SPIN RESONANCE MEASUREMENT SOFTWARE

User manual

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INTRODUCTION

The software is designed for Spin Resonance (SR) measurements (such as ESR, EDMR, ODMR, EDSR, etc.) using ESR spectrometer, PC with installed Windows'98, PCI-2024E model AD converter from "National instruments" (http://www.ni.com) and model 2010 gaussmeter from "Magnetic Instrumentation Inc" (http://www.maginst.com).

Output signal from a lock-in amplifier (build-in or additional) and an analog signal from magnetic field sweep (-5-0-+5V) are connected to the second and first input channels of the AD converter respectively. Signal from the gaussmeter is fed to a PC through serial (RS-232) interface. All application software related to the gaussmeter and the AD converter should be preliminary installed on the PC. For the details of the software and hardware installation see related manuals of the gaussmeter and the AD converter.

The main procedure of the present software gives possibility to register spin resonance related spectra on the PC and save measured data to the ASCII format file. Besides that the software gives possibility to monitor magnetic field intensity and related signal from the lock-in amplifier (LI) and make chronological measurements (timer measurements) of the signal from the LI. It is also possible to make high precision calibration of the magnetic field (H) related analog signal using spectrometric standard containing Mn^{+2} paramagnetic atoms, load previously measured SR spectra and compare it to that under investigation, print out measured spectra on the printer, balance zero level of the LI, change channel settings and amplification parameters of the AD converter, check H settings. Many measurement parameters can be changed and controlled by the software.

In the following we will present a procedure of standard SR measurement using the software and detailed explanations of possible regimes and functions.

STANDARD SPIN RESONANCE MEASUREMENTS

Prepare the ESR installation for the measurement: insert a sample, set desired temperature and microwave power, find resonator deep and switch on auto control of microwave frequency. You may also set desired value of the H, the LI amplification coefficient and phase and modulation strength at this stage.

- After starting the program, first check all connections and system functionality by clicking on "Signal monitor" button. If everything is connected correctly, you will see the H values (measured by gaussmeter) and signal values from both channels of AD converter on indicators positioned in "Current value and error monitor". Simultaneously, the progress bar below the spectra screen will indicate graphically current H value within the sweep range and the points indicating LI signal value will appear inside the spectra screen. If the level of the signal from LI ("Y signal, V") is out of "–1" – "+1" range (signal points are out of the screen in this case) use the LI level compensation to bring it close to zero. Alternatively, you can click "Balance Y" button to bring the signal value to the central-horizontal position of the screen.
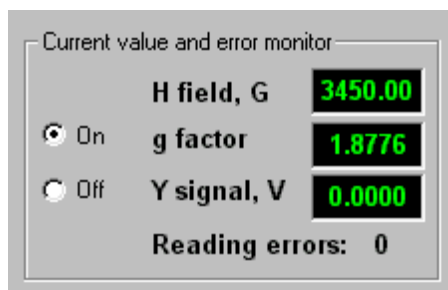
IMPORTANT: AD converter <u>input channel diapason</u> for default gain=1 is from +5 to -5V (for gain=0.5: from +10V to –10V; for gain=10 from +0.5V to –0.5V; for gain=100: from +0.05V to –0.05V). Thus out of this range the AD does not respond to the analog signal, giving same, extreme digital value. Thus, it is preferable to use zero-level position control of LI for general correction of the signal level. "Balance Y" button should be used generally for the small corrections of the signal level during measurement process.

- In the "Signal monitor" mode you can precisely set desired magnetic field central position and sweep diapason value, amplification coefficient and other parameters of installation, thus preparing all of equipment for the recording of SR spectra.

  Signal from LI appears on the spectra screen if option "Down" is checked inside the "Pen" frame. Clicking on the neighbor "Up" option stops Y signal output to the screen. You also can choose the "pen" color from the listbox. Click on the "Clear Memory" button to clear the screen.


IMPORTANT: Besides the erasure of the spectra screen, "Clear Memory" button sets to zero all previously measured Y(H) values. Thus, if you do not want to loose measured points be sure to save spectra to the file before clicking on the button.

- For your convenience you can place up to four visible markers (crosses) on the spectra screen. These markers are useful when you want to mark some position or line amplitude during monitoring. To set marker you need to double click on the desired position. Next double-click after the fourth one will cause the first marker to disappear and the new one will be placed at the place of the recent double-click. Markers reside even after "Clear Memory" command. You can remove all markers choosing "Hide markers" command from the buffer menu.

- Click the "Stop" button when all preparatory settings are finished. Fill in the text fields in the "Measurement details" frame. The values and comment printed in the text fields will be saved to an ASCII file together
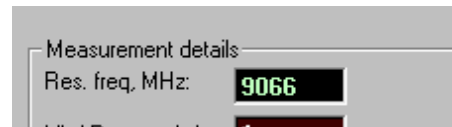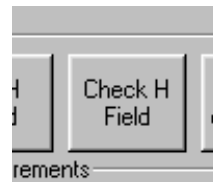

  with the measured data, when the "Save" command will be executed. Besides that the resonator frequency value ("Res. Freq, MHz") is used to calculate current value of the g-factor during the measurement and review of the data, thus, be sure to input the correct value in [MHz] from the frequency meter in the appropriate field.

- The magnetic field should be related to analog signal from the X channel of the ESR spectrometer prior to the SR measurements. Click on the "Set H field" button in the "main controls" screen. A message box will appear asking you to make field settings. At this stage you can make changes to the position of central H field and sweep diapason (if changes are not needed just click "OK"). The next message asks you to change field value within the sweep range and click on "OK" after.

__IMPORTANT:__ The first and the second positions for the H field settings can be any but the same within the sweep range – the H field is calibrated inside and outside the positions within the whole sweep range. For higher precision choose the end positions of desired measurement region within the sweep range for H calibration. Do not forget to make H calibration after each change of central H field position or/and sweep range (previous calibration is valid until the new one will be made). You can check the precision of current calibration by clicking on "Check H field" button for several positions inside the sweep range and make new calibration desired number of times. Clicking the "Check H field" button causes the message to appear, which compares actual H field (measured by gaussmeter) to the one calculated using the recent setting constants. The acceptable difference between the two values should not exceed 0.1% of the sweep range.

- On this stage you have also to choose the H field step between the data points of the forthcoming SR measurement. Click and change the step value [G] inside the "Hstep, G" frame, if desired.
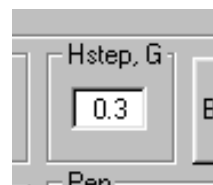
NOTICE: Although the value of the step is not limited, please keep in mind that the maximal number of points, which can be memorized by the software, is N_{MAX}=10000. Thus, the value of sweep diapason divided by the step value (N=sweep/step) should not exceed this number. Besides that, the precision of AD converter - sweep generator system

is limited. That means, that the N should not exceed 4000 for all data points to be reliably detected. These parameters are checked by the software while transferring to the measurement mode, so if the limits are exceeded, you will be warned in advance.

- To start the SR measurement procedure click on the "Measure" button inside the "Main controls" frame. The progress bar below the spectra screen will indicate the current H value graphically; the side and center values of H sweep diapason [G] will be also indicated. The current measurement values (H, g-factor and Y) will appear in the "Current value and error monitor". Three regimes are possible inside the measurement mode: memorizing, monitoring and blank sweep.

- Click on the "OFF" button inside the "Store" frame to start memorizing the data (the caption of the button will change from "OFF" to "ON" after the click). The LI Y signal values are assigned to the data with step number corresponding to the H value in this regime and temporarily memorized. If the H does not change, the Y data for the current number is averaged. Besides that, Y data for the relevant step number is averaged each and every time when the appropriate H value is reached, allowing further data averaging. Click on the same button if you want to stop memorizing the data.

- The graphical data monitoring is possible if the caption on the store button is "OFF. The "Down" option of the "Pen" frame should be checked for monitoring to proceed. Similarly to the "Signal Monitor" mode, during the data monitoring the point on the spectra screen indicates current Y value. Already memorized data are not affected. Clicking the "OFF" button inside "Store" frame will stop monitoring and start memorizing from the current H position.

- If the "Store" button is in "OFF" position and the "Pen" is "Up" the program will be set in blank sweep mode. This mode allows changing H value without memorizing/monitoring data. Control the H field position using the progress bar and H field indicator during such mode. This mode can be used when several scans of same spectra are to be taken for noise minimizing.

IMPORTANT: If during the memorizing sweep, data for the successive point(s) was not acquired the error event is generated. The number of such error events is indicated in the "Current value and error monitor" as a "Reading errors". Appearance of "Reading errors" during the measurement may occur by many reasons and is not dangerous if the number does not exceed 10% of the total number of measured points. Large number of error readings generally indicates that the H sweep speed is high for the actual H step value and appropriate correction in the settings will be favorable. The absence of data for some points inside the measured spectra will be checked by software also after finishing of the measuring mode and warning message will be generated. Still the measured data can be used as usual.

NOTICE: at every moment the stored data can be erased from the temporal memory by clicking on the "Clear memory" button. The spectra screen will be also cleared in such a case.

- Click on the "Stop" button inside the "Main controls" frame to stop the measurement. The memorized data will appear in the spectra screen. You can inspect the data by moving mouse inside the spectra screen: position of cross in values of H field/g-factor/Y signal will be indicated in <u>"current value and error monitor"</u> frame.
- Saving of measured data can be accomplished by clicking on the "Save As" ("Save All") command from the "File" menu. You will be prompted for the name of the ASCII format ("*.dat") file. "Save As" command saves measured data in two columns, H and Y correspondingly. "Save All" additionally saves statistical weight for each Y value and quasi g-factor ($=h\nu/\mu_B H$). Thus, under such a command data are saved in four columns (H, Y, W, g).

IMPORTANT: The program does not check for similar file names, thus be sure not to overwrite existing data file.

TIMER MEASUREMENTS

Timer measurements can be used to monitor change in the SR signal for the constant H value caused by change in sample illumination, temperature, applied microwave power, etc. Thus, on the first stage you should choose appropriate resonance conditions (H value for signal amplitude maximum) using "Signal monitor" mode and click "Stop" button. Time measurements will allow you to detect changes in signal amplitude caused by external factors.

- First, the values for time step and total measurement interval should be set. Insert appropriate values in the related text fields inside the "Timer Measurements" frame.


IMPORTANT: Previously mentioned limitations on the total number of measured points still apply here, thus ratio of interval/step should be less than 10000. Depending on specifications of the PC used, reliable minimal time step interval will be also limited. For detection safety it is not recommended to use step value <0.05sec.

- Click on ">" button to start timer measurements.
- Clicking on "II" button during the timer measurements will pause timer measurements.
- Click on the "[]" button to stop the procedure.
- After finishing the procedure you may save Y(t) data in the same way as Y(H) data is usually saved, although "Save All" command should not be used in this case.

CALIBRATION OF MAGNETIC FIELD

Additional, high precision calibration of magnetic field will be needed only in the case when position of gaussmeter tip inside the magnet gap was changed or there exists supposition about such a change and you want to check actual field measurement precision. In such a case calibration is done using spectrometric standard, which contains Mn^{+2} ions. Mn^{+2} ions give ESR spectra with well-documented values of g-factor for resonance lines (for more details see Manual for ESR installation). Before calibration procedure the powder containing Mn^{+2} ions should be placed inside the resonator in the place of the sample or using additional holder (see ESR installation manual). Different samples should be removed from the resonator. Calibration is done at room temperature.

- To start calibration process click on "H field calibration" button inside the "Main controls" frame. Step by step explanations of calibration procedure are given in the messages, which appear during the calibration and will not be repeated here. At any moment during the calibration the process can be aborted by clicking at "Cancel" button in the right-top corner of the spectra screen. This button is visible only during calibration process. In the last message of calibration process you will get resultant information about difference between H field calculated from the position of 4th resonance line of Mn^{+2} spectra and that measured by gaussmeter for the same position. If the difference is less than 1-1.5 G, recalibration is not necessary and you may click on the "Cancel" button inside the message field (you may accept calibration as well, but such an error is inside the statistical error of gaussmeter and actually does not need correction). If the error is out of named diapason, you should accept the calibration results and calibrate the field every time you switch on the ESR installation. Only finding correct position for gaussmeter tip and making corresponding changes to the initialization parameters of the software will finally remove the H calibration problem.
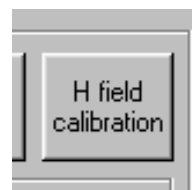
MEMORY BUFFER

Previously saved date can by viewed and compared to the recent measurements using buffer menu commands. Only one spectrum can be stored in the buffer memory. Thus the next loaded spectra will erase the previous. You can clean buffer by the "Clean" command from the same menu.

- To load existing file to the buffer memory click on the "Load file" command from the buffer menu. If you click on the "Load data" command, the recently measured spectra will be transferred to buffer for comparison with future measurements. Files saved or loaded to the buffer during the current session can be chosen from "Loaded specs" and "Saved specs" submenus.

- After loading the spectra will be shown in different window. Mowing mouse pointer inside the buffer window will give you values of H field and amplitude relevant to the loaded spectra in the "Cursor values" frame. You can hide the buffer window by clicking on "Hide" button (Click inside the main window to bring it to the front and keep buffer window opened). To open buffer window again (or to bring it to the front) click on the "Buffer window" command from the buffer menu.

- To overlay buffer data on the main spectra screen click on the "Overlay buffer → Yes" command from the buffer menu. The loaded spectrum will be overlaid only in the part where H field positions of loaded spectra coincide with H field range settings of the spectra screen.

- To remove buffer spectra from the spectra screen click on the "Overlay buffer → No" command from the buffer menu. If applicable, clean the spectra screen by clicking on the "Clear memory" button.
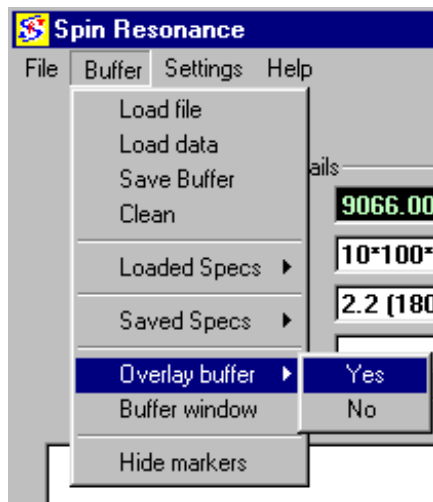
Zooming and browsing spectra in buffer window; obtaining peak positions, intensity and width.

- To zoom inside the spectra placed into the buffer, push the left button and drug mouse pointer across the area of interest. You will see black empty rectangle, which follows mouse pointer. When you release the button, area selected by the rectangle will expand to the whole spectra window.
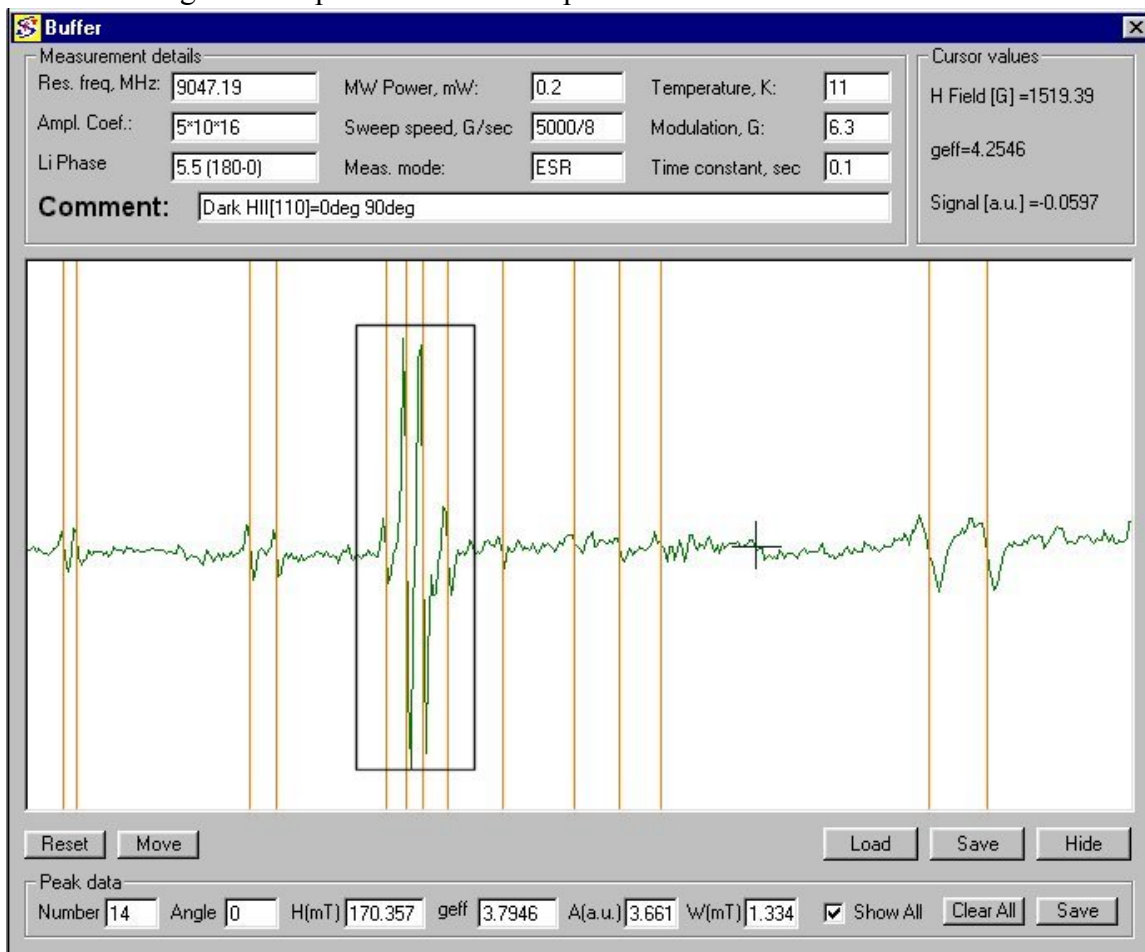

- You can zoom further inside the already selected area or reset the spectra by clicking on the "Reset" command button.

- To browse the spectra with zoomed magnification, click on the "Move" button. Red arrows will appear, showing possible directions of browsing. Clicking on the arrow will cause spectra to translate in the relevant direction on ¾ of actual scale. To eliminate the arrows click on the same command button (the name of the button will be changed to "Fixed" at that time).

- You can save the part of the spectra shown in the window to new file by clicking on the "Save" command button positioned just beneath the spectra window. The "Save Buffer" command from the "Buffer" menu saves entire data loaded to buffer.

- You can memorize the position corresponding to mouse pointer inside the spectra window by clicking the left mouse button. The field and signal values will be shown in the "Peak data" frame and temporarily stored to PC memory. Related vertical line will appear in the spectra window. This function can be used for characterization of peak positions in the spectra.

- For more precise peak characterization you can click right mouse button at peak minima and maxima (two points). In such case besides peak position and effective g-factor, the amplitude and width of the peak will be memorized and shown in the "Peak data" frame. Vertical line will pass through the center between the two chosen positions (*this function works for the first derivatives of the signal!*).

- Besides a peak number and characterization data, a textbox corresponding to sample rotation angle is presented in the "Peak data" frame. You can set the rotation angle by clicking the "Angle" box and printing inside. Data presented in the "Angle" textbox will be assigned to the peak.

- To save the data related to the characterized peaks click at the "Save" command button inside the "Peak data" frame. Peak related data will be saved in ASCII format file with ".dat" extension and include five columns related to the angle, magnetic field value, effective g-factor, peak amplitude and peak width. If the peak was left-clicked, "-1" will be assigned to the peak amplitude and width.

- If the "Show all" option is not checked, the vertical lines showing peak positions will disappear after the spectrum is moved, zoomed or reset. If the option is checked positions corresponding to all characterized peaks will be shown in the spectra window.

- You can discard all characterized peaks by clicking on the "Clear all" command button inside the "Peak data" frame. Also you can add/change/delete specified peak data directly using the related textboxes.

PRINTING OUT THE MEASURED DATA

Two modes of printing are available. In the first mode ("Graph") software prints out recently memorized data in the form of a plot (with axes, tics and tick labels), while in the second ("Image") - image of spectra screen will be printed "as is". The image printing is helpful when you want to print out some data detected during the monitoring process and can be used at any moment. The "Graph" mode printing can be used only after the "Measure" procedure was finished. In the both cases all information contained in the "measurement details" frame will be printed beneath the image.
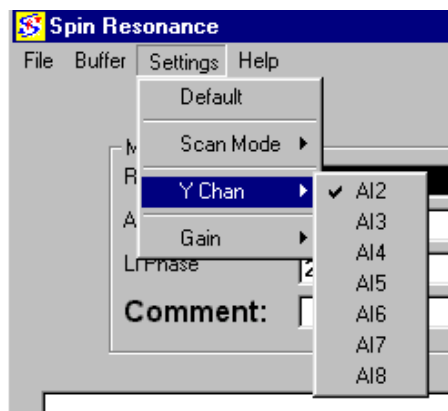
- To start "Image" mode printing click on the "Print→Image" command from the "File" menu.

- To start printing in the "Graph" mode click on the "Print→Graph" command from the "File" menu after you finished the SR spectra of Timer measurements.

- Click on the "Printer setup" command from the "File" menu for access to printer settings.
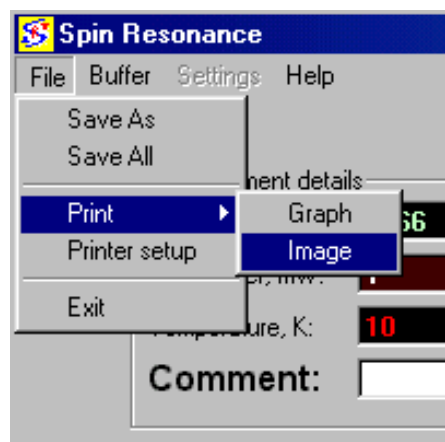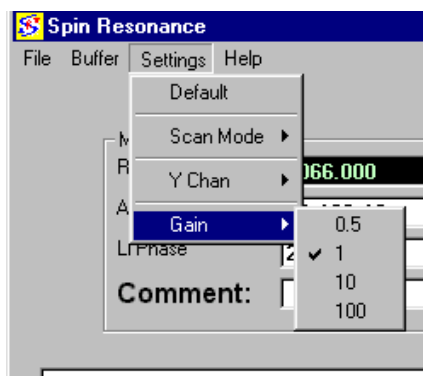
PROGRAM SETTINGS

The software allows setting the number of a channel to which "Y signal" is connected and setting a gain value for the connected channel.

- By default setting the Y-signal is connected to the 2nd channel. It is possible to use any channel from 2 to 8 to connect Y-signal to PC. After you have completed physical connection, click on relevant channel number from the settings menu. Channel, which is connected to the PC is checked in the menu.

- You can change gain of each channel independently from the settings menu. Gain setting changes the "voltage resolution" of the AD converter channel. The relevant gain settings, input ranges and AD channel conversion precisions are shown in the table below. Default setting for the 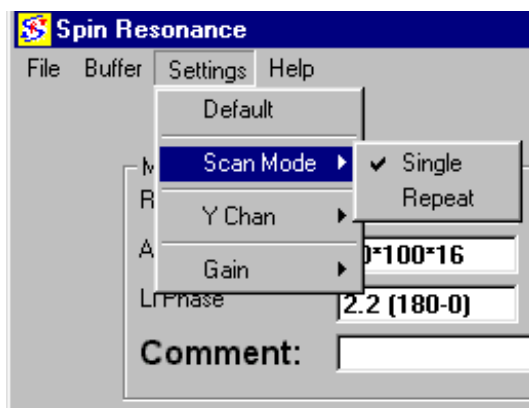gain is "1". If you change the channel gain by clicking the "Gain" menu item, currently chosen gain will be checked in the menu. The setting of the gain for every channel is not affected by the switching of channels. The default settings (gain=1 for all channels) are restored at the next start-up of the software.

Gain	Input Range	Precision *
0.5	−10 to +10 V	4.88 mV
1.0	−5 to +5 V	2.44 mV
10.0	−500 to +500 mV	244.14 µV
100.0	−50 to +50 mV	24.41 µV

*The value of 1 LSB of the 12-bit ADC; that is, the voltage increment corresponding to a change of one count in the ADC 12-bit count.

- The default settings for the software are: channel No.2 for Y-signal and Gain=1 for the 2nd channel. You can easily restore the default settings by clicking on "Default" command from the "Settings menu.

To accumulate spectra in the automatic mode by the sweeping the same magnetic field range several times, you can click on the "Settings → Scan Mode → Repeat " command. In this measurement mode, data acquisition will automatically stop each time when the magnetic field will reach maximal value and started again after the field will reach the minimal value in the current sweep range. Obtained signal levels for the same value of the field will be averaged taking in account the measured data weight.