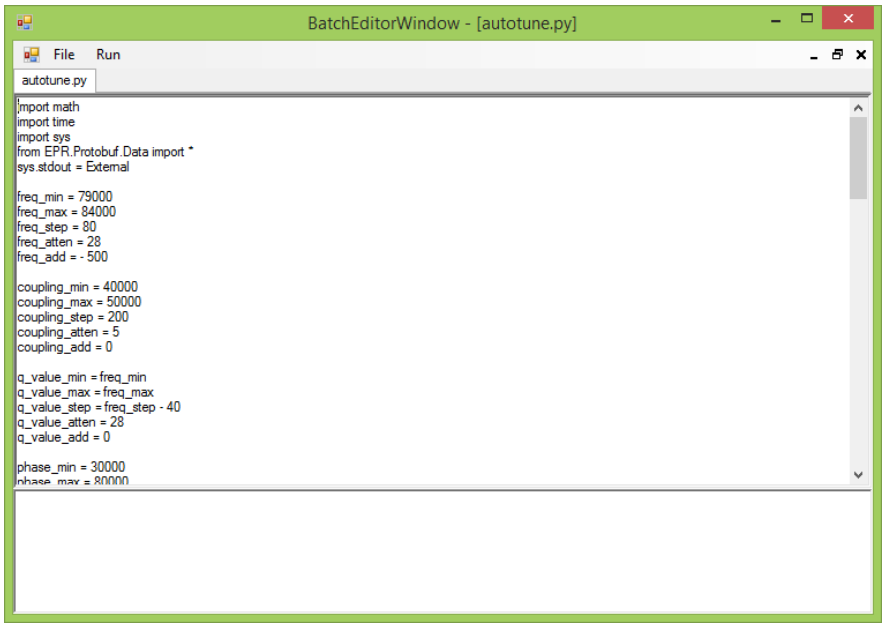


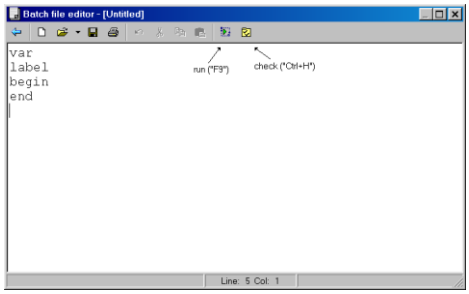

Figure 5.19

| | |
|---|---|
| NOTICE | *Script files are very helpful. If you do not want to repeat the same operations several times manually, you can create a script file, run it and wait for its completion.* |

**Tabs:**

**Run** – opens and runs the batch file;

**Editor** – open a batch file for editing (refer to Appendix A of this manual for the description of script editor).




Batch is a simplified language for editors designed specifically for older EPR spectrometers.

This language is easy to use and helps to automate all manual operations.

# OPERATION PROCEDURES

## SECTION 6

# 6 OPERATION PROCEDURES

Glass ampoules are used to perform measurements in the EPR-spectrometer:

- sample ampoules 3, 4, 5 and 6 mm OD;

- 100 mm capillaries;

- 100 ul “flat cell” cuvette;

The measured volume along the resonator vertical axis is 15 mm. Gauss distribution of the signal intensity occurs along this 15 mm distance. It means that 100% intensity is registered in the center, 95% intensity is 1 mm below and above the center, 90% intensity is 2,5 mm below and above the center. Hence, there are two options to get reproducible and comparable measurements:

1. Filling the sample ampoule along all the active volume of the resonator;

2. Providing approximately 5 mm sample height, but the sample should be precisely centered inside the resonator.

“Flat cell” cuvette can be oriented parallel or normal to the spectrometer front panel. The user should check both orientations and choose the one with maximal signal intensity.

Similar samples should be analyzed using one and the same ampoule for best results.

Capillaries are standard calibrated vials for sample preparation. They are usually used with the glass tube to place the capillary precisely vertically in the center of an ampoule. For aqueous solutions the maximum capillary volume is 50 ul. For organic solvents capillaries of larger volume or ampoules can be used.

Ampoules are used for liquid and solid samples. We offer ampoules of various diameter and length.

Solvent permittivity determines the ampoule diameter necessary for measurement. You may use ampoules with 3, 4, 5 and 6 mm OD. For solid samples the diameter is determined by the humidity of the sample.

If the sample cannot be tuned You may use the ampoule of lower diameter or take lesser amount of the sample.

Flat cells are often used for liquid samples with high permittivity. Uniform distribution of the sample inside the resonator allows for intensity enhancement several times vs the 50ul capillaries. Cell orientation, vertical positioning and filled volume are the most important factors for the flat cell usage.

50 and 150 ml liquid nitrogen finger flasks are recommended for low-temperature (77 K) measurements.

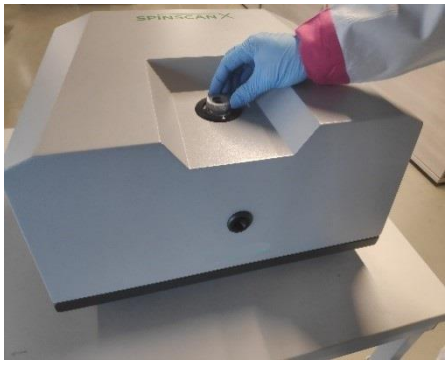
## 6.1 SAMPLE PREPARATION AND INITIAL SETUP

1. Start the **e-Spinoza** program on the operator's computer;

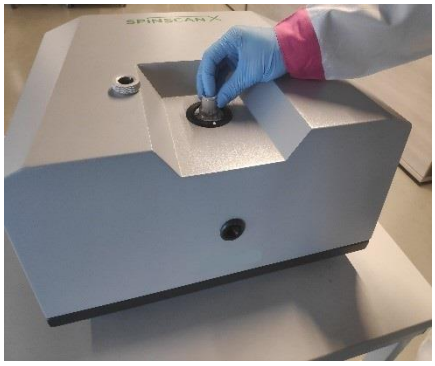
2. Switch on the SPINSCAN X spectrometer – the green LED indicator lights up;

3. Press the **Connect** icon to connect the spectrometer to the computer, and the spectrometer light indicator will light up green. The warm-up time of the spectrometer required for its stable operation is 90 minutes after the connection;

4. Unscrew the hold-down nut of the metal holder and take out compression paddle (Fig.6.1a, b);



a



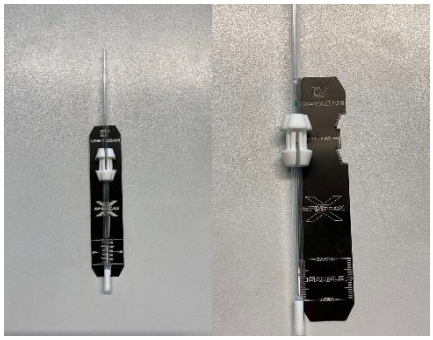
b

Figure 6.1 – a – Hold-Down Nut of the Metal Holder; b – Compression Paddle of the Metal Holder

5. Before placing the sample into the resonator thoroughly wipe and clean the outer surface of the sample tube to avoid contamination of the resonator;

6. For an ampoule with a sample, choose a Teflon holder to match the diameter of the ampoule. The ampoule should be able to move freely in the holder and not interfere with the fixation of the hold-down nut (Fig.6.2);

a



b

Figure 6.2 – a – Ampoule with A Sample, Teflon Holder; b – Teflon Holder Position

7. Place the ampoule with the sample in the Teflon holder. The distance (h) between the center of the sample and the top of the Teflon holder should be 95 mm. In this case, the center of the sample will be in the center of the resonator, as shown in Fig.6.4;

8. In case of using the holder for precise positioning, loosen the Teflon inserts at the top and bottom of holder, place the sample the sample ampoule with the outer diameter corresponding to the Teflon inserts and gently clamp back in. The distance between the center of the sample and the bottom of the metal holder should be 69 mm (Fig.6.3);

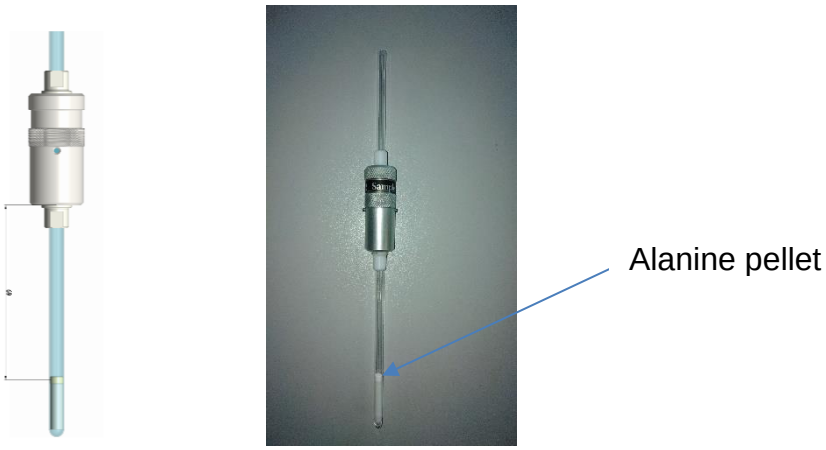



Figure 6.3

**NOTICE**

*The height of the active area in the resonator is 15 mm. Make sure that the sample minimum height conforms to that value. In case of small sample amounts (height less than 15 mm), carefully adjust the distance between the center of the sample and the Top of the Teflon holder.*

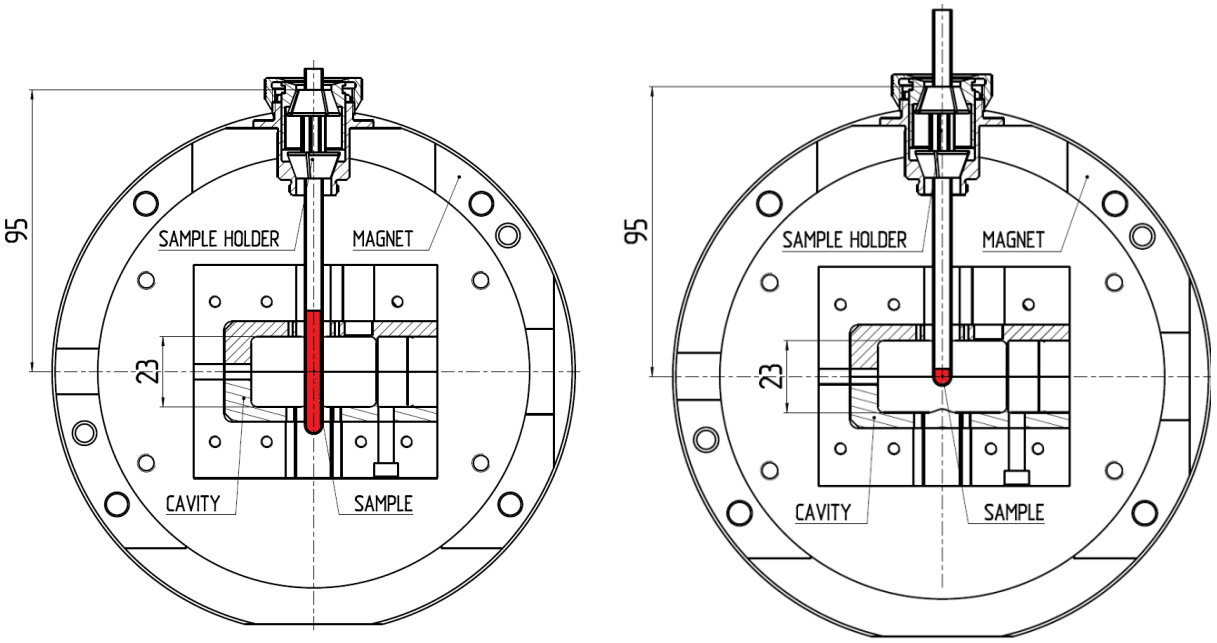
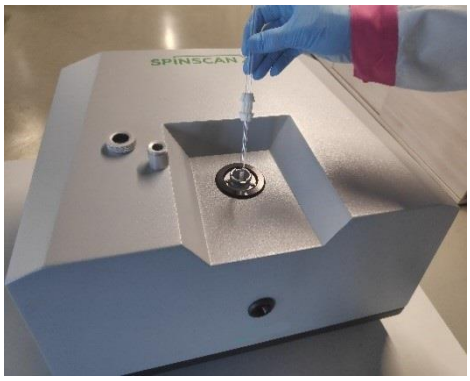
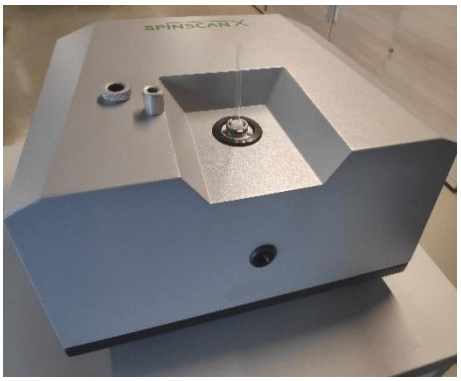



Figure 6.4 – Scheme of The Sample Test-Tube in the EPR Resonator:

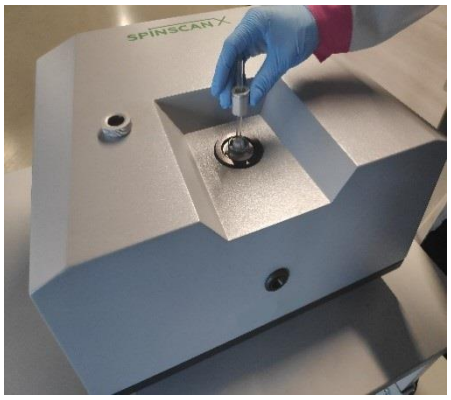
9. Insert the test tube with the sample into the Teflon holder (Fig.6.5 a);



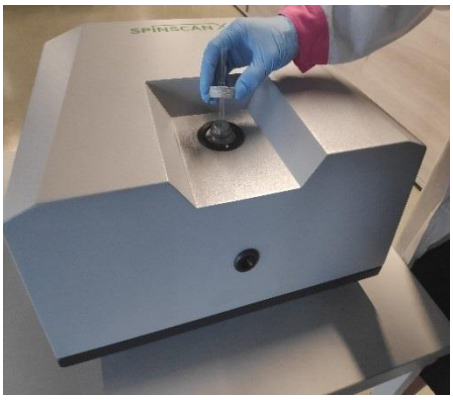
a



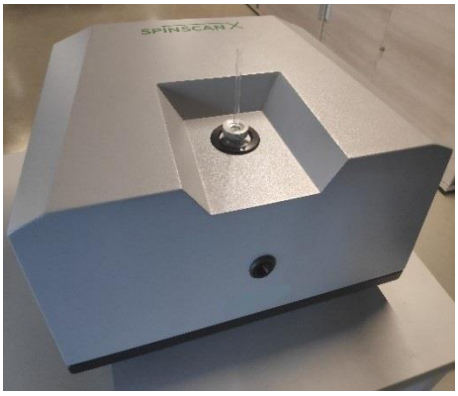
b



c



d



e

Figure 6.4

10. Fix the tube tightly with the metal hold-down screw (Fig.6.4 e);

11. Set the measurement parameters using **Spectrometer parameters** section (center field, sweep width, sweep time, modulation amplitude, time constant; number of smoothing points; number of points, number of scans);

12. Set the microwave **attenuation** (or **power**) in the drop-down menu **Microwave**;

The parameters for recording the reference sample can also be set as follows:

- open the previously recorded spectrum of the reference sample;

- click **Apply** in the drop-down report to the spectrum. The values of the spectrum parameters are automatically transferred to the corresponding cells of the **Spectrometer parameters** section and the **Microwave** section.

13. After all parameters are assigned press **Set parameters**. It will load the settings to the device to run the measurement ;

14. Before starting the measurement, connect the microwave bridge with the resonator – either automatically or manually (this procedure is described in Section 6.2);

15. As soon as Autotune is completed, the corresponding dialog box will appear on the screen. Click **OK,** if the tuning was successful

16. To start the scan, press the button  .

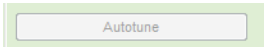
| NOTICE | *You can skip the repeated **autotune** procedure in case of performing a series of experiments under the same conditions without changing the position of the sample.* |
|---|---|

## 6.2 MICROWAVE BRIDGE TUNING

Click the icon 

on the toolbar to open the microwave bridge tuning window and leave the main window of the current measurement on the screen to be able to control all processes during connection. When the connection is completed, you can minimize the microwave bridge settings window or close it.

### 6.2.1 MICROWAVE BRIDGE TUNING (AUTOMATIC)

The automatic tuning algorythm provides matching of all parameters: frequency, connection and phase shift of the signal. Press the 

button and wait till the matching is completed.

| NOTICE | *We recommend performing a full auto-tuning before starting measurements and after each change of sample position in the resonator (when changing or moving the sample).* |
|---|---|

After the connection process is complete, the screen will show that the procedure is over.

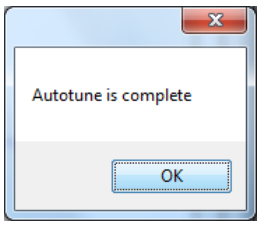



Figure 6.5 – Information window

## 6.2.2 MICROWAVE BRIDGE TUNING (SEMI-AUTOTUNE)

For semi-automatic and manual tuning of the microwave bridge press icon MW. **Microwave bridge monitor** (Fig.6.6) will be opened in separate window.

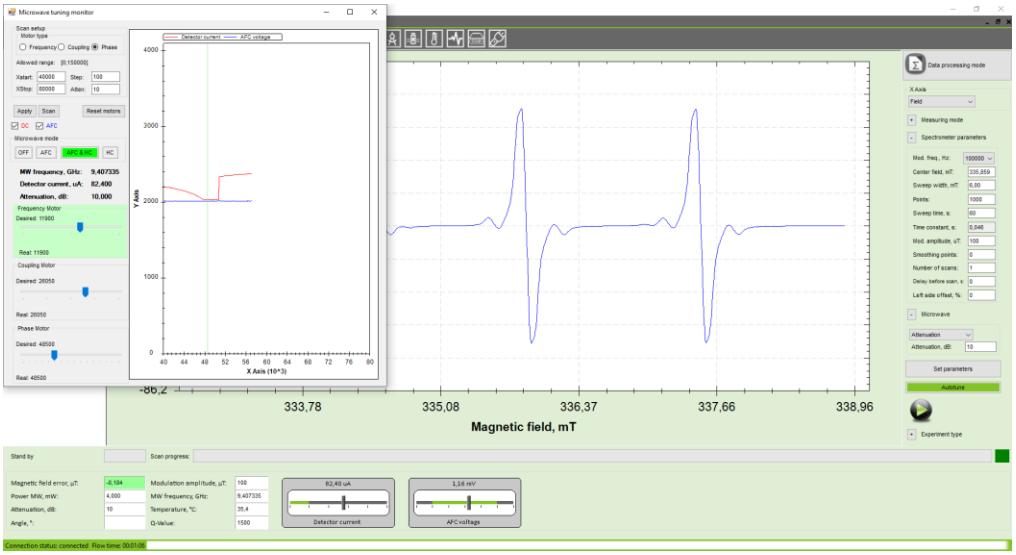


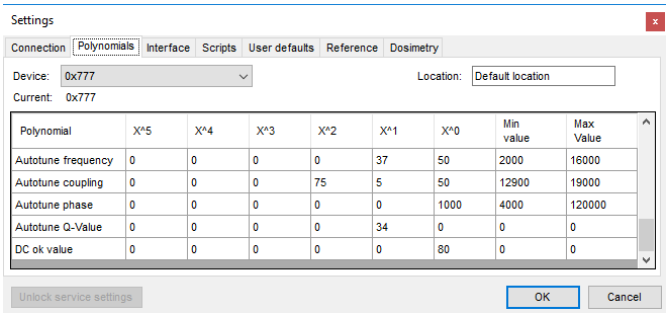

Figure 6.6 – Microwave bridge monitor

Keep the two windows on the monitor screen to follow the microwave bridge tuning process: the main window and the microwave tuning monitor window.Open the script in Batch menu editor in the folder Batch/File/Open system script/ full _ autotune.py and keep it opened on screen too.

Use the parameters which have been recorded and setup by manufacturer in **Polynomials tab**: Xmin, Xmax for all type of motors; number of steps and MW attenuation.

### 6.2.2.1 SEMI-AUTOMATIC (ROUGH) FREQUENCY TUNING

1. Switch on the **Main window**. Setup all parameters (except MW attenuation) what you will use to run real experiment (or use default parameters);
2. Setup **Attenuation** for Autotune frequency: column X^1 (30 – 40dB);




| Polynomial | X^5 | X^4 | X^3 | X^2 | X^1 | X^0 | Min value | Max Value |
|---|---|---|---|---|---|---|---|---|
| Autotune frequency | 0 | 0 | 0 | 0 | 37 | 50 | 2000 | 16000 |
| Autotune coupling | 0 | 0 | 0 | 75 | 5 | 50 | 12900 | 19000 |
| Autotune phase | 0 | 0 | 0 | 0 | 0 | 1000 | 4000 | 120000 |
| Autotune Q-Value | 0 | 0 | 0 | 0 | 34 | 0 | 0 | 0 |
| DC ok value | 0 | 0 | 0 | 0 | 0 | 80 | 0 | 0 |

Figure 6.7

3. Click **Set parameters**;
4. Open the **Microwave tuning monitor** window (Fig.6.8);

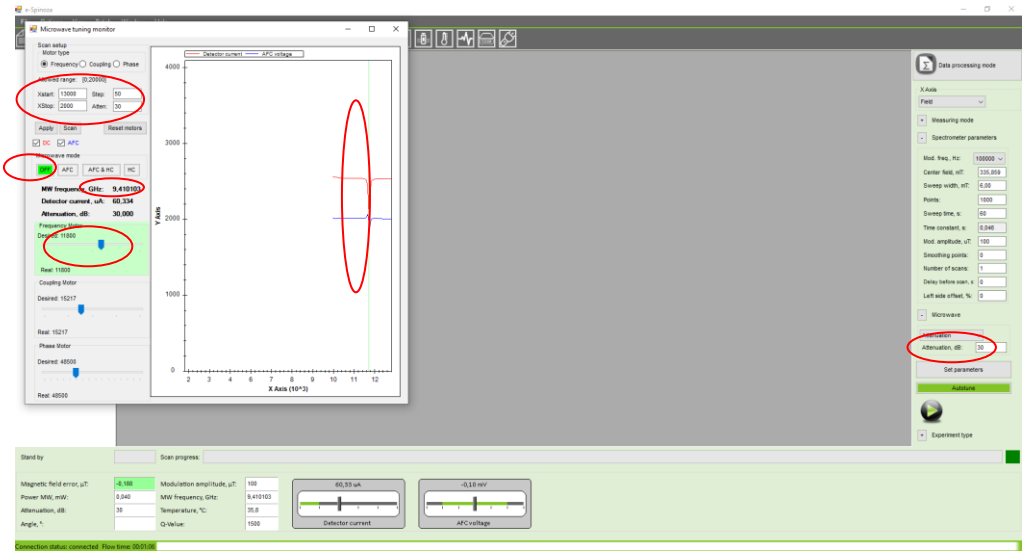


Figure 6.8

5. Check the type of motor. Motor for frequency adjustment is set by default;
6. Set **Xmin** and **Xmax**; step (see the **Polynomials tab** – Autotune frequency: Min value, Max value, X^0);
7. Press **Apply**;
8. Press **OFF**. The button will be green. Auto frequency control (AFC) and homodyne channel (HC) will be off;
9. Press **Scan**.

The graphical window on the right will display the frequency tuning process.

Once the scanning process is complete, find the resonance minimum microwave power on the detectors (the generator frequency is equal to the resonator frequency) (Fig. 6.8) and set the marker to this minimum by moving the engine slider. For subsequent manual tuning of the frequency, use the obtained value of the minimum Xfrq.

### 6.2.2.2 MANUAL (FINE) FREQUENCY TUNING

1. Move the **Frequency motor** cursor with mouse in position approximately equal to **Xfrq**;
2. Move the cursor slowly, while controlling the **Detector Current** indicator in the main window;
3. The detector current will change. Once the cursor of the detector current is in the green zone, stop moving the cursor of the motor;
4. **AFC voltage** indicator should be in the green zone.

| NOTICE | *The frequency is usually in the range of 9.300-9.550 GHz* |
|---|---|

### 6.2.2.3 SEMI-AUTOMATIC COUPLING TUNING

1. After completing the frequency tuning select the motor type mark '**Coupling**' and click the **AFC** (auto frequency control) button - it gets green (Figure 6.9);

2. Check the **AFC voltage** indicator, which should be in the green zone. Otherwise (out of green zone) repeat the frequency tuning;

3. Set the **Microwave attenuation** value in the e-Spinoza main window. The value is in the range of 2-10dB. It should be noted that tuning at lower power simplifies the procedure for finding the optimal value, and tuning at higher power increases its accuracy;

4. Click **Set parameters**;

5. Set all parameters (**Min value, Max value, X^0**) in the **MW tuning monitor** window (for the parameters limits see the **Polynomials tab)**;;

6. Click **Apply**;

7. Click **Scan**. The motor for Coupling will move in the selected range Xmin - Xmax.

The graphical window on the right will display the coupling tuning process.

After the scan is completed, you will see that the detector current at the resonant frequency is minimal (optimal coupling) (Figure 6.11). Set the marker to this minimum by moving the motor slider.

For subsequent manual fine tuning of coupling, use the minimum Xcpl value

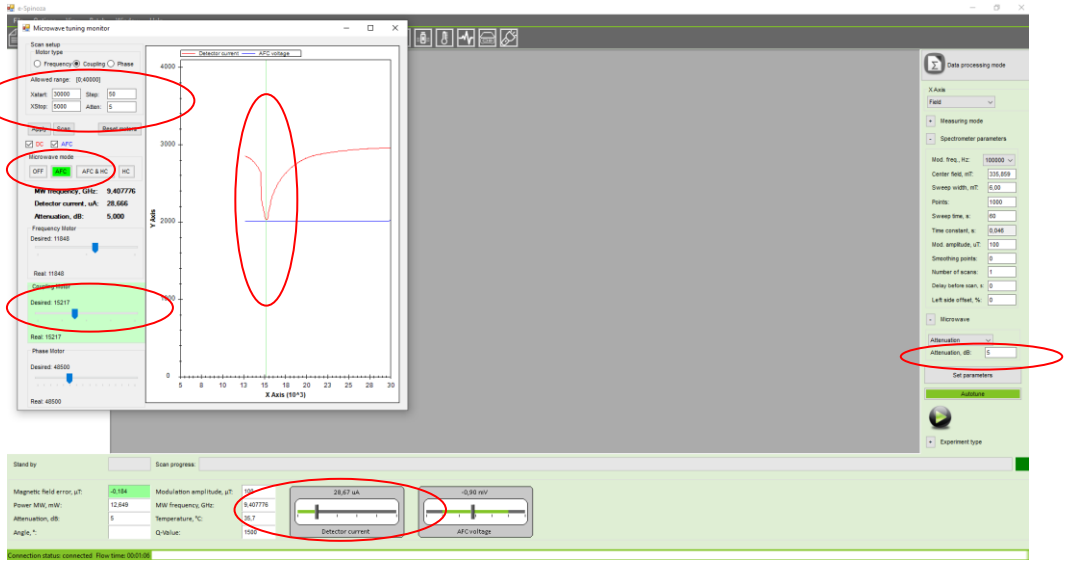


Figure 6.9

### 6.2.2.4 MANUAL (FINE) COUPLING TUNING

1. Set the Coupling motor cursor to a position approximately equal to Xcpl;
2. Move the cursor slowly and check the **Detector Current** Indicator;
3. Detector Current will be changing. As soon as the detector current reaches 0, the indicator will become red, stop the cursor for the motor in this position. The **Coupling** tuning is completed.

### 6.2.2.5 SEMI – AUTOMATIC PHASE SHIFTING TUNING

1. After the coupling is tuned, set the **Microwave attenuation** in the **e-Spinoza main window** to the phase tuning. This value is the current value for the measurement;
2. Press **Set parameters**;
3. Set parameters from **Polynomials – Autotune phase: Min value, Max value, X^0**;
4. Select motor type - **Phase**;
5. **AFC&HC** – automatic frequency control with homodyne channel enabled. The button will become green;
6. Press **Apply**;
7. Press **Scan**. The motor responsible for the phase tuning will start moving in the selected Xmin - Xmax range.

The graphical window on the right will display the phase tuning process.

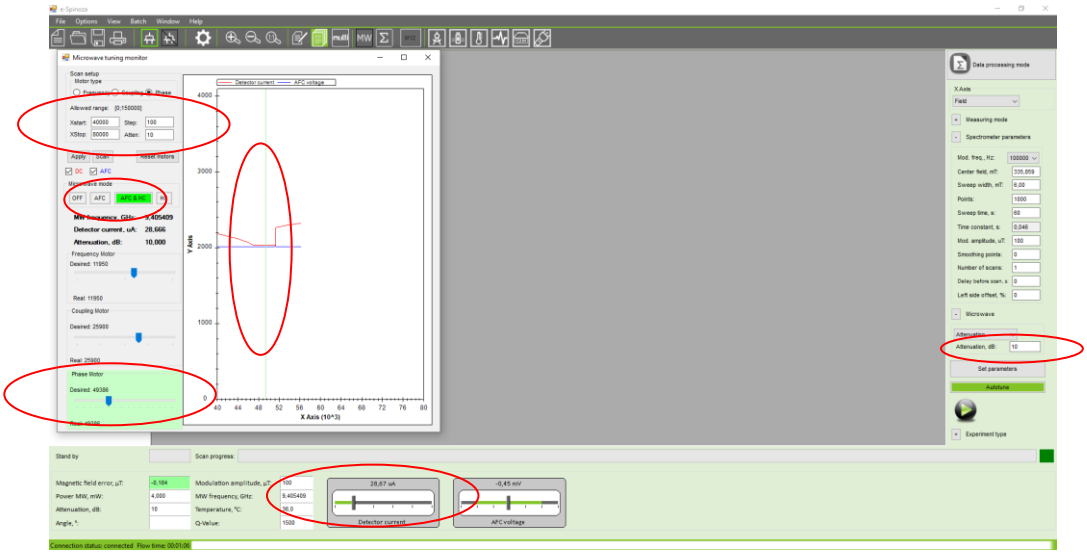


Figure 6.10 – Semi-autotune is completed.

After completing the scanning process, find the position of the minimum detector current (optimal phase) (Fig. 6.10). Place the marker on this minimum by moving the engine slider. Please use this minimum value Xcpl for subsequent manual tuning of phase shift.

### 6.2.2.6 MANUAL (FINE) PHASE SHIFTING TUNING

1. Place the Phase motor cursor in a position approximately equal to Xcpl;

2. Move the cursor slowly and control the process with **Detector Current Indicator** in the **Main window**.

Detector Current will be changing. By moving the motor control slider, the minimum value of the detector current will be obtained (this minimum is not equal to zero). The phase shift tuning has been successfully completed.

| NOTICE | *Instead of semi-automatic tuning, you can adjust all motor types manually. Scroll the position of each motor in the recommended range to find the approximate position of the motor in resonant conditions. Follow the instruction for accurate tuning.* |
|---|---|

## 6.3 DATA ACQUISITION

When the MW bridge tuning is successfully completed, you can start the measurement. Press the button  to start the scanning process. Once the measurement process starts, the other two **Stop** and **Pause** buttons will appear on the right side of the screen instead of **Scan**  .

**Scan** – starts the measurement;

**Stop** – stops the measurement; after that you cannot continue the measurement. The data will not be recorded in the file;

**Pause** – stops the magnetic field sweep, but the measurement continues over time. Press  to continue measurement during magnetic field scanning.

When the measurement is finished, the system will ask you to save the data, and a dialog box will appear:

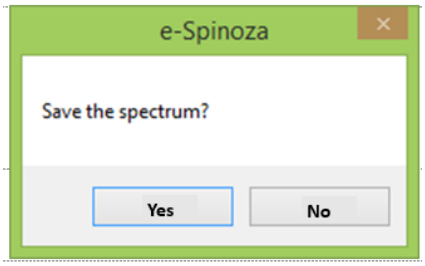



Figure 6.11

If you want to save, press **Yes**. In this case, you can choose a folder in the dialog box and change the file name, or you can find the file in the default auto-saved folder (**Settings** /**Connection** Tabs). If you do not need to save this spectrum, press **No.**

**NOTICE** *All scanned spectra are automatically saved in the default folder (* see the **Settings/Interface** tab *).*

All data files are saved as temporary and displayed in stack bar (Fig.6.12). You can save spectrum at any time before closing the program, but we recommend doing it as soon as the measurement is complete.

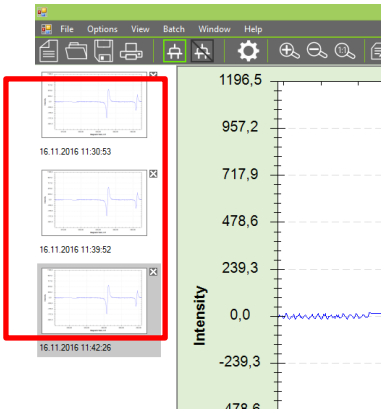



Figure 6.12

## 6.4 SPECTRUM VIEW MODES

A right mouse click in the graphic window allows you to open standard functions that can be used to work with the spectrum (Fig.6.13). A dialog boxes will appear:

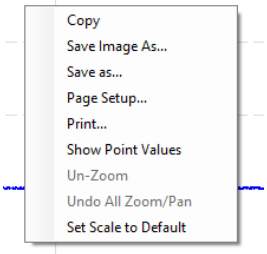



Figure 6.13

**Copy** – copies the spectrum image to the clipboard;

**Save Image as…** – saves the spectrum image under a new name;

**Save as…** – saves the spectrum under a new name;

**Page setup…** – tunes page settings for printing;

**Print…** – prints the current spectrum;

**Show point values** – displays the coordinates of the selected point on the screen (Fig.6.14);

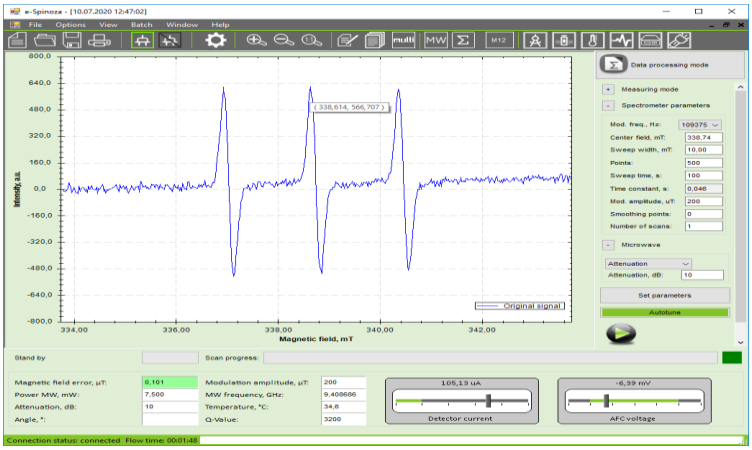
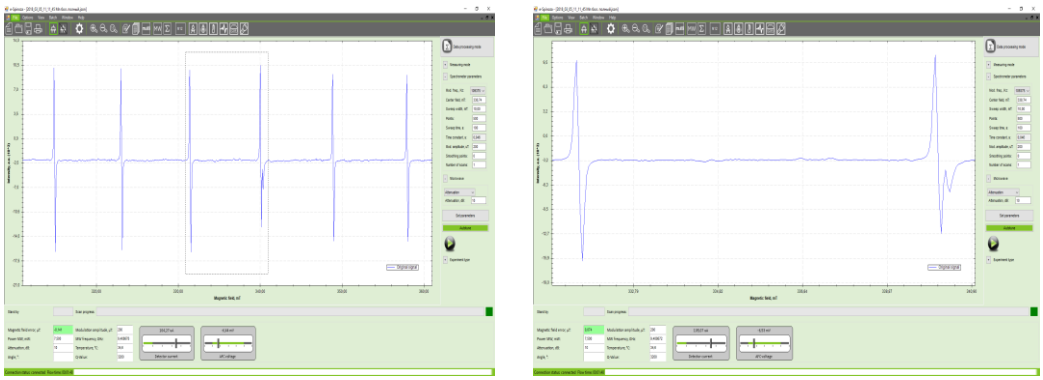



Figure 6.14

**Un-Zoom** – un-zooms in the picture;

**Undo All Zoom/Pan** – returns to the initial spectrum view;

A left click chooses a fragment of the spectrum and holds the zoom of the spectrum image. To zoom only X or Y axis, select full displayed scale Y or X axis and move cursor selecting fragment you want to zoom. Only one axis will be stretched (Fig. 6.15).



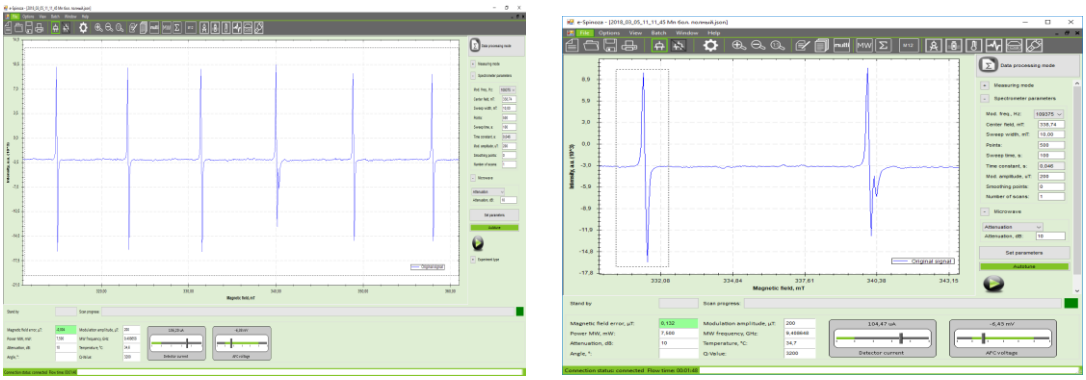


Figure 6.15 – Zooming only X or Y axis

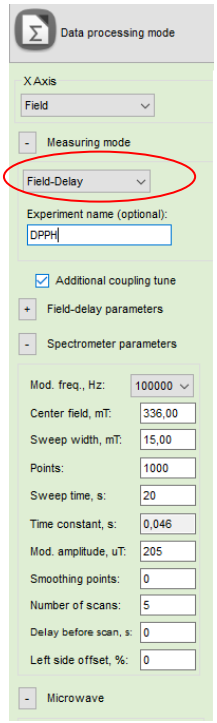
## 6.5 MEASUREMENT SERIES (SIGNAL ACCUMULATION)

In case you want to perform more than one scan for a sample, you should specify the required number of scans in the **Settings** menu of the main window. After one scan has been completed, subsequent measurements (scans) will be performed one by one under the same conditions.

All measured spectra are stored in a buffer and only one spectrum obtained after averaging is displayed in a graphic window. You can see all recorded spectra ONLY in the mathematical processing mode (**Average** function). The removal of any failed spectrum from the series is described in chapter 7.6

## 6.6 MEASUREMENT MODES

### 6.6.1 FIELD-DELAY




If you need to observe the change in signal intensity over time, you need to:

- Select the type of **Field-Delay** experiment from the drop-down list;
- Set the name of the experiment in the **Experimental name (optional)** window, which will be displayed in its name (optional);
- Set the **delay time** in the **Field-Delay parameters** window;
- Set the **number of scans** in the **Spectrometer parameters** window;
- Press the **Set parameters** button;
- Press the button  to start the scanning process with the above requirements (Fig.6.16).

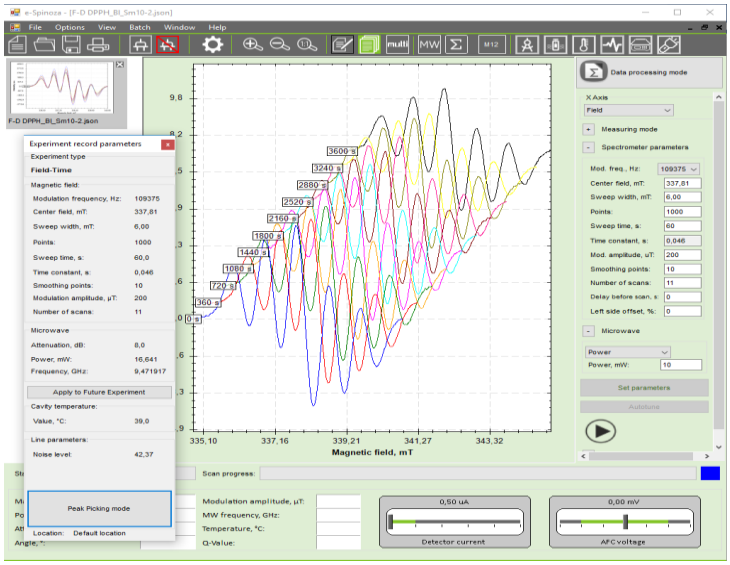



Figure 6.16 – Field-Delay experiment

To display the intensity change over time of any peak in the recorded signal, it is necessary:

- Switch to **peak picking mode** by pressing the corresponding button in the experiment report (Fig.6.17),;
- In the peak picking mode window on the graph, select the studied peak in the signal, thereby implementing the peak picking mathematical function;

- The change of signal in numerical (in the table) and graphical versions will display at the windows below;

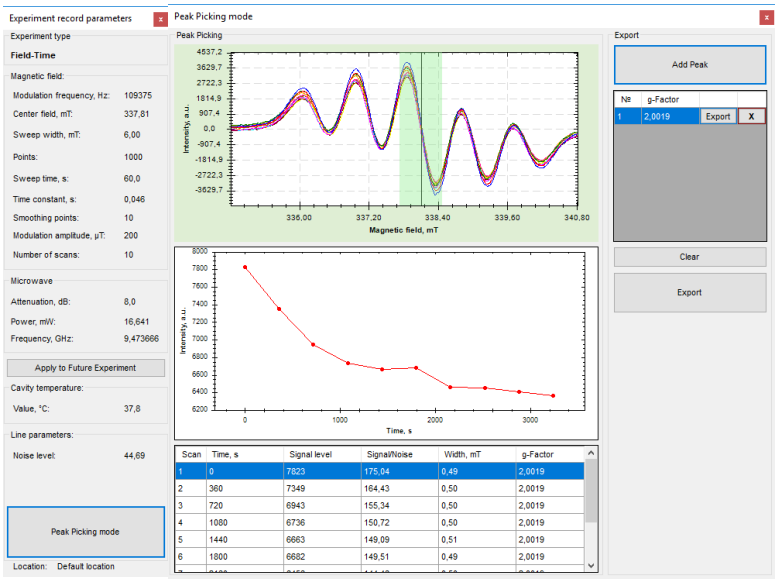


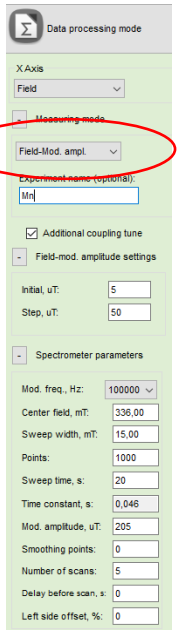

Figure 6.17 – Field-Delay experiment report

• To export the necessary information about the signal, select a peak on the chart, click the **Add Peak** button in the Export subwindow and the results will be displayed in the table;

• To export data on changes in the intensity of a single peak in the signal, select the desired peak in this table and press the **Export** button for this row. To export data on changes in peak intensities in the signal, click the **Export** button in the Export subwindow. To delete peak data, press either **x** (single peak) in the corresponding row of the table, or the **Clear** button (clearing the entire table).

## 6.6.2 FIELD-MODULATION AMPLITUDE

When conducting the Field-Modulation amplitude experiment, it is necessary:




- Select the type of **Field- Mod. ampl** experiment from the drop-down list;
- Set the name of the experiment in the **Experimental name (optional)** window, which will be displayed in its name (optional);
- Set the initial value of the modulation amplitude and the step of changing this peremeter in the **Field- Mod. ampl settings** window;
- Press the **Set parameters** button;
- Press the button  to start the scanning process with the above requirements (Fig.6.18).

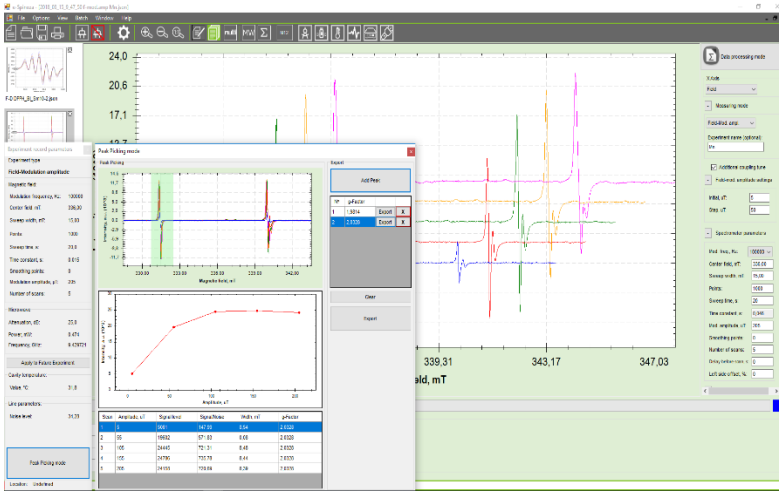
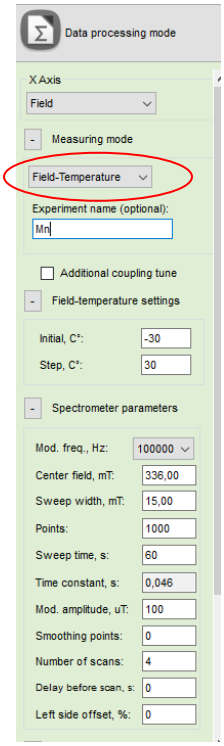



Figure 6.18 – Field- Modulation amplitude experiment

## 6.6.3 FIELD- TEMPERATURE




When conducting **the Field-Temperature experiment**, it is necessary:

- Select the type of **Field-Temperature** experiment from the drop-down list;
- Set the name of the experiment in the **Experimental name (optional)** window, which will be displayed in its name (optional);
- Set the initial value of the Temperature and the step of changing this peremeter in the **Field- Temperature settings** window;
- Press the **Set parameters** button;
- Press the button  to start the scanning process with the above requirements (Fig.6.19).

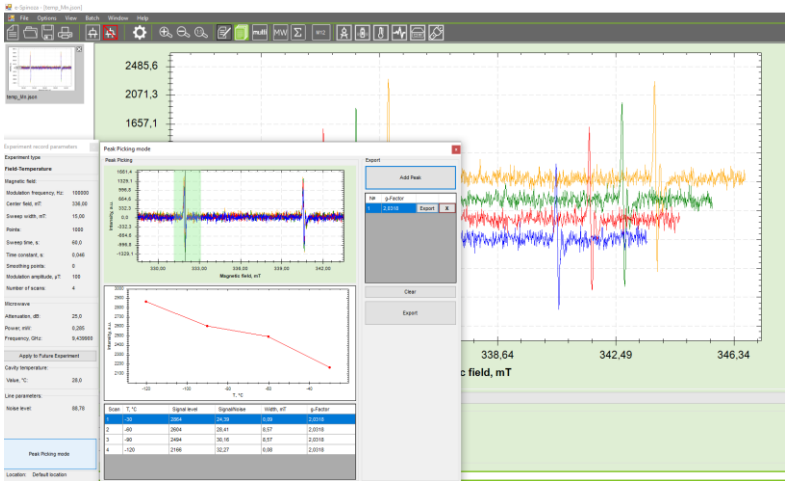


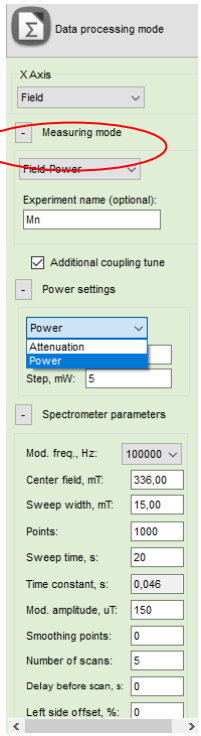

Figure 6.19 – Field- Temperature experiment

## 6.6.4 FIELD- POWER

When conducting **the Field- Power experiment**, it is necessary:

- Select the type of Field- Power experiment from the drop-down list;
- Set the name of the experiment in the **Experimental name (optional)** window, which will be displayed in its name (optional);
- Select the type of experiment (**Power** or **Attenuation)** in **Power setting** window;

- Set the initial value and the step of changing this paremeter in the **Field- Temperature settings** window;
- Press the **Set parameters** button;
- Press the button  to start the scanning process with the above requirements (Fig.6.20).



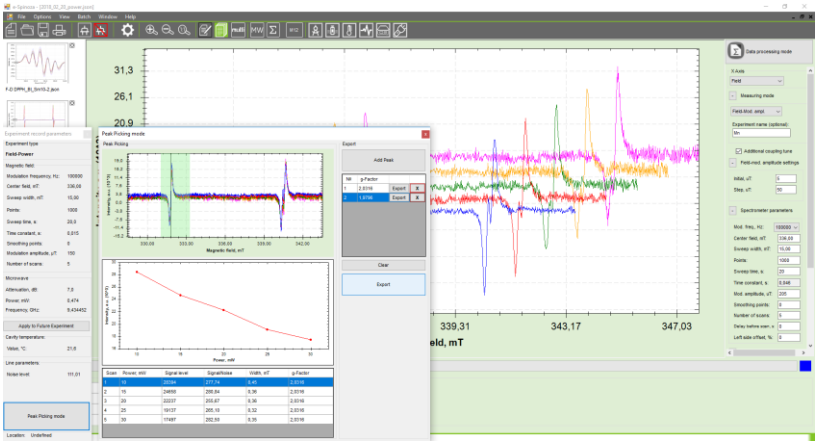
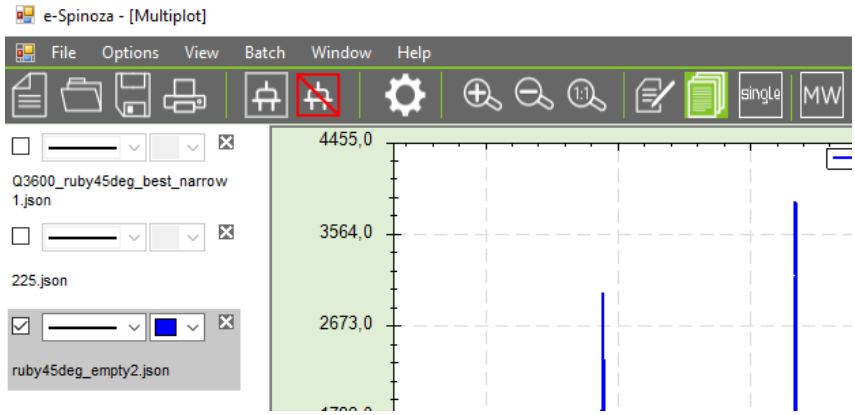




Figure 6.20 – Field- Power experiment

## 6.7 MULTI PLOT FUNCTION

To activate the multiplot function, press the button on the control panel.

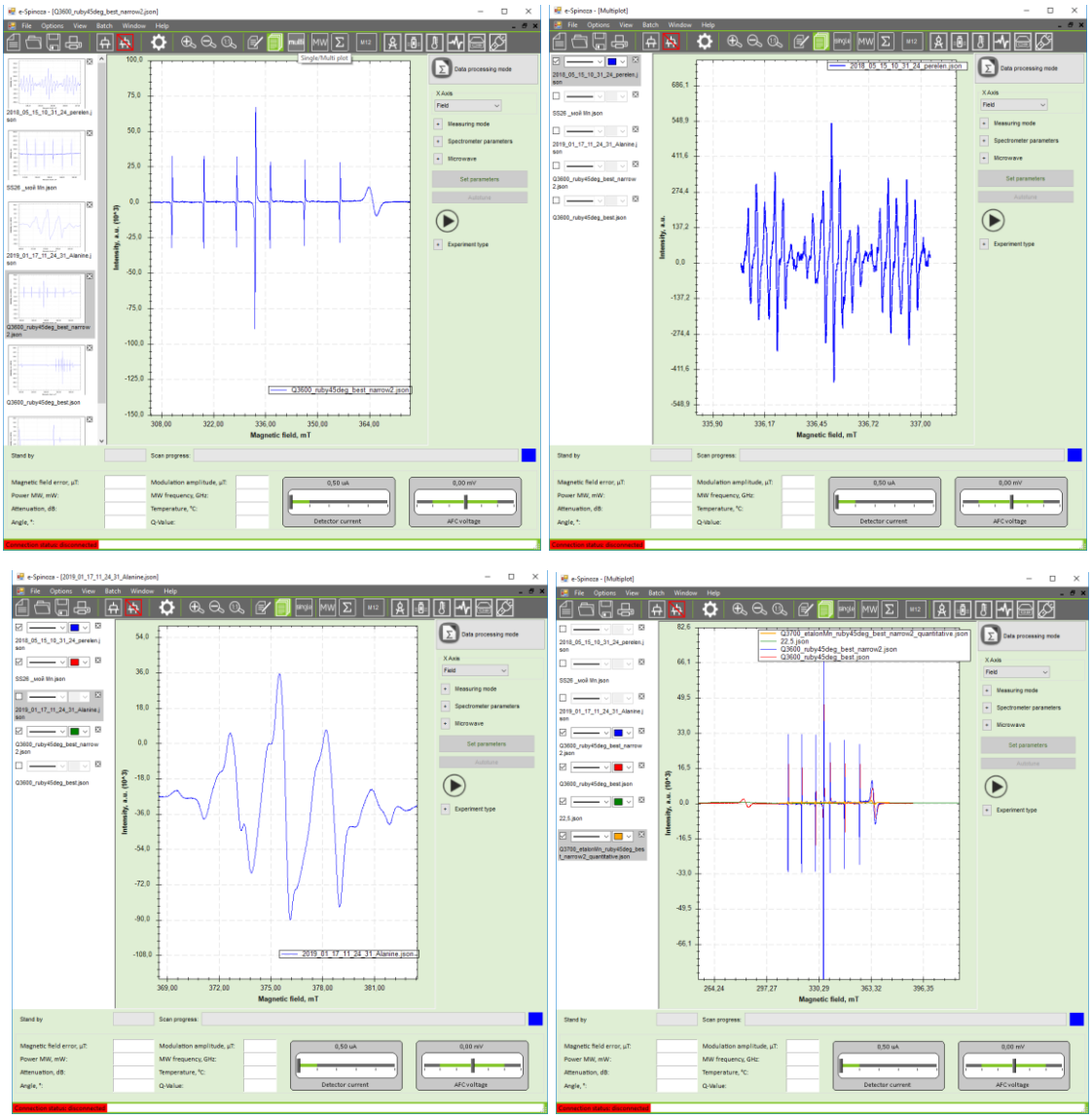
This function is implemented only for 2D experiments. The display of recorded spectra changes on the slide panel when using this function.




To select the spectra to be displayed in the graphics window in **multiplot mode**, check the box on the right for the spectra found in the slide panel. The signals are displayed in the selected type and color, which can be changed individually.

When using multiplot function you can view the spectra both in this mode and in single (ordinary mode).

At the graphical window will display only those spectra that are highlighted with a check mark on the slide panel (a check mark in the window on the right). To do this, highlight the required spectra in color and right-click on the name of the corresponding spectrum.



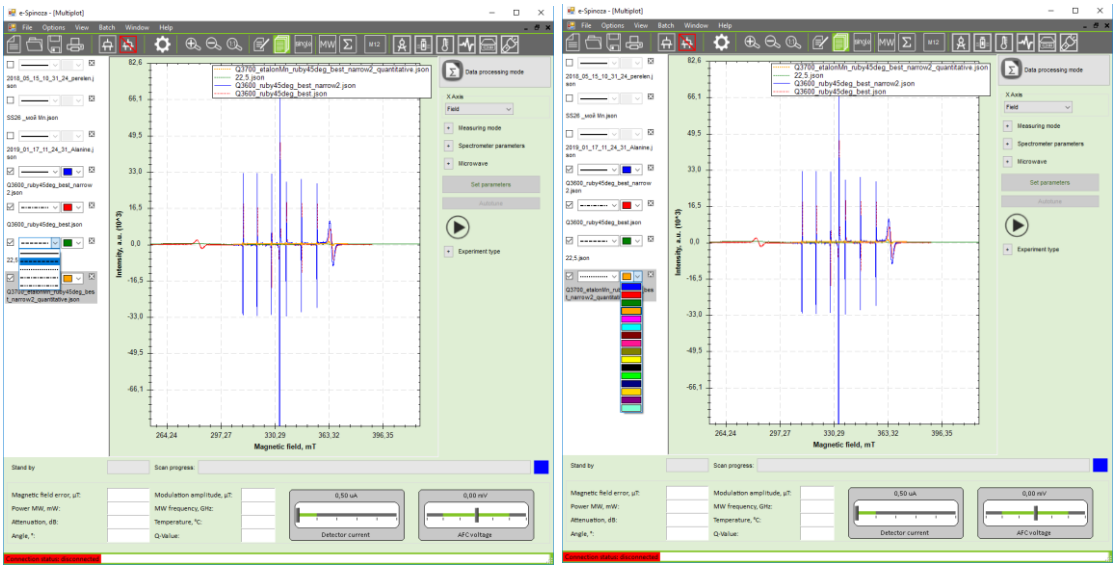


Figure 6.21 – Multi plot function

## 6.8 SPECTRUM DISPLAY MODE

The spectrum of the sample can be displayed in a graphical window depending on the selected value of the X-axis: Field, g-factor, Frequency, time.

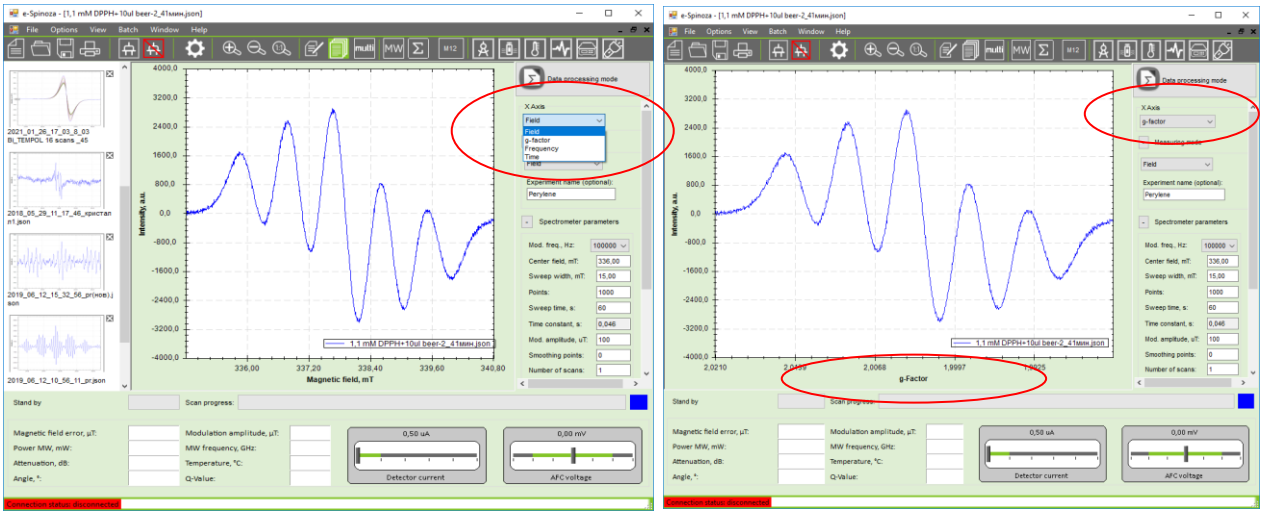


Figure 6.22 – Spectrum display mode (g-factor)

# DATA PROCESSING

## SECTION 7

# 7 DATA PROCESSING

This chapter describes the opportunities of the data processing functions. Press the button **Data processing mode** (or button in tool bar) to switch to the main window of data processing interface. On the graphic window the last measured spectrum will be displayed. Continue to work with this spectra or open new one which you want to process from the folder or from stack bar.

In the Data Processing mode, the following list of functions for processing the obtained spectrum is proposed:

Phase adjusting, Smoothing, Differentiation, Integration, Baseline correction, Averaging, Peak Picking, Curve Fitting, algebraic operations with spectrum lines (Linear Algebra).

**NOTICE**

*You can use the functions of the **Data Processing mode** when the spectrometer is off.*

## 7.1 PHASE ADJUSTING

Phase adjusting is a function which mathematically changes the value of the phase shift between the modulation signal and the reference signal of the synchronous detector.

To perform the phase adjusting:

- use the current spectrum or load one from the folder;
- choose **Phase adjusting** in the drop-down list;
- when moving the cursor, the modified spectrum will be displayed on the screen;
- press **Apply** button (Fig.7.1).

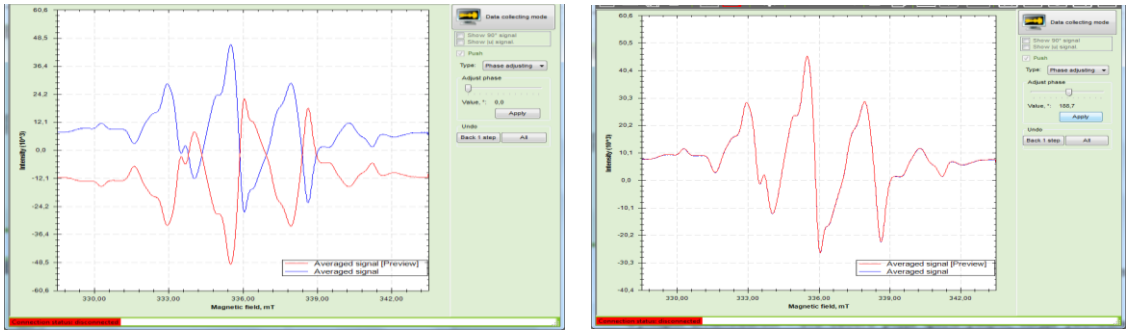


Figure 7.1 – Spectrum Before and After the 180º Phase Adjustment

## 7.2 SMOOTHING

Smoothing function is a kind of filtering when the noise is reduced. Moving a scroll bar Adjust smoothing you choose a number of points for binomial smoothing. The more points you choose the smoother spectrum you get.

The number of smoothing points is selected so that the intensity of the smoothed signal decreases by no more than 10% (Fig. 7.2). For a given number of smoothing points **n**, the intensity of each point in the spectrum will be given by the weighted average of **2n + 1** points (**n** before a specific point, **n** after the point itself). Weighting coefficients are given by binomial $C_{2n}^{k} = \frac{2n!}{k!(2n-k)!}, k = \overline{0,2n}$. So after smoothing, the intensity of each point will be equal to: $I_0 = \frac{\sum_{k=0}^{2n} C_{2n}^{k} I_{k-n}}{2^{2n}}$, where $I_{k-n}$ is the intensity of the points on the left (with negative index) and to the right (with positive) from the current one. $2^{2n}$in the denominator is necessary for normalization. For example, for **n = 2**, the intensity of the spectrum points is as follows: $I_0 = \frac{1 \cdot I_{-2} + 4 \cdot I_{-1} + 6 \cdot I_0 + 4 \cdot I_1 + 1 \cdot I_2}{16}$.

To perform the smoothing:

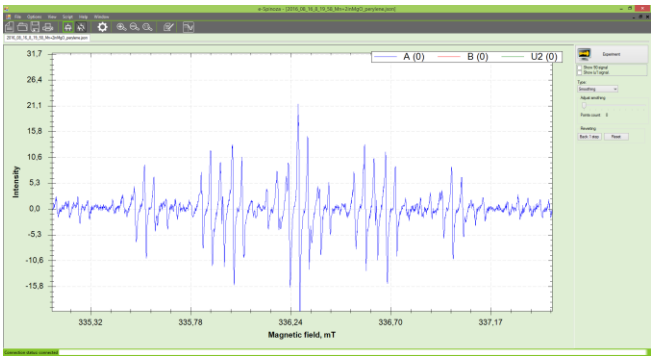
- use the current spectrum or load one from the folder;

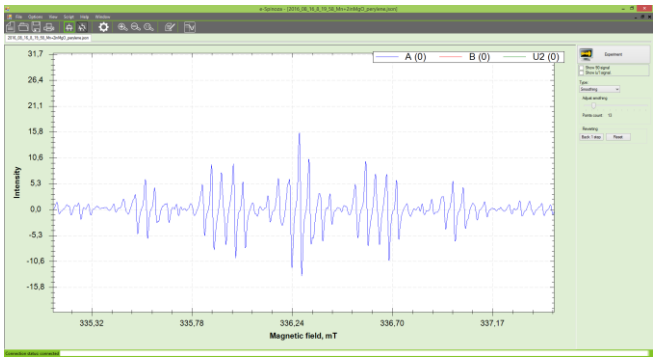
- choose **Smoothing function** in the drop-down list;

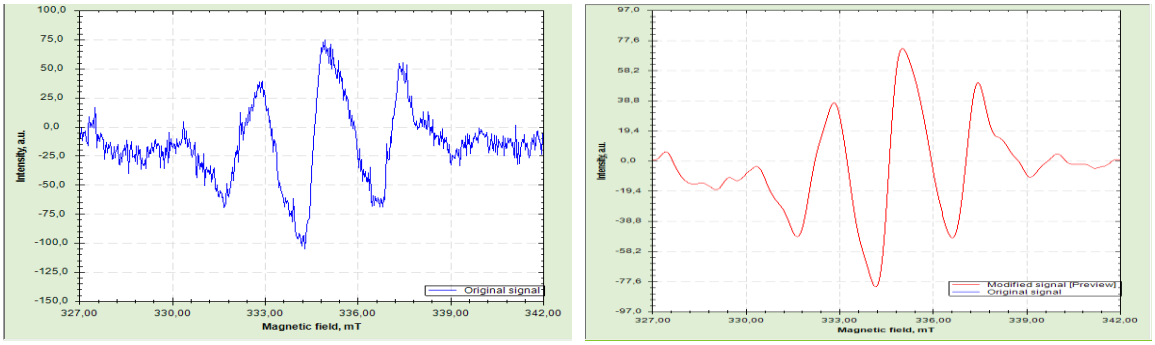
- move the cursor in the scroll bar to choose the required number of smoothing points or enter the number manually;

- Click **Apply**, if you think the smoothing result is sufficient.

Click **Back 1 step,** if you need to go back after smoothing. Press **All,** if you want to return to the initial spectrum (Fig.7.2).






Initial spectrum | After smoothing

Figure 7.2 – Spectrum before and after smoothing

## 7.3 BASELINE CORRECTION

If EPR spectra may have offsets, sloping baselines or background signals from other species before doing data processing it is necessary to do a baseline correction. To perform baseline correction:

- use the current spectrum or load one from the folder;

- choose **Baseline** in the drop-down list;

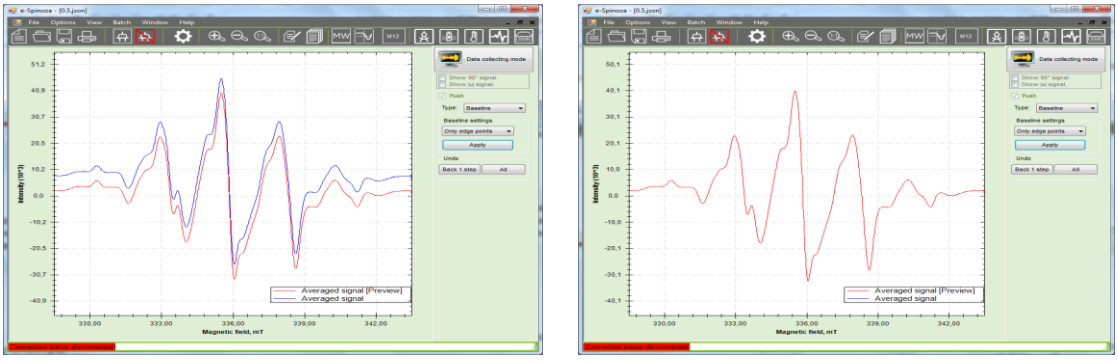
- choose **Baseline settings**: automatically only edge points or manually;

- in manual mode, choose the points on the spectrum on which the baseline will be corrected;

- click **Apply**.

| NOTICE | *For manual base line correction, the end points of the recorded spectrum are selected automatically.* |
|---|---|

If after the base line correction, you need to return to 1 step, press **Back.** If you want to return to the initial spectrum, press **All** (Fig. 7.3).




Initial spectrum | After Baseline correction

Figure 7.3 – Spectrum before and after Baseline correction.

## 7.4 INTEGRATION

For quantitative characterization of an EPR signal, a double integration of the first derivative of the EPR spectrum is usually used. Since most spectrometers record the EPR signal as the first derivative of the absorption signal, we must integrate the spectrum once to restore the absorption spectrum and then integrate again to obtain the area under the absorption curve.

To use the Integration function to process the obtained spectrum:

- use the current spectrum or load another one from the folder;

- choose **Baseline** in the drop-down list and follow the recommendations described in section 7.3. Even slight baseline drifts, background signals, or a very low signal to noise ratio can effect on the accuracy of the integration;

- choose **peak picking** in the drop-down list and follow the recommendations described in section 7.7;

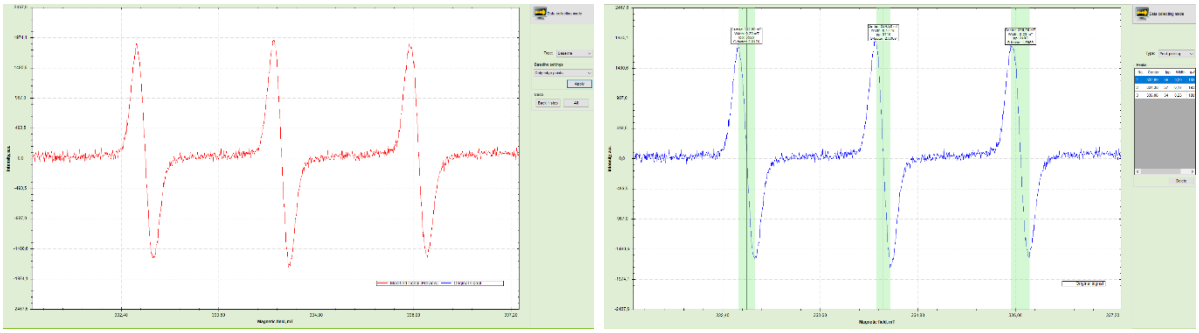
- choose **curve fitting** in the drop-down list and follow the recommendations described in section 7.8;

- choose **Integration** in the drop-down list;

- click **Apply** 2 times;

- click **None**;

If you need to come back after **Integration** on 1 Step press **Back**. If you want to come back to initial spectrum press **All** (Fig. 7.4).



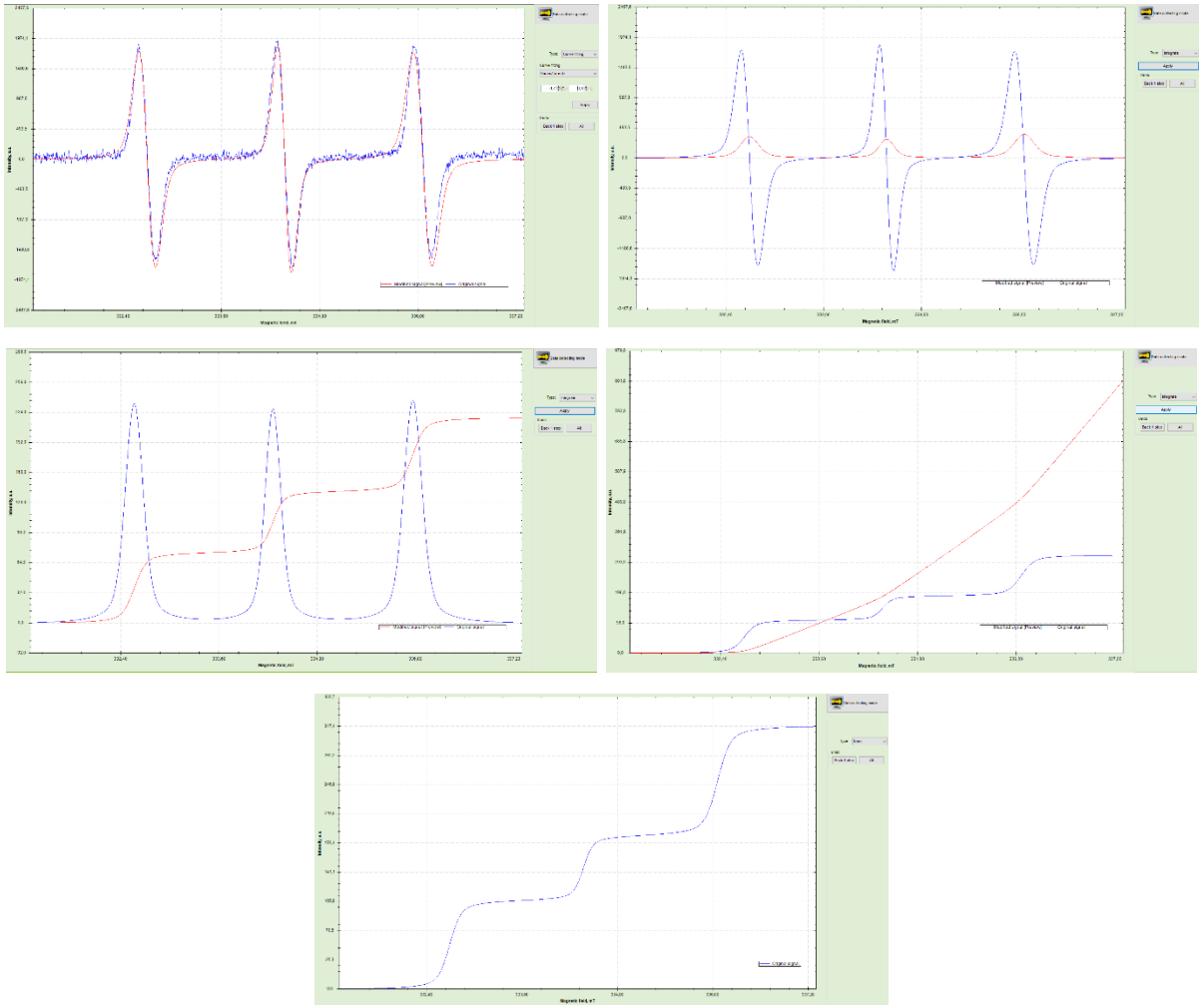


Figure 7.4 – Operation with Integration mode

## 7.5 DIFFERENTIATION

To use the **Differentiation** function to process the obtained spectrum:

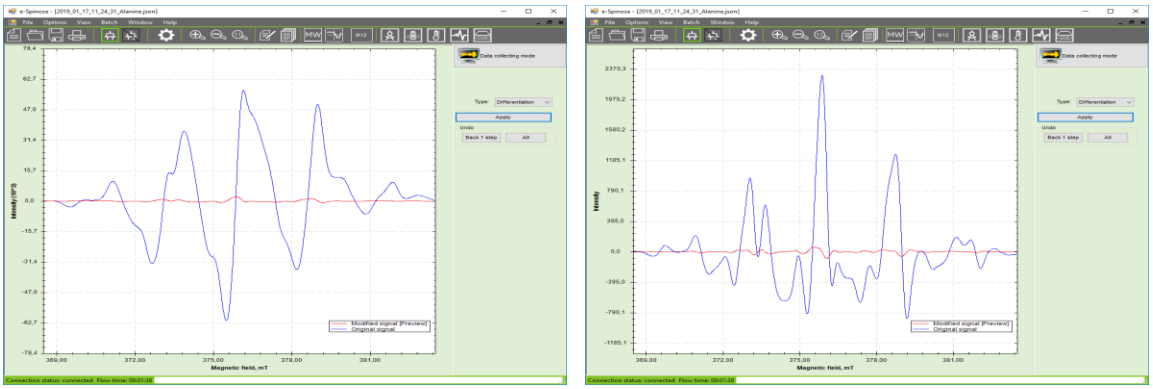
- use the current spectrum or load another one from the folder;

- choose the **Smoothing** in the drop-down list and follow the instruction described above in chapter 7.2;

- choose **Differentiation** in the drop-down list;

- click **Apply**.

If you need to return after **Differentiation** on 1 Step press **Back**. If you want to return to the initial spectrum, press **All** (Fig.7.5).



Initial spectrum | After differentiation

Figure 7.5 – Spectrum before and after differentiation.

## 7.6 SIGNAL AVERAGING

Signal averaging can be used to improve the signal\noise ratio of an EPR spectrum.

It is possible to see here the results of individual scans during accumulation.

To perform the averaging:

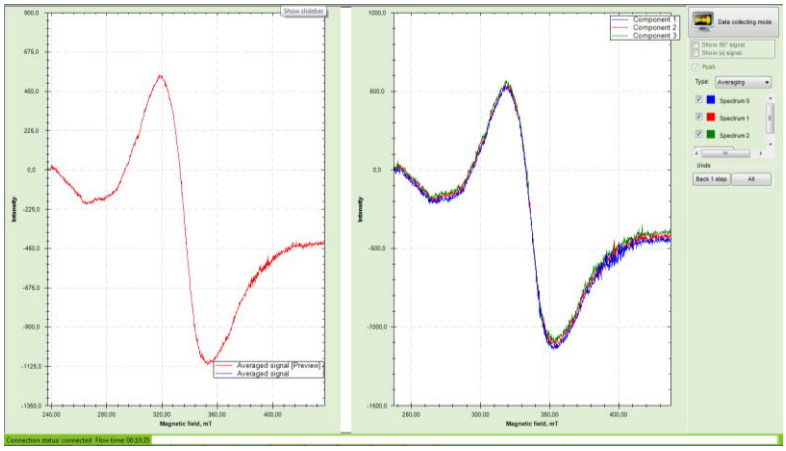
- use the current spectrum or load another one from the folder;

- select **Averaging** in the drop-down list;

Two graphic windows will appear on the screen. The left graphic window shows the averaged spectrum after measurement, on the right graphic window you will see all original spectra what were used for averaging (Fig.7.6).

- in the panel on the right you will see a list of check boxes with marks. The color of window corresponds to the color of spectra in the right graphic window;

- a tick in the box indicates the participation of this spectrum in the averaging process. Remove the ticks if you do not want to use the selected spectra for averaging. On the left graphic window you will immediately see the result of averaging.



Average spectrum window  Individual scans window

Figure 7.6 – Operation with averaging mode.

## 7.7 SPECTRUM LINE SELECTION (PEAK PICKING) AND CHARACTERIZATION

To use the **peak picking** function to process the obtained spectrum:

- use the current spectra or open any one you need from the folder;
- choose the **Peak picking** in the drop-down list
- select the desired peak (spectrum line) in the graphical window of the spectrum.

The corresponding dialog box will appear on the screen. After confirmation all marked peaks will be displayed on the right in the corresponding table (Fig. 7.7);

Figure 7.7 – Operation with Peak Picking

After confirmation all selected picks will be displayed in the table on the right panel. Select and press **Delete** if you want to remove it from list.

## 7.8 CURVE FITTING

An analytical curve can be selected for the current spectrum, which will exactly repeat the shape of the lines.

One of the options is a combination of Gaussian and Lorentzian $kG + (1 - k)L$, where $k$ = *[0;1]*, *G* and *L* are the sum of Gaussians and Lorentzians for each line respectively, described by the formulas:

$$G(x) = \frac{Ipp}{2} \cdot a \cdot e^{-\frac{a^2-1}{2}},$$

$$L(x) = \frac{16 \cdot \frac{Ipp}{2} \cdot a}{(3+a^2)^2},$$

$a = \frac{x - H_0}{0{,}5 \cdot \Delta H_{pp}}$, where $Ipp$ is the line intensity, $H_0$ is the center line, $\Delta H_{pp}$ is the line width, $x$ is the magnetic field in mT.

To use the **curve fitting** function for processing the spectrum:

- use the current spectrum or open any one you need from the folder;

- choose **baseline** in the drop-down list and follow the recommendations described in chapter 7.3;

- perform **peak picking** to determine the line's parameters;

- choose **curve fitting** in the list of functions;

- to provide the best fitting of the approximated curve to experimental spectrum choose the best ratio coefficients for Gauss and Lorentz analytical functions. The values of L and G coefficients are chosen from the range 0 – 1;

- upon completion of the selection procedure, click **Apply** (Fig. 7.8).

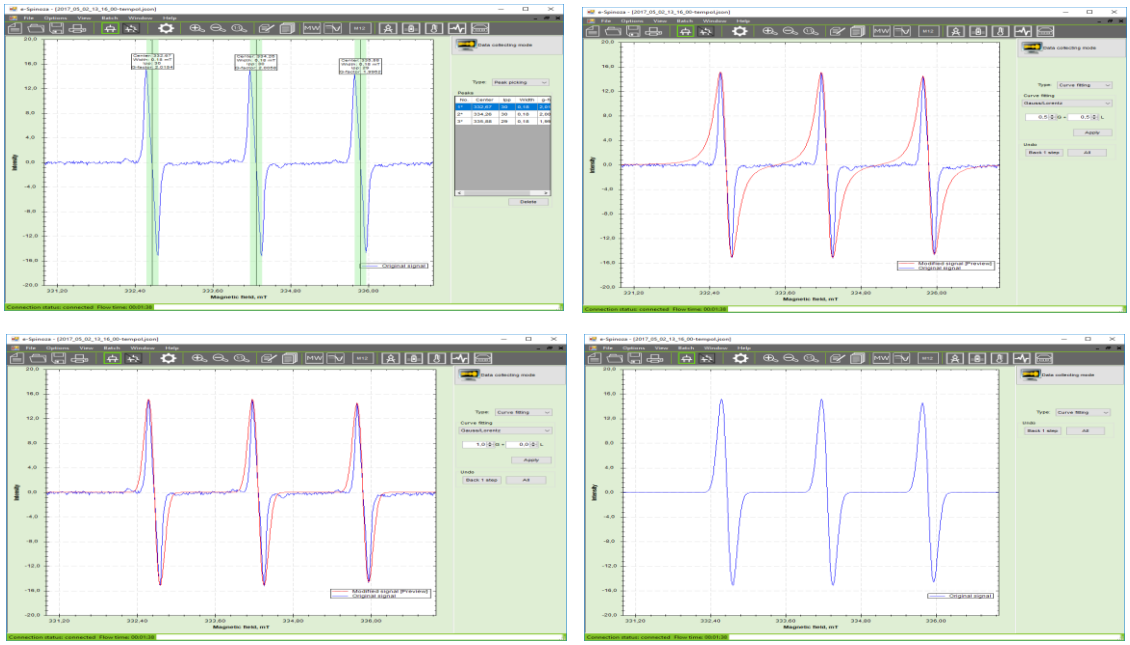


Figure 7.8 – Operation with curve fitting

## 7.9 LINEAR ALGEBRA

To perform the **linear algebra** function to summarize or subtract the spectra follow the instruction:

- for the first spectrum use the current spectrum or open any one you need from the folder;

- select **linear algebra** in the drop-down list, the graph will be divided into two windows and the first spectrum will be displayed in the top window in blue color - **X1** (Fig. 7.9)

- select the second spectrum - press **Open X2**;

- the second spectrum **X2** will be displayed in the lower window in green color, and the upper window will display: the first spectrum **X1** in blue color; the resulting spectrum **X1 + X2** in red color. The summation coefficients are specified in the corresponding input fields X1 and X2. The shift of the X2 spectrum in the magnetic field in mT relative to X1 for the summation operation is set in the Margin window;

- press **Apply** to complete the process.

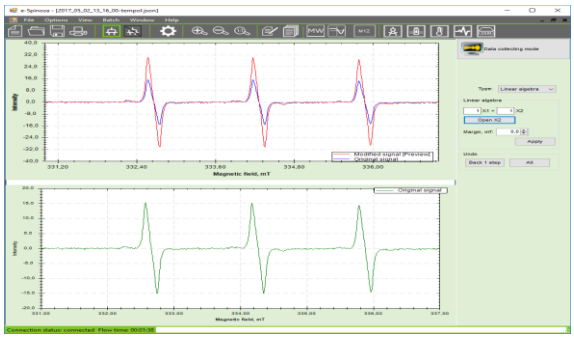


Figure 7.9 – Operation with linear algebra

## 7.10 LINEAR COMBINATION OF MULTIPLE HARMONICS

When recording narrow signals at high modulation amplitude the signal/noise ratio may be enhanced by using the **multiple harmonics** function**.**

Intensity *f* of a magnetic field in a particular point is calculated as:

$$f = \sum_{k=0}^{n-1} a_k \sin\left(\frac{2m\pi}{n}k + \varphi\right), \text{ where}$$

*($a_0$, $a_1$,.., $a_{n-1}$)* – data array coming from the device;

*m* – harmonic number;

*φ* – phase.

At *m=1* the first harmonic is being calculated, at *m=2* – the second one, etc.

To perform the harmonics addition/subtraction:

- record the spectrum in the **ADC data** mode. Select the required mode in **User defaults** tab of the spectrometer settings window (fig. 7.9)

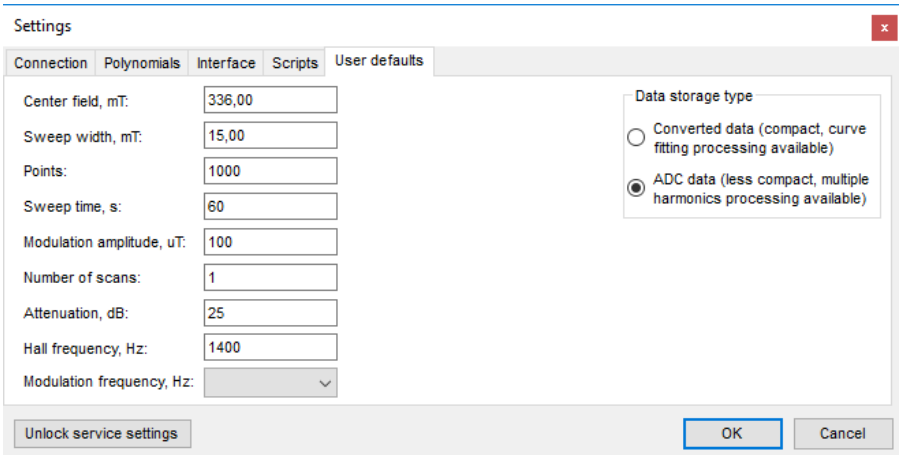
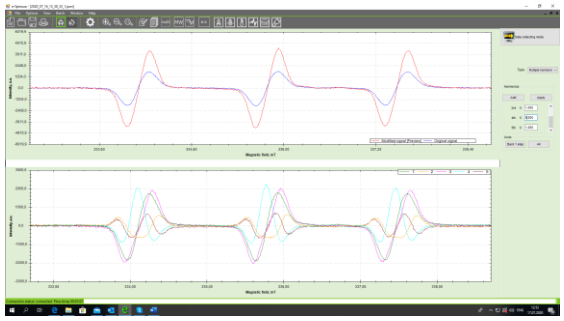



Figure 7.10 – **User defaults** tab.

- Select **multiple harmonics** in **Data processing mode**. The window will be divided into two subwindows, the initial spectrum will be shown in the upper subwindow;

- the **Add** button will add new harmonics;



- the summation coefficients are entered into the corresponding fields either with «+(plus)», or with «- (minus)» signs;

- end the process by clicking **Apply**.

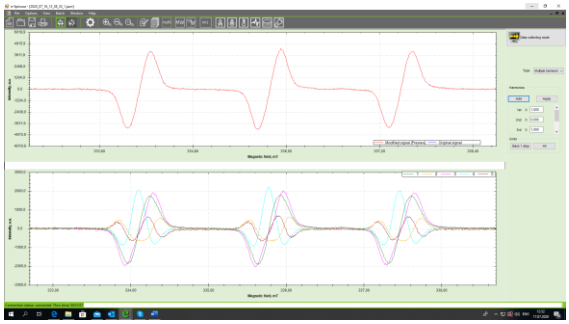


Figure 7.10 demonstrates the effect of multiple harmonics combination. Yellow line is the initial (1st harmonic) spectrum S1, green line is the combination of the 1st, 3rd and 5th harmonics $S_H$=S1+S3+S5. The signal/noise ratio is enhanced 1,5 times compared to the initial spectrum S1.

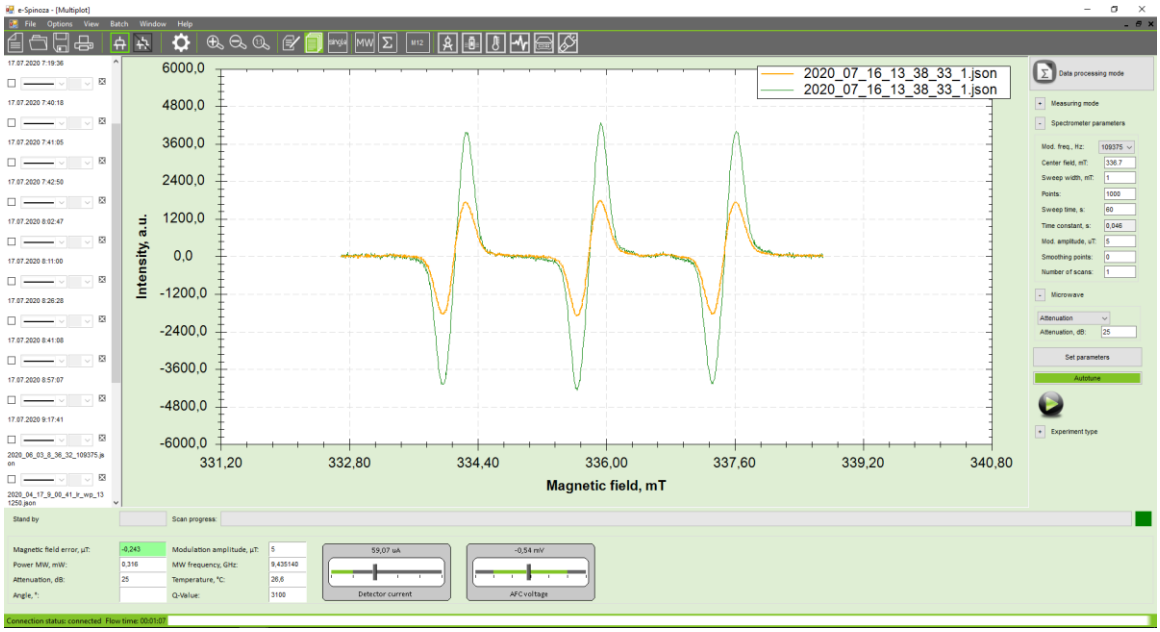



Figure 7.11 – **multiple harmonics** application.

## 7.11 EXTRACTION OF SINGLE SPECTRA FROM AN ARRAY

This function is only possible for 3D experiments.

In order to process a single spectrum from the entire array of spectra recorded in a single 3D experiment, you should use the **Extraction function**:

- use the current 3D spectrum or open any one you need from the folder;
- select the **Extraction** from the drop-down list;
- select the desired spectrum in the panel on the right, for example Scan 1;
- press the **Extract** button and save the selected spectrum

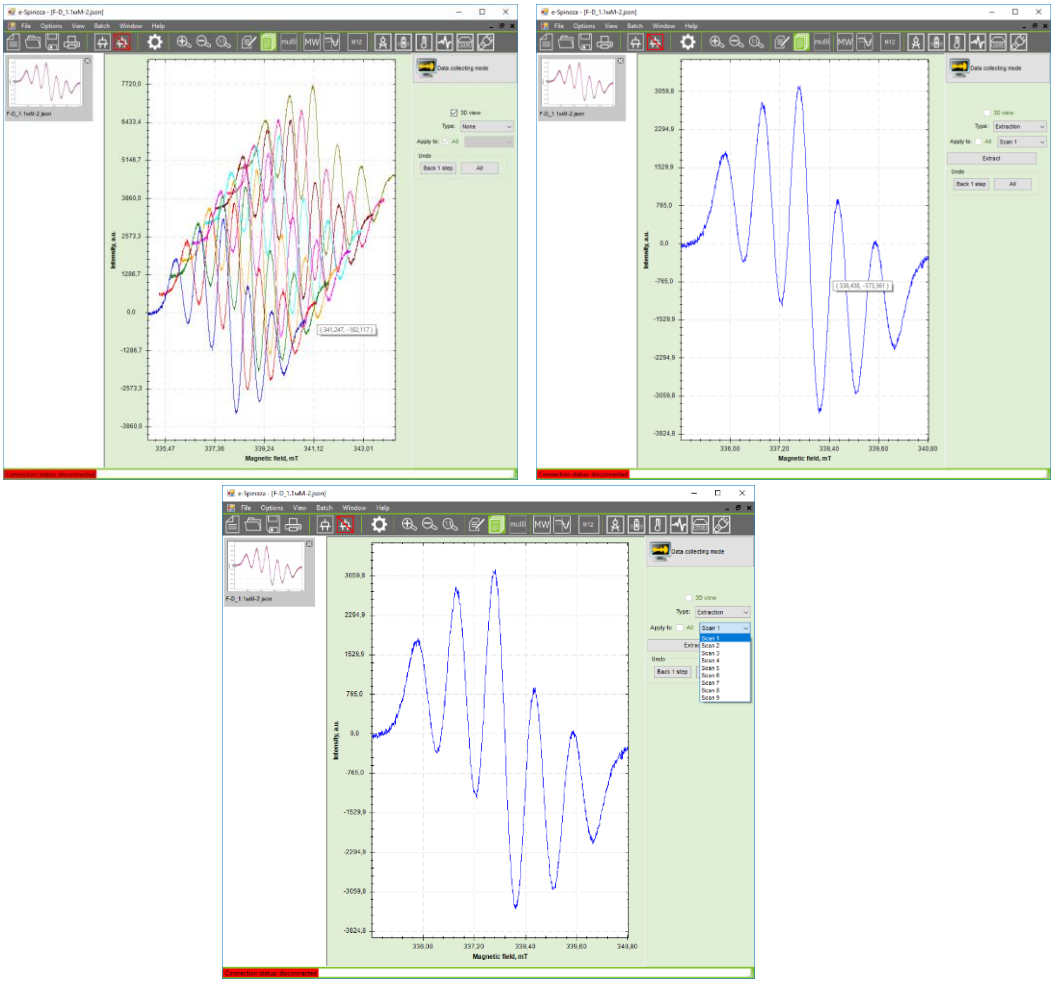


Figure 7.12 – **Extraction** function application

# TROUBLESHOOTING

## SECTION 8

# 8 TROUBLESHOOTING

## 8.1 GENERAL ERRORS

The list below shows some of the problems and errors you may encounter with spectrometer, their description and ways to resolve them.

### 8.1.1 SWITCHING ON

| Error | Description | What to do |
|---|---|---|
| No power | Power cable is faulty or not connected<br>Fuses are absent or blown | Replace the power cable and try again<br>Two supply-line fuses (6.3 A) are located under SPINSCAN power socket |
| No connection with PC | Conflicting IP address on LAN | Contact your local network administrator |

**WARNING** **DO NOT REPLACE THE FUSES MORE THAN ONCE. IF THE FUSES BURN OUT AGAIN, CONTACT THE MANUFACTURER.**

### 8.1.2 MICROWAVE BRIDGE ADJUSTMENT

The microwave bridge adjustment should be performed each time you set the microwave bridge power attenuation or when you use the full auto-tune command.

| Error | Description | What to do |
|---|---|---|
| Unable to tune the frequency | Too much glass in the resonator | Use thinner glass vials or smaller sample volumes |
| Unable to match the resonator | Loose sample (sample with high dielectric losses)<br>This is usually caused by water in the resonator | In case of a solid sample, drying usually helps. In case of a liquid sample, it is preferable to use a non-polar solvent. If water should be used as a solvent, reduce the volume or use a capillary tube (max. 50 µl) |

## 8.1.3 SPECTRUM SCAN

| Error | Description | What to do |
|---|---|---|
| No spectrum<br>(only noise) | Magnetic field sweep amplitude (scanning bandwidth) is too narrow. Field offset is too large | Adjust microwave power or modulation amplitude |
| No spectrum (only a horizontal line at the button or top of the display) | Incorrect sample position.<br>EPR signal high intensity | Adjust microwave power or attenuation |
| The spectrum clips top or button | The gain ratio is too high | If the spectrum clips even with the minimum gain ratio, reduce the microwave power or the modulation amplitude |

## 8.1.4 EVALUATION OF RESULTS

| Error | Description | What to do |
|---|---|---|
| The results are inconsistent – presence of phantom signals | The tube with a sample in the resonator is not positioned correctly (not tightened down properly) | Tighten the sample holder |
| EPR signal intensity is too low | The sample is not positioned correctly | Always make sure that the sample is positioned vertically in the center of the cavity.<br>When loading a new sample, always make sure you have loosened the sample holder. Tighten the holder again as soon as a new position has been set |
| The results are inconsistent – large baseline signal intensity | The resonator protection tube is dirty | Remove the metal part of the sample holder with the protection tube having removed three screws. Clean the protection tube with pure acetone, dry and insert it into the resonator |

# MAINTENANCE

## SECTION 9

# 9 MAINTENANCE

## 9.1 GENERAL

The purpose of this Maintenance (M) is to ensure continuous safe performance of the spectrometer, increase its operability and ensure safety (personal risk).

The following inspection and maintenance procedures, and recommended intervals, are the manufacturer's recommendation regarding the most effective maintenance schedule of the spectrometer.

The service tasks must only be performed by factory-trained technicians.

The maintenance procedure consists of regular periodic maintenance and unplanned repairs in case of malfunctions.

**WARNING**

**THE MANUFACTURER DOES NOT ACCEPT ANY RESPONSIBILITY FOR THE SPECTROMETER THAT HAS NOT BEEN SERVICED AND MAINTAINED IN ACCORDANCE WITH THE MANUFACTURER INSTRUCTIONS, OR WHICH HAVE BEEN AUTONOMOUSLY CHANGED.**

The Maintenance should be provided for each spectrometer since its commissioning. It can be of three types: daily (M1) and periodical (M2, M3).

The first M2 should be performed six (6) months after installation and the subsequent M3 procedures at twelve (12) month intervals.

**CAUTION**

**When any important component is replaced in the spectrometer, perform the corresponding Configuration, Calibration and Acceptance procedures**

### 9.1.1 OPERATOR TASKS

Tasks of the daily maintenance includes the following items (M1):

- Check for external damages;
- Inspect protective housings, fastenings, plugs and power cords and cables;
- Clean exterior surfaces.

**CAUTION**

**Do not try to disassemble and clean any part of the spectrometer when it is activated.**

Do not use cleaning agents or solvents when cleaning the spectrometer surface.

## 9.1.2 SERVICE TASKS

Servicing should be carried out by qualified personnel.

The tasks of M2 should include the following points:

- Visually inspect the spectrometer, remove dust from components and clean them, adjust and restore mechanical connections;
- Check the connections, the insulation of the electric wires, and the ground circuit resistance;
- Calibrate the central value of polarization magnetic field;
- Check the spectrometer sensitivity.

Tasks of M3 shall include the following items:

- Visually inspect the spectrometer, remove dust from components and clean them, adjust and restore mechanical connections;
- Check the connections, the insulation of electric wires and cables, and ground circuit resistance;
- Open housings, inspect the spectrometer PCBs, and clean card connectors with rectified alcohol;
- Check the operation of the spectrometer;
- Calibrate the spectrometer;
- Calibrate the operating modes;
- Check the spectrometer sensitivity.


**When the cover of the spectrometer is removed and the integrity of the power cord is checked, disconnect the device from the mains to avoid the risk of electric shock.**

# TECHNICAL SPECIFICATION

## SECTION 10

# 10 TECHNICAL SPECIFICATION

Main parameters and characteristics of the spectrometer are given in the Table 10.1.

Table 10.1

| Description | Value |
|---|---|
| Sensitivity, spin/T (max.) at least | $2x10^{14}$ |
| Absolute resolution, mT (max.) at least | 0.006 |
| Microwave oscillator working frequency range, GHz | 9.2-9.55 |
| Working frequency relative instability, 1/h (max.) | $1 \cdot 10^{-6}$ |
| Microwave oscillator output power, Mw (min.) | 90 |
| Maximum microwave power attenuation at resonator, dB (min.) | 40 |
| Polarization magnetic field induction central value variation range, T | 0.05-0.65 |
| Polarization magnetic field sweep amplitude assignment relative error, % (max.) | 1.5 |
| Polarization magnetic field induction sweep amplitude variation range, T | $10^{-4}$- 0.65 |
| Minimum sweep time, s (max.) | 1 |
| Polarization magnetic field magnetic induction central value instability, μT/h (max.) | 2 |
| Maximum polarization magnetic field modulation amplitude, mT (min.) | 1 |
| Spectrometric unit overall dimensions, mm (max.)<br>– Length<br>– Width<br>– Height | <br>500<br>400<br>300 |
| Spectrometric unit weight, kg (max.) | 48 |
| Time to set the operating mode, minutes (max.) | 60 |
| Operability after setting the operating mode, hour (min) | 6 |
| Maximum power consumption, VA | 400 |

Minimum technical characteristics of operator's AWS are given in Table 10.2.

Table 10.2

| | |
|---|---|
| CPU | core i3 |
| DDR3 | 1Gb |
| HDD | 160 Gb |
| Display | At least 19" |
| UPS | APC Smart 1000 VA |
| Operating system | Windows 7, 8 or 10 |
| Interface | Ethernet |

**NOTICE**

*The recommended configuration of the operator' AWS is optimal in terms of quality / price. In addition, the configuration can be tailored to better meet the individual needs of the customer.*

**Operating limitations**

It is recommended to place the spectrometer on the table and protect it from draughts, direct sunlight, heat sources, vibrations and power cables.

**Operating conditions**

- Ambient temperature: from plus 10 to plus 35 ºC.
- Relative air humidity: from 45 to 70 % at plus 25 °C.
- Atmospheric pressure: from 84.0 to 106.7 kPa.

# COMPLETENESS

## SECTION 11

# 11 COMPLETENESS

The completeness of the ADN23.00.00.000-06 spectrometer is shown in the table 11.1.

Table 11.1

| Indication | Description | Quantity, pieces |
|---|---|---|
| ADN23.00.00.000 | The EPR spectrometer SPINSCAN X | 1 |
| ADN77.00.00.000 | Automated working station * (system unit, monitor, keyboard, mouse, power supply, software) | 1 |
| – | Set of glass capillaries and cuvette** | 1 |
| ADN23.00.00.000 SP | Set of spare parts, tools and accessories according to the spare parts list | 1 |
| ADN23.00.00.000 OM | Set of operational documentation in accordance with the statement | 1 |
| | Package | 1 |

* Completeness of AWS - in agreement with the customer. The minimum technical characteristics of the arm operator are shown in the table 10.2

** Standard set of glass capillaries and cuvette:

- Quartz tube Hilgenberg GmbH® L 200±1 OD=2,95±0,05 S=0,30±0,05 mm;
- Quartz tube Hilgenberg GmbH® L 200±1 OD=3,95±0,1 S=0,30±0,05 mm;
- Quartz tube Hilgenberg GmbH® L 200±1 OD=4,95±0,1 S=0,38±0,05 mm;
- Capillary Intramark Blaubrand® L=125 mm, 50mcl.

*It is allowed to use the above capillaries and cuvettes of the specified manufacturers, or analogs with the same characteristics*.

# APPENDIX A. ALANINE DOSIMETRY

## A1 PURPOSE

The alanine-EPR dosimetry system provides a means for measuring the absorbed dose. It is based on the measurement of specific stable free radicals in crystalline alanine generated by ionizing radiation. The Alanine free radical gives a characteristic EPR-signal and its intensity is proportional to the absorbed dose, but doesn't depend on power and energy of radiation.

An EPR spectrometer SPINSCAN X can be used as routine dosimetry system for read out of the irradiated dose.

## A2 COMPONENTS AND DELIVERY SET

The alanine-EPR dosimetry system consist of:

I. Electron Paramagnetic Resonance (EPR) Spectrometer model SPINSCAN;

II. Software package e-Spinoza v X. (including dosimetry software for read out of dose (for pellet and strips);

III. Temperature controller (optionally);

IV. Desk ( or machine support);

V. Analytical balance (210 g capacity, 0.1mg readability with automatic internal calibration function);

VI. Uninterruptible power supply (UPS);

VII. Bar code reader (available on request);

VIII. Work Operating Station (WOP) - Personal computer with min. configuration CPU Core i3, DDR3 1Gb, HDD 160Gb, 19”, Wind. 7/8/10, Ethernet, Wi-Fi, USB.

IX. Calibration set (N doses x 3 pellets);

X. Alanine dosimeters pellets and tape tabs (the quantity on request);

XI. Dosimetry package accessories:

- Alanine pellet holders (quartz tubes) ();
- Alanine films (strips) holders (quartz tubes) (the quantity on request on request);
- EPR sample holder tubes for alanine pellet dosimeters (the quantity on request);
- EPR sample holder tubes for alanine strip (the quantity on request on request);
- Multipurpose EPR sample holder tubes for different type of alanine dosimeters the quantity on request

XII. EPR intensity reference material.

# A3 TECHNICAL SPECIFICATIONS

**Alanine dosimeters pellets and tape tabs** (*the spec depends on producer):

| | |
|---|---|
| Batch number of dosimeter | as specified on the package |
| Dosimeter mass | 66 ± 0.5 mg; ± 0.1 mg ( may vary) |
| Dosimeter diameter; height | 4.80 ± 0.1 mm, 3.00 ± 0.3 mm |
| Mass ratio alanine/binder | 0.96/0.04 (may vary) |
| Permissible temperature range | -10°C up to 80°C linear relationship |
| Batch homogeneity of ESR signal | < ± 1 % (k=2), (mass corrected) |
| Temperature coefficient | 0.001 ± 0.0002 K-1 (-10°C up to 80°C) |
| Accuracy of dose measurement | typically ± 3 %, depending on calibration ESR equipment and measuring parameters |
| Foil dimensions ( For tape tabs) | 150 mm x 9 mm (may vary) |
| Carrier material For tape tabs | PET (may vary) |

## Calibration dosimeters set

Dose range: from 0.5 Gy to 200 kGy

Dose levels: For a dose range of less than one decade, at least four dose values forming an arithmetic progression are used. For a dose range of more than one decade, at least 5 dose values per decade are used, so that they form a geometric progression (ISO/ASTM51900).

A set of dosimeters includes 3 to 4 dosimeters for each dose.

The calibration dosimeters set supplied by manufacturer in accordance with dosimetry standard (the doses range and dose levels can vary depends on irradiation condition of User facility) and should be performed at an accredited international or national calibration laboratory and confirmed by the certificate of calibration of the irradiation laboratory.

The parameters for irradiations conditions of calibration dosimeters (the source type, temperature, irradiation ratio etc.) should be agreed with the User before to perform the irradiation of calibration set.

## Analytical balance

| | |
|---|---|
| Ambient temperature: | 10°C to 30°C |
| Relative humidity | 15 % to 80 % at 31°C non-condensing, decreasing linearly to 50% at 40°C |
| Protection: | Protected against dust and water |
| Pollution degree | 2 |

| | |
|---|---|
| Installation category | Class II |
| Readability (g) | 0.0001 |
| Repeatability (sd) | 0.0001 |
| Linearity (g) | ±0.0003 |
| Stabilization | 3s |

**The SPINSCAN X DOSIMETRY SYSTEM** meets following specifications:

- operates at a microwave frequency of 9.2 to 9.55 GHz;
- has automatic frequency locking;
- Microwave power to 150 mW (leveled);
- field scan range up to 650 mT;
- a magnetic field modulation amplitude - 0.1 to 1 mT at 100 kHz frequency modulation;
- Adjustable sweep time constant, and AUTO gain according to absorbed dose;
- Experimental parameters set up and can be controlled in real time;
- The sensitivity - $2x10^{14}$ spins for EPR line width of 0.1 mT;

## A4 MEASURING AND ANALYSIS

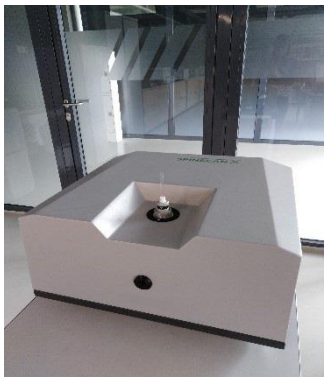
### SAMPLE PREPARATION.

For the alanine dosimetry the precise height adjustment of the sample is a critical point as the dosimeter must be in the middle of active area of the resonator. Using the alanine dosimeters holder follow the next rule: the distance between the edge of the metal sample holder and the center of dosimeter should be 69 mm. The alanine dosimeter should be inserted into the sample tube as shown on Figure. When installing the holder in the resonator, make sure that its installation is always the same: the mark in the inscription on the holder must face the operator

**NOTICE**

*In a case you need to adjust the height loose off the Teflon screws from both sides of metal holder and move the sample tube along the holder on proper height.*





AMBIENT CONDITIONS:

The temperature into resonator can effect on the measurement results. It may change the amplitude of signal and the peak of its position as well. The alanine dosimetry measurements require stable conditions so we advise to follow the next recommendations:

1. Adjust the ambient temperature in the range T=25±2℃ and control it during the day;

2. Warm-up the spectrometer after first initialization up to 2 hours until the temp of cavity will stabilized;

3. Use the temperature control system (optionally) (read the Appendix B of user manual for its installation); for alanine dosimetry we recommend to adjust the temp 28±0.5℃;

4. Use the water cooling (optionally) (read the Appendix C how to install it).

**NOTICE**

a) Switching on the power supply will NOT start the warm-up. Do CONNECT and SEND PARAMETERS to set up the connection and start to warm up the instrument. Use the special parameters for a fast heat-up of the system.

The initialization status: green is connected; red - disconnected.

b) Switch off the instrument only if you do not do the measurements for a long time.

## A5 USING THE SOFTWARE

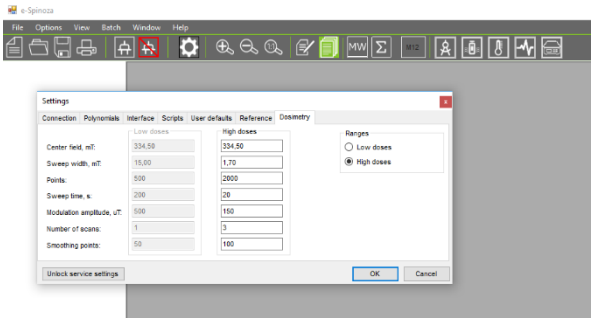
### BEFORE TO USE

Before to USE the DOSIMETRY apps of e-SPINOZA software package you need to add into the interface of the main window the buttons what will run automatically every type of experiment **Verification CC, Calibration, Dosimetry, Irradiation**. For every type of experiment

is the signed the script what supplied with package of software. You may learn how to do this in the Part 3 of Operation Manual.

After you will modify the interface for dosimetry on the right side of setting panel in **Experiment type** section you will see new buttons - **Verification CC, Calibration, Dosimetry, Irradiation**.

In the settings window, select the dose range in which the dosimetric system will work. By default, the system uses the settings for **High doses** mode. Select the dose range **Low doses**, if necessary, change the operating mode.




If the computer is supplied with spectrometer, the manufacturer will develop the interface before the shipment.

**NOTICE**

*Verification CC, calibration and measurements must be carried out with the same tube in the holder. When changing it, you need to repeat these processes.*

## A6 CALIBRATION CURVE VERIFICATION TEST

The calibration curve applied to the spectrometer received from the supplier should be considered only as general (preliminary) information and should not be used without testing its applicability.

Prior to use of a calibration curve, a calibration verification should be performed to assess the suitability of the calibration curve for the conditions of use.

The verification of calibration curve should be conducted at least at one dose level from the calibration dose range. It may be dose in the middle range or a frequent dose.

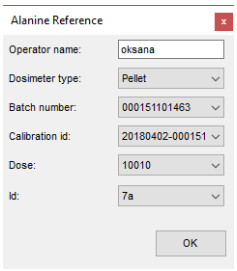
The routine dosimeter for the calibration verification should be selected from the same dosimeter batch as the calibration dosimeters. Verification test should be carry out before routine measurements every day and during the day when temperature of the resonator is changed to 1℃.

**NOTICE** *The work with alanine dosimeters is carried out only with gloves*

1. Press the button **Verification CC** to carry out the calibration curve verification;

2. Fill in the dialogue box, click **OK**, and the measurement will begin;

**Dosimeter type** - select the type of alanine dosimeter (pellet or tape tabs);




**Batch number** - select a unique (batch) number of the dosimeter from the list provided;

**Callibration ID** – id of used calibration;

**Dose** - choose the appropriate dose from the list

**Id** – Id of current dosimeter (given by user);

3. Insert the sample and press **OK**, then the measurement will start;

3. Upon completion of the measurement, the message appears on the screen **"Reference test completed successfully. Ratio: N."** The results are saved in the data base **View -> Dosimetry -> tab Verification**.

If the measured dose is over the tolerance, the corresponding message will appear on the screen. Please call the service office or the manufacturer's service center to fix the problem.

**NOTICE** *Monitor the resonator temperature throughout the day. It is recommended that when the resonator temperature changes by more than 1 °C, repeat the verification test.*

4. Remove the reference sample from the resonator and place it in the appropriate storage cell.

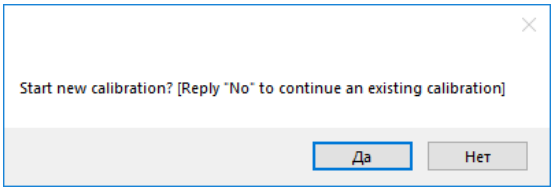
**NOTICE** *When operating the spectrometer, the holder for dosimeters should always be in the resonator of the device, and only when changing the sample under study does it get from the resonator.*

## A7 CALIBRATION

The Alanine EPR dosimetry system calibration should be performed by manufacturer before shipment and verified during installation. However in some cases the User can do it by yourself with calibration dosimeters set supplied by manufacturer in accordance with dosimetry standard (the calibration set should be performed at an accredited international or national calibration laboratory) or with new dosimeter batch.

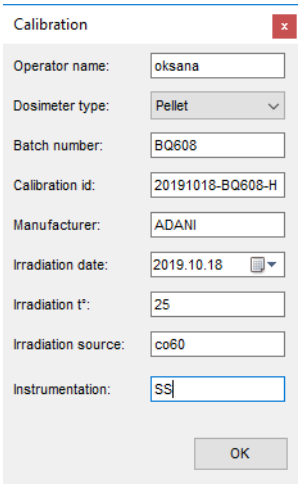
To perform **Calibration procedure** follow the next instruction:

1. Press the **Calibration** buttons to run the Calibration procedure and follow the instruction with dialog windows what will appear on the screen.



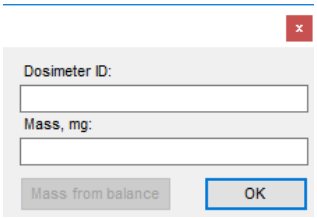

2. First fill in the **Mass** line. Remove the pill from the weighing pan and place it in the resonator. Continue filling the dialog box and press **Ok**;

3. The system start to generate the calibration file with unique ID.




**Operator name** – the authorized name of User who is performing the system calibration
**Dosimeter type**- choose one (pellet or tape tabs)
**Batch number**- insert the in unique number of batch of dosimeters
**Calibration ID** – the system generates the unique ID of calibration curve what will be saved in the data base (DB).
**Manufacturer**- the name of manufacturer of dosimeters
**Irradiation date**- the date of origin
**Irradiation temp** – the temperature at irradiation
**Irradiation source**- the type of irradiation source
**Instrumentation**- the name of instrumentation ( model)

4. Follow the instruction and start to measure the calibrated dosimeters one by one;




5. Insert the next sample, fill the requested fields and press **OK**. The program will run an experiment;

6. Repeat the procedure as much as needed.

7. While working in the **low doses range (up to 1 kGy)** the dose measurement starts either with new registration of unirradiated pellet and verification pellet spectra, or with already registered spectra. The verification pellet should correspond to the selected dose range.

8. After the measurement of every dosimeter of the calibration set you will see on screen the table with the results (mass, weight, intensity). The program ask you to apply the data and continue the measurement. Press **OK** if you agree to apply the results. If you discover that the data are failed and over the range you can answer No and repeat the measurement one more time.

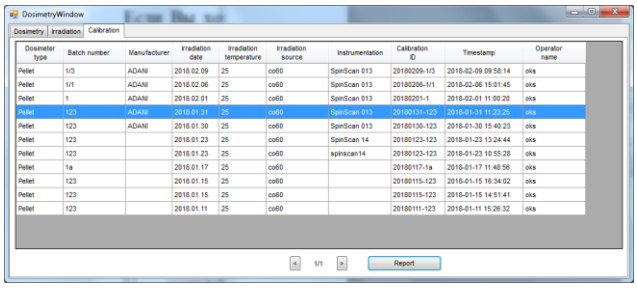
Choose **YES** to continue the measurements of the next dosimeter when the system ask you to continue. If you want to finish press **NO** and the calibration procedure will be completed. You will see the sign **“Calibration is complete”**.

Press **ok** and the **Calibration editor module** will show up in the window. At the same time the file with calibration data will add to Data Base Calibration (BDC).

You can find it here: **View -> Dosimetry -> tab Calibration**. The file has ID and save the information about the batch number, temperature, means od calibrated dose etc.

The rule for the calibration ID generation as follow - | irr date|_ |batch #|

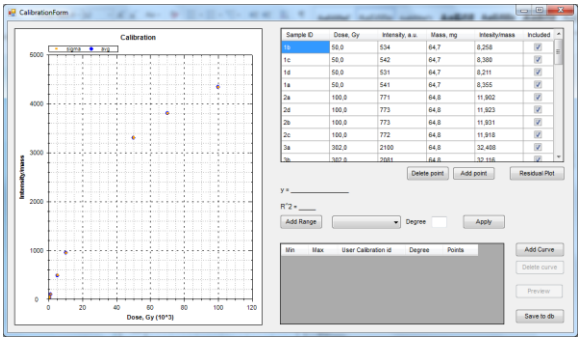
You can close the window of **Calibration editor** if you do not want to use it. How to work with Editor module you can find in the Editor Module description of this Manual.

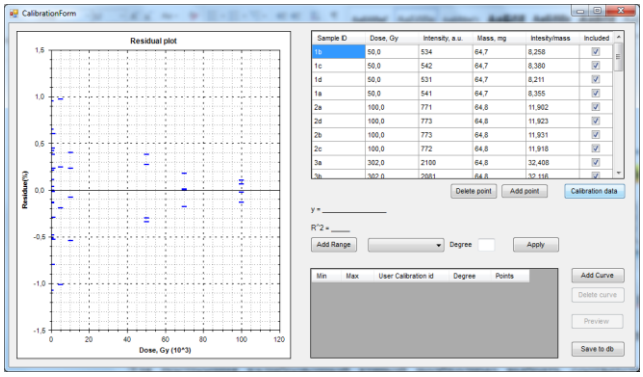

## A8 CALIBRATION EDITOR MODULE

The Calibration editor module allow to modify and create the list of calibration curves based on the original one. You can open it as follow: **View -> Dosimetry -> tab Calibration-> ->** select the original ID file and press **Report.**

Calibration editor will be opened in new window. On the graph window and on the right there is the table with the data of measurements. You can switch the graph window with calibration curve on the residual plot window or switch back**.**

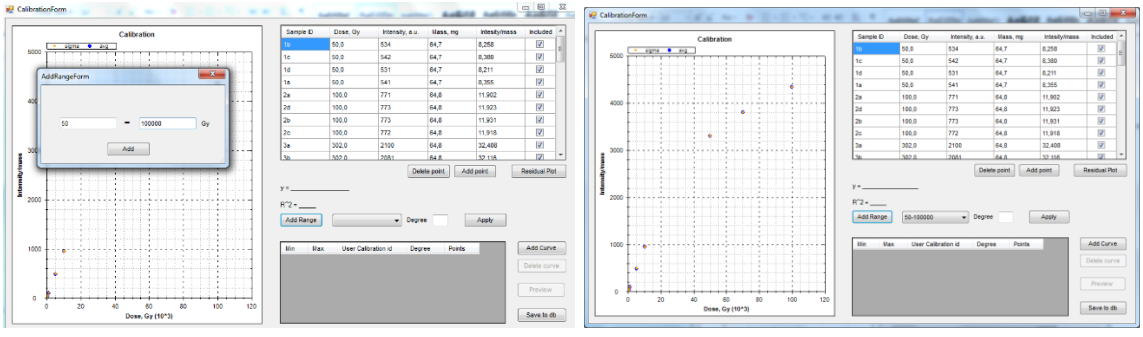


In the mode **Calibration curve** the graph intensity/mass vs dose is shown. (blue points – average means, yellow points – the original means).



1. To build the calibration curve in chosen dose range press **Add Range**, choose the dose range what you wish to use ( what is more typical for your dose range irradiation). The chosen range will appear in the window;

| NOTICE | *At the beginning, you should generate the calibration curve for whole dose range to assure that all measurements have been done correct.* |
|---|---|

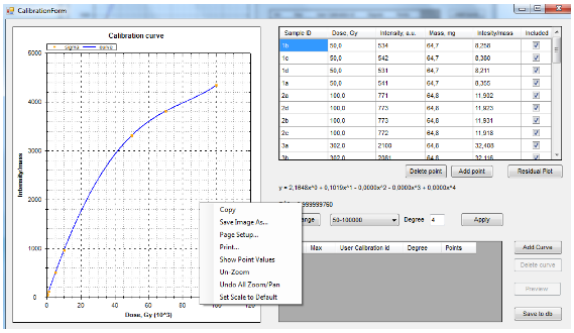


2. Choose the best degree for analytical equation for fitting.

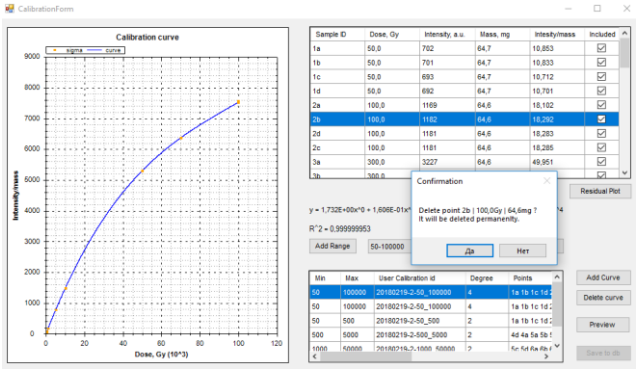
3. It is possible to remove any failed point from the list of measured doses. You need to unmark the box in front of point (column **Included**). After that press **Apply** and the calibration

curve in the selected range and built with the selected pointed in the window **Calibration curve** will be created;

With right click of mouth you can activate menu to work with the graph window.



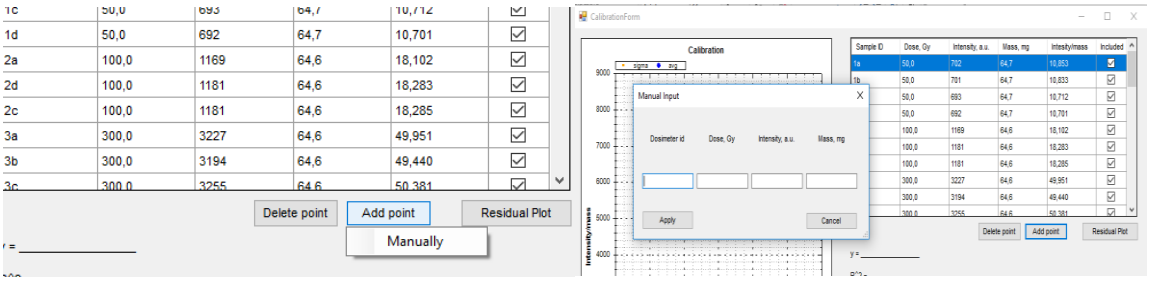
When **Delete point** is pressed the measured dose data is irreversibly deleted from the table. Choose the corresponding row in the calibration data table, press **Delete point** and confirm the deletion.



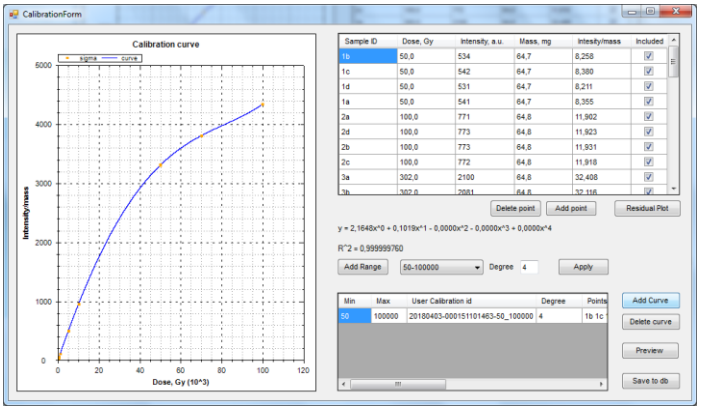
Additional calibration points can be included into the calibration data table using the **Add point** button:

- Press the **Add point** button, choose **Manually**;
- Fill the empty fields in the dialog window to characterize the calibration point;
- Press **Apply**.

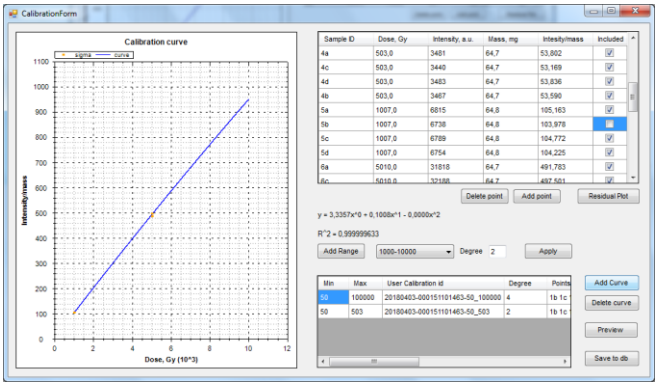
The newly added calibration point will appear in the calibration data table and the calibration curve will be rebuilt.



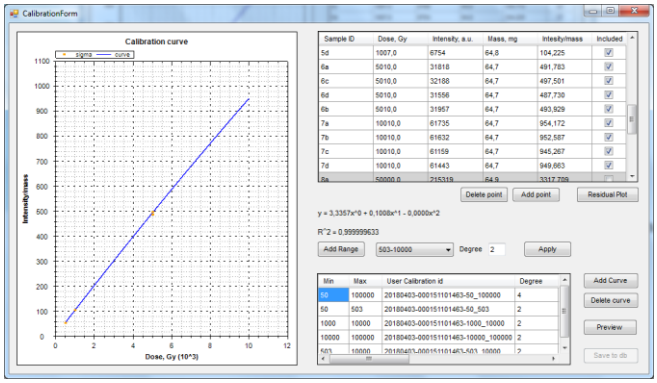

4. Press **Add Curve** and the calibration curve for specifies dose rage will add to the list;




5. The User can modify and build the list of the calibration curves in the dose ranges what is more typical for routine irradiation practice by repeating the items 1-5;




6. After you finish with the list of calibration curves – press **Save to db** and this list will be add to the archiving Data base of calibration curves under ID calibration;




7. Close it or minimize a window and go to main window of *e-Spinoza*.

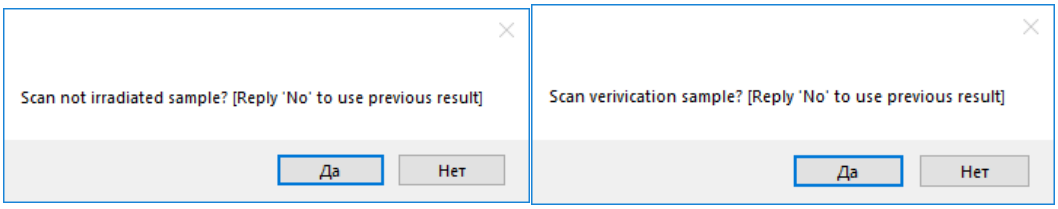
## A9 DOSIMETRY

For routine absorbed dose measurements follow the next algorithm:

1.Select the sample to be tested and place it on the scale (working with alanine dosimeters is carried out only with gloves);

2. Click the **Dosimetry** button and follow the online instructions;

3. While working in the low doses range (up to 1 kGy) the dose measurement starts either with new registration of unirradiated pellet and verification pellet spectra, or with already registered spectra. The verification pellet should correspond to the selected dose range.




4. Fill in the sample data with the free fields in the drop-down dialog box and click **ok**.

Follow the interactive instruction.

First fill in the **Mass** line. Remove the pill from the weighing pan and place it in the resonator. Continue filling the dialog box:

**Operator name –** the authorized name of User who is performing the current test;

**Dosimeter type**- choose one (pellet or tape tabs);

**Batch number**- choose the in unique number of batch of dosimeters from the list;

**Calibration id** - id of used calibration;

**Calibration range** – choose the range for the specific doses range of calibration curve saved in database (DB).

**Dosimeter id**- ID of current dosimeter (given by user);

**Irradiation date**- the date of dosimeter irradiation;

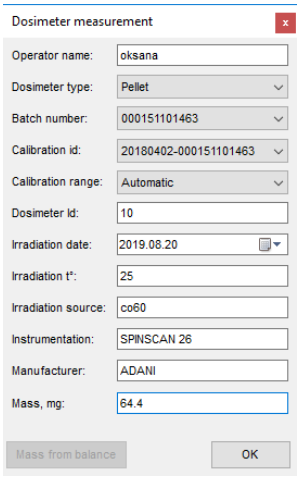
**Irradiation t◦** – the temperature at dosimeter irradiation;

**Irradiation source**- the type of irradiation source;

**Instrumentation**- the name of instrumentation (model);

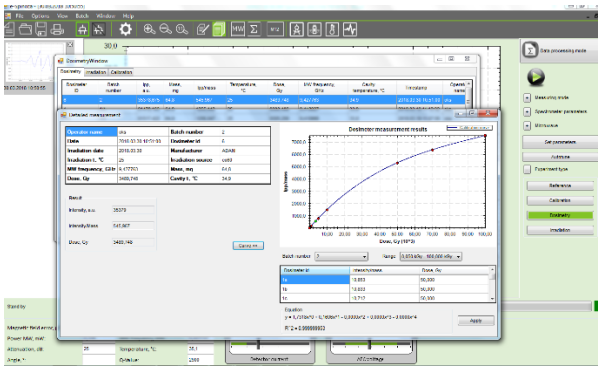
**Manufacturer**- the name of manufacturer;

**Mass**- weight of dosimeter from balance or manually;




5. Upon completion of the measurement, the summary on the completed experiment will appear (intensity, dose, weight). Click **Ok**. The results are saved in the data base **View -> Dosimetry -> tab Dosimetry**.

6. Choose the line with dosimeter ID and press **Report.** Press the button **Export** to create the measurement report in pdf format, save or print it.

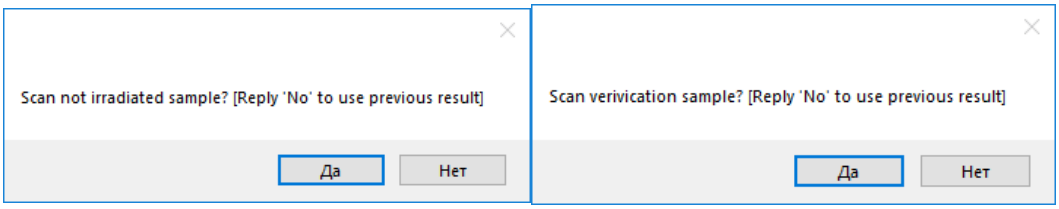


## A10 IRRADIATION

For dose mapping of absorbed dose follow the next algorithm:

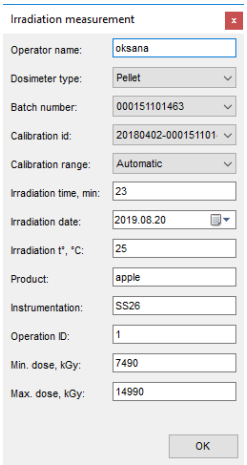
1. Press **Irradiation**. Follow the interactive instruction.
2. While working in the low doses range (up to 1 kGy) the dose measurement starts either with new registration of unirradiated pellet and verification pellet spectra, or

with already registered spectra. The verification pellet should correspond to the selected dose range.




3. Fill the requested fields about the sample:

**Operator name** – the authorized name of User who is performing the test;




**Dosimeter type** - choose one (pellet or tape tabs);

**Batch number** - choose the unique number of batch of dosimeters from the available list;

**Calibration id** - id of used calibration;

**Calibration range** – choose the range for the specific doses range of calibration curve saved in database (DB).

**Irradiation time** - time of irradiation of dosimeter;

**Irradiation date** - the date of dosimeter irradiation;

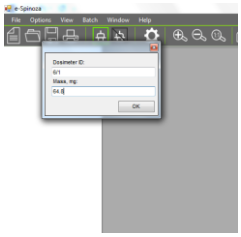
**Irradiation t◦** – the temperature at irradiation

**Product** - the name of irradiation subject;

**Operator ID**- the unique number of dosimeter position on irradiation setup (given by user);

**Min.dose and max.dose** - the dose range of irradiation;

4. Insert the sample and fill the information about the dosimeter;



5. Press **OK** and it runs the measurement;

6. Repeat the steps 3-4 for next dosimeters;

7. The results are saved in the data base **View -> Dosimetry -> tab Irradiation**;

8. To see the full report, select the line with necessary **operator id** and press **Report**. Press the button **Export** to create the measurement report in pdf format, save or print it.

**REPORT in PDF**

From DB you can generate the report and export it in PDF, Excel or ASCII file and then print it.

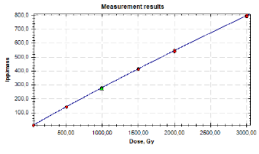
## Dosimetry report

19.10.2018 13:45

Spectrometer: SpinscanX-013
Operator name: oksana

Dosimeter type: Pellet
Batch number: 1
Dosimeter ID: 1.0
Irradiation t°, °C: 25
Irradiation date: 2018.07.20

Measurement date: 2018.07.20 17:00:35
Calibration curve ID: 20180720-1-506_3000




| ID | Intensity | Int/mass | Dose, Gy |
|---|---|---|---|
| 1a | 702 | 10,853 | 50,000 |
| 1 | 9235 | 142,735 | 506,000 |
| 1/2 | 9247 | 142,927 | 506,000 |
| 1/3 | 9229 | 142,641 | 506,000 |
| 2/1 | 17758 | 272,783 | 992,000 |
| 2/2 | 17818 | 273,818 | 992,000 |
| 2/3 | 18117 | 280,453 | 992,000 |
| 3/1 | 26628 | 412,840 | 1498,000 |
| 3/2 | 26619 | 412,698 | 1498,000 |
| 3/3 | 26931 | 417,527 | 1498,000 |
| 4/1 | 34755 | 540,514 | 2000,000 |
| 4/3 | 35302 | 549,023 | 2000,000 |
| 5/1 | 51302 | 791,690 | 3000,000 |
| 5/2 | 51935 | 801,466 | 3000,000 |
| 5/3 | 51605 | 796,366 | 3000,000 |

Δ - measured point(s)
o - calibration points

y = - 3,606E+00x^0 + 2,932E-01x^1 - 1,074E-05x^2 +
+ 6,323E-10x^3
R^2 = 0,999995241

| Mass, mg | Intensity, a.u. | Intensity/mass | Calc. dose, Gy |
|---|---|---|---|
| 64,6 | 17908 | 277,221 | 991,604 |

# APPENDIX B. TEMPERATURE CONTROLLER OPERATION

## B1 ASSEMBLY AND INSTALLATION

(The temperature control system TC-01L)

The temperature control system is used to maintain the constant value of the sample temperature inside the resonator in the temp range -170 … + 180 °C.

| | |
|---|---|
| Gas carrier | gas $N_2$ |
| Consumption, l/min | 30 |
| Sample tube diameter, mm | 4 |
| Controlled temperature range ºC | -170….+180 |
| Temp setting accuracy, ºC | ±0,5 |
| Temperature meter | Thermocouple type K |
| Time to target temperature, min | Up to 3 |
| Heater power, W | 80 |
| Dimensions:<br>Control unit, mm<br>Desk, mm | <br>160 x 175 x 295<br>710 x 520 x 890 |

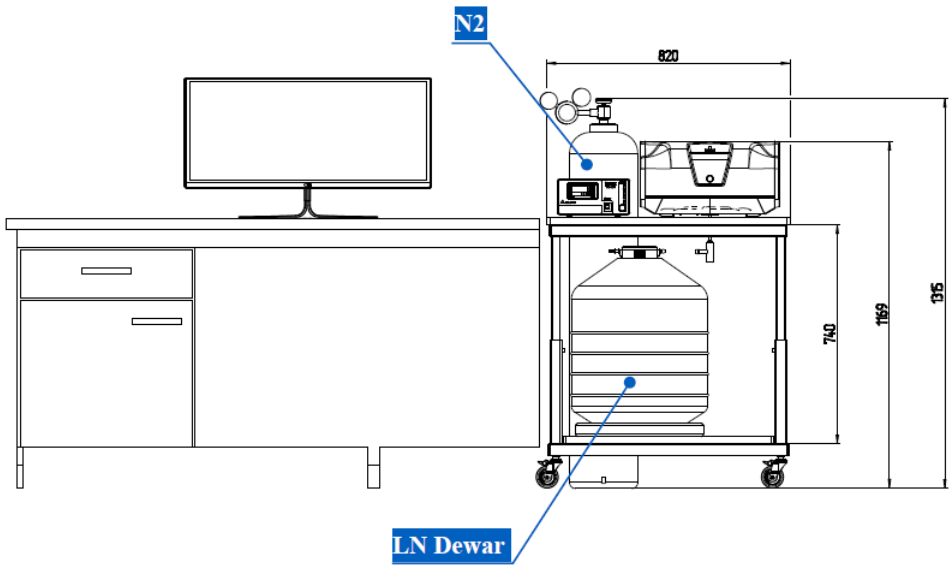



Figure 1 – General view of the fully assembled system

1. Assembly the desk (attach the wills to the bottom of the desk);

2. Place the spectrometer on the desk. The spectrometer sample opening should be adjusted in such a way to be in the middle of the countertop hole position. Fig 2.

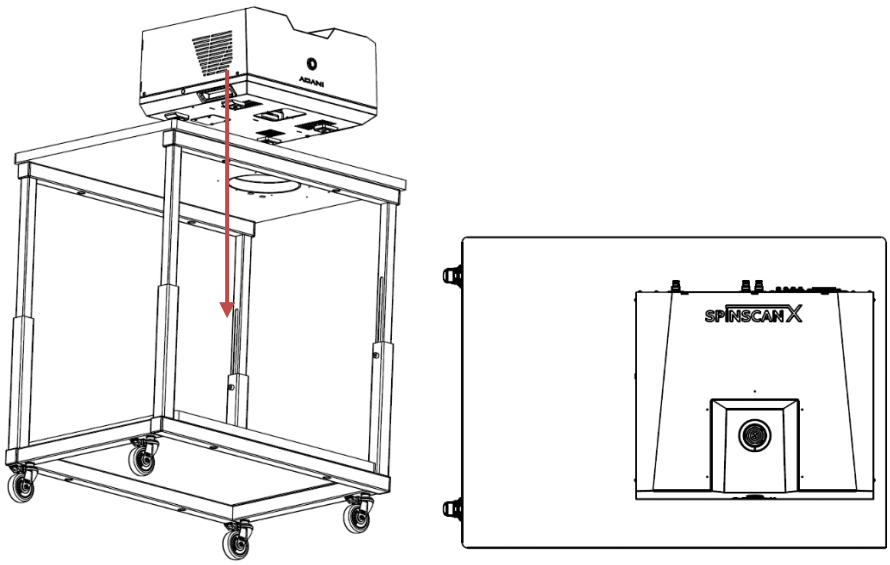



Figure 2 – Spectrometer positioning



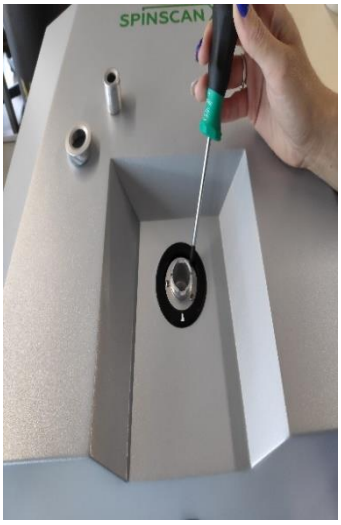
1- heater switch;
2 - flow meter;
3 - temperature control;

1 - Heater connector;
2 – I/O power switch;
3 - Power socket;
4 - Thermocouple connector;
5 - USB connector;
6 - Nitrogen input Hose2;
7 - Nitrogen output Hose1.

Figure 3 – Temperature controller

3. Connect the following wires to the spectrometer: power cable, Ethernet cable. Connect the TC-01L thermocontroller to a PC using a USB Type-A - USB Type-B cable (included in the kit);

4. Unscrew 3 screws of the holder and remove the holder with protective tube as shown below:




5. Detach the protective quartz tube of the resonator;

6. Place the holder back without the quartz tube;

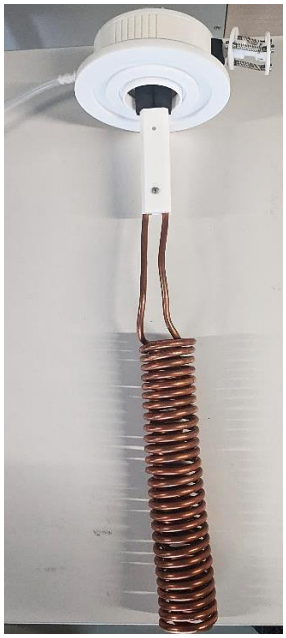
7. Insert into the hole from beneath the desk the VT Quartz Dewar insert and fix it using the Teflon holder of ID 10 mm (similar like the sample tube is fixed). Please mind the black line on the Dewar Insert external wall – this line indicates the proper positioning of the Dewar Insert inside the spectrometer. The black mark should be on the same level as the top metal screw fixing the Dewar insert.

8. Connect the cables and hoses to the controller as shown on photo:


9. Install the gas pressure regulator on the $N_2$ balloon;

10. Connect the gas regulator and the controller with the hose ($N_2$ input);

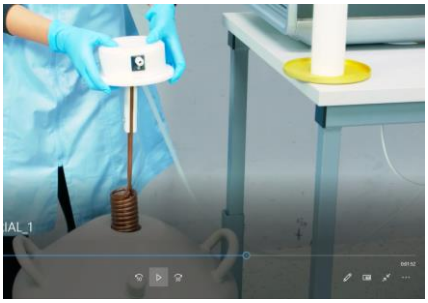
11. Connect the $N_2$ output of the controller to the input of the cooling unit with the hose and install the gas connector on the output of the cooling unit as shown on photo:



12. Before proceeding further make sure that the **HEATER** mode is **OFF** and all the wires and hoses are connected as described above;

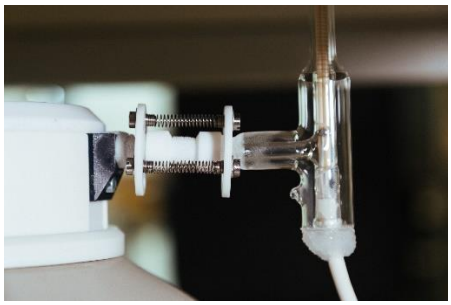
13. Enable the nitrogen gas to be supplied to the system. Adjust the gas flow to 2-4 l/min;

14. Immerse the cooling unit carefully and slowly into the 25L Dewar while keeping the nitrogen gas flow through the cooling unit; **Be careful and use protective tools to avoid contact with liquid nitrogen;**



15. After inserting the cooling unit into the Dewar place it on the bottom shelf of the desk under the spectrometer. Move the Dewar carefully to bring the connector to the quartz Dewar insert;

16. Be **VERY CAREFUL** while connecting the cooling unit to the quartz Dewar insert with the connector;



17. Set the nitrogen flow rate 10-15 l/min using the flow meter on the front panel of the temperature control unit. For the temperature range -130…+20°C the nitrogen flow rate is enough. In a case of temp -170…-130°C set up the flow rate 20-25 l/min;


18. Switch on the control unit using the I/O button on the rear panel; the current and the set temperature values will be displayed;

19. Switch ON the **HEATER**; Wait until the temperature reaches the set value;

**CAUTION** **Check the gas flow rate. Do not use the HEATER if the gas is not supplied to the system!**

20. Set the temperature using the "▲" and "▼" buttons located on the front panel of the control unit, and press the **PROG** button 5 times. The set and current temperatures are displayed by green and red LED indicators, respectively.


25. The Controller can be tuned either manually (on the TCS instrument panel) or using the PC software for faster and more accurate temperature control system operation. This procedure is performed when the temperature control system is first started, or when the operating modes are changed (transition from one mode to another). Autotuning is performed under the experimental conditions and at the average temperature of the experiment.

To start the autotune of PID coefficients from the thermocontroller panel, please do the following:

- press the **PROG** button on the display;

- the inscription **r-S** (controller start / stop) will appear;

- press the **PROG** button again to switch to the **At** mode - start / stop autotune;

- use the arrow buttons to select the **rUn** mode - start autotune.

The **AH** indicator lights on during this procedure. When it finishes, the indicator turns off, which indicates the Autotune completed successfully.

It is easier to carry out the Autotune using the application for controlling the thermal controller of the e-Spinoza software, therefore we recommend opening the program and performing autotune in accordance with the instructions below.

## B2 CONTROL WITH e-SPINOZA SOFTWARE PACKAGE

For different types of temperature control systems, different temperature controller calibration coefficients are used. Changes to the operating modes of the temperature control system, as well as the control of the temperature controller, can be performed using the e-Spinoza software;

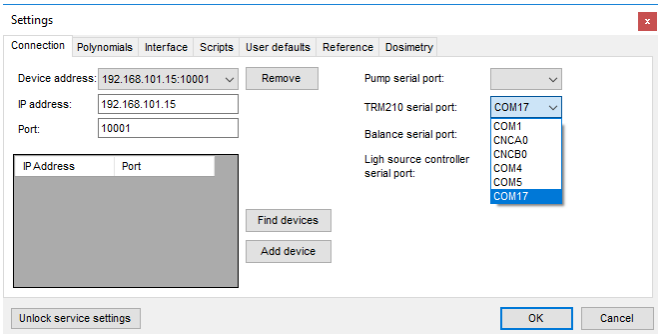
1. At first it is necessary to install the PL2303_Prolific thermal controller device driver, which is located on the flash drive in the installation package in the Prolific folder (or is already saved on the computer when the software is installed at the factory);

2. Open the command execution window by pressing **Win + R**, enter the command **devmgmt.msc** and press **ENTER**;

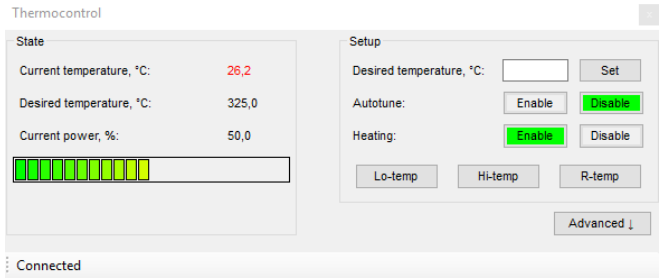
3. Select **Ports (COM and LPT)** in the device manager on the PC;

4. Define the **COM port number** of the Prolific USB-to-Serial Comm Port from the list provided. This COM port will be used further when installing the device;

5. In the e-Spinoza interface click the **Settings** button  and set the previously defined COM port for **TRM210 serial port**. If the driver USB – COM is installed correctly you will see the new COM port in the ports list. Close the window;



6. Press the icon **Thermocontrol** to open the window for control unit;




**State** – The current status of the controller is displayed: current temp and Desired temp;

**Setup** – Set up the desired temperature of experiment;

**Autotune** – Automatically adjust the parameters of the control unit.

It is recommended that autotune be performed under experimental conditions and under the temperature set up for the experiment.

**Heating** – switch on (Enable) and switch off (Disable) the heater. It operates the same like the HEATER switch on the front panel of the control unit.

For correct operation both buttons (in the software interface and on the panel) should be ON.

7. To activate the type of the temperature control system press the appropriate button (**Lo-Temp** - low-temperature thermo-system, **Hi-temp** - high-temperature thermo-system or **R-temp** - biological thermo-system). The button will be highlighted in green;

9. Set the value of the desired temperature in the **Desired temperature** window and press **Set**;

10. Press **Enable** to turn the heater on and **Disable** to turn it off. Please note that the **Enable / Disable** buttons for Heating are active only when the **HEATER** switch on the front panel of the thermocontroller is on.

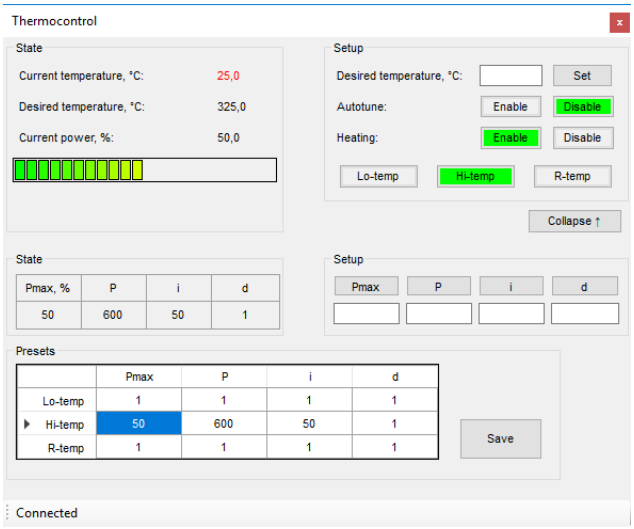
The assembled temperature control system will operate with the PID coefficients set by the manufacturer (the Thermocontrol_coef.txt file with the coefficients for the thermocontroller is located on the flash drive in the installation package).

If necessary, these coefficients can be changed automatically or manually.

1. To autotune the PID coefficients of the temperature controller, in the **Setup** sub-window in the **Autotune** section, press **Enable**. In this case, the **Enable** button will light up green. At the end of this procedure, the **Disable** button will turn green.

The resulting new PID coefficients are not automatically saved in the **Presets** table. In order for the temperature control system to operate in the new mode in the future, transfer the obtained values of the PID coefficients manually to the corresponding windows and click **Save**;

2. Press **Advanced** to manually set the controller calibration coefficients;




3. Correct the PID gain values manually and click **Save**;

4. The lower section **State** displays the coefficients of the currently active system;

5. Section **Setup_Service** is used by a service engineer to fine-tune the temperature controller. Do not use this table without consulting the manufacturer.

## B3 DISASSEMBLING AND SWITCHING THE SYSTEM OFF

1. Switch off the heating using the **HEATER** button;
2. Reduce the flow rate using the flow meter regulation down to 2-4 l/min;
3. CAREFULLY disconnect the cooling unit of quartz insert;
4. Move LN Dewar and place it on the floor;
5. Very slowly take out the cooling unit from the LN Dewar. Do not stop the gas flow through the cooler;
6. Place the cooling unit on the bottom shelf of the table. After 2-3 min the gas flow can be stopped;
7. Close the LN Dewar with the cover;
8. Disconnect the wires and hoses from the control unit;
9. Make sure that the quartz insert is securely clamped in the collet clamp;
10. Hold the quartz insert at the bottom and loosen the collet clamp;
11. Remove the quartz insert from the cavity;
12. Place the quartz insert into the safe packaging.

# APPENDIX C. ELECTROMAGNET COOLING

To reach the 20°C cavity temperature you have to use the water-cooling system:

- take the plastic Sockets Nitto-Kohki (for water cooling) from the service kit;
- put them on Nitto Kohki Cube Cupla Plug on the rear panel of the EPR spectrometer (see the figures below);

- connect the Silicone tubes I.D. 6 mm to the input and output plugs;
- connect the water-input silicone tube to the cold water tap;
- put the water-output tube into the sink and release the water flow.

Approximately 35-40 min is required for the 0,5 l/min water flow rate to change the cavity temperature from 32°C to 20°C (the magnetic field was set at 350 mT).

# APPENDIX D. FLAT CELL INSTALLATION

To install flat cell you have to:

- take the heater with the thermocouple (fig. 1) and check if there is a hole in the middle part (fig.2). If not create a hole (diameter is approx. 3mm);

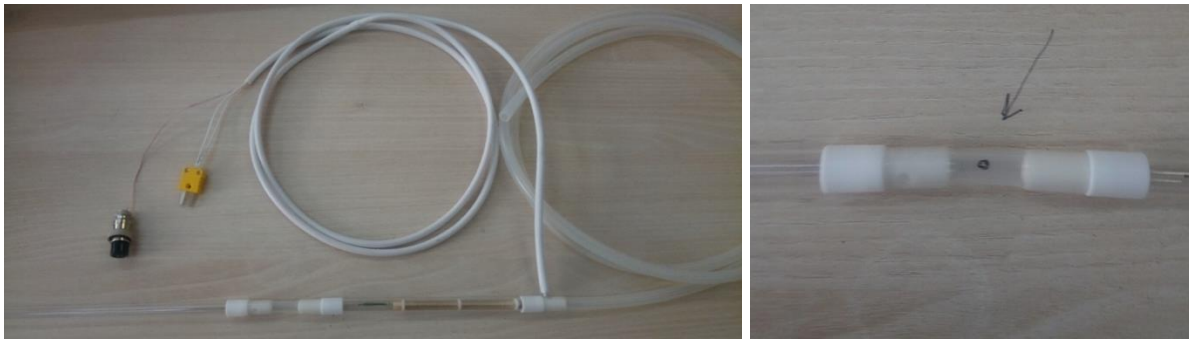


Figure 1

- Insert the thin tube in this hole in the side opposite from the thermocouple like is shown on the fig. 2. This tube have to pass through the glass tube out and the other side of the tube you will put later in some glass for returning a liquid (fig. 3);

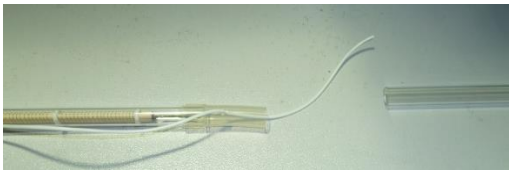


Figure 2

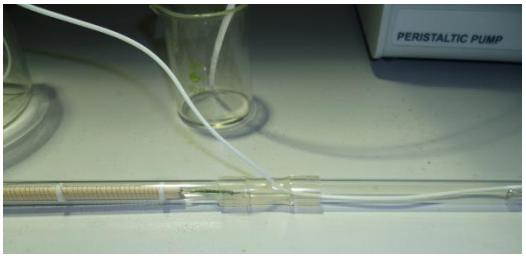



Figure 3

- Unscrew the metal holder and get out together with the protection tube (fig.4). Get out the protection tube (fig.4);


Figure 4

- Check the spectrometer position on the table. The hole into the resonator should be directly under the hole on the table. Insert the heating system from the bottom part of the table. Please do it very carefully. Take the glass part of the heating system from the top of spectrometer (fig.5);

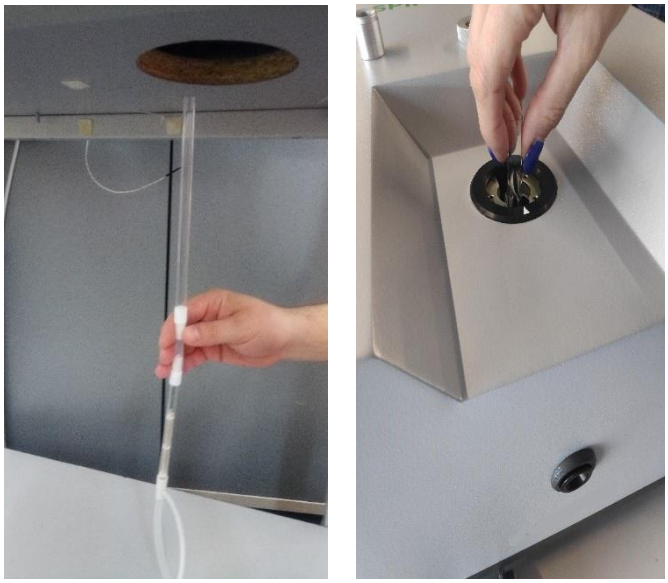


Figure 5

- Put the Teflon holder on the top part (the thin tube should be still out) and fix the metal holder (fig.6);

Figure 6

- Connect the flat cell with the thin tube. Put the Teflon folder on flat cell in the position in order to have 95 mm from the upper part of the holder to the central part of the working area of the flat cell;

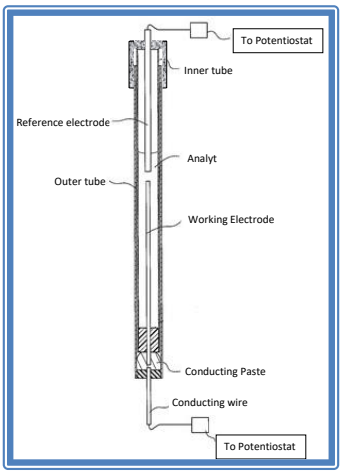
- Put flat cell into resonator;

- Put the other end of the thin tube into some glass for returning a liquid.

# APPENDIX E. ELECTROCHEMISTRY-EPR EXPERIMENT PROTOCOL

**I Materials**: Electrochemical Cell (EC) -EPR Cell:

- Quartz tubes (outer and inner)
- Conducting liquid/paste
- Pt/Cu/Graphite Working Electrode
- Ag-(Ag-AgCl*)Counter/Reference Electrode




*** How to make Ag / AgCl electrode.**

Immerse the pre-cleaned silver wire into the acidified 0.25M $FeCl_3$ solution (ferric chloride (III), prepared in 0.1M HCl or Sulfuric acid to avoid $Fe^{3+}$ hydrolysis) for 10 seconds to form a dark coating.

**II Equipment:** EPR spectrometer SPINSCAN X LAB and potentiostat.

**III Experiment**

1. Run the e-Spinoza program for EPR spectrometer control;
2. Turn on the EPR spectrometer and warm up for 1.5-2 hours;
3. Start the PSTrace program for potentiostat control;
4. Prepare the test solution (for example CTZ solution: 0.2 g CTZ in 0.1 M $H_2SO_4$):

- Prepare 0.1 M sulfuric acid (2 mL of concentrated acid in 200 mL Water)

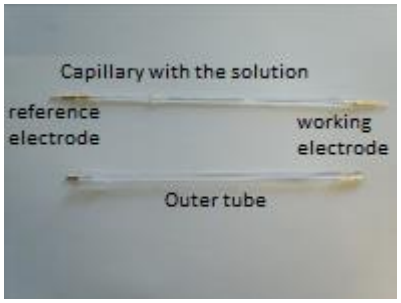
- Grind very well 5 tablets to make fine powder

- Dissolve 0.2 g of the powder into 100 mL of the prepared sulfuric acid solution

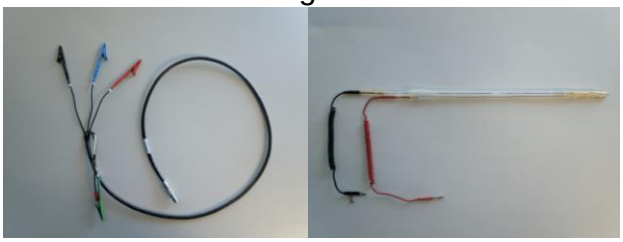
- Use stirrer for proper dissolving of powder contents

- Filtrate the turbid solution to get clear solution containing the active material well dissolved). Fill the capillary OD 0.8 mm with the sample;

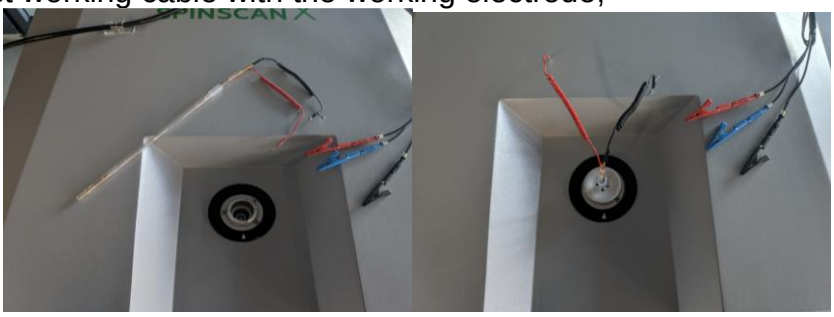
5. Put the reference and working electrodes into the capillary with the test solution and then insert it into the outer tube. The distance between electrodes should be 3-10mm.




6. Install the assembled EC-EPR cell into the resonator using the Teflon holder. The cell should be centered well (for adjustment you can rotate the cell around axis so the copper wire inside of outer tube was to the left or to the right of the cell with test solution;



7. Connect the cable to the potentiostat using the 4-pin outlet;
8. Connect reference and auxiliary cables to the - to the Ag / AgCl electrode;
9. Connect working cable with the working electrode;



10. Set up the experiment parameters for e-Spinoza. Recommended parameters are as follows (may vary):

| | |
|---|---|
| Mod. frequency, Hz | 100000 |
| Center field, mT | 336 |
| Sweep width, mT | 10 |
| Points, s | 1000 |
| Mod. amplitude, uT | 150 |
| Smoothing points | 0 |
| Sweep time, s | 20 |
| Scans | 10 |

11. Press **Autotune** to tune the microwave bridge with the resonator when the EC-EPR tube is inserted in the resonator;

12. On the **PSTrace** window left panel choose the ***Connection*** and update the list of devices. In case the Device has not been recognized automatically select the correct COM port number in the drop down menu; press ***Connect***;

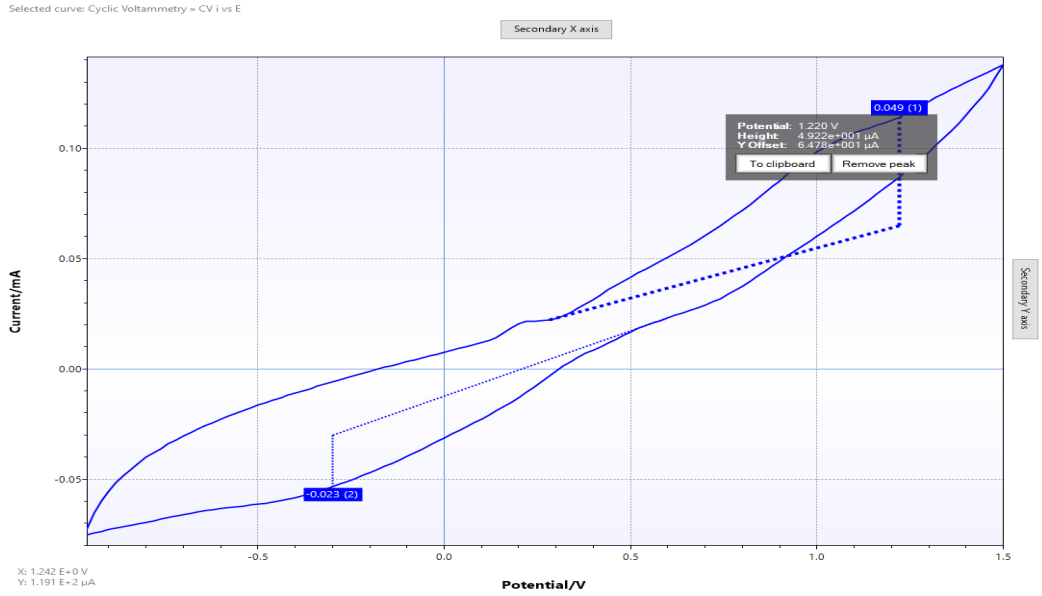
13. Choose the mode ***Measurement*** and set up the experiment parameters;
In the mode ***cyclic VA*** run ***Cyclic – Voltammetry*** *(CV)* test.

Recommended parameters are follow:

*Voltage - from -1.0 to 1.3V*

*Scan rate - 0.1 V/s*

*Step potential - 0.01V*

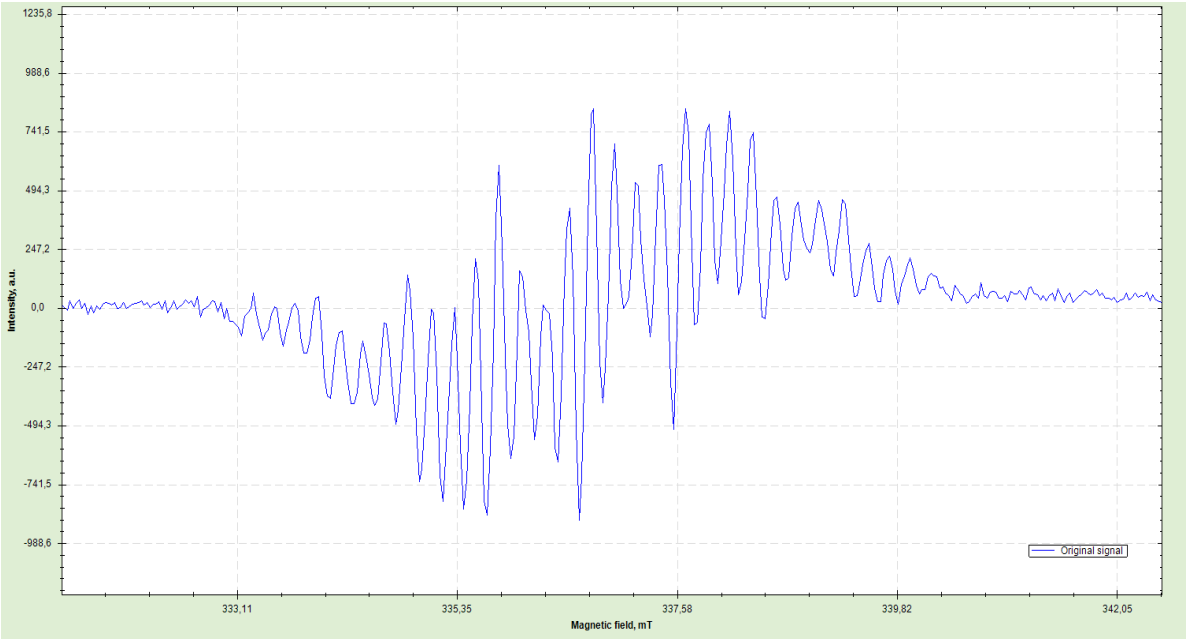


14. Switch to modes ***Chronoamperometry*** or ***Cyclic VA***, set up the fixed potential (for example 1,2 V) to start the electrolysis reaction; if you choose chronoamperometry – then set the parameters values: t equilibration – 1000s (or any value suitable for you), E dc – 1,2V (recommended value, you may vary it depending on the CV test performed in §13); if you choose Cyclic VA – then set the parameters values: t equilibration – 1000s (or any value suitable for you), E begin – 1,2V (recommended value, you may vary it depending on the CV test performed in §13);

| Chronoamperometry Settings | | |
|---|---|---|
| t equilibration | 1000 | s |
| E dc | 1.2 | V |
| t interval | 0.1 | s |
| t run | 1000.0 | s |

| Cyclic Voltammetry Settings | | |
|---|---|---|
| t equilibration | 1000 | s |
| E begin | 1.2 | V |
| E vertex1 | 1.2 | V |
| E vertex2 | 1.25 | V |
| E step | 0.01 | V |
| Scan rate | 0.01 | V/s |
| Number of scans | 10 | |

15. Press ***Start measurement*** to run the electrolysis reaction;
16. Wait 1-2 minutes to accumulate enough electrolysis product and start the EPR spectrum acquisition.



# APPENDIX F. PHOTOCHEMISTRY-EPR EXPERIMENT PROTOCOL

F1 REACTANTS

DMPO or DEPMPO spin traps may be used for hydroxyl radicals detection.

1) 200 mM stock solution of DMPO (3,4-dihydro-2,3-dimethyl-2H-pyrrole 1-oxide);

2) 200 mM stock solution of DEPMPO P-(3,4-dihydro-2-methyl-1-oxido-2H-pyrrol-2-yl)-phosphonic acid, diethyl ester).

3) 25 mM $H_2O_2$ in a buffer solution (buffer: 0,1 mM KCl, 0,1 mM $CaCl_2$, 1 Tris / 2 MES pH 6.0)

F2 MATERIALS

1) Micropipettes 200-1000 ul, 20-200 ul, 10-100 ul, 2-20 ul;

2) Micropipette tips and polypropylene microtubes;

3) Capillaries, BLAUBRAND®, DE-M marking, intraMark;

4) Wax for sealing the capillaries, Marienfeld.

F3 EQUIPMENT

1) EPR-spectrometer SPINSCAN X;

2) Light source - DEUTERIUM TUNGSTEN HPOWER / LS-DWHP, spectral range 190-3000 nm, Sarspec;

3) Optical filter for λ=395 nm "Sarspec LDA" SLPF-395-LSP;

4) Optical fibre, UV/VIS, 50 cm length, 1000 um diameter, PVC protective coating.

F4 EXPERIMENT

Preparation of a mixture for hydroxyl radicals generation

1. Hydroxyl radicals can be generated in a reaction mixture, containing 10 mM $H_2O_2$ and 100 mM DMPO.

Mix the following solutions:

| Solutions: | 200 mM DMPO | Buffer solution | 25 mM $H_2O_2$ |
|---|---|---|---|

| | | | |
|---|---|---|---|
| V= 50 ul | 25 ul | 5 ul | 20 ul |

2. Hydroxyl radicals can be generated in a reaction mixture, containing 10 mM $H_2O_2$ and 50 mM DEPMPO

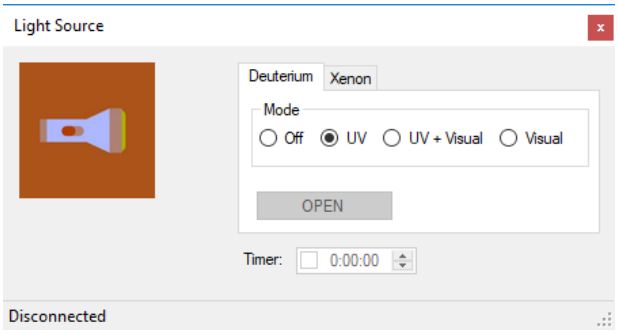
Mix the following solutions:

| Solutions: | 200мM DEPMPO | Buffer solution | 25 mM $H_2O_2$ |
|---|---|---|---|
| V= 50 ul | 12,5 ul | 17,5 ul | 20 ul |

Carefully shake and immediately fill the capillary with the obtained solution. Seal the capillary with wax.

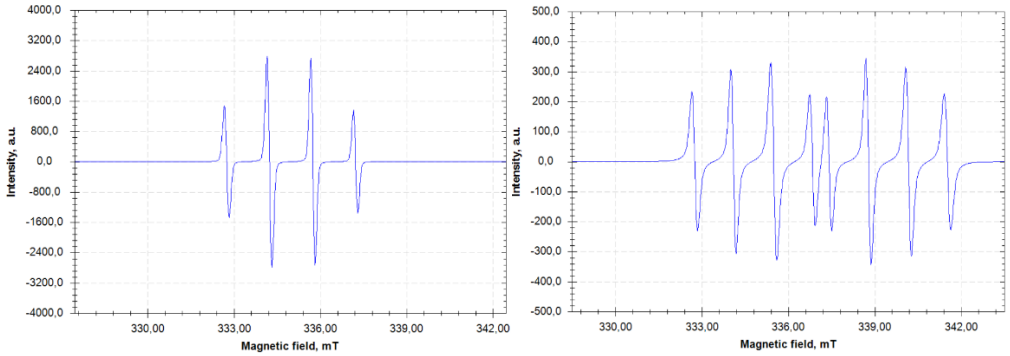
Measurements

1. Connect the light source to the EPR-Spectrometer SpinScanX illumination opening using the optical fibre;
2. Open the **Connection** menu and renew the list of connected devices. If the **Device** cannot be found automatically – select the corresponding COM port number in the drop-down menu. Click **Connect**;
3. Insert the sample into the spectrometer;
4. Click the **LIGHT SOURCE** button  ;




5. Select the required irradiation mode (**UV**) and irradiation time (3 min as an example);
6. EPR-spectra are recorded at room temperature. Set the spectrometer parameters. Recommended parameters are given below (you may optimize the parameters for a particular device and experimental setup):

| | |
|---|---|
| Mod. frequency, Hz | 93750 |
| Center field, mT | 336 |
| Sweep width, mT | 10 |
| Points, s | 1000 |
| Mod. amplitude, uT | 150 |
| Smoothing points | 0 |
| Sweep time, s | 40 |
| Scans | 1 |
| Attenuation, dB | 10 |




**a)** **b)**

EPR spectra of DMPO-OH (a) and DEPMPO-OH (b) UV-generated radicals

# APPENDIX G. LIQUID NITROGEN DEWAR FINGER-FLASK

1. Turn the spectrometer ON and launch the *e-Spinoza* software.

2. The liquid nitrogen (LN) Dewar finger flask must always be clean. Please check its purity and dryness prior to performing measurements.

3. Install the flask inside the spectrometer and fill it with LN.

> NOTIC
>
> *Use protective gloves and glasses while operating with LN.*
>
> *Use a gauze filter while filling the flask to prevent contamination. When the flask is filled with LN use a piece of cotton wool to wipe the flask inner surfaces to stop the bubbles formation.*

4. Insert the sample tube into the flask and wait until the formation of bubbles stops.

5. Perform the tuning and spectrum record.

6. Refill the LN inside the flask if necessary.

> **NOTICE**
>
> *Note that nitrogen bubbles are periodically being formed – it disturbs the spectrum registration and may cause the sample "throw-out" of the tube. Fast spectrum acquisition is recommended.*

# APPENDIX H. INTRODUCTION TO PYTHON

Many of the examples in this manual include comments. Comments in Python start with the hash character, #, and extend to the end of the physical line. A comment may appear at the start of a line or following whitespace or code, but not within a string literal. A hash character within a string literal is just a hash character. Since comments are to clarify code and are not interpreted by Python, they may be omitted when typing in examples.

Some examples:

```
# this is the first comment
spam = 1  # and this is the second comment
        # ... and now a third!
text = "# This is not a comment because it's inside quotes."
```

## 1.1 Using Python as a Calculator

Let’s try some simple Python commands.

### 1.1.1 Numbers

The interpreter acts as a simple calculator: you can type an expression at it and it will write the value. Expression syntax is straightforward: the operators +, -, * and / work just like in most other languages (for example, Pascal or C); parentheses (()) can be used for grouping. For example:

```
print 2 + 2
print 50 - 5*6
print (50 - 5.0*6) / 4
print 8 / 5.0
```

output:

```
4
20
5.0
1.6
```

The integer numbers (e.g. 2, 4, 20) have type int, the ones with a fractional part (e.g. 5.0, 1.6) have type float. We will see more about numeric types later in the tutorial.

The return type of a division (/) operation depends on its operands. If both operands are of type int, floor division is performed and an int is returned. If either operand is a float, classic division is performed and a float is returned. The // operator is also provided for doing floor division no matter what the operands are. The remainder can be calculated with the % operator:

```
print 17 / 3  # int / int -> int
print 17 / 3.0  # int / float -> float
print 17 // 3.0  # explicit floor division discards the fractional part
print 17 % 3  # the % operator returns the remainder of the division
print 5 * 3 + 2  # result * divisor + remainder
```

output:

```
5
5.6666666666667
5.0
2
17
```

With Python, it is possible to use the ** operator to calculate powers [1]:

```
print 5 ** 2  # 5 squared
print 2 ** 7  # 2 to the power of 7
```

output:

```
25
128
```

The equal sign (=) is used to assign a value to a variable:

```
width = 20
height = 5 * 9
print width * height
```

output:

900

If a variable is not “defined” (assigned a value), trying to use it will give you an error:

print n  # try to access an undefined variable

Message: name 'n' is not defined

There is full support for floating point; operators with mixed type operands convert the integer operand to floating point:

print 3 * 3.75 / 1.5

print 7.0 / 2

output:

7.5

3.5

In addition to int and float, Python supports other types of numbers, such as Decimal and Fraction. Python also has built-in support for complex numbers, and uses the j or J suffix to indicate the imaginary part (e.g. 3+5j).

### 1.1.2 Strings

Besides numbers, Python can also manipulate strings, which can be expressed in several ways. They can be enclosed in single quotes ('...') or double quotes ("...") with the same result [2]. \ can be used to escape quotes:

print 'spam eggs'  # single quotes

print 'doesn\'t'  # use \' to escape the single quote...

print "doesn't"  # ...or use double quotes instead

print '"Yes," he said.'

print "\"Yes,\" he said."

print '"Isn\'t," she said.'

output:

spam eggs

doesn't

doesn't

"Yes," he said.

"Yes," he said.

"Isn\'t," she said.

If you don’t want characters prefaced by \ to be interpreted as special characters, you can use *raw strings* by adding an r before the first quote:

```
print 'C:\some\name'  # here \n means newline!
print r'C:\some\name'  # note the r before the quote
```

output:

C:\some

ame

C:\some\name

String literals can span multiple lines. One way is using triple-quotes: """..."""" or '''...'''. End of lines are automatically included in the string, but it’s possible to prevent this by adding a \ at the end of the line. The following example:

```
print """\
Usage: thingy [OPTIONS]
    -h                  Display this usage message
    -H hostname         Hostname to connect to
"""
```

produces the following output (note that the initial newline is not included):

```
Usage: thingy [OPTIONS]
    -h                  Display this usage message
    -H hostname         Hostname to connect to
```

Strings can be concatenated (glued together) with the + operator, and repeated with *:

```
# 3 times 'un', followed by 'ium'
print 3 * 'un' + 'ium'
```

output:

unununium

Two or more *string literals* (i.e. the ones enclosed between quotes) next to each other are automatically concatenated.

```
print 'Py' 'thon'
```

output:

Python

This only works with two literals though, not with variables or expressions:

```
prefix = 'Py'
print prefix 'thon'  # can't concatenate a variable and a string literal
```

Message: invalid syntax

```
print ('un' * 3) 'ium'
```

Message: invalid syntax

If you want to concatenate variables or a variable and a literal, use +:

```
prefix = 'Py'
print prefix + 'thon'
```

output:

'Python'

Strings can be *indexed* (subscripted), with the first character having index 0. There is no separate character type; a character is simply a string of size one:

```
word = 'Python'
print word[0]  # character in position 0
print word[5]  # character in position 5


output:
P
n
```

Indices may also be negative numbers, to start counting from the right:

```
print word[-1]  # last character
print word[-2]  # second-last character
print word[-6]


n
o
P
```

Note that since -0 is the same as 0, negative indices start from -1.

In addition to indexing, *slicing* is also supported. While indexing is used to obtain individual characters, *slicing* allows you to obtain a substring:

```
print word[0:2]  # characters from position 0 (included) to 2 (excluded)
print word[2:5]  # characters from position 2 (included) to 5 (excluded)


output:
Py
tho
```

Note how the start is always included, and the end always excluded. This makes sure that s[:i] + s[i:] is always equal to s:

```
print word[:2] + word[2:]
print word[:4] + word[4:]
```

Python

Python

Slice indices have useful defaults; an omitted first index defaults to zero, an omitted second index defaults to the size of the string being sliced.

```
print word[:2]   # character from the beginning to position 2 (excluded)
print word[4:]   # characters from position 4 (included) to the end
print word[-2:]  # characters from the second-last (included) to the end
```

output:

Py

on

on

One way to remember how slices work is to think of the indices as pointing *between* characters, with the left edge of the first character numbered 0. Then the right edge of the last character of a string of *n* characters has index *n*, for example:

```
 +---+---+---+---+---+---+
 | P | y | t | h | o | n |
 +---+---+---+---+---+---+
 0   1   2   3   4   5   6
-6  -5  -4  -3  -2  -1
```

The first row of numbers gives the position of the indices 0...6 in the string; the second row gives the corresponding negative indices. The slice from *i* to *j* consists of all characters between the edges labeled *i* and *j*, respectively.

For non-negative indices, the length of a slice is the difference of the indices, if both are within bounds. For example, the length of word[1:3] is 2.

Attempting to use an index that is too large will result in an error:

print word[42]  # the word only has 6 characters

Message: index out of range: 42

However, out of range slice indexes are handled gracefully when used for slicing:

print word[4:42]

print word[42:]

output:

on

Python strings cannot be changed — they are immutable. Therefore, assigning to an indexed position in the string results in an error:

word[0] = 'J'

Message: 'str' object does not support item assignment

word[2:] = 'py'

Message: 'str' object does not support item assignment

If you need a different string, you should create a new one:

print 'J' + word[1:]

print word[:2] + 'py'

output:

Jython

Pypy

The built-in function len() returns the length of a string:

s = 'supercalifragilisticexpialidocious'

print len(s)

output:

34

### 1.1.3 Lists

Python knows a number of *compound* data types, used to group together other values. The most versatile is the *list*, which can be written as a list of comma-separated values (items) between square brackets. Lists might contain items of different types, but usually the items all have the same type.

```
squares = [1, 4, 9, 16, 25]
print squares
```

output:

[1, 4, 9, 16, 25]

Like strings (and all other built-in sequence type), lists can be indexed and sliced:

```
print squares[0]  # indexing returns the item
print squares[-1]
print squares[-3:]  # slicing returns a new list
```

output:

1

25

[9, 16, 25]

All slice operations return a new list containing the requested elements. This means that the following slice returns a new (shallow) copy of the list:

```
print squares[:]
```

output:

[1, 4, 9, 16, 25]

Lists also supports operations like concatenation:

```
print squares + [36, 49, 64, 81, 100]
```

output:

```
[1, 4, 9, 16, 25, 36, 49, 64, 81, 100]
```

Unlike strings, which are immutable, lists are a mutable type, i.e. it is possible to change their content:

```
cubes = [1, 8, 27, 65, 125]  # something's wrong here
print 4 ** 3  # the cube of 4 is 64, not 65!
cubes[3] = 64  # replace the wrong value
print cubes
```

output:

```
64
[1, 8, 27, 64, 125]
```

You can also add new items at the end of the list, by using the append() *method* (we will see more about methods later):

```
cubes.append(216)  # add the cube of 6
cubes.append(7 ** 3)  # and the cube of 7
print cubes
```

output:

```
[1, 8, 27, 64, 125, 216, 343]
```

Assignment to slices is also possible, and this can even change the size of the list or clear it entirely:

```
letters = ['a', 'b', 'c', 'd', 'e', 'f', 'g']
print letters

# replace some values
```

```
letters[2:5] = ['C', 'D', 'E']
print letters

# now remove them
letters[2:5] = []
print letters

# clear the list by replacing all the elements with an empty list
letters[:] = []
print letters
```

output:

```
['a', 'b', 'c', 'd', 'e', 'f', 'g']
['a', 'b', 'C', 'D', 'E', 'f', 'g']
['a', 'b', 'f', 'g']
[]
```

The built-in function len() also applies to lists:

```
letters = ['a', 'b', 'c', 'd']
print len(letters)
```

output:

```
4
```

It is possible to nest lists (create lists containing other lists), for example:

```
a = ['a', 'b', 'c']
n = [1, 2, 3]
x = [a, n]

print x
```

```
print x[0]
print x[0][1]


output:
[['a', 'b', 'c'], [1, 2, 3]]
['a', 'b', 'c']
'b'
```

## 1.2 First Steps Towards Programming

Of course, we can use Python for more complicated tasks than adding two and two together. For instance, we can write an initial sub-sequence of the *Fibonacci* series as follows:

```
# Fibonacci series:
# the sum of two elements defines the next
a, b = 0, 1
while b < 10:
   print b
   a, b = b, a+b


output:
1
1
2
3
5
8
```

This example introduces several new features.

- The first line contains a *multiple assignment*: the variables a and b simultaneously get the new values 0 and 1. On the last line this is used again, demonstrating that the expressions on the right-hand side are all evaluated first before any of the

assignments take place. The right-hand side expressions are evaluated from the left to the right.

- The while loop executes as long as the condition (here: b < 10) remains true. In Python, like in C, any non-zero integer value is true; zero is false. The condition may also be a string or list value, in fact any sequence; anything with a non-zero length is true, empty sequences are false. The test used in the example is a simple comparison. The standard comparison operators are written the same as in C: < (less than), > (greater than), == (equal to), <= (less than or equal to), >= (greater than or equal to) and != (not equal to).
- The *body* of the loop is *indented*: indentation is Python's way of grouping statements. At the interactive prompt, you have to type a tab or space(s) for each indented line. In practice you will prepare more complicated input for Python with a text editor; all decent text editors have an auto-indent facility. When a compound statement is entered interactively, it must be followed by a blank line to indicate completion (since the parser cannot guess when you have typed the last line). Note that each line within a basic block must be indented by the same amount.
- The print statement writes the value of the expression(s) it is given. It differs from just writing the expression you want to write (as we did earlier in the calculator examples) in the way it handles multiple expressions and strings. Strings are printed without quotes, and a space is inserted between items, so you can format things nicely, like this:

```
i = 256*256
print 'The value of i is', i
```

output:

```
The value of i is 65536
```

A trailing comma avoids the newline after the output:

```
a, b = 0, 1
while b < 1000:
    print b,
    a, b = b, a+b
```

output:

```
1 1 2 3 5 8 13 21 34 55 89 144 233 377 610 987
```

Note that the interpreter inserts a newline before it prints the next prompt if the last line was not completed.

Footnotes

[1] Since ** has higher precedence than -, -3**2 will be interpreted as -(3**2) and thus result in -9. To avoid this and get 9, you can use (-3)**2.

[2] Unlike other languages, special characters such as \n have the same meaning with both single ('...') and double ("...") quotes. The only difference between the two is that within single quotes you don't need to escape " (but you have to escape \') and vice versa.

## 1.3 Special variables

There are two special variables: Device and MW. 'MW' variable represents mw tuning monitor functionality. 'Device' variable is used to get or set spectrometer state. See detailed description about these variables below.

### 1.3.1 'Device' variable.

'Device' variable has methods listed below.

Device.SetModulationFrequency(index)

Sets desired modulation frequency from the list with number *index*. *Index* starts from 0. Available frequencies (Hz): 6250, 9375, 10253, 10937, 11718, 12500, 13671, 15625, 16406, 18750, 20507, 21875, 23437, 25000, 27343, 31250, 32812, 37500, 41015, 43750, 46875, 50000, 54687, 62500, 65625, 75000, 82031, 87500, 93750, 100000, 109375, 125000, 131250, 150000, 164062, 175000, 187500, 200000, 218750, 250000, 262500, 300000, 328125, 350000, 375000, 400000, 437500, 500000.

Usage:

Device.SetModulationFrequency(29) # sets 100000 Hz.

Device.GetMWFrequency()

Returns current microwave frequency in Hz or None if impossible to get the frequency.

Usage:

```
a = Device.GetMWFrequency() #only with connected device
print a

output:
15998479
```

Device.SetAttenuation(att)

Sets attenuation att in dB. att is float value.

Usage:

```
Device.SetAttenuation(20)#Sets attenuation 20.0 db
```

Device.SetMWSpeed(speed_type)

Sets speed of microwave frequency measuring. speed_type has next definition:

enum MWSpeedType { mstNormal, mstFast }

Setting speed to mstFast reduces precision but increases speed of getting new microwave frequency value. Setting to mstNormal restores default parameters.

Usage:

```
Device.SetMWSpeed(MWSpeedType.mstFast)      # settings fast speed
Device.SetMWSpeed(MWSpeedType.mstNormal)    # setting default parameters
```

Device.GetMagneticFieldInfo()

Returns MagneticFieldInfo struct about current magnetic field or None if this call was unsuccessful. MagneticFieldInfo has next definition:

```
struct MagneticFieldInfo
{
    int StartMagneticField,
    int CurrentMagneticField,
```

int EndMagneticField

}.

Each field in this struct represents device code (not physical unit in milliTesla)

usage:

info = Device.GetMagneticFieldInfo()

print info

print info.StartMagneticField

print info.CurrentMagneticField

print info.EndMagneticField

Possible output:

{ "startMagneticField": 100000, "currentMagneticField": 100000, "endMagneticField": 200000 }

100000

100000

200000

Device.GetAttenuation()

Returns attenuation value in dB or None if this call was not successful.

Usage:

att = Device.GetAttenuation()

print att

Possible output:

19.9996948242

Device.ResetMotors()

Sets required and current position for every motor to 0. This command only sets values but does not perform any real moving.

Usage:

```
Device.ResetMotors()
print Device.GetMotorsPosition()
```

Possible output:

{ "frequencyMotorCurrentPosition": 0, "frequencyMotorDesiredPosition": 0, "couplingMotorCurrentPosition": 0, "couplingMotorDesiredPosition": 0, "phaseMotorCurrentPosition": 0, "phaseMotorDesiredPosition": 0, "isFrequencyTrailerActive": true, "isCouplingTrailerActive": true, "isPhaseTrailerActive": true, "trailer": 7 }

Device.MoveMotor(motor_type, move_type, value)

Performs desired motor moving to desired position. Parameters have next definition:

enum MotorType { MtFrequency, MtCoupling, MtPhase }

enum MotorMoveType { MmtReverse, MmtForward, MmtAbsolute }.

Last parameter 'value' has type int.

Usage:

```
# Moving frequency motor backward on 10 steps.
Device.MoveMotor(MotorType.MtFrequency, MotorMoveType.MmtReverse, 10)
```

Device.GetMotorsPosition()

Returns *MotorsPosition* struct about motors. *MotorsPosition* struct has next definition:

```
struct MotorsPosition
{
    int FrequencyMotorCurrentPosition,
```

```
        int FrequencyMotorDesiredPosition,
        int CouplingMotorCurrentPosition,
        int CouplingMotorDesiredPosition,
        int PhaseMotorCurrentPosition,
        int PhaseMotorDesiredPosition
}
```

Device.GetADCValues()

Returns ADCValues struct containing information about detector current, input AFC voltage, output AFC voltage, magnetic field error codes or None if this call wasn't successful. ADCValues has next definition:

```
struct ADCValues
{
        int InputAfcVoltage,
        int OutputAfcVoltage,
        int DetectorCurrent,
        int MagneticFieldError
}
```

Usage:

```
info = Device.GetADCValues()
print info
print info.InputAfcVoltage
print info.OutputAfcVoltage
print info.DetectorCurrent
print info.MagneticFieldError
```

Output:

```
{ "inputAfcVoltage": 1784, "outputAfcVoltage": 2014, "detectorCurrent": 3011, "magneticFieldError": 2285 }
1784
2014
```

3011

2285

Device.GetDACValues()

Returns DACValues struct or None if this cal wasn't successful. DACValues definition:

```
struct DACValues
{
    int HallFrequency,
    int HallAmplitude,
    int ModulationAmplitude,
    int AfcFrequency,
    int Attenuation
}
```

Usage:

```
info = Device.GetDACValues()

print info.HallFrequency
print info.HallAmplitude
print info.ModulationAmplitude
print info.AfcFrequency
print info.Attenuation
```

Output:

0

5120

0

0

0

Device.SetDiodeTapeColor(color)

Sets color for the device tape. color has next definition:

enum DiodeTapeColor { dtcDisabled, dtcBlue, dtcRed, dtcPurple, dtcGreen, dtcAqua, dtcYellow, dctWhite }

Usage:

Device.SetDiodeTapeColor(DiodeTapeColor.DtcBlue) #sets blue color

Device.SetMWMode(mode)

Enables/disables AFC and Homodyne. mode has next definition:

enum MWMode { MmOff, MmAfcOn, MmHomoOn, MmBoth }

Usage:

Device.SetMWMode(MWMode.MmOff) # disables AFC and Homodyne
Device.SetMWMode(MWMode.MmAfcOn) # enables AFC and disables Homodyne
Device.SetMWMode(MWMode.MmHomoOn) # disables AFC and enables Homodyne
Device.SetMWMode(MWMode.MmBoth) #enables AFC and Homodyne

### 1.3.2 'MW' variable.

'MW' variable is used to perform microwave scan and get the results. It has methods listed below.

MW.Scan(start, end, step, motor_type, reserved)

Performs microwave scanning with specified motor and range. start, end, step have type int. motor_type has type MotorType. See Device.Move motor for MotorType definition. reserved has type bool. It's reserved for future purposes. Must be always True.

Usage:

```
# Perform scan for frequency motor in range [10000, 20000] with step 100.
MW.Scan(10000, 20000, 100, MotorType.MtFrequency, True)
```

MW.GetAFCValues()

Returns AFC values from last microwave scan containing AFC codes.

Usage:

```
MW.Scan(10000, 20000, 100, MotorType.MtCoupling, True)
afc_values = MW.GetAFCValues()
print afc_values.Count      # list count
print afc_values[33]        # element access
```

Output:

```
101
1083
```

MW.GetDetectorCurrentValues()

Returns detector current values from last microwave scan containing detector current codes.

Usage:

```
MW.Scan(10000, 20000, 100, MotorType.MtFrequency, True)
dc_values = MW.GetDetectorCurrentValues()
print dc_values.Count       # list count
print dc_values[33]         # element access
```

Output:

```
101
2997
```

MW.GetMWFrequencyValues()

Returns microwave frequency values from last microwave scan containing values for any element of AFC list or detector current list.

Usage:

```
MW.Scan(10000, 20000, 100, MotorType.MtFrequency, True)
mw_values = MW.GetMWFrequencyValues()
print mw_values.Count          # list count
print mw_values[33]            # element access
```

Output:

101

9450123

MW.GetMotorPositionValues()

Returns motor position values from last microwave scan containing values for any element of AFC, detector current or microwave frequency lists.

Usage:

```
MW.Scan(10000, 20000, 100, MotorType.MtFrequency, True)
m_values = MW.GetMotorPositionValues()
print m_values.Count           # list count
print m_values[33]         # element access
```

Output:

101

13300

MW.SetQValue(value)

Sets current q-value and displays it on control panel. value has type float

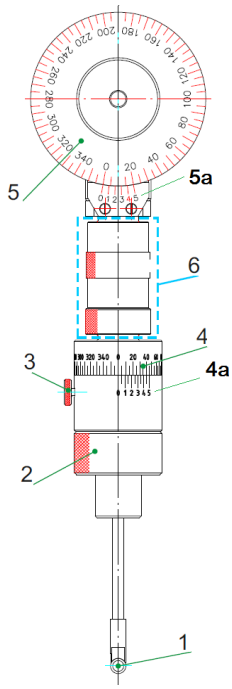
Usage:

```
MW.SetQValue(300.5)
```

# APPENDIX I. GONIOMETR

To study the anisotropy of the g – factor of solid samples a goniometer can be used.

The design of the goniometer is shown in Fig.1.




1- sample holder (diameter 5 mm);
2- goniometer fixing nut;
3- horizontal orientation fixing screw;
4- horizontal adjustment scale;
5- vertical adjustment scale;
6- thread tension knot

Figure 1

1. The crystal (Fig. 2) should be fixed in the sample holder (1) (Fig.1) at the bottom of the goniometer (Fig.3). The size of the crystal used for analysis is limited by the inner diameter of the ring (d=5mm). For a tighter fixation of the sample in the holder, it can be placed in cotton wool or in another material that does not give an EPR signal (Fig.4).

Figure 2

Figure 3

Figure 4

2. To combine the goniometer with the spectrometer it is necessary to unscrew the nut of the sample holder as shown in Fig.5. and insert the goniometer into the holder so that the lower Vernier scale is placed facing the user (Fig.6);

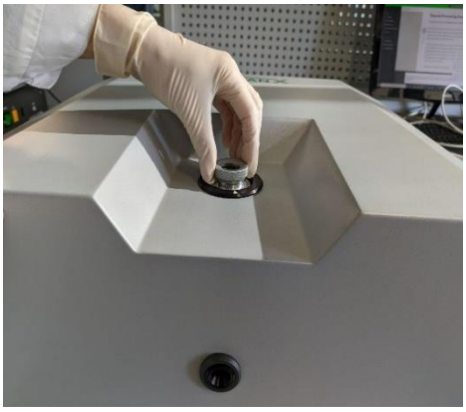


Figure 5

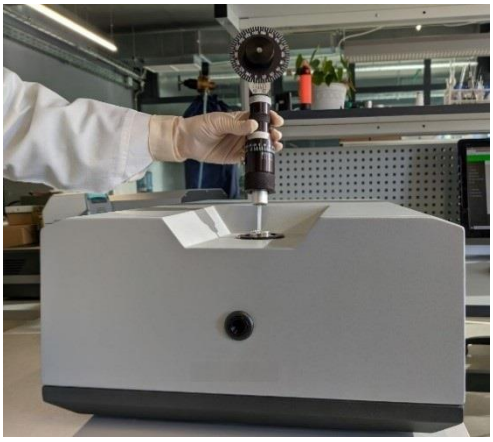


Figure 6

3. To fixation of the goniometer in the spectrometer it is necessary to tighten the goniometer fixing nut (2) (Fig.7 and 8);

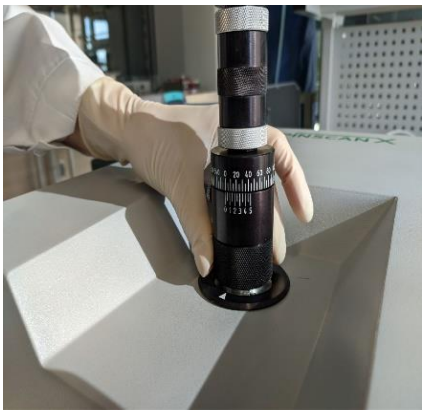


Figure 7

Figure 8

4. The anisotropy of the g-factor of a crystal can be studied in two planes: horizontal and vertical. You can work both with one plane with a fixed second, and with the change of two at the same time. Using the lower screw of the scale the crystal sample rotates in the horizontal plane (Fig.9). The crystal sample rotates in the vertical plane using the upper limb with the Vernier scale (Fig.10).

Before removing the spectrum set the appropriate angles on the Nonius scales (upper and lower). The selected values of the angles are fixed when the zero of the lower scale is combined with the value of the upper scale;

Figure 9

Figure 10

***Note*** ☞ Don’t rotate the gray nuts located in node (6) which responsible for the tension of the threads fixing the ring for samples. It cause to unbalance the entire goniometer structure

5. To record the spectrum of the test sample it is necessary:
- set the parameters of spectrum in the e-Spinoza program;
- press the **Set parameters** button;
- perform the **Autotune**;
- record the spectrum of the sample.

7. The **Autotune** procedure must be repeated after each change of the goniometer angle before recording spectrum.