DIASPEC 18

Low field NMR relaxometry equipment

User manual

Summary

I. System overview 3

1. Diagram of the equipment 3

2. Technical specifications 4

Static induction of the sensor 4

Radiofrequency structure (RF) 4

Structure geometry (Diaspec 18) 4

II. Measuring equipement connection 5

III. DIALOG software presentation 6

1. Starting the sofware 6

2. Expert mode 7

New sequence 8

New calibration 11

New measurement 14

Options 19

Curve compare 20

Automatic calibration 21

Open measurement 22

Export 24

3. Operator mode 26

Operator mode 27

Porosity mode 32

4. Administrator mode 35

User manager 36

Database manager 38

Options 39

I. System overview

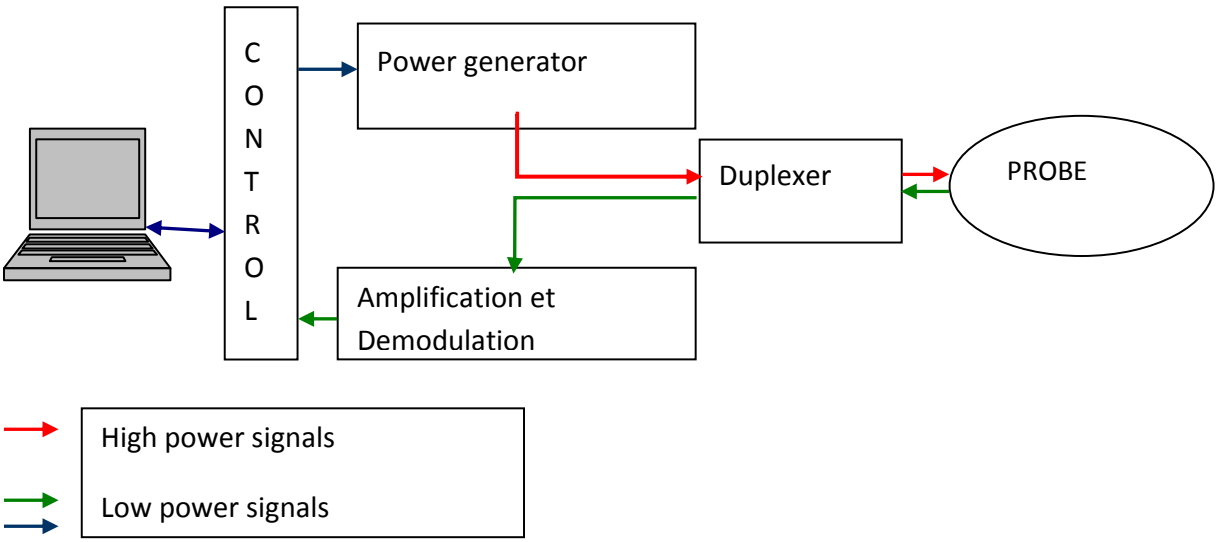
ARTEC SYSTEM propose a non-destructive measurement system based on low field magnetic resonance technology to measure quantity of water, oil or grease, establish quality criteria and characterize porosity.

Materials covered by these analysis can take the form of solids, liquids or pastes.

DIASPEC is a transportable measuring instrument, ideal for rapid characterization of samples in the laboratory or quality control. The measurement and results analysis software is easily handled by non-expert operators in NMR.

1. Diagram of the equipment

The general structure of the equipment is represented in the figure below.



2. Technical specifications

Static induction of the sensor

The material used for the generation of static and uniform magnetic field, required to operate the sensor, is neodymium-iron-boron. It presents a good thermal stability up to 80 ° C. In the configuration of Diaspec devices, the volume of measurement corresponds to a cylinder, with an axis perpendicular to the static field, a diameter of 18 mm and a height of 20mm.

The value of the static induction (B0) is approximately 0.44 T (varies around this value, depending on the device).

The inhomogeneity of the magnetic field is less than $dB0/B0 = 10^{-4}T$ in the measuring volume.

Radiofrequency structure (RF)

The radiofrequency structure, integrated inside the sensor, includes an antenna and a transmission line coupling. It generates an RF induction perpendicular to the static induction in the measuring volume.

Structure geometry (Diaspec 18)

- Measuring tube: diameter 18mm external
- Radial resolution: 18mm diameter
- Vertical resolution: 20mm height

The external metallic case of the measuring instrument is covered with an anti-corrosion coating.

a) b)

a) overview of equipment with sensor and spectrometer

b) top view of the NMR diameter 18 mm sensor

II. System connection

For a first powering of the equipment, please follow the connection method:

1. Connect the 50 ohm coaxial cable from the sensor to the back of the instrumentation.
2. Connect the USB cable from the back of the instrumentation to one USB 2.0 socket on the computer.

   Caution, be sure to connect the instrumentation to a **USB2 port** of the computer. Connection to a USB 3 port (rated USB SS or blue plastic on some PC) will result in the non recognition of the device.

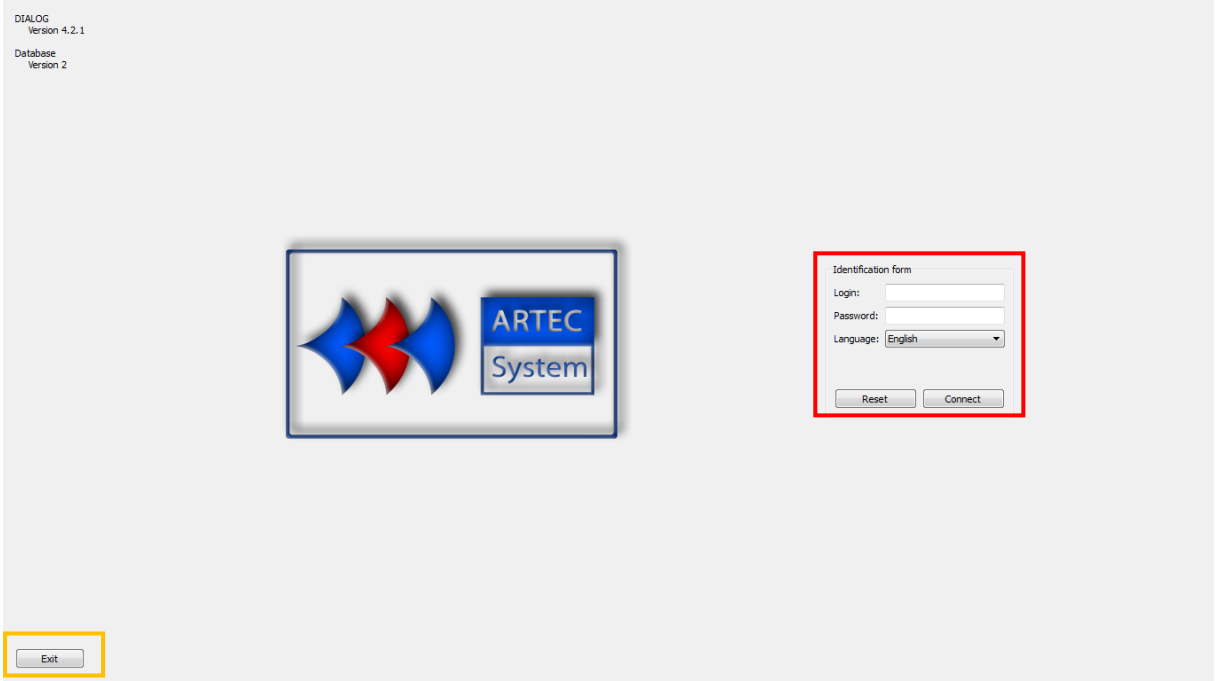
3. Turn on the computer.
4. Turn on the instrumentation from the switch on the front panel of the instrumentation and one on the rear face. Please wait 15 seconds before the first measurement.

CAUTION TO THE PRESENCE OF VOLTAGE IN THE CAPACITORS IN THE AMPLIFIER AFTER EXTINCTION OF THE DEVICE. DO NOT OPEN THE BOX OF INSTRUMENTATION.

III. DIALOG software presentation

1. Starting the software

1. Check the connections of the device (power and signal).
2. Turn on the instrumentation rack.
3. Launch Dialog by double-clicking on the shortcut icon.
4. Software open the login window



Login zone

login : User login. A login give a type of access privileges (operator, expert, or administrator).

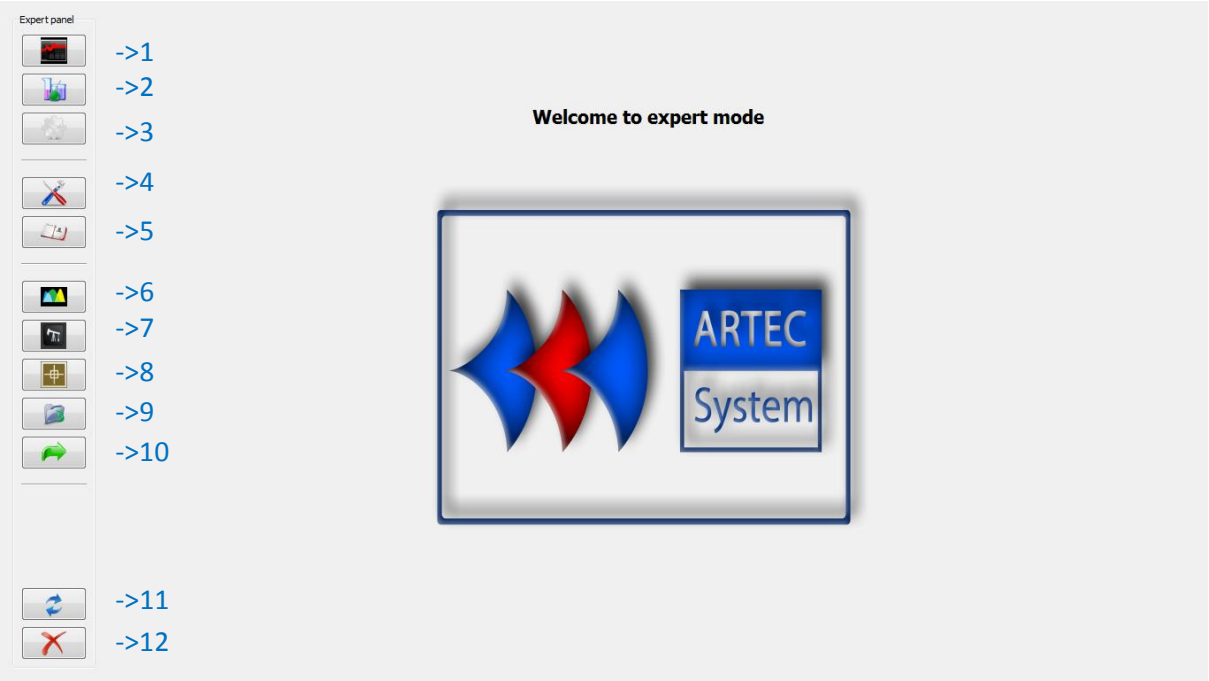
Password: Captures the password. Secure the identification.

Language: Choice of language, the software restarts with the chosen language.

Application exit button

2. Expert mode

There is, by default, an expert login implemented in the software (identifier and password : Exp). It is possible, for the administrator, to create other users with Expert rights.

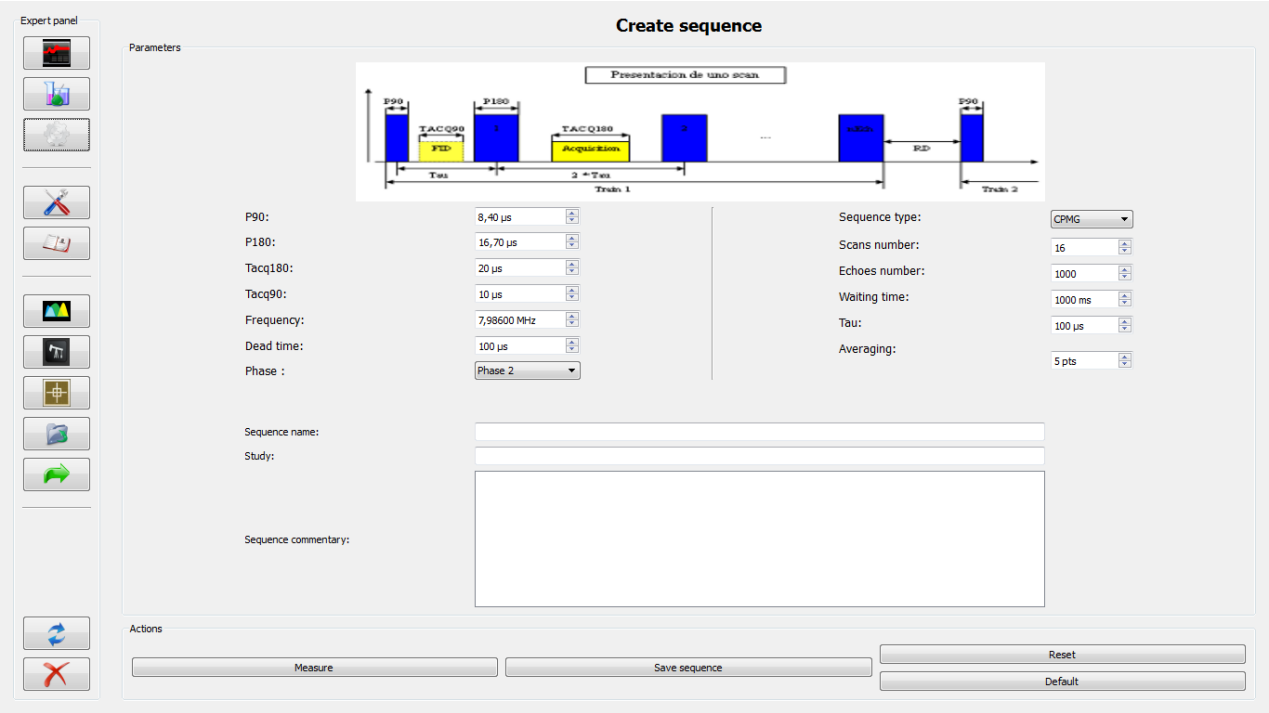


Depending on customer options, some of these features may be inaccessible.

1. New measurement
2. New calibration
3. New sequence
4. Expert options
5. Summary
6. Curve comparison
7. Well comparison
8. Auto calibration
9. Opening measurement
10. Export
11. Disconnection
12. Closing the application

New sequence

This window allows editing of the NMR (CPMG or FID) sequences in order to perform measurements.



A sequence is composed of the following parameters:

P90 : Duration of the P90 pulse.

P180 : Duration of the P180 pulse. P180 is close to 2 * P90.

TACQ180 : Duration of the acquisition of a CPMG echo.

TACQ90 : Duration of the acquisition of the decay of magnetization after a pulse P90 (choice FID).

Frequency : Working frequency of the instrument.

Dead time (DT) : Dead time before and after each pulse. Prevents the registration of erroneous data due to residues of pulses. (FID)

Phase : Phase cycling. Recommended value: SEQ2.

Sequence type : choice of the type of the NMR sequence to launch (CPMG or FID)

Number of scans (nScan) : The number of repetitions ('scans') of the sequence. The measured values are accumulated and then divided by the number of scans. To get a better signal to noise ratio. Recommended value: a multiple of 4 with the choice of phase "phase 2".

Number of echoes (nEch): The number of echoes defines the number of measured points of the signal. For quantities of water measurements, a small number is sufficient (50). For relaxation times of the sample, increase the number of echoes in order to obtain the complete decay of the signal. The signal is recorded on a time interval 2 * Tau * nEch.

Waiting time (RD) : Time between two scans. This time must be at least equal to 5 times the system dominant relaxation time.

Tau : Time between pulses P90 and P180. The signal is recorded on a time interval 2 * Tau * nEch.

Averaging : Number of points at the top of the echo used to filter the value of the maximum of the echo.

Sequence name : Name of the sequence (must be unique).

Comment : This field can be left blank or contain the information that the user considers as useful.

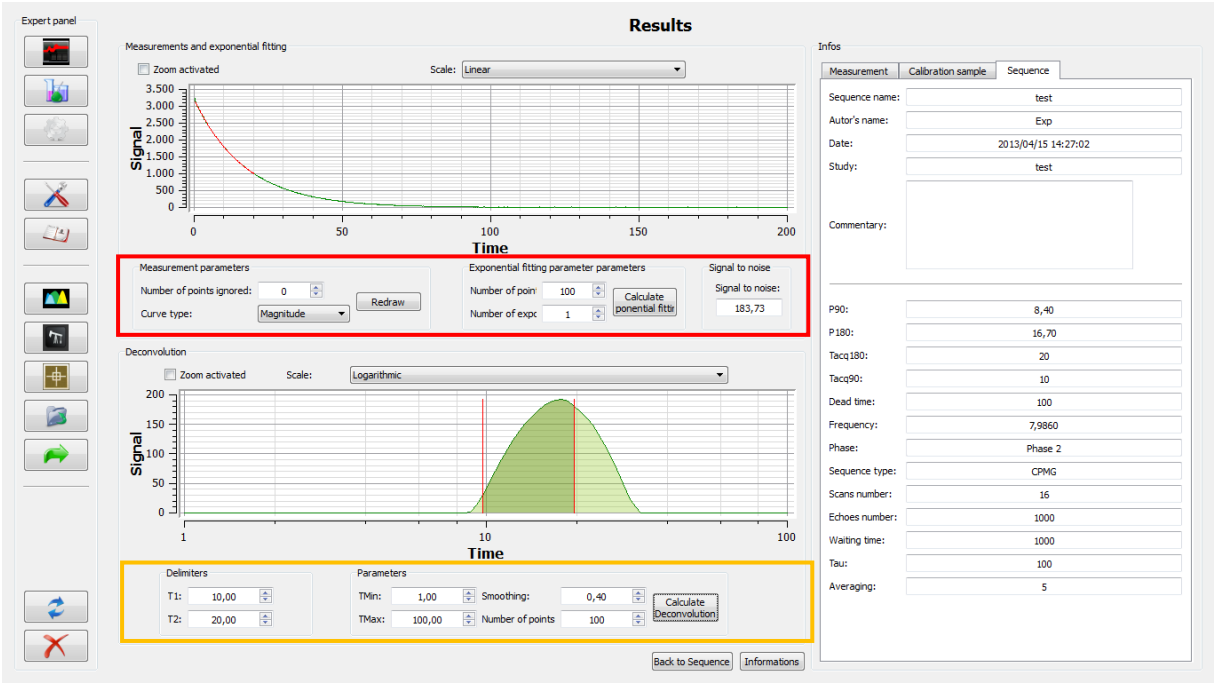
Buttons at the bottom have the following actions:

Reset: This button empty boxes in the window.

Default: This button filled the box containing the parameters of the sequence by the saved default settings.

Save sequence: Allows you to save the sequence in the database.

Measure: This mode allows to test the sequence before saving it. Pressing this button launches a measure from the filled sequence.



On this result window, it is possible to recalculate exponential and deconvolution processing on measurement. Two display modes are available for each curve, the time scale can be either linear or logarithmic.

It is possible to show or hide the information of the measure. This panel contains information about the sequence. This function not calling calibration, the calculations are made for visual analysis of the good treatment. The numerical results are not given.

Measurements and exponential processing setting

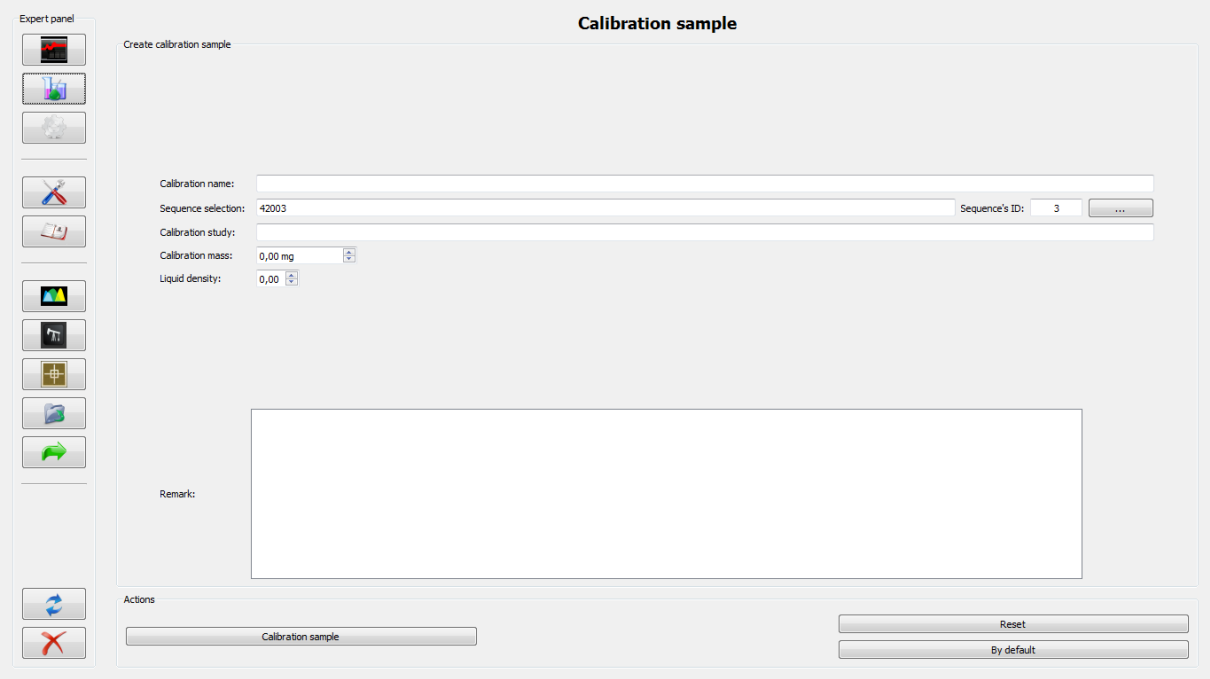
It is possible to display different curves from the measurement: absolute value, rephase or noise (angle correction following the imaginary). This menu allows the deletion of point at the beginning of the curve.

It is also possible to modify the multi-exponential processing parameters. These parameters are the number of points to use (these points are systematically taken early in the curve) and the number of exponentials to calculate.

Deconvolution setting

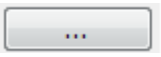
It is possible from this mode to verify the proper calculation of deconvolution with the result of the test sequence. If necessary, it is possible to test a modification of start and end times of the calculation, the number of calculated points and the curve smoothing.

New calibration

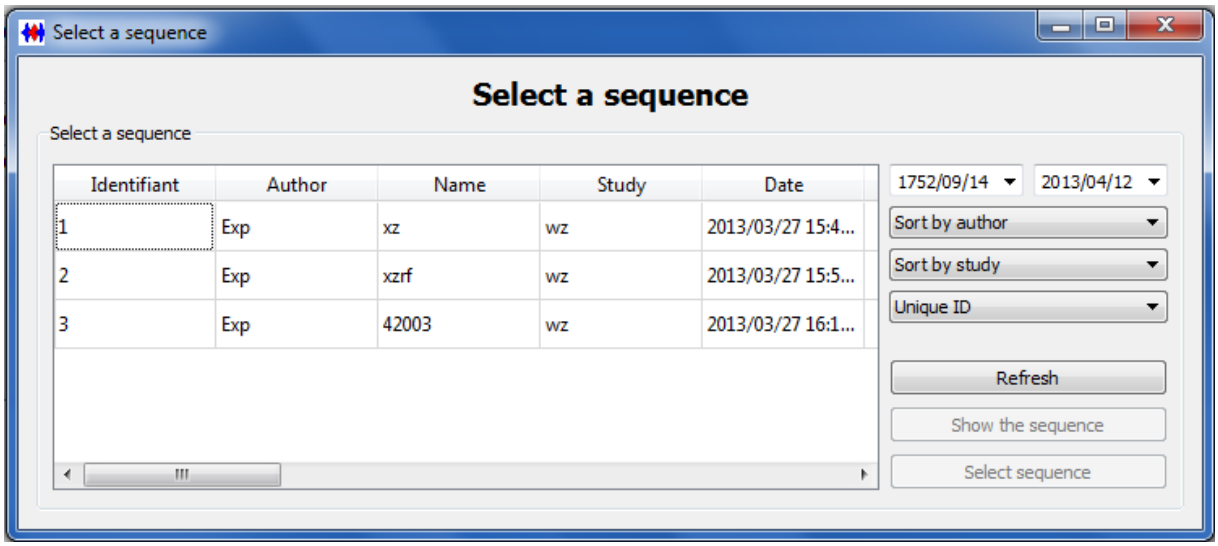


This window allows to fill the parameters used for the creation of a new standard. The parameters are the following:

Calibration name : This field contains the name of the future standard. This name must be unique.

Sequence selection : This field contains the name of the sequence to use. To change the sequence to use, click on 

. A window appears. It list all the sequences in the database



To select the sequence you want, click on the corresponding ID (the integer, highlighted on the image) then click on select the sequence.

It is possible to use the filters, on the right, to simplify the search for the desired sequence.

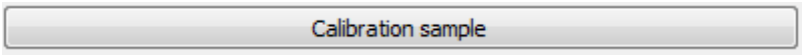
Calibration study : Fills in the study name for which the calibration was created. Several calibration can belong to the same study.

Mass of the calibration sample : Fill in the precise mass of the sample used for the calibration. This value is used for calculation of humidity measurements using this calibration.

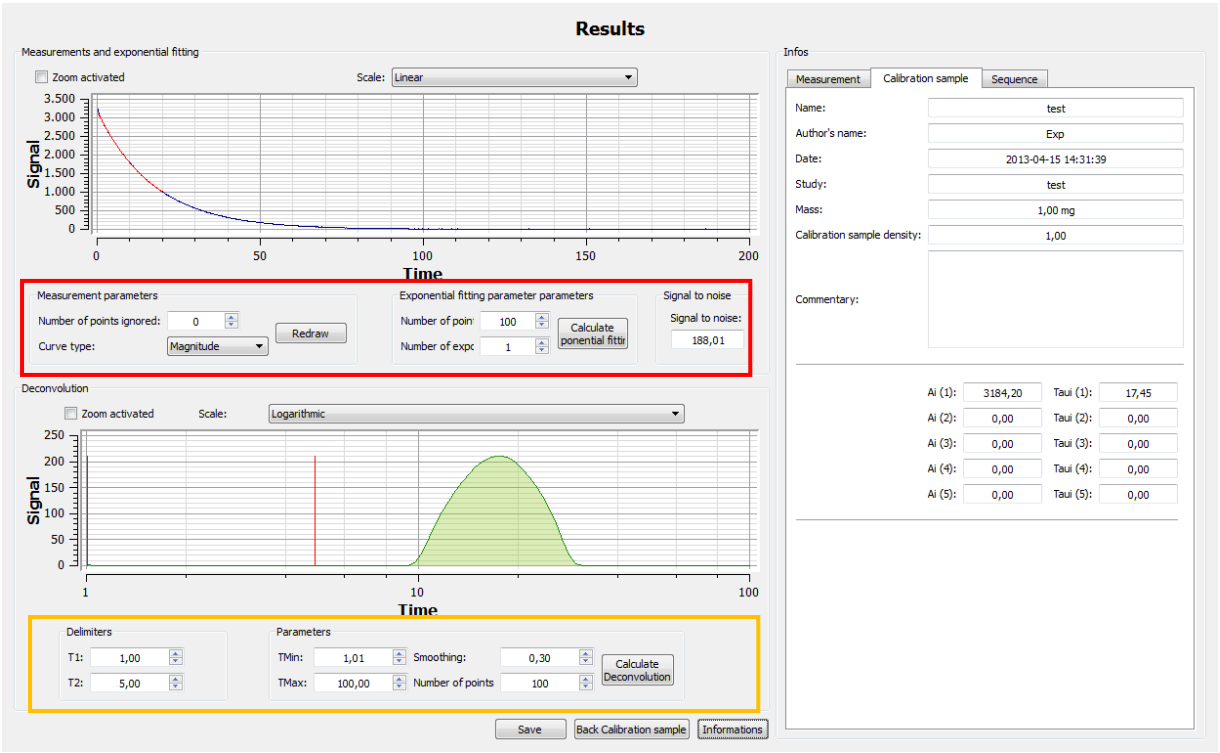
Density of the liquid's saturation: Density of the liquid having served to saturate the sample.

Comment: This field can be left blank or contain the information that the user considers as useful.

To initiate the creation of the standard, click on



After the measurement is carried out, the following window appears.



On this result window, it is possible to recalculate exponential and deconvolution processing on measurement. Two display modes are available for each curve, the time scale can be either linear or logarithmic.

It is possible to show or hide the information of the measure. This panel contains information about the sequence and the calibration.

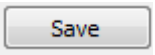
Measurements and exponential processing setting

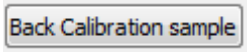
It is possible to display different curves from the measurement: absolute value, rephase or noise (power factor correction following the imaginary). This menu allows the deletion of point at beginning of the curve.

It is also possible to modify the multi-exponential calculation parameters. These parameters are the number of points to use (these points are systematically taken early in the curve) and the number of exponentials to calculate.

Deconvolution setting

It is possible from this mode to verify the proper calculation of deconvolution with the result of the calibration. If necessary, it is possible to test a modification of start and end of the calculation, the number of calculation time and the curve smoothing.

If the standard is correct, Store it by clicking on 

otherwise come back to the settings window by 

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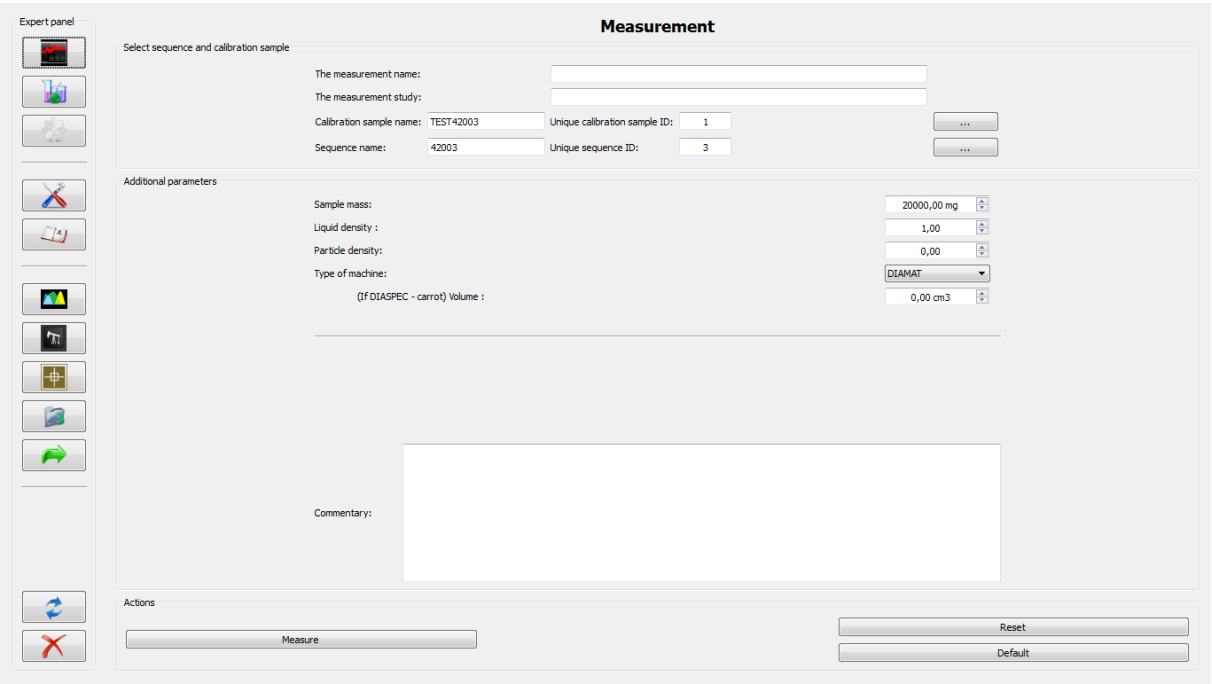
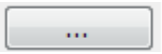
New measurement



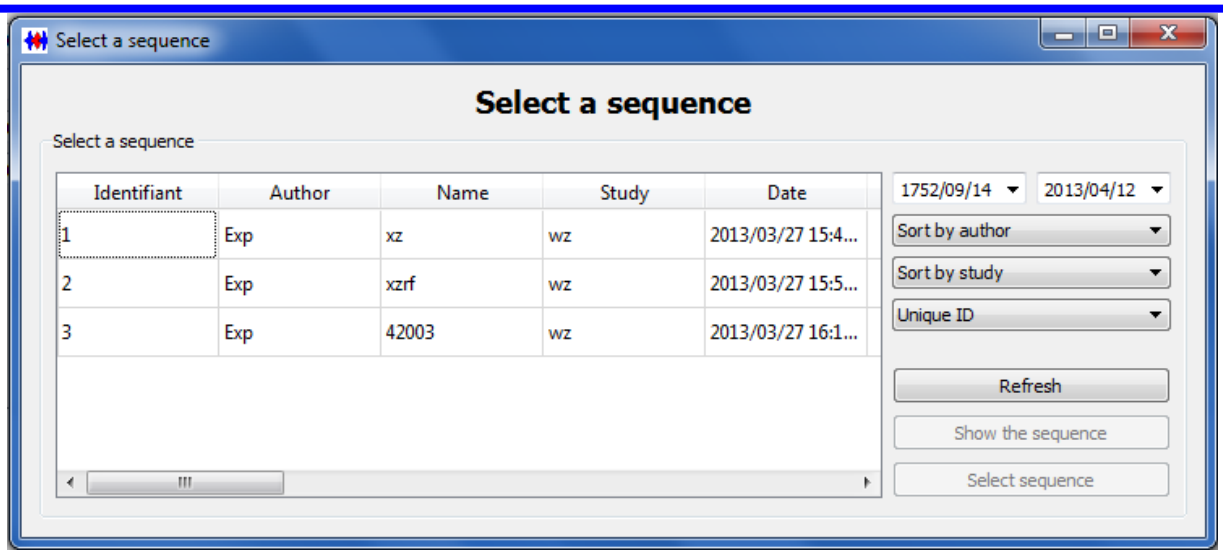
This window allows to fill parameters for the creation of a new sequence. The parameters are as follows:

Measurement name : This field contains the name of the measurement. This name must be unique.

Study: Fill in the study name the measurement was created for. Several measurement may belong to the same study.

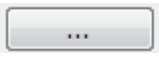
Sequence selection: This field contains the name of the sequence to use. To change the sequence to use, click 

. A selection window appears. This lists all the sequences in the database.

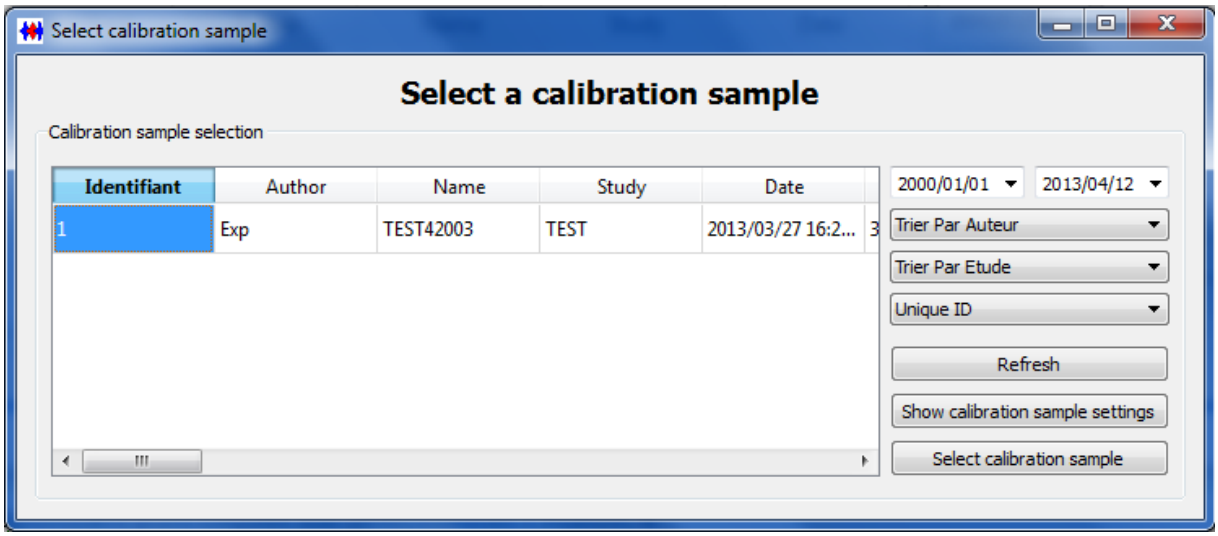


To select the sequence, click on the corresponding ID (the integer, highlighted on the image) then click on select the sequence.

It is possible to use the filters on the right to simplify the search for the desired sequence.

Calibration sample : This field contains the name of the calibration sample to use. To change the calibration sample to use, click 

. A selection window appears. This lists all the calibration sample in the database.



To select the calibration sample, click on the corresponding ID (the integer, highlighted on the image) then click on select calibration sample.

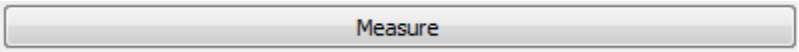
It is possible to use the filters on the right to simplify the search for the desired calibration sample.

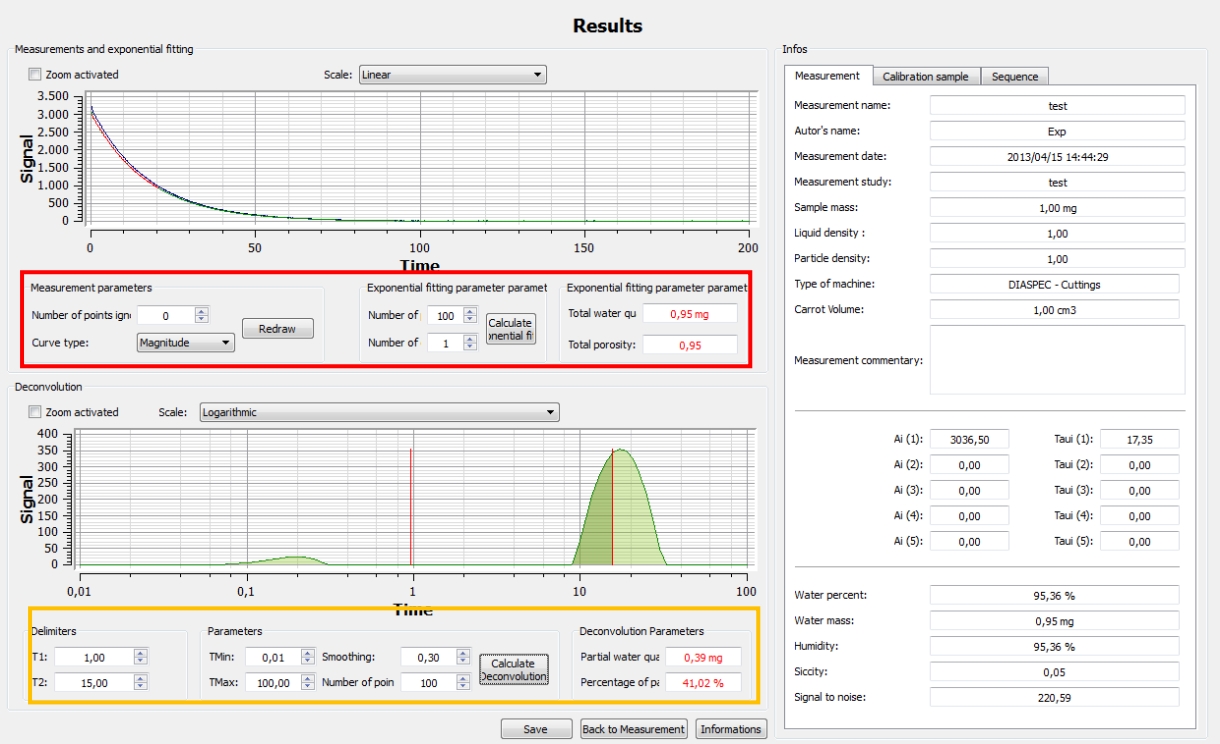
Sample mass : Fill in the precise weights of the standard. This value will be used for calculation of moisture

Liquid density : Density of liquid used in the saturation of the sample.

Particle density: Granularity of the sample in the case of measurements on "cuttings" (approx.2.7). For measurements on other sample types, this value has no importance but must be> 0.

Comment: This field may be left blank or contain information that the user considers useful.

To start the measurement, click 



On this result window, it is possible to recalculate treatments and exponential deconvolution of the measurement. Two display modes are available for each curve, the time scale can be either linear or logarithmic.

It is possible to show or hide the information from the measurement. This section contains information about the sequence and the standard achieved. More he finds the results of calculations on the measurement.

Measurement and exponential processing setting

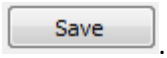
It is possible to display different curves from the measurement: absolute value, rephase or noise (angle correction following the imaginary). This menu allows the deletion of point at beginning of the curve.

It is also possible to modify the multi-exponential calculation parameters. These parameters are the number of points to use (these points are systematically taken early in the curve) and the number of exponentials to calculate.

Deconvolution setting

It is possible from this mode to verify the proper calculation of deconvolution with the result of the calibration. If necessary, it is possible to test a modification of start and end of the calculation, the number of calculation time and the curve smoothing.

Furthermore, it is possible to use the delimiter function to know the partial porosity of the sample at an interval deconvolution (part of the curve in dark green).

Measurement can be saved by clicking on 

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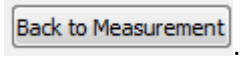
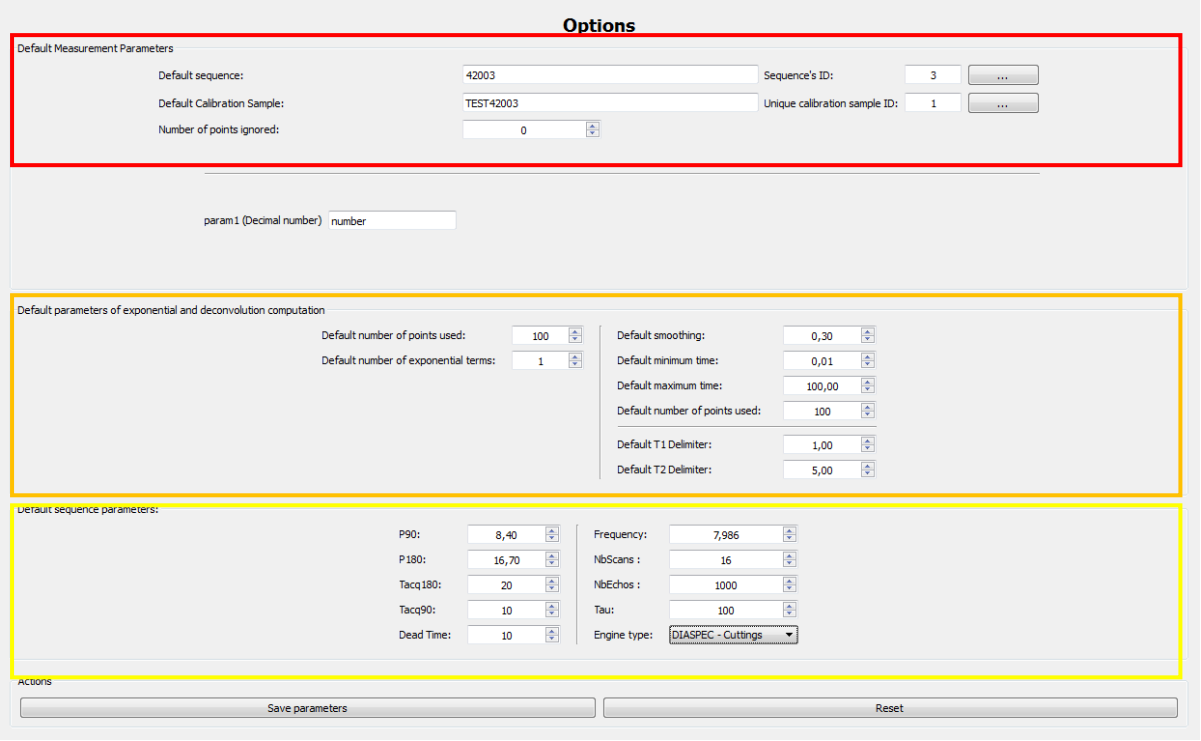
To do another measurement, use 

. Warning: if the previous measurement has not been saved, it will be lost.

Options



Use this page to change the default settings. There are three groups of settings.

Measurement settings

Fill in this block the settings to used by default for measurement: the sequence and the calibration.

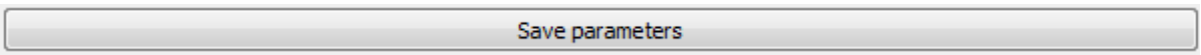
Processing settings

These are the parameters defined by the expert. At the return of measurement, multi-exponential and deconvolution processing shall be performed with these settings. These parameters will be used for the calculations of operator mode.

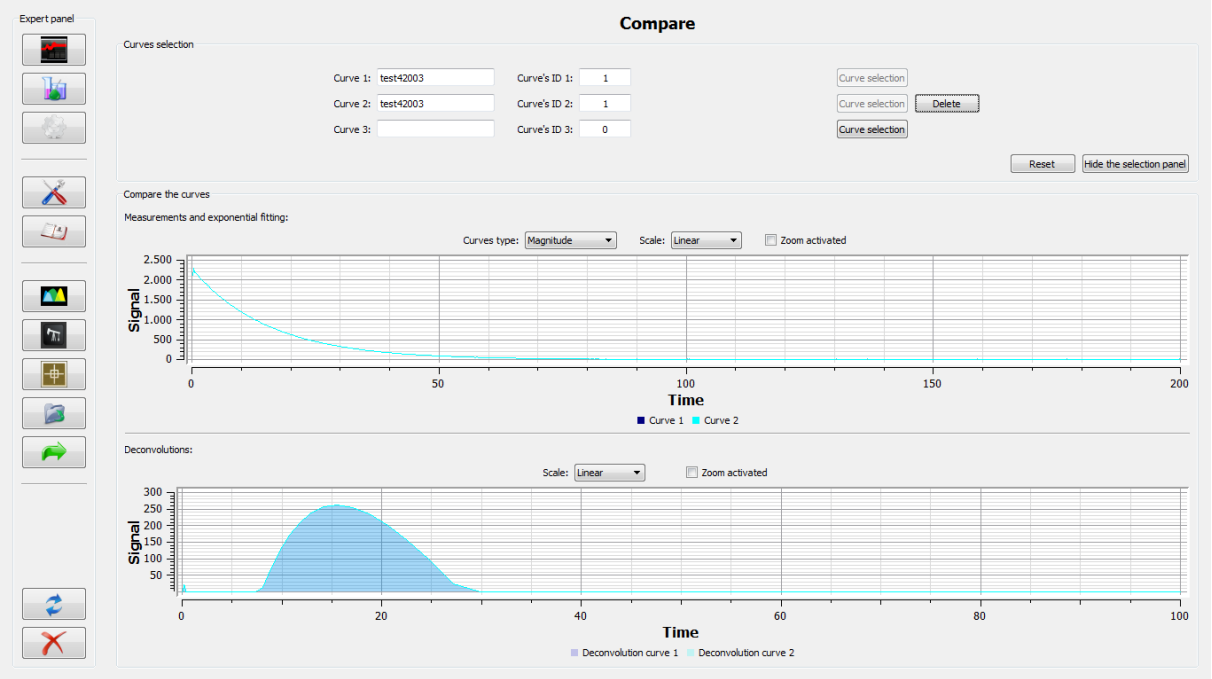
Sequence parameters

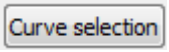
These settings are loaded by default for creating sequence. They correspond to the optimal sequence parameters for the machine.

These settings are saved by pressing



Curve compare

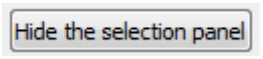


This mode provides a graphical comparison of several curves. To add a curve, click 

. It is then possible to remove the last added curve 

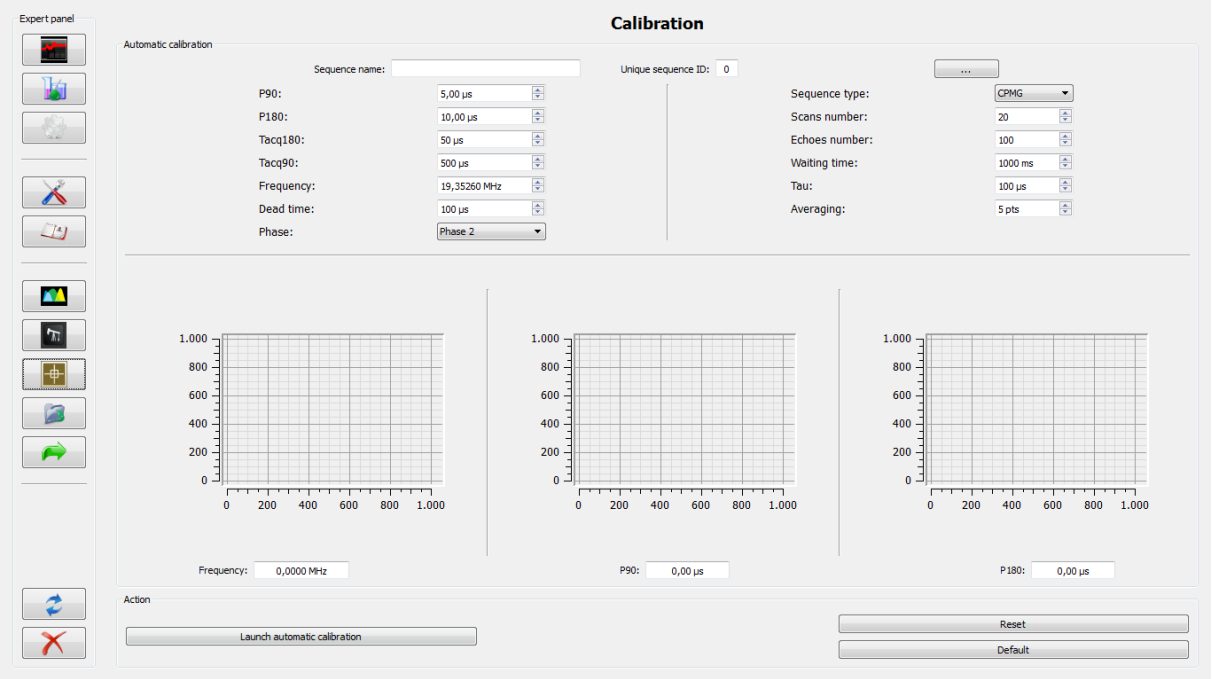
or all curves presents 

.

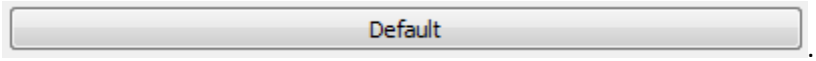
Button 

reduces the area occupied by selection to increase The area covered by the display of curves.

Automatic calibration

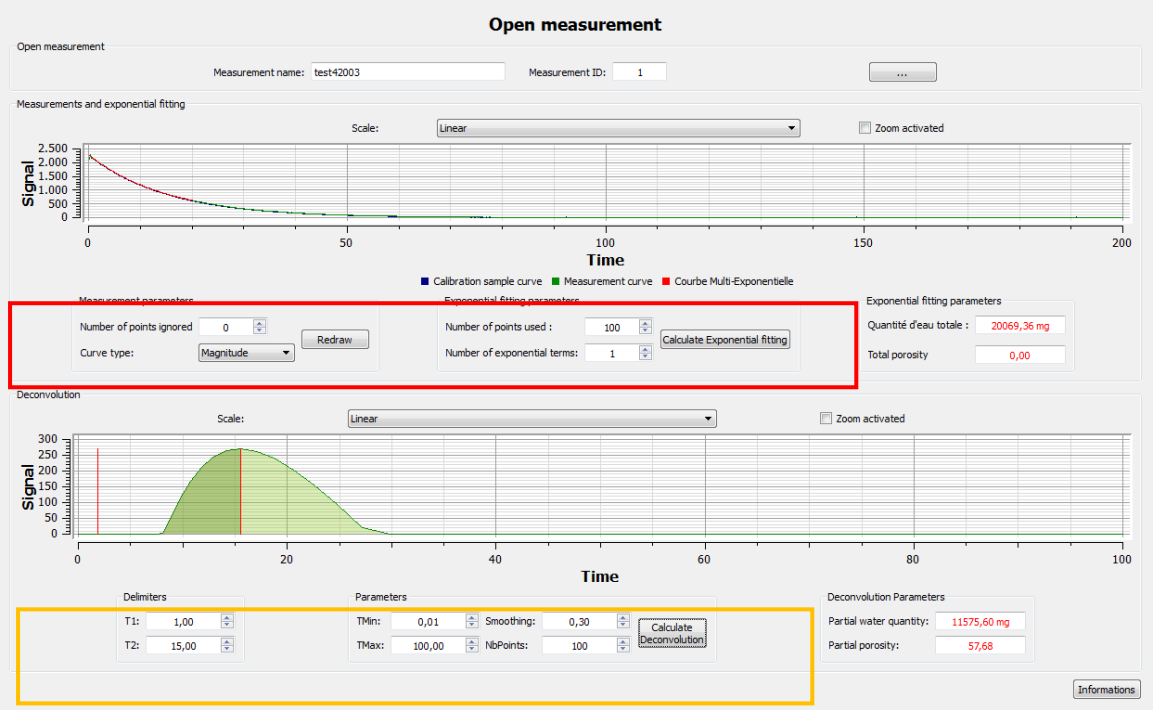


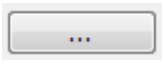
The automatic calibration tool allows a search of the optimal parameters of the measurement. The calibration is carried out in three phases: searching for the resonant frequency of the device, then search for P90 and P180 research.

To start a calibration, enter the parameters of the sequence near optimal settings. It is possible for simplicity choose a sequence database  or use the default settings  .

Caution, is recommended for the successful completion of the calibration, using a sufficient number of scans (at least 20), a sample wet enough and low number of echoes (one hundred). Once the calibration is complete, a window appears providing the backup settings as default settings found. These settings can be changed later (see Expert Options).

Open measurement



This window allows the reopening of a measurement database. To select the measurement, click  .

It is possible to recalculate exponential and deconvolution processing of the measurement. Two display modes are available for each curve, the time scale can be either linear or logarithmic.

It is possible to show or hide the information from the measurement. This section contains information about the sequence and the standard achieved. Moreover, he finds the results of calculations on the measurement.

Measurment and exponential processing setting

It is possible to display different curves from the measurement: absolute value, rephase or noise (power factor correction following the imaginary). This menu allows the deletion of points at beginning of the curve.

It is also possible to modify the multi-exponential calculation parameters. These parameters are the number of points to use (these points are systematically taken early in the curve) and the number of exponentials to calculate.

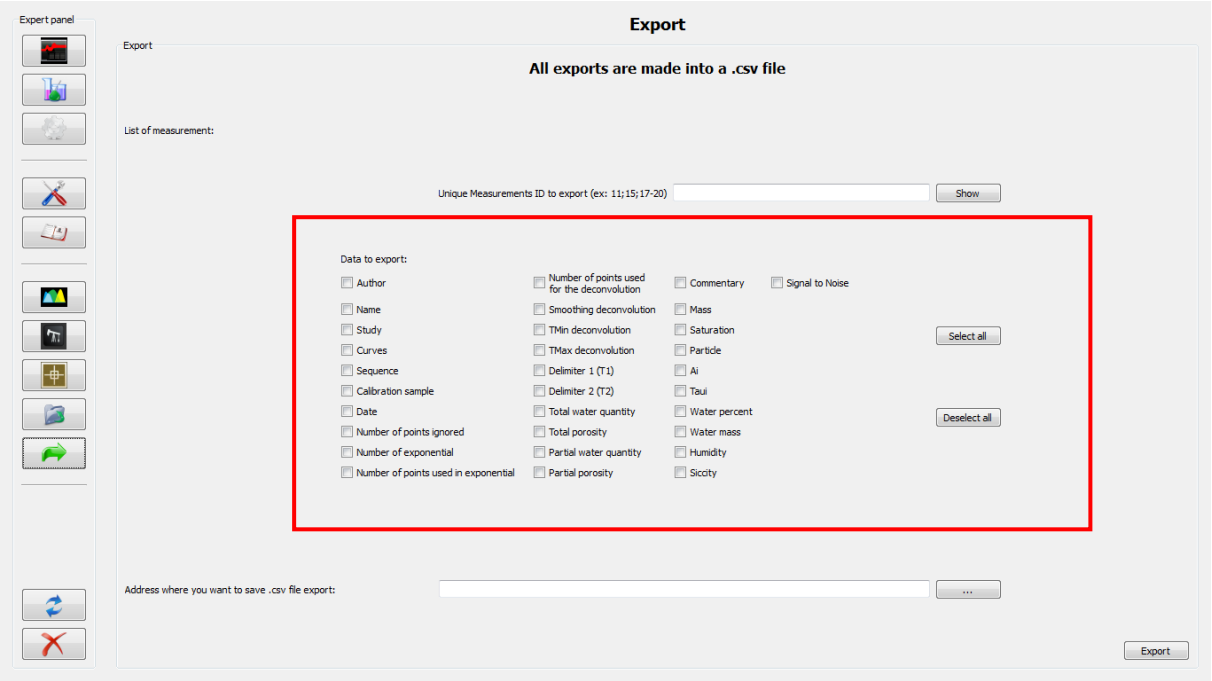
Deconvolution setting

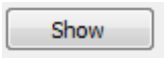
It is possible from this mode to verify the proper calculation of deconvolution with the result of the calibration. If necessary, it is possible to test a modification of start and end of the calculation, the number of calculation time and the curve smoothing.

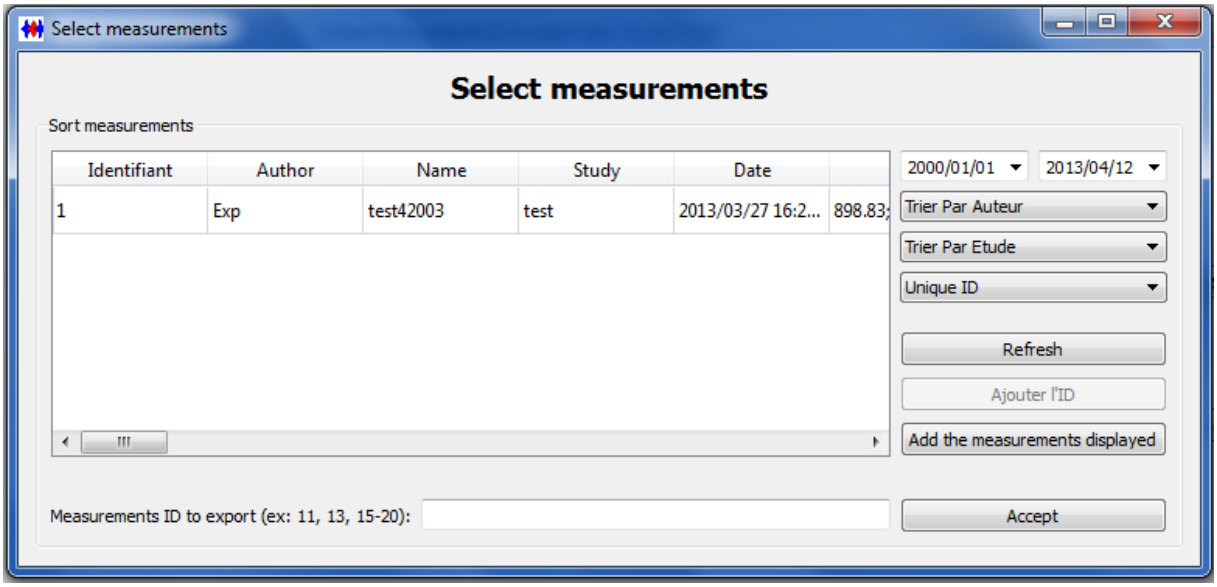
In addition it is possible to use the delimiter to know the partial porosity of the sample at an interval deconvolution (part of the curve in dark green).

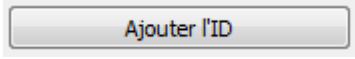
Information about these calculations are shown in the Information panel.

Export



This function allows you to export to CSV of a part or all the information relating to a measure in the database. Select the measurement to export using 



Select the measurement ID and click 

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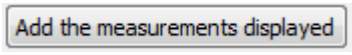
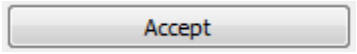
It is possible to add multiple measures. In addition the use of the filters allows the selection of a measurement assembly with the same properties. After selecting the filter (date, author, study) click 

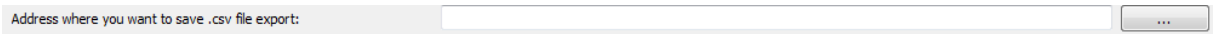
.

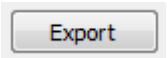
When all measurements are selected, click 

to validate the choice.

Select the area indicated by a red strap which attributes to export measures.

Choose a directory for the extraction:

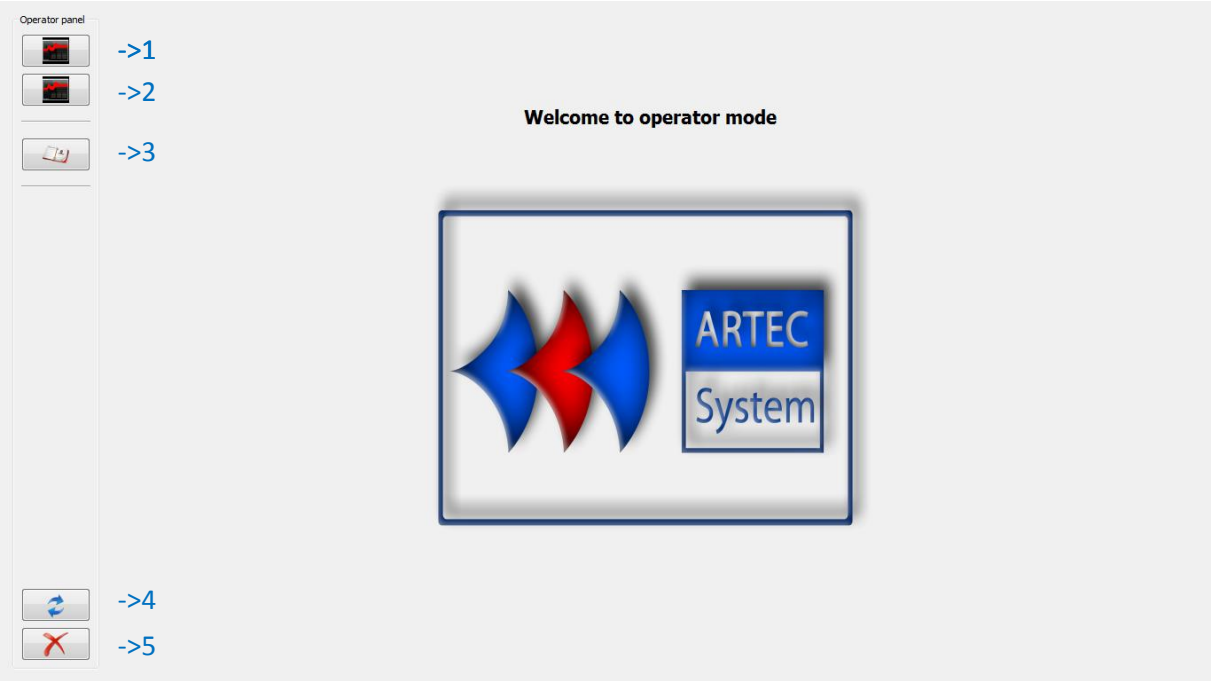


CSV files will be created in the desired destination by clicking on 

.

3. Operator mode

There is one default operator identifier implemented in the software (username and password Ope). Administrator can create other operator mode access according the operator users.

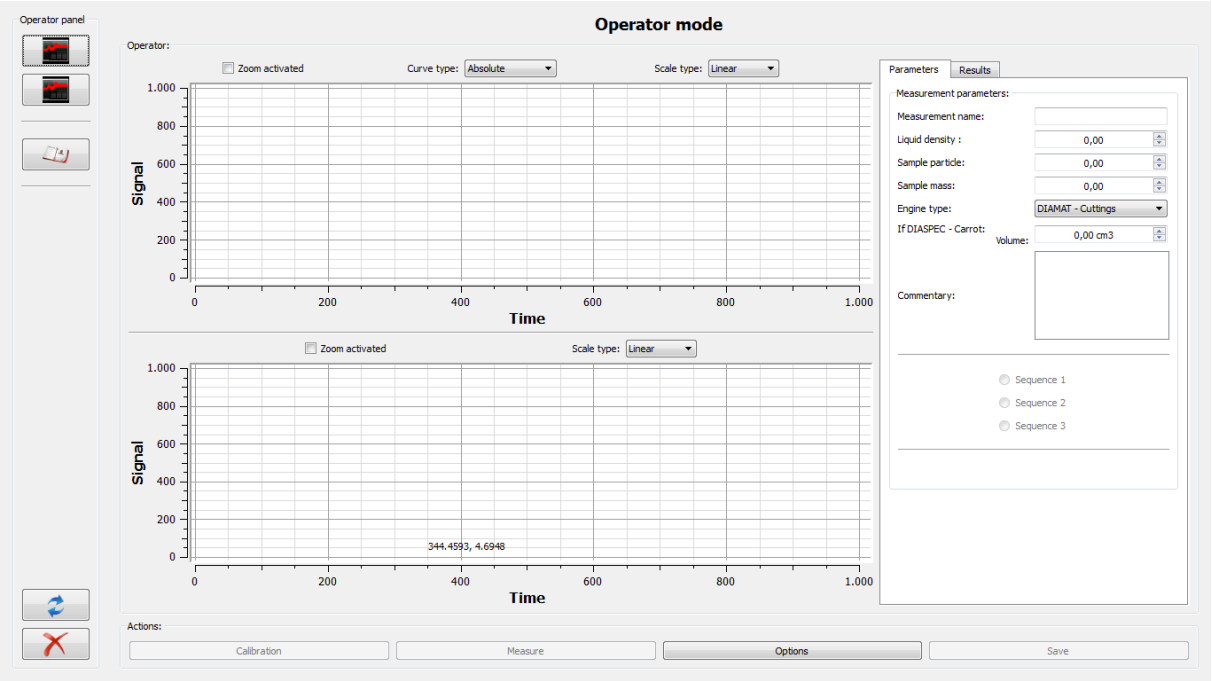


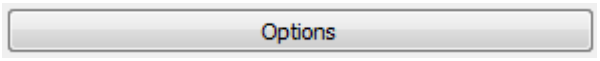
Depending on customer options, some of these features may be inaccessible.

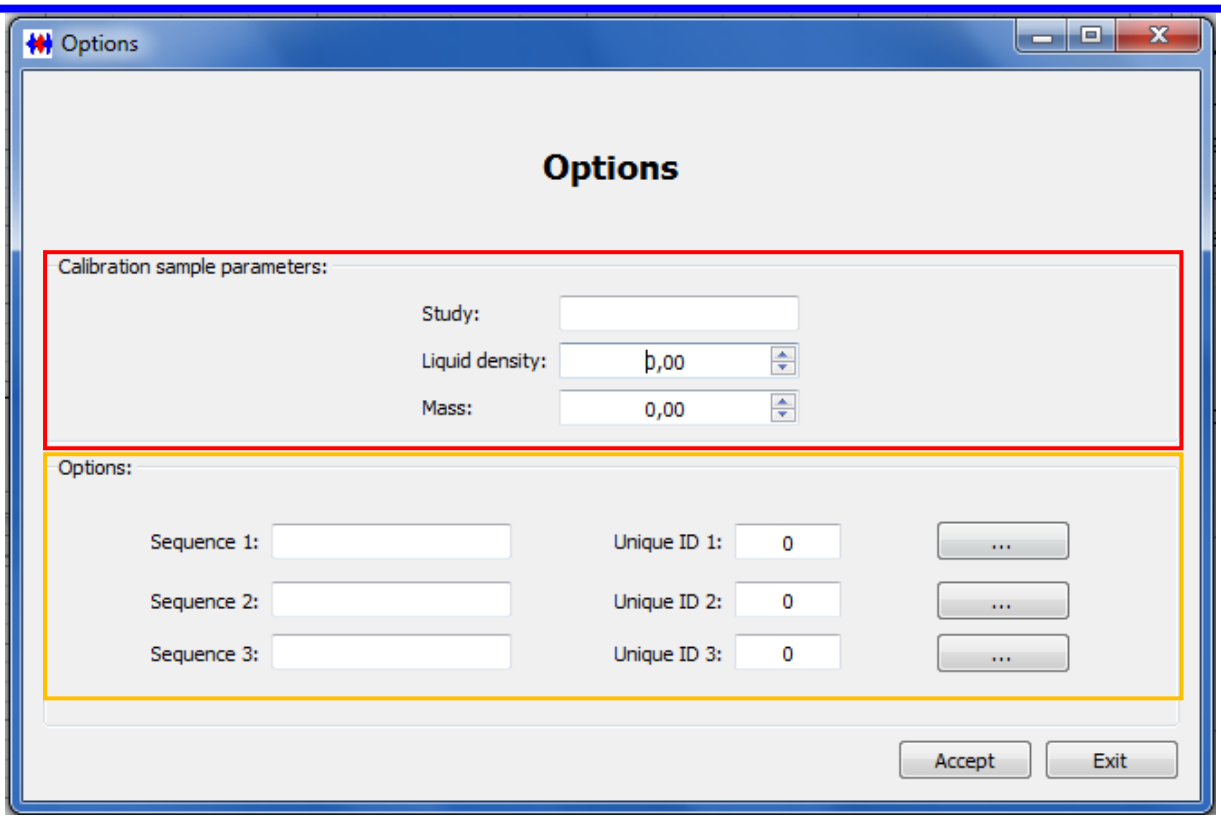
1. Operator mode
2. Porosity mode
3. Summary
4. Disconnection
5. Closing the application

Operator mode

Operator mode allows quick and easy access to the NMR measurements.



Fill options. Open the settings panel by clicking 



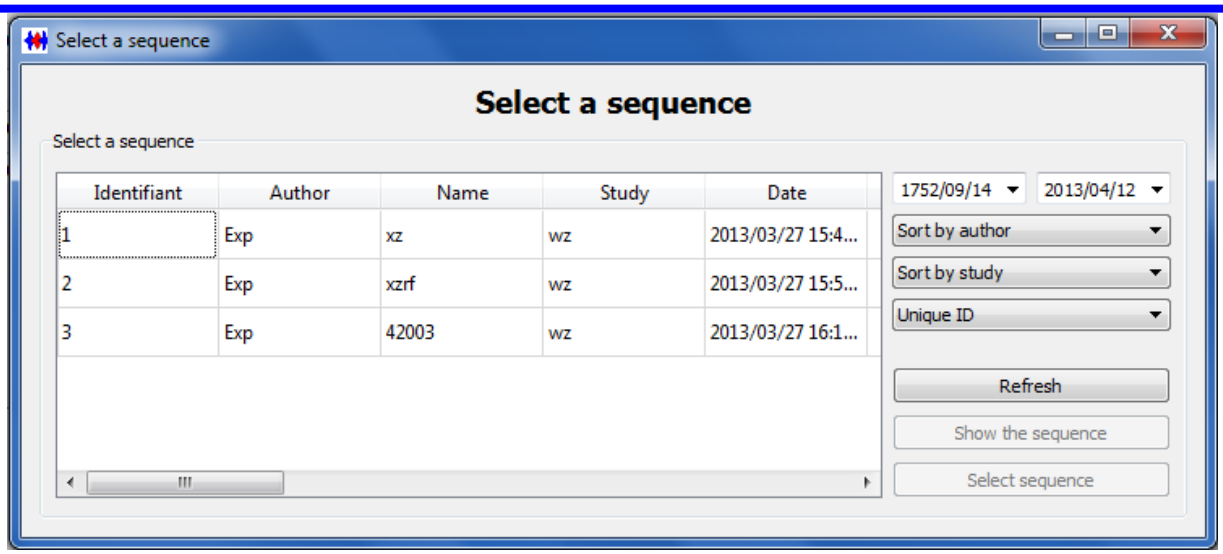
Calibration settings

To achieve a proper calibration, it is important to fill the mass of the sample used for calibration and its density.

Sequence choice

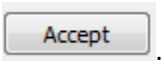
Operator mode provides quick access to three different sequences (though it is possible to use only one). Selecting sequences thereby by pressing the button 

.



To select the sequence, click on the corresponding ID (the integer, highlighted on the image) corresponding then click on select the sequence.

It is possible to use the filters on the right to simplify the search for the desired sequence.

When all parameters are filled (one sequence is sufficient), validate options by 

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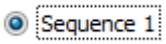
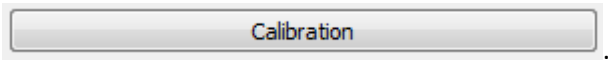
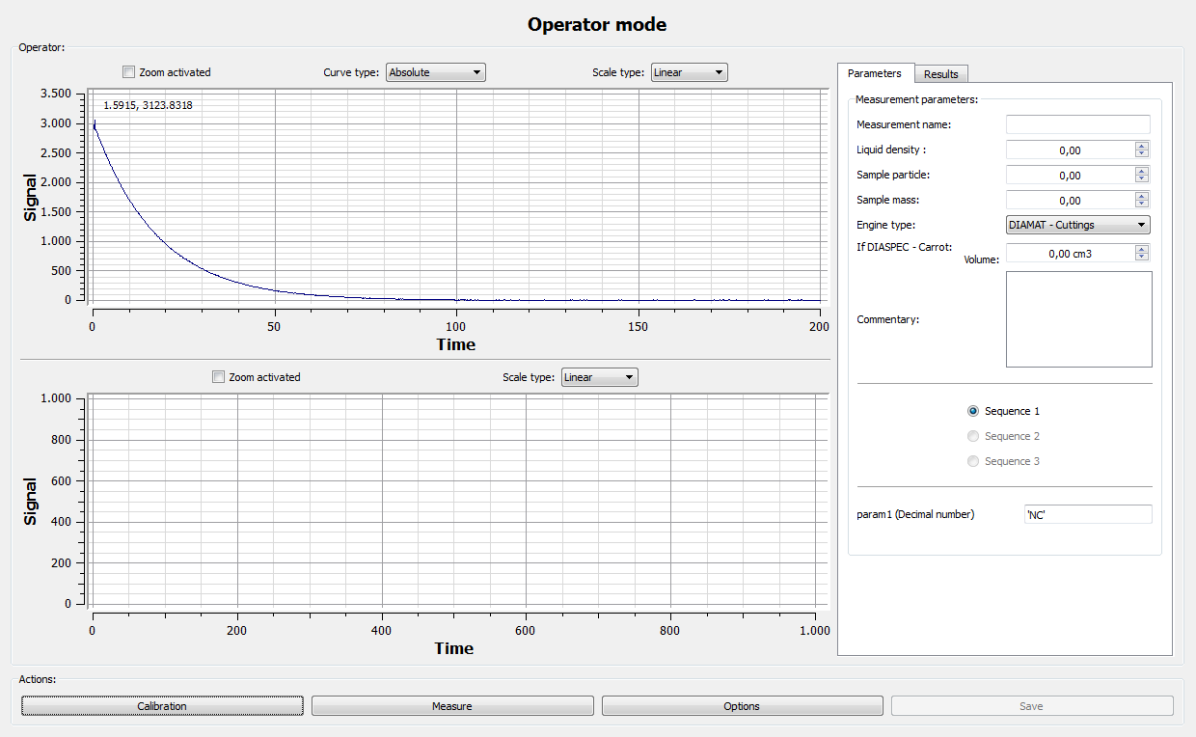
In the Parameters tab on the right, choose a sequence 

.

Launch calibration 

.



If the calibration is correct, it is possible to make a measurement. Fill the parameters related to the sample:

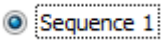
Measurement Name: The name of the measurement will be automatically filled to each measurement.

Liquid density : Density of the liquid having used for the saturation of the sample.

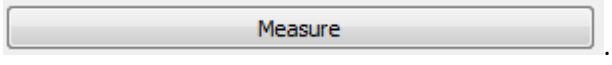
Sample particle : Granularity of the sample in the case of measurements on "cuttings" (approx .2.7). For measurements on other sample types, this value has no importance but must be> 0.

Sample mass: Total mass of the sample for the calculation of moisture.

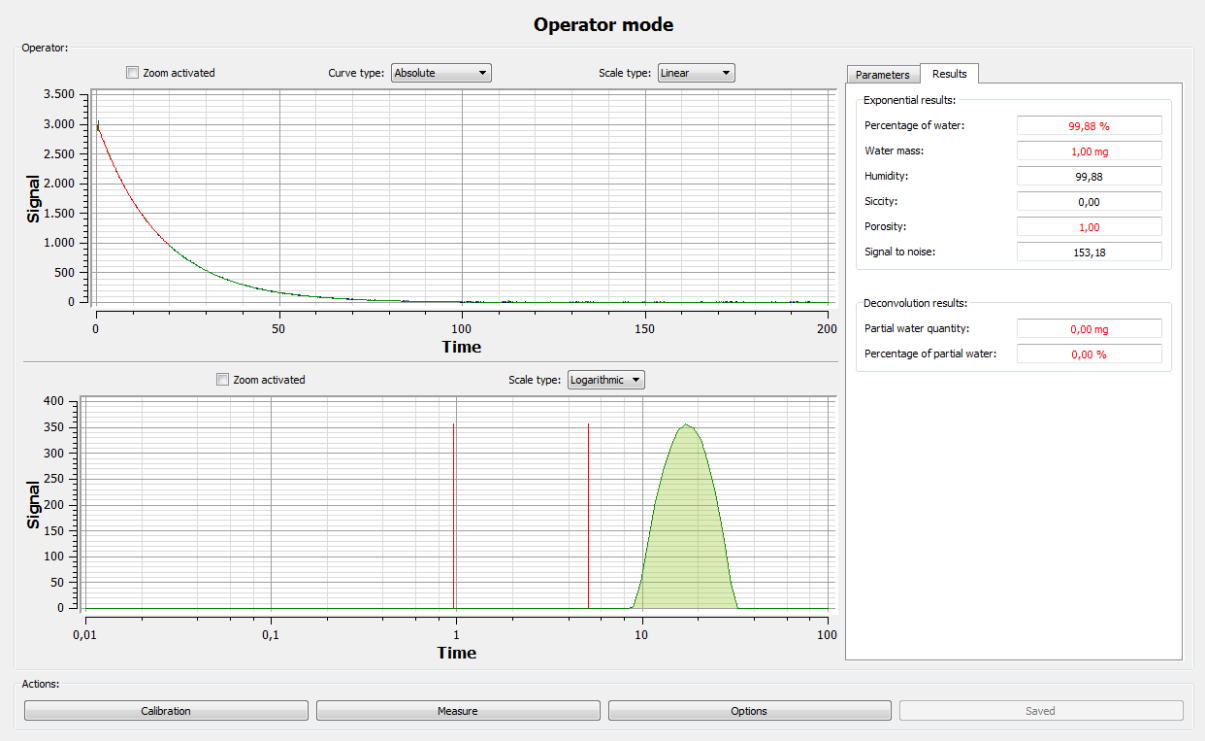
Machine type: Choose DIASPEC-Cuttings for DIASPEC 18.

Choose a sequence 

.

Launch measurement 

.



At the end of the measurement, the calculations are done automatically according to the parameters specified in the expert options. Calculation results are displayed in the right panel.

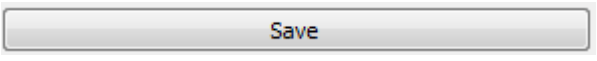
Percentage of water: Water mass in the sample relative to the calibration.

Water mass: Mass of water present in the sample.

Humidity: Relative humidity of the sample

Partial water quantity: Water quantity shown between the delimiters deconvolution (dark green).

Percentage of partial water: Ratio of the amount of water between the delimiters relative to the total quantity of water.

The measure must be saved by pressing 

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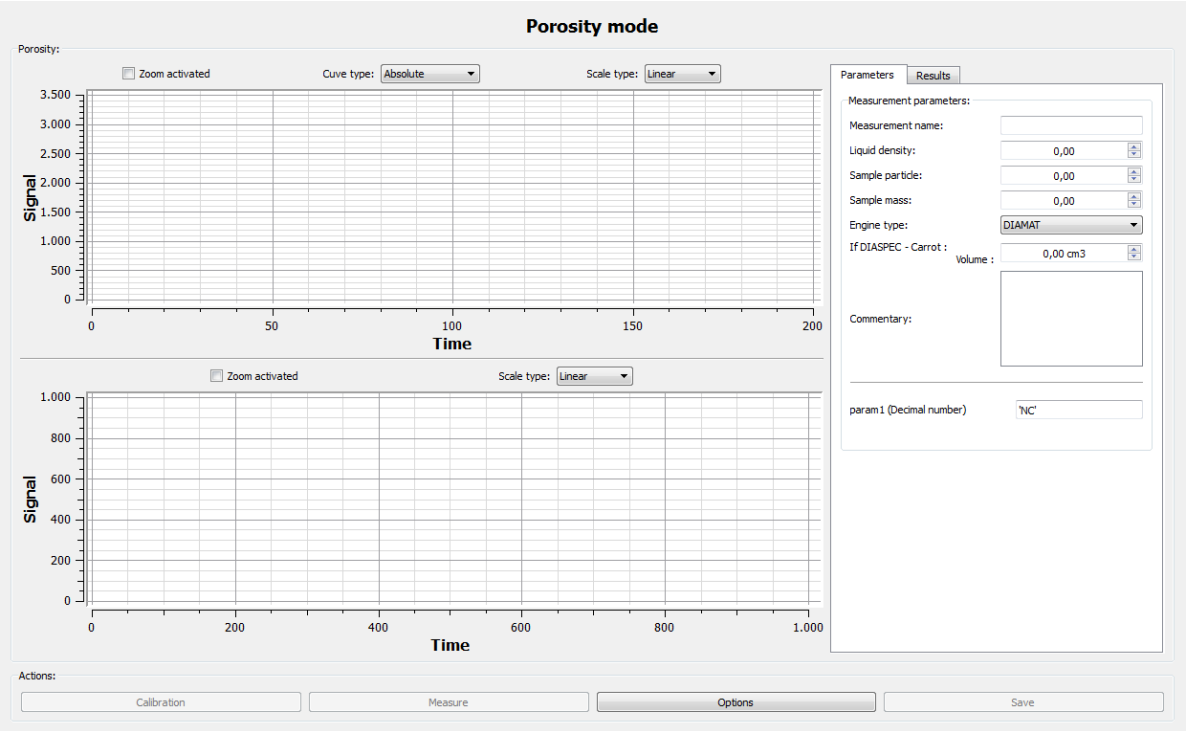
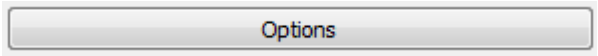
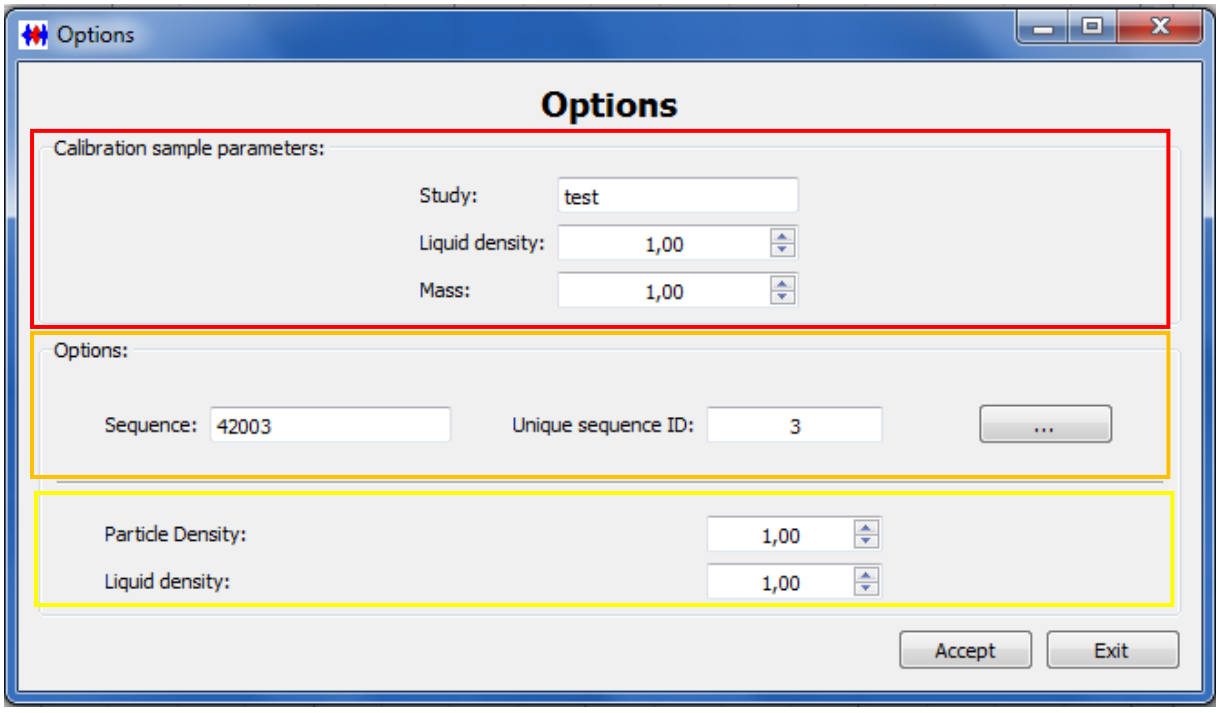
SARL au cap de 75.000 euros. Siège social : 45 Avenue Rhin Danube-07100 Annonay France
Tel : 33 (0) 4 75 69 22 21 Fax :33 (0) 4 75 69 22 19 Mail : info@artec-system.com
RCS Annonay 505 395 293 – Banque Société Générale Annonay : 30003 02161 00020419085 20

Porosity mode

Porosity mode allows quick and easy access to porosity measurements



Fill options. Open the settings panel by clicking 



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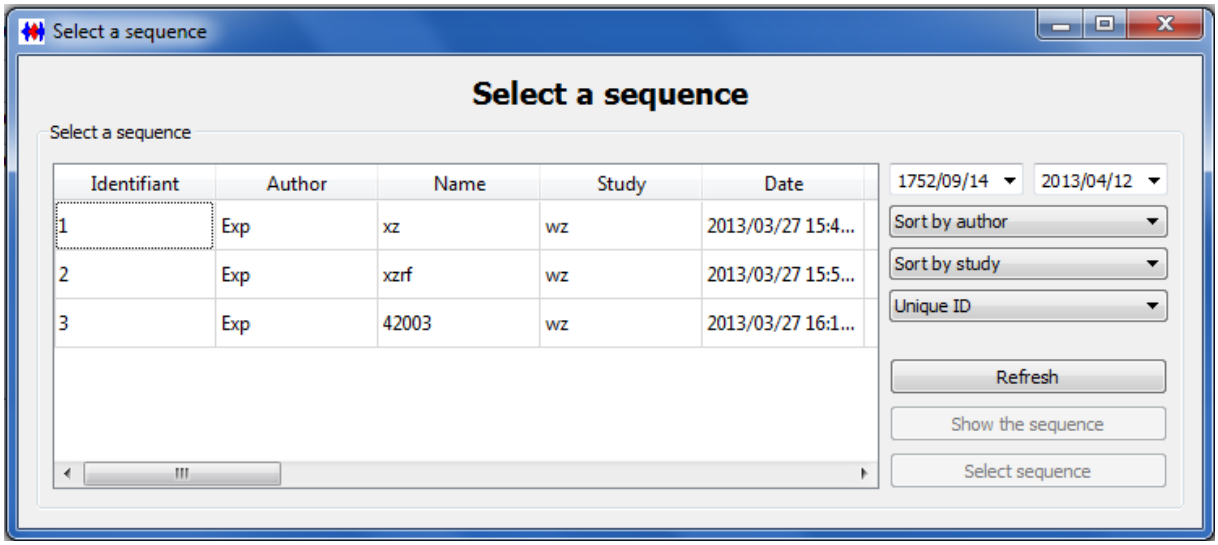
RCS Annonay 505 395 293 – Banque Société Générale Annonay : 30003 02161 00020419085 20

Calibration settings

To achieve a proper calibration, it is important to fill the mass of the sample used for calibration and its density.

Sequence choice

Operator mode provides quick access to three different sequences (though it is possible to use only one). Selecting sequences thereby by pressing the button ...



To select the sequence you want, click on the corresponding ID (the integer, highlighted on the image) then click on select the sequence.

It is possible to use the filters on the right to simplify the search for the desired sequence.

Validate by clicking Select sequence.

Click on Accept for validate the options.

Launch calibration Calibration.

Fill the parameters related to the sample:

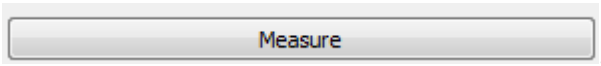
Measurement Name: The name of the measurement will be automatically filled to the measurement.

Liquid density : Density of the liquid having served for the saturation of the sample.

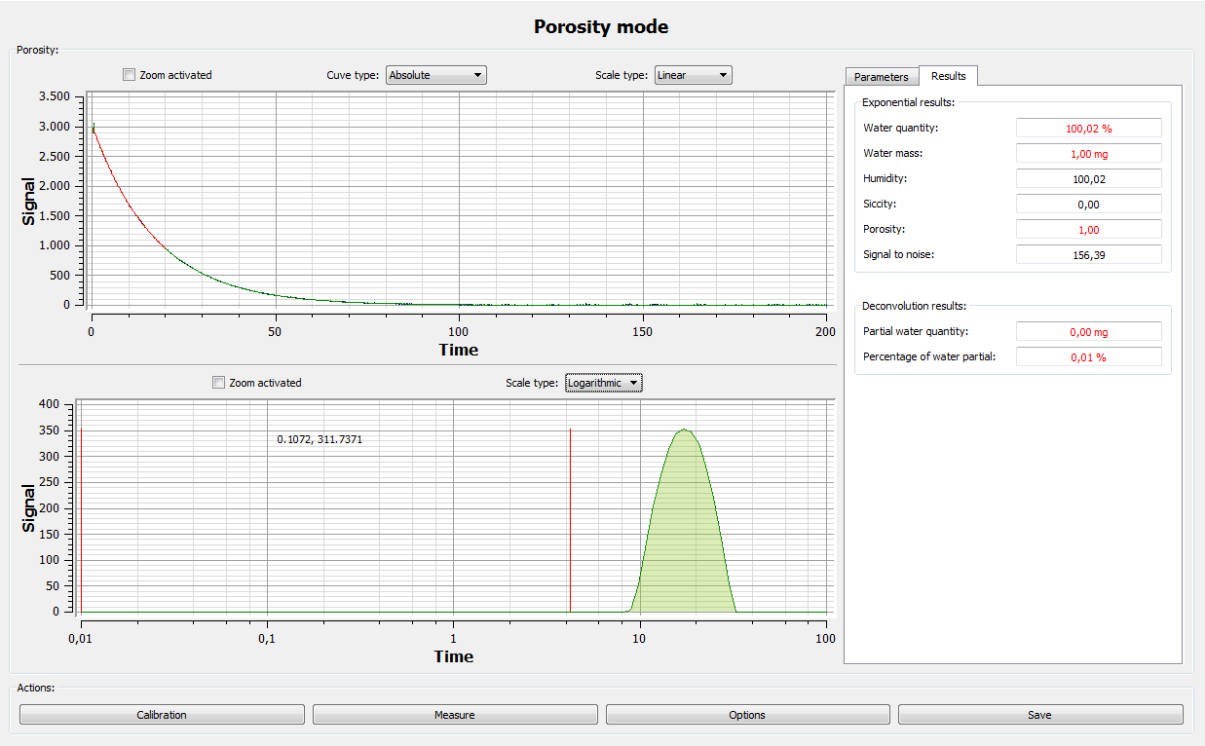
Sample particle : Granularity of the sample in the case of measurements on "cuttings" (approx.2.7). For measurements on other sample types, this value has no importance but must be> 0.

Sample mass: Total mass of the sample for the calculation of moisture.

Machine type: Choose DIASPEC-Cuttings for DIASPEC 18.

Start measurement 

.



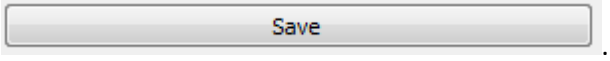
Percentage of water: Water mass in the sample relative to the calibration.

Water mass: Mass of water present in the sample.

Humidity: Relative humidity of the sample

Partial water quantity: Water quantity shown between the delimiters deconvolution (dark green).

Percentage of partial water: Ratio of the amount of water between the delimiters relative to the total quantity of water.

The measure must be saved by pressing 

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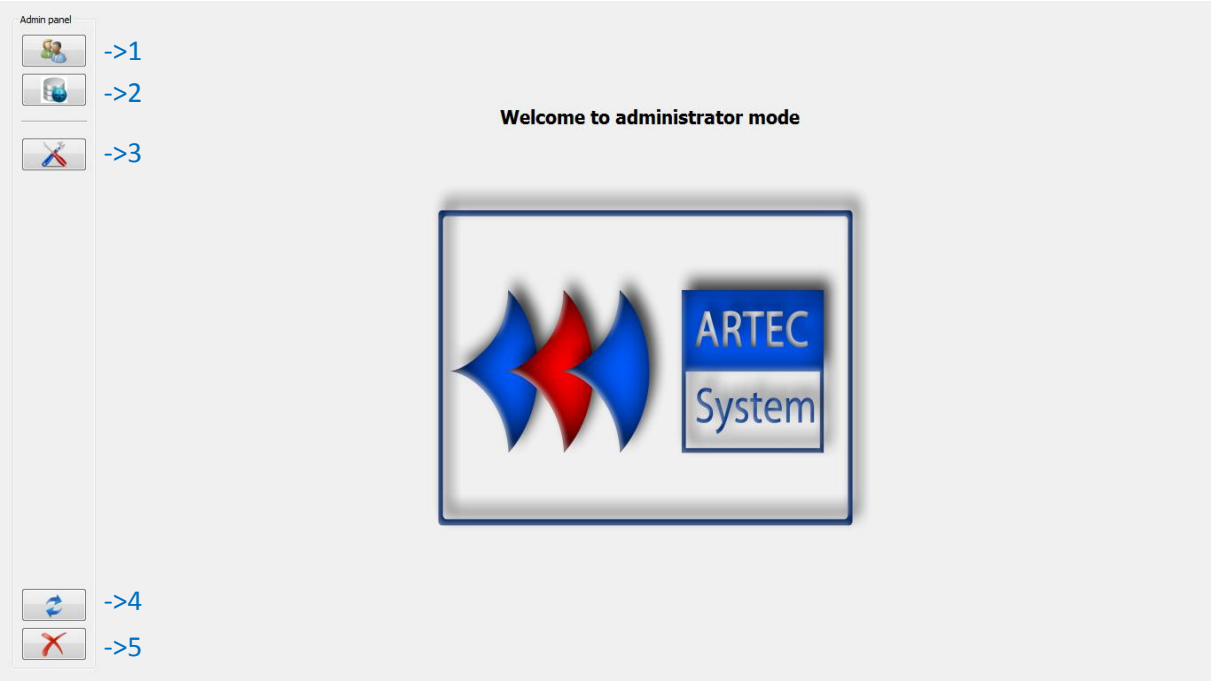
4. Administrator mode

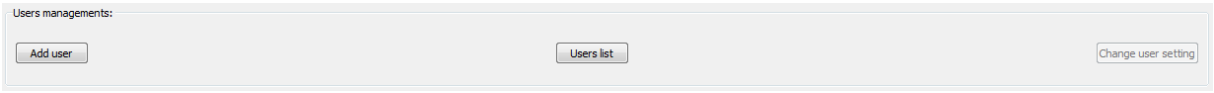
Administrator mode allows database and users managing. A default identifier is available (username and password: Admin).



The following functions are available :

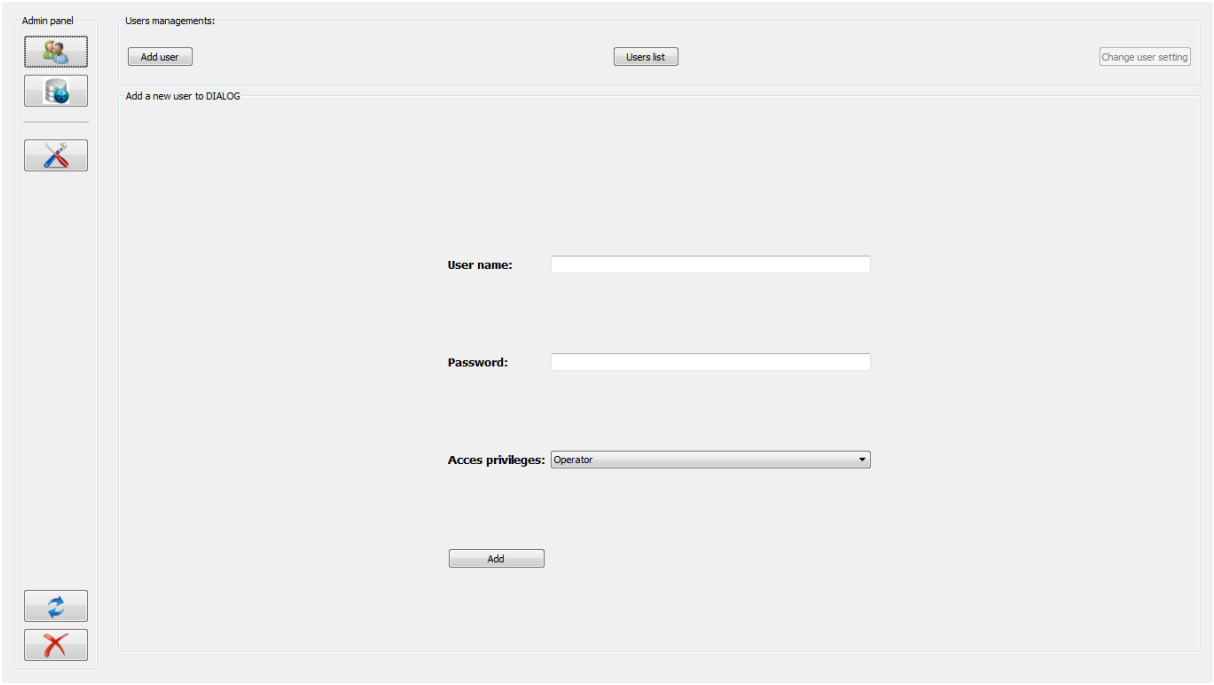
1. User manager
2. Database manager
3. Change options
4. Disconnection
5. Closing the application

User manager



Three actions are possible from this panel.

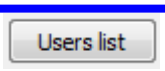
Add user 

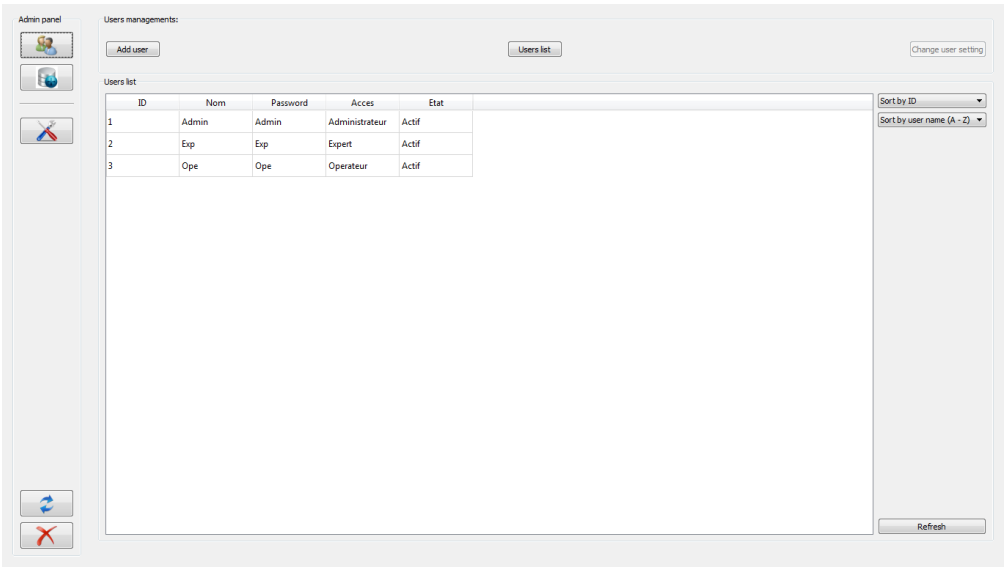


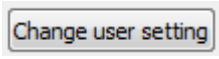
To add a user, you must fill its name, this will serve as an identifier, a password to protect the user account and an access level. Indeed, an account gives access only to a single mode (administrator, operator or expert).

Validate account by clicking on 

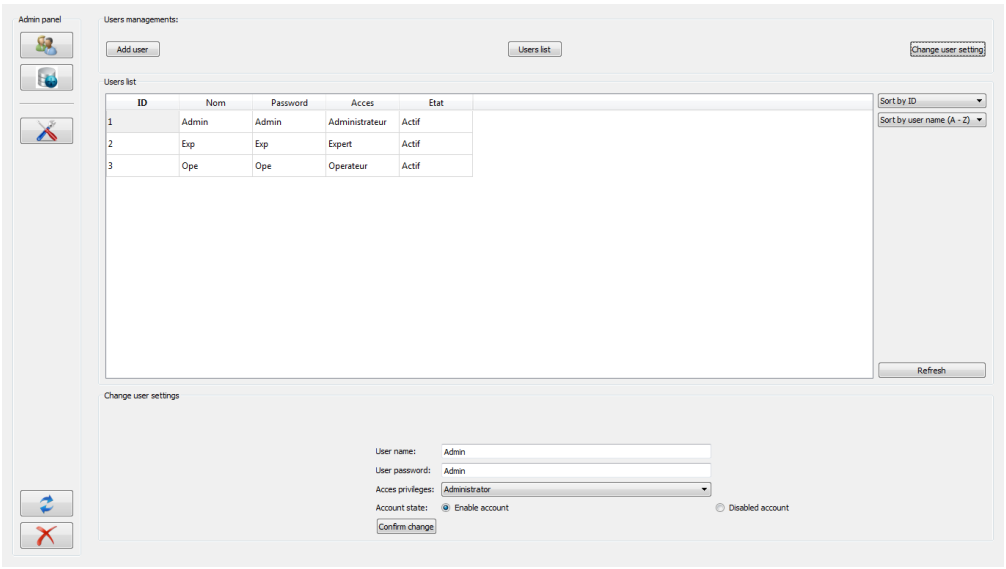
.

List user 

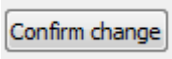


From this list, select a user by ID allows access to modify function 

.



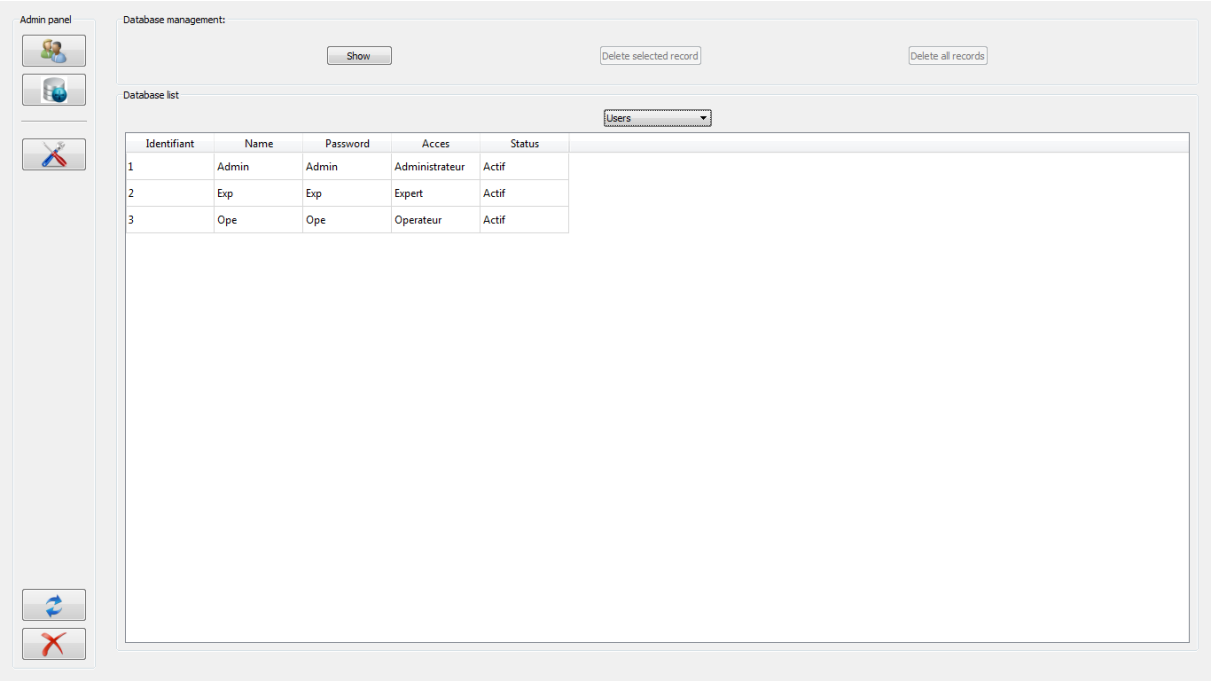
This function allows changing the selected user account. Change the password, type of access and the ability to disable the account.

The account will never be deleted, but if given inactive, the connection will be impossible. Changes are validated by clicking 

.

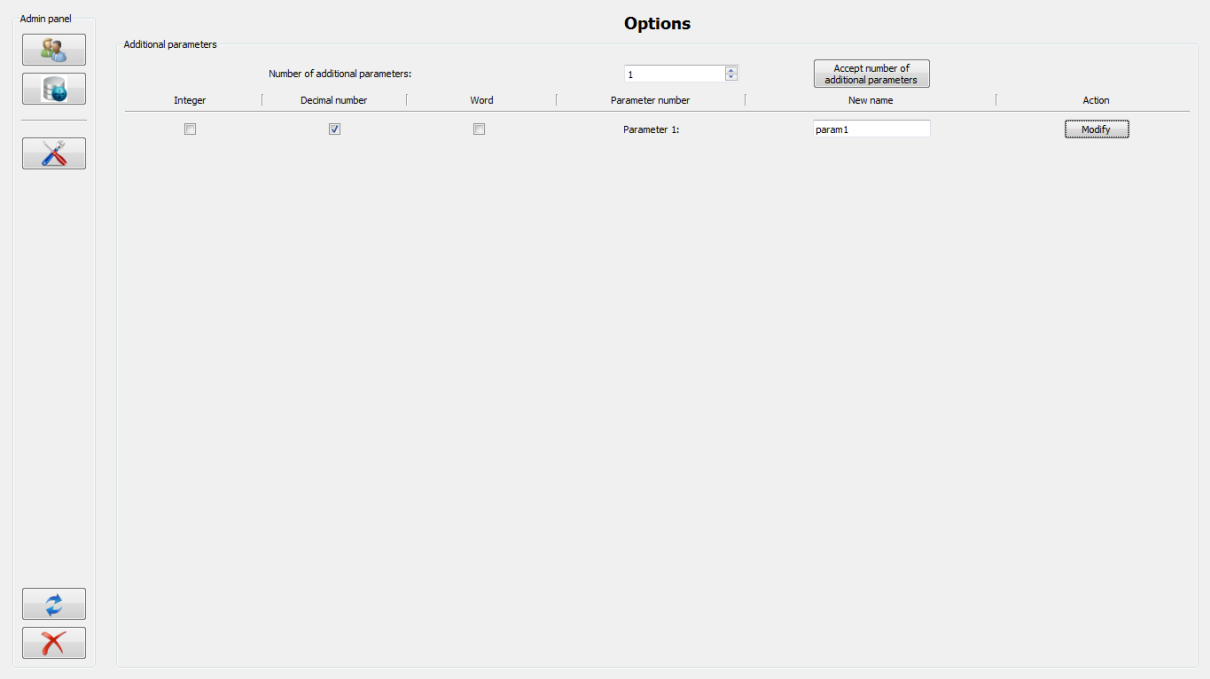
Database manager

It is possible from this menu to display software database.

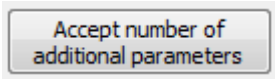


For some base (calibration, measurement and sequence) it is possible to delete the records one by one or emptying the entire database. Careful records are permanently deleted.

Options



This function allows you to add additional parameters to the measurements. The administrator informs the desired number of parameters and their types and names to display.

To add parameters, increment the number and click 

.

After information on the type and name parameters, validate its implementation by 

.

You need to fill the value of each parameter before launch a measurement.

The default value will be entered by the expert in the options menu.