DYNOMAG

Instruction manual

Software version: 2.0
Manual revision 32, August 2023

Compliance information

EU:

DynoMag Instrument complies with the essential requirements of the following applicable European Directives, and carries the CE marking accordingly:

EMC Directive (2014/30/EU)

and conforms with the following product standards:

Aspect	Standard	Version	Title	Selected levels in standard
EMC	EN 61326-1	2013	Electrical equipment for measurement, control and laboratory use – EMC requirements - Part 1: general requirements	emission: EN 55011, Group2, Class B immunity: basic requirements

US:

NOTE: This equipment has been tested and found to comply with the limits for a Class B digital device, pursuant to Part 15 of the FCC Rules. These limits are designed to provide reasonable protection against harmful interference in a residential installation. This equipment generates, uses and can radiate radio frequency energy and, if not installed and used in accordance with the instructions, may cause harmful interference to radio communications. However, there is no guarantee that interference will not occur in a particular installation.
If this equipment does cause harmful interference to radio or television reception, which can be determined by turning the equipment off and on, the user is encouraged to try to correct the interference by one or more of the following measures:

- Reorient or relocate the receiving antenna.
- Increase the separation between the equipment and receiver.
- Connect the equipment into an outlet on a circuit different from that to which the receiver is connected.
- Consult the dealer or an experienced radio/TV technician for help.

Installation requirements

The DynoMag is delivered with the following necessary accessory:

1. Power supply unit with power outlet cable
2. Laptop PC with USB cable to DynoMag
3. Calibration sample

The specifications of DynoMag as well as the above stated "Compliance information" and as printed in the Declaration of Conformity, is valid only when DynoMag is operated utilizing all of the accessory 1-3 above.

Contents

1 Introduction

In order to fully optimize a system in which magnetic materials are used, it is crucial to understand the magnetic properties of the material. To obtain this information, AC susceptometry can be used and allows for probing the dynamic magnetic properties.

The dynamic magnetic properties give information on how fast the magnetization is building up in the material. Different types of magnetization processes, for instance internal magnetization reversal in magnetic single-domain crystals (Néel relaxation), randomly rotation of particles containing thermally blocked single-domain crystals (Brownian relaxation) or domain wall motion as in poly-domain materials can be detected with AC susceptometry. All of these magnetization processes create a specific pattern in the dynamic magnetic properties for instance in the AC susceptibility. Measuring the AC susceptibility is almost the same as a spectroscopic detection where the different relaxation processes is visualized as relaxation peaks at their specific frequencies. In a magnetic nano-particle system the magnetic susceptibility at low magnetic fields is sensitive to the size of the magnetic single domain crystals, the number of magnetic single-domain crystals per particle (multi-core particles), the positions and orientations of the single-domain crystals in the total particle, the material in the single-domain crystals, the concentration of the single-domain crystals and also on the total particle size (when the particle includes thermally blocked single-domain crystals). This makes AC susceptometry as a very good magnetic tool in order to have a good control of the manufacturing process of magnetic nano-particle systems.

AC susceptometry can be found in vast area of applications, from magnetic particle manufacturing to measuring soils in geophysical applications. In the case of detecting Brownian relaxation of particles including slowly relaxing single-domain (with respect to the stochastic particle rotation) crystals the DynoMag system is able from the experimental data to determine the size distribution of the particles using an algorithm included in the DynoMag software. With the same instrument it is also possible to study any clustering process of the particles and to follow the binding reactions of different substances to the surface of the particles.

DynoMag is a portable magnetic instrument where it is possible to measure the AC susceptibility of liquids, powders or solid samples. The frequency range is from 1 Hz up to 500 kHz) with a resolution in magnetic moment typically $8 \cdot 10^{-10}$ Am^2 or in volume susceptibility 10^{-5} at 1 kHz and excitation amplitude of 0.5 mT [1]. With the software it is possible to set measurement parameter, visualize the data, store the data, calibrate the instrument, perform data fittings of the result and to determine the

[1] The resolution in magnetic response is determined from the standard deviation of the magnetic signal of the Dy_2O_3 calibration sample at excitation frequency 1 kHz and 0.5 mT field amplitude using 20 data points.

hydrodynamic size of particles containing domains with slow (compared to the Brownian relaxation) internal magnetic relaxations.

In the DynoMag system induction technique is used in order to detect the AC susceptibility. The magnetic flux change from the sample is detected with a well-balanced detection coil system. The detection coil system is centred in an excitation coil system connected to an AC - source that delivers the time dependent current to the excitation coil that gives the time dependent excitation field. The signals from the detection coil system are fed into a low noise Lock-In amplifier. All the electronics are integrated in the DynoMag instrument. A schematic picture of the DynoMag instrument can be seen in Figure 1 below.

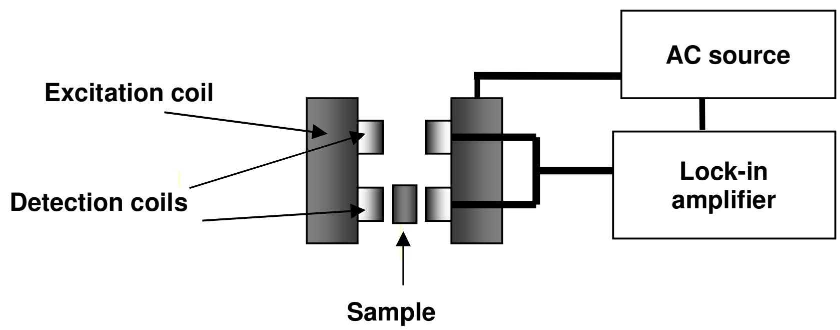


Figure 1. Schematic picture of the excitation and detection coil system, the AC source and the lock-in amplifier.

The DynoMag system software governs the whole experiment, for instance optimum measurement time and amplifier gain settings (automatically adjusted in the DynoMag system), frequency range, number of data points etc. and the collection of the data and presentation of the result in a user-friendly interface. For the detection of magnetic particles including slowly relaxing single-domain crystals (with respect to the particle Brownian rotation), it is also possible to determine the size distribution of the particles with a set of models included in the software. In the DynoMag system there is also a calibration procedure that is performed in the frequency range from 20 Hz up to 500 kHz.

The detection coil system detects the differential induced voltage which is dependent on the change of the differential magnetic flux (Φ_1 - Φ_2 from upper and lower detection coils) with time. The differential induced voltage from an ideal balanced detection coil system can be expressed as:

$$\Delta V = N\frac{d}{dt}\left(\Phi_1 - \Phi_2\right) = \mu_0 NA\alpha\frac{d}{dt}\left(H + M - H\right) = \mu_0 NA\alpha\frac{d}{dt}M \qquad [1]$$

where N is the number of turns in the two identical detection coils (that is forming the detection coil system), A the cross-sectional area in the detection coils, H the magnetic field produced by the excitation coil, α a magnetic coupling factor which depends on the geometry and dimensions of the sample with respect to the detection coil dimensions and M is the magnetization of the sample. The coupling factor α is determined from the calibration of the induction coil system where a calibration sample, with a known and well-defined susceptibility and the same dimensions as for the measured sample, is used.

For a sinus excitation field the magnetization can be expressed by:

$$M = (\chi' - j\chi'')H \qquad [2]$$

where χ' is the real part and χ'' is the imaginary part of the dynamic magnetic volume susceptibility. The amplitude of the differential induced voltage from the detection coils then becomes:

$$\Delta V = \mu_0 NA\alpha(\chi' - j\chi'')\, j\omega H_0 = \mu_0 NA\omega H_0 \alpha(\chi'' + j\chi') \qquad [3]$$

where H_0 is the amplitude of the excitation magnetic field and ω is the angular frequency ($2\pi f$ where f is the excitation frequency).

For a real detection coil system, the coil induction voltage is dependent on both imbalance of the coil and the electric properties of the coil. This is handled by the automatic sample movement in the coil system and the calibration procedure using the Dy_2O_3 calibration sample.

2 System overview

This chapter describes the included hardware and software of the DynoMag system.

2.1 Hardware

2.1.1 Instrument exterior and interfaces

The front panel of the DynoMag instrument has a logo text and a blue LED showing the status of the system (power ON/OFF).

The top panel has a sliding lid that protects the instrument interior. The sample is mounted by open this lid and insert the glass vial containing the sample to be measured.

Together with the DynoMag system there are glass sample vials for mounting the sample under investigation. New sample vials can be purchased from VWR international (Cat./Art. No 548-0042).

Figure 2. Pictures showing the top and front panel of the DynoMag instrument. The top panel has a sliding lid that protects the opening where the sample is mounted. A blue LED indicates the status of the system (power ON/OFF).

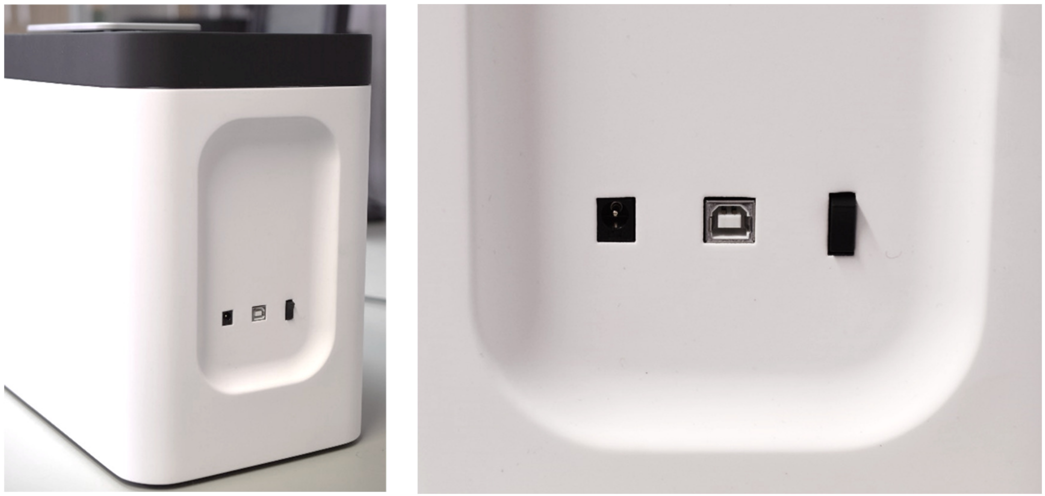


Figure 3. The back panel of the DynoMag instrument has a power supply connector, a USB connector and an on/off switch.

On the back panel there are two connectors; one for the power supply unit (DC IN plug) and one for the USB cable (Standard Type-B) that connects the instrument to the PC. There is also a power on/off switch.

2.1.2 System overview of the electronics

The electronics in the DynoMag system represented as a schematic block diagram can be seen in Figure 4 below.

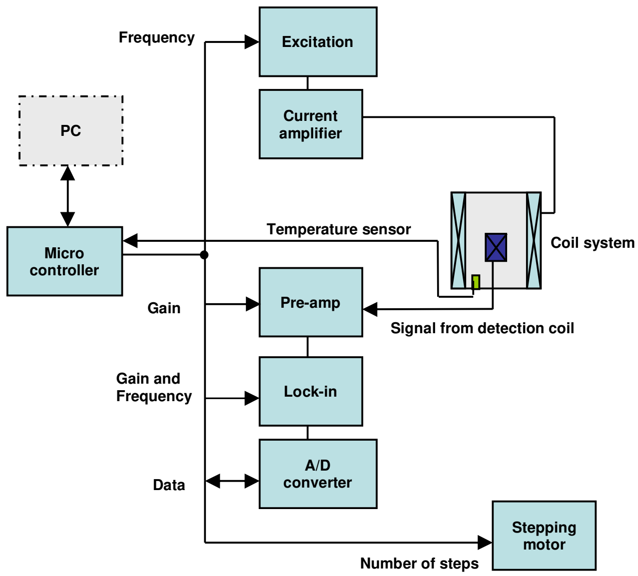


Figure 4. Schematic block diagram of the electronics in the DynoMag system.

The PC software communicates with the microcontroller in the DynoMag, and sets all measurements parameters to be used in a measurement such as:

- Frequency of the excitation field .
- Gain in the pre-amplifier.
- Gain in the lock-in amplifier.

During a measurement the PC software receives the following data:
- Induced voltage sampled by A/D converter.
- Temperature measurement close to the sample.
- Sample position in the coil system.
- An indication of “Overload” in case of too high signal in the A/D converter from the detection coils.

The software also controls the number of steps to the stepping motor (that controls the length of the sample movement); stores data to a data file, calibrates the DynoMag system and can determine the hydrodynamic size distribution of magnetic nano-particles that undergo Brownian relaxation. The measurement parameters and modeling of the data points are described in more detail in the software chapter.

2.1.3 The excitation and detection coil system

The excitation coil is wound as an ordinary solenoid with a specific length, diameter and number of turns of the winding. The excitation coil produces the excitation field, H, and have the effect on the signal response as described in equation 1 – 3. The number of turns per unit length of the solenoid and the current in the excitation coil determines the excitation field strength. A detection coil system in a form of a first order gradiometer coupling is placed in the center of the excitation coil. The detection coil system is formed by two well-balanced coils coupled together to detect the difference in magnetic flux of the two coils. The detection coil system is formed by positioning the two coils with their length axis co-linear to the length axis of the excitation coil.

Without a magnetic material in the detection coils, no detection coil signal should be present. Inevitably, small variations in geometry between the detection coils are present, giving rise to a small background signal. In order to measure small magnetic signals from samples with low content of magnetic material or low magnetic susceptibility, these small variations of the two coils have to be compensated for. This is done by calculating the difference value of the magnetic signal from a measurement with the sample in the lower detection coil and then another measurement of the sample in the upper detection coil.

The excitation field amplitude is set to 0.5 mT (5 Gauss) at lower frequencies and decreases at higher frequencies. The field amplitude is low enough for the magnetization of a typical magnetic nano-particle system to be in the linear field region (ensuring right low field susceptibility), but still high enough to yield a good signal. The variation of the field amplitude is compensated for in the software when determining the susceptibility, or performing the calibration procedure.

2.2 Measurement procedure

Below the different steps in a standard measurement procedure are described:

1. A sample is mounted at the top of the instrument.
2. User sets the measurement parameters and start the measurement from the PC.
3. The sample moves to the initial position.
4. DynoMag sets the first frequency.
5. The sample moves to the upper detection coil and takes measurement points according to the measurement parameter settings. The optimum amplifier gain is regulated.

6. The sample moves to the lower detection coil and takes measurements points according to the measurement parameter settings.

7. The signal difference between sample in upper and sample in lower detection coil is calculated.

8. The obtained signal difference is first subtracted from the background level and then multiplied with the calibration factor which corrects the amplitude and phase of the signal.

9. The second frequency point is set according to the measurement parameters.

10. The measurement starts again in point 5. The measurement procedure goes on until the last frequency point. The data is stored continuously in a data file containing 13 data columns:
    - *Frequency* (Hz)
    - *Real susceptibility* (volume susceptibility, unitless in SI units, or mass susceptibility, m^3/kg in SI units, depending on the susceptibility choice in the software)
    - *Imaginary susceptibility* (volume susceptibility, unitless in SI units, or mass susceptibility, m^3/kg in SI units, depending on the susceptibility choice in the software)
    - *H-field* (A/m)
    - *Temperature* (deg C)
    - *Real induced voltage* (V/Hz) of upper coil
    - *Imaginary induced voltage* (V/Hz) of upper coil
    - *Real induced voltage* (V/Hz) of lower coil
    - *Imaginary induced voltage* (V/Hz) of lower coil
    - *Time (s)* Time of frequency point relative to the starting time of the measurement
    - *Detection gain* preamplifier gain setting (used for status check of instrument)
    - *I gain* in-phase gain setting (used for status check of instrument)
    - *Q gain* out-of-phase gain setting (used for status check of instrument)

    The header of the data file contains the information about instrument settings and user comments (see chapter 2.3.6.1).

11. If the 'Curve fit' procedure is activated according to the instructions in chapter 2.3.6, the data is fitted to the susceptibility model in order to determine the hydrodynamic size of the particles as explained in detail in chapter 2.5.

2.3 PC Software

This chapter describes the DynoMag PC Software. The graphical user interface (GUI), showed in Figure 5 below, has a main control panel to the left and to the right of this panel, several tabs can be selected to show the instrument settings, calibration files, measurement results and the data analysis can be found. Each part of the program will be described in the following sections.

A software installation guide can be found in chapter 2.4.

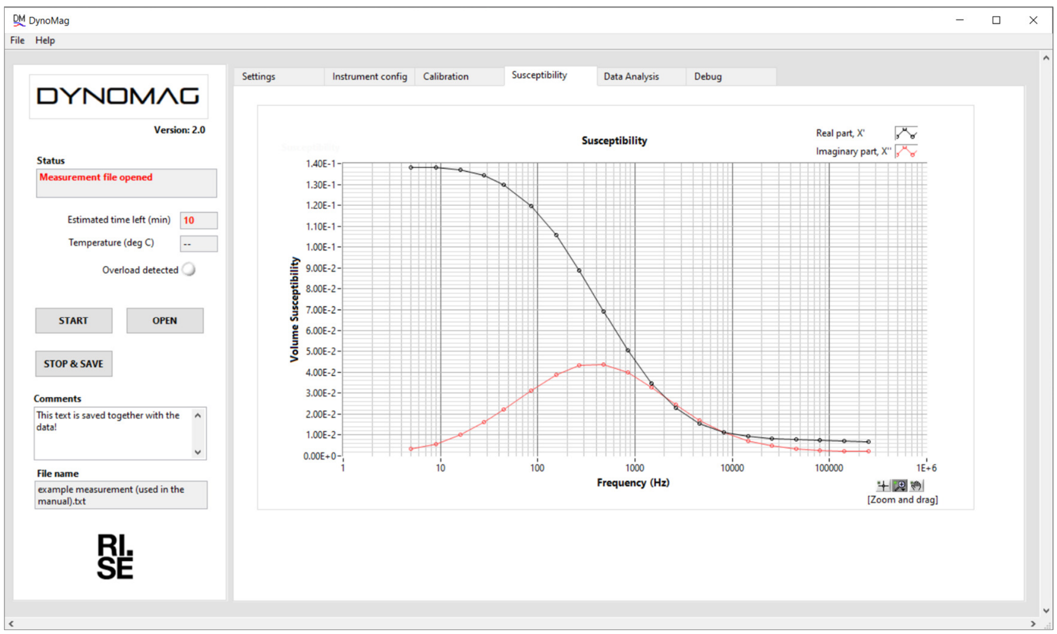


Figure 5. The DynoMag Software GUI.

2.3.1 Main control panel

The main control panel is always visible to the user and it contains the controls and indicators listed below (and showed in the Figure 6 below).

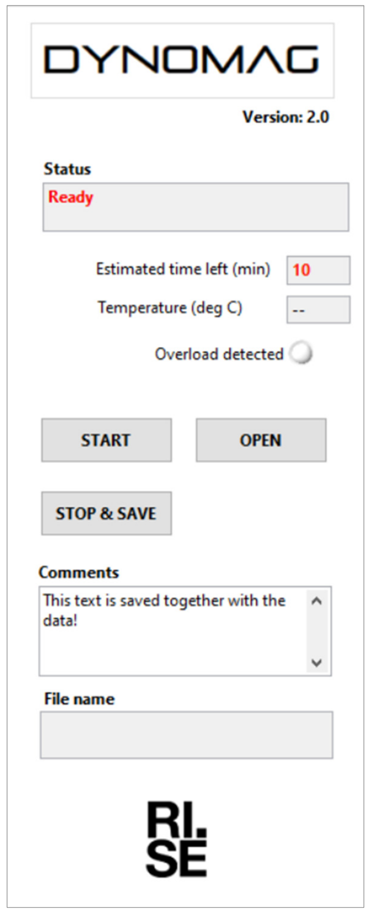


Figure 6. Main frame contains controls and indicators that are always visible.

- The **Start** button starts the measurements. When the **Start** button has been activated, a save prompt will let the user to choose a file name and save location before the measurement starts. The name of the measurement file will be automatically set to *"measurement DATE TIME.txt"* as default. The default file path for saving the measurement file (the working directory) is *~/measurements/*.
- The **Stop/Save** button stops measurements and enables saving the data to a data file with specific name.

- The **Temperature** is the temperature in the coil system which is close to the sample. This temperature is measured at each data point and is stored in the data file.
- The **Status indicator** shows the current status of the DynoMag instrument.
- **Estimated time left** is the time left before the measurement is finished.
- In the **Comment** box the user can write comments in connection to the measurement. These comments are stored in the header of the data file. The comments must be written in the text box prior to the measurement starts.
- The **File name** button shows the file name of the current measurement or the last performed measurement. If a measurement is opened, the file name will also be shown in this textbox.
- The **Open measurement** button can be used to open an old measurement. A dialog will then appear with two choices; "Open" or "Re-calc" a measurement file.

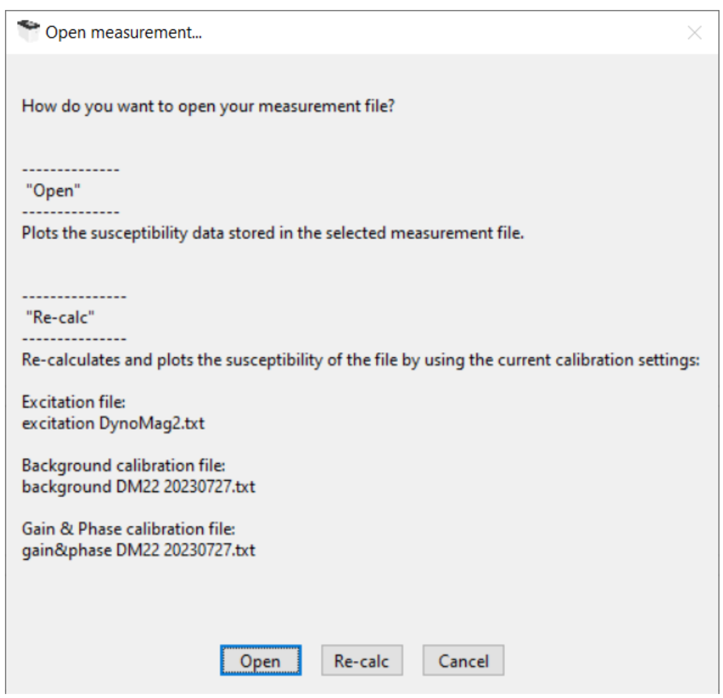


Figure 7. The "Open measurement" dialog.

1. By selecting **"Open",** a file dialog will appear. After selecting a measurement file (saved by the DynoMag system) the susceptibility will be plotted in the "Susceptibility" tab.

2. By selecting **"Re-calc"** a file dialog will appear. After selecting a measurement file (saved by the DynoMag system) the susceptibility will be recalculated using the current selected *background* and *gain&phase* calibration files. This is possible because all measurement files also include raw data (induced voltages). The resulting susceptibility will be plotted in the "Susceptibility" tab.

Opened data can be analyzed in the same way as when measured; by pressing the button "RUN CURVE FIT" in the "Data analysis" page, which is described in chapter 2.3.6.

Opened files can be saved as new files by clicking on the "Stop & Save" button. If the curve fit algorithm has been run, the new file header will consist of the original header of the opened file plus information about the curve fit input parameters together with the results of the curve fit. Also, the resulting susceptibility from the curve fit (both real and imaginary part) will be added as two extra columns in the new data file.

Recalculated files can also be saved by clicking the "Stop & Save" button. The new file header will then consist of the original file header plus a new header containing information about the new background- and gain/phase calibration files used for the calculation.

2.3.2 Settings

Under this tab, the user can set different measurement parameters before the measurement starts, such as the frequency interval, the number of data points and the sample volume or sample mass.

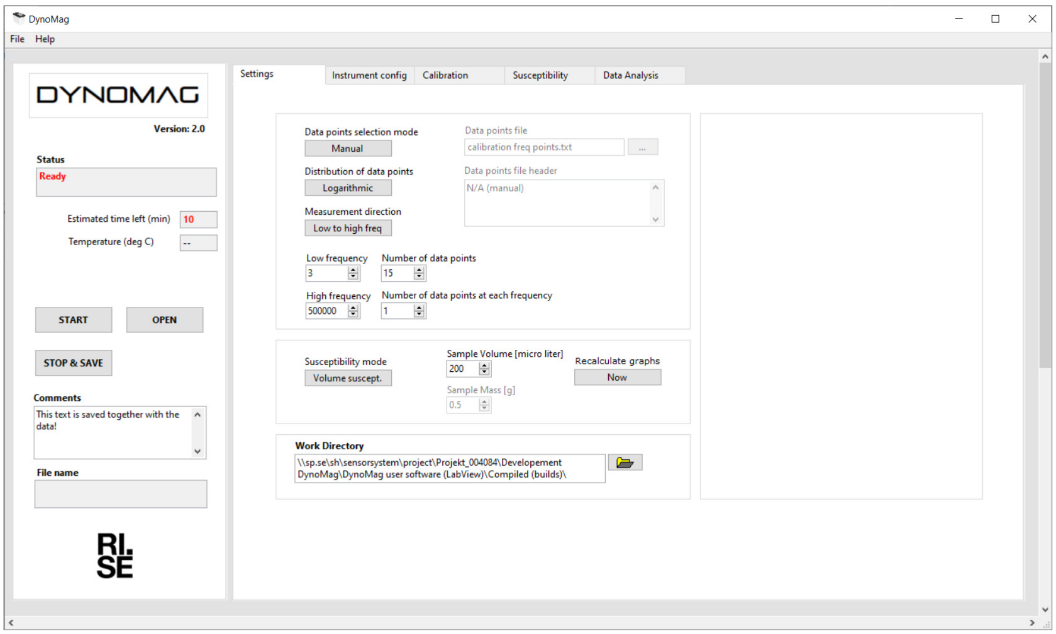


Figure 8. Measurement settings such as the frequency interval, number of data points and sample volume can be set under the "Settings" tab.

- **Data points selection mode.** Using this control, the frequency points are either manually selected (**Manual**), or predefined frequency points are chosen from a data file (**Automatic**). In manual mode, the frequency interval is entered using the Low- and High frequency controls (see below). In Automatic mode, the data points file is selected by pressing the **Select data points file** and selecting an appropriate file in the following dialog. The selected file is displayed in the **Data points file** indicator.

- **Distribution of data points** sets if the frequency points will be distributed logarithmic (recommended if the plots are logarithmic in frequency) or linear.

- **Measurement direction** sets if the measurements shall start at the low or high frequency.

- **Low frequency** (≥1 Hz) gives the lowest frequency point in the measurement series. **High frequency** (≤ 500 kHz) gives the highest frequency point in the measurement series. **Number of data points** (≥2) is the number data points between the low and high frequency. **Number of data points at each frequency** (≥1) is the number of data points that is measured at each frequency point.

- **Susceptibility mode**. The susceptibility can be measured in either volume or mass susceptibility. This control sets the desired susceptibility mode for the measurement.

- **Sample mass**. If mass susceptibility is measured, the sample mass in grams is entered here.

- **Sample volume**. If volume susceptibility is chosen, the sample volume in µl is entered here. The calibration sample that follows with the DynoMag system is adjusted for 200 µl samples. If the user wants to use smaller sample volumes the height of the calibration sample must be adjusted to be the same as for the measured sample (see the calibration chapter 4).

- **Recalculate graphs**. If the susceptibility mode has been changed while the susceptibility graph contains data, pressing this button recalculates the susceptibility of the graphs to the new susceptibility mode. The button is disabled if no change of susceptibility mode has been made.

- **Working directory** shows the path to folder where the last measurement was saved. When starting a new measurement the “User Save dialog” will start in this folder. The working directory path can also be changed by pressing the “browse button” to the right of the text box.

2.3.3 Instrument configuration

In this tab, the instrument configuration parameters are set. The graph shows the excitation field amplitude versus the excitation frequency. The determined susceptibility is compensated in the software for the field amplitude variation with frequency.

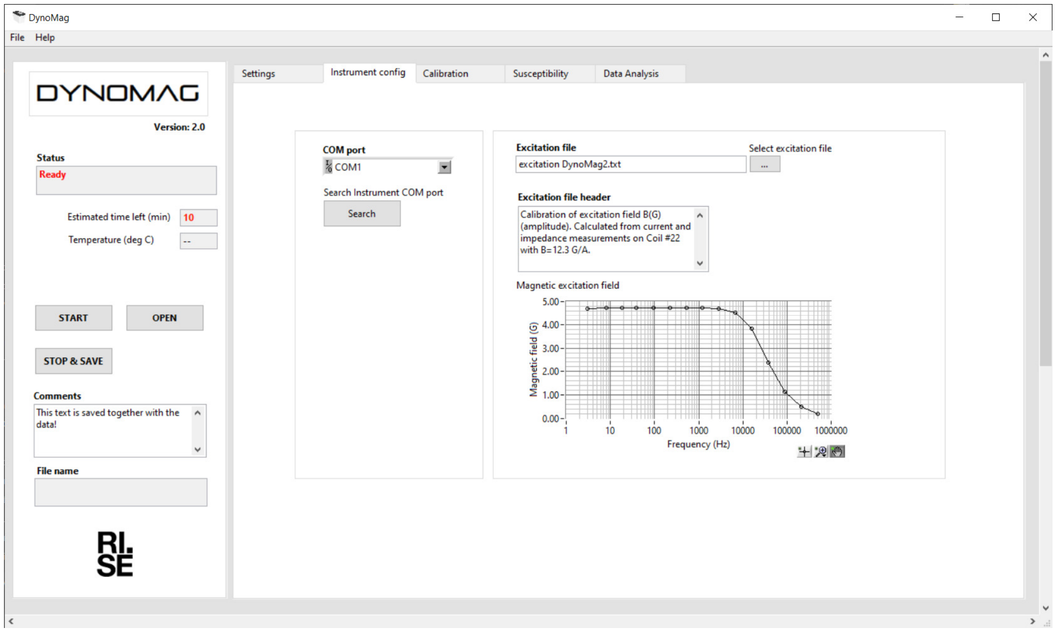


Figure 9. The instrument configuration (COM port settings, excitation file settings) is located under "Instrument config" tab.

- **COM port** sets the COM port number for USB communication.
- By pressing the button **Search Instrument COM-port** the software will automatically try to find which COM-port the DynoMag system is connected to. The DynoMag system must be powered on.
- **Excitation file** selection. This file contains data that describes the excitation field (in Gauss) as a function of the measurement frequency. The excitation field has been calibrated before shipment of the system and it is not recommended to edit or change this file. Changing the data in the file will not change the actual excitation field generated by the system; instead the data is used in the calculation following a measurement.

2.3.4 Calibration

In this panel the calibration files are selected. Also, the two calibration routines (background and gain/phase) can be started here. The two graphs show the data from the two selected calibration files. The calibration procedures are explained in more detail in chapter 4. The first figure is a plot of the background subtraction data points and the second the gain/phase calibration. Either new calibrations can be performed or be selected from previous calibrations by listing the corresponding calibration files.

Figure 10. In the calibration panel, user can select which calibration files to use. The calibration routines (background and gain/phase) are also started from here.

- **Background calibration file** sets the name of the file for the background subtraction. The background calibration file is selected by pressing the **Select background calibration file** and by selecting a proper file in the following dialog. The selected file is displayed in the **Background calibration file** indicator.

- **Gain&Phase calibration file** sets the name of the file for the gain and phase correction. The gain&phase calibration file is selected by pressing the **Select gain&phase calibration file** and by selecting a proper file in the following dialog. The selected file is displayed in the **Gain&phase calibration file** indicator.

- **Test calibration** can be performed in order to control if the instrument needs to be recalibrated. For more details see chapter 4.3.

- **Start Background Calibration** and **Start Gain and Phase Calibration** start a new calibration. See chapter 4.3 for more details on the calibration procedures.
- The **Calibration sample properties,** is where the mass of the calibration sample must be defined when a new gain&phase calibration is performed.
- In the **Background calibration header** and **Gain Phase calibration file header** the user can find parameters that are saved in the calibration file**.**

2.3.5 Susceptibility

In this panel the result of the real and imaginary part of the measured volume (or mass) susceptibility (depending on unit chosen with the "susceptibility mode"-switch) versus the excitation frequency. The real- and imaginary part of the complex susceptibility is displayed in the graph and in the data files as the volume or mass susceptibility.

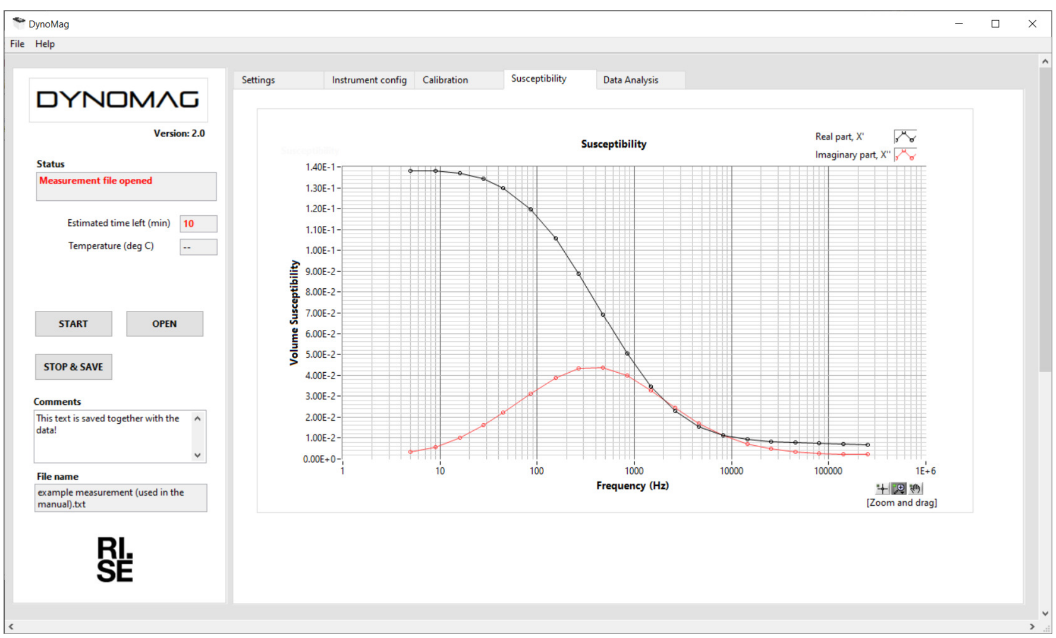


Figure 11. The susceptibility panel shows the result of the measured susceptibility.

2.3.6 Data analysis

The data analysis panel handles the curve fitting of the measured susceptibility to the models described in chapter 2.5. It is also possible to switch to a "Plot tool mode" which allows the user to plot the different models included by manually enter the parameter values. A screen shot of the program window under this tab is shown in Figure 12 below. The graph to the left shows the measured data points (red circles and crosses) for the real and imaginary part of the susceptibility, together with the fitted data for both parts (black solid and dashed line).

The graph to the right is the result of the size distribution of the particles versus particle radius. The size distribution is obtained from fitting of both the real and imaginary part of the susceptibility. The final size distribution curve is the mean value of these two curves weighted with the goodness of each fit.

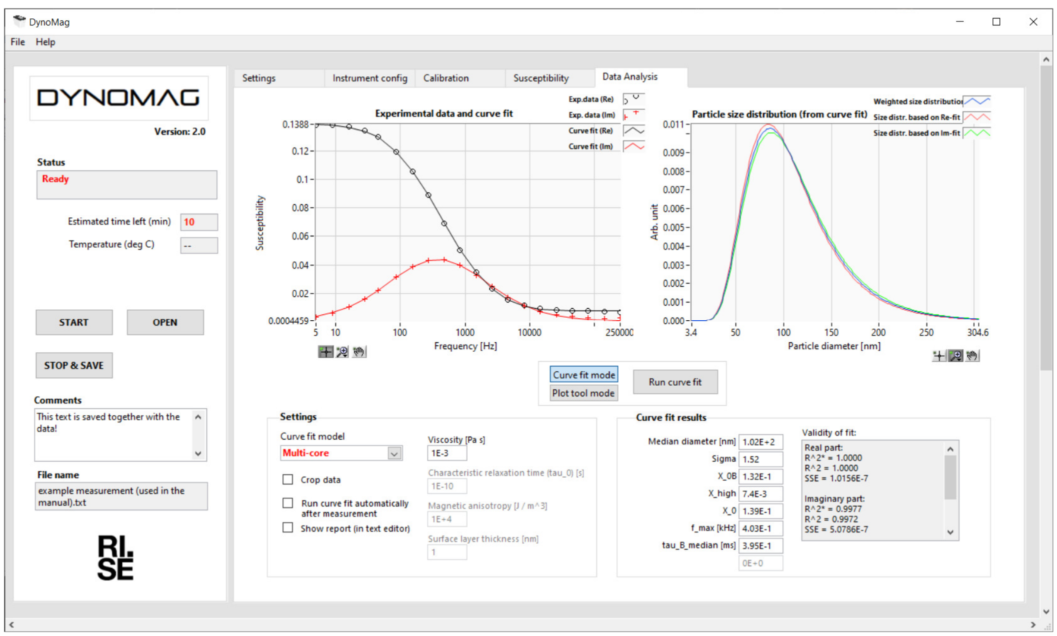


Figure 12. The data analysis panel.

2.3.6.1 *Running the Curve fit*

After a measurement is ready, or after a measurement file is opened, the susceptibility can be fitted to an AC susceptibility model (described in chapter 2.5), using the built in curve fit algorithm. The curve fit is started by pressing the "RUN CURVE FIT" button. By activating the checkbox "Run curve fit automatically after measurement" the user does not have to press the "RUN CURVE FIT" button to start the curve fit algorithm.

When the curve fit algorithm is finished the fit results will be shown in the "Data Analysis" window (see Figure 12). If the curve fit has been performed after a measurement, the user will be asked if the curve fit results should be added to the current measurement file. By selecting "Yes" curve fit data will be added to the current measurement file. The default name for the file will be *"measurement DATE TIME (curve fit data incl.).txt"*. Information about the model used in the curve fit and the values of the input parameters (such as viscosity) will be saved together with the results of the curve fit parameters in the file header (see example below). The fitted data will also be added as two extra data columns after the measurement data.

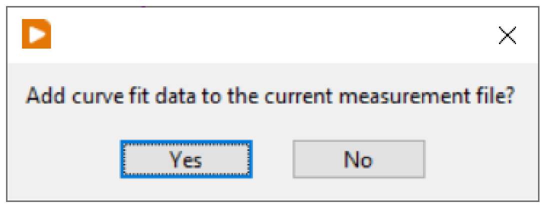


Figure 13. Save dialog shown after the curve fit has been run.

If the curve fit algorithm has been run on data from an "Opened" file, the user can save the results as a new file by clicking the "Stop & Save" button. The new file header will then consist of the original header of the opened file, plus information about the curve fit input parameters, model used and the results of performed the curve fit (see example below). The fitted susceptibility data (both real and imaginary part) will also in this case be added as two extra columns after the measurement data. The default filename will now be *"-original filename- (curve fit data incl.).txt"*

Example of a **measurement file** header where curve fit data has been added:

```
File saved: 2012-04-01 12:00:00
Comments: Sample #
DynoMag version: B1j
Data points file: N/A (manual)
Excitation file: excitation_example.txt
Background calibration file: background_example.txt
Gain & phase calibration file: gain&phase_example.txt
Time constants file: time constants.txt
Sample volume [micro liter]: 200
Curve fit model: Multi-core
Input parameter value(s):
Viscosity [Pa s]
1.0000E-3
Calculated curve fit parameters:
Median diameter (nm) Sigma X_0B X_high X_0f_max (kHz)
tau_B_median (s) Re [R^2] Im [R^2]
1.0153E+2    1.5200E+0    1.3180E-1    7.4050E-3    1.3920E-1    4.0300E-1
3.9493E-1m   9.9996E-1    9.9723E-1
---END OF HEADER---
[Names of data columns]
<< DATA >>
```

Example of a file header of an **opened file** where curve fit data has been added:

```
File saved: 2010-10-02 13:00:00
Comments:
Curve fit results has been added to measurement file: example measurement.txt
---------------  Original file header ----------------
File saved: 2012-04-01 12:00:00
Comments: Sample #
DynoMag version: B1j
Data points file: N/A (manual)
Excitation file: excitation_example.txt
Background calibration file: background_example.txt
Gain & phase calibration file: gain&phase_example.txt
Time constants file: time constants.txt
Sample volume [micro liter]: 200
[Names of data columns]
---------------  End of Original file header ----------------
Curve fit model: Multi-core
Input parameter value(s):
Viscosity [Pa s]
1.0000E-3
Calculated curve fit parameters:
Median diameter (nm) Sigma X_0B X_high X_0f_max (kHz)
tau_B_median (s) Re [R^2] Im [R^2]
1.0153E+2   1.5200E+0   1.3180E-1   7.4050E-3   1.3920E-1   4.0300E-1
3.9493E-1m  9.9996E-1   9.9723E-1
---END OF HEADER---
[Names of data columns]
<< DATA >>
```

If the measured data fits poorly to the chosen model, a warning message will appear to the user (Figure 14). The condition for this message is set to R^2 < 0.9. The curve fit results will still be shown and added to measurement file.

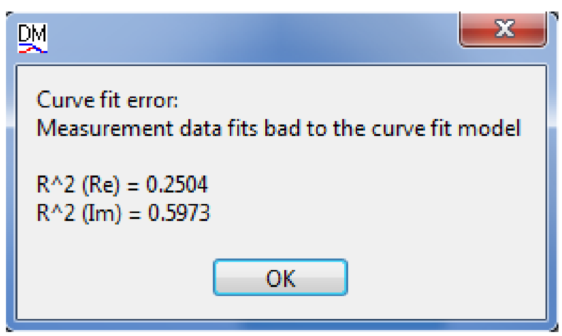


Figure 14. A warning message is shown if the measured data fits poorly to the chosen curve fit model.

A *Curve fit log*, containing output information and errors from the Curve fit function (matlab script), is stored in a text file. The file is located at *"~\config\curve fit log.txt"*, and is written every time the *Curve fit* has completed. The log file is overwritten each time.

2.3.6.2 *Curve fit settings*

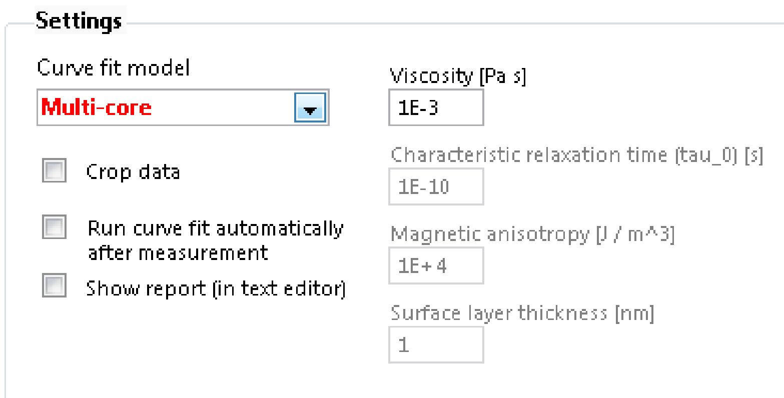


Figure 15. Curve fit settings can be found in the Data analysis tab.

In the settings box, located down to the left in the program window, user can define some different settings regarding the Curve fit algorithm:

- There are three different models that can be used to fit the susceptibility data; “Multi-core model”, “Extended multi-core model”, “Single-core model” and “Cole-Cole model. The desired model is chosen from the drop-down list called “Curve fit model”. The models will be described more in detail below and in chapter 2.5.

- Depending on selected model there are some input parameters that must be set:
    - *Viscosity [Pa s]*: Viscosity of the liquid surrounding the magnetic particles. (default value = 1E-3, corresponding to water at room temperature)
    - *Characteristic relaxation time [s] (τ_0)*: (default value = 1E-10)
    - *Magnetic anisotropy [J / m^3] (K)*: (default value = 1E+4)
    - *Surface layer thickness [nm]*: Thickness of the outer surface layer of the particles, outside of the magnetic core radius (default value = 1)

  In the “Multi-core” and the “Extended multi-core” model only *Viscosity* parameter is included and therefore the only one that needs to be set. In the “Single-core” model all four parameters are included.

- “Crop data” checkbox activates data cropping. This allows the user to manually select the frequency interval from which measurement data should be included in the curve fit algorithm. The frequency interval is selected by two graph cursors that could be dragged along the measured susceptibility curve (see Figure 16).

- The checkbox called "Run curve fit automatically after measurement" can be used to start the curve fit algorithm directly when the measurement is ready.

- If the checkbox "Show report (in text editor)" is activated the *"Curve fit log.txt"* file will be opened after the curve fit is ready. The log contains some additional information about the curve fit algorithm.

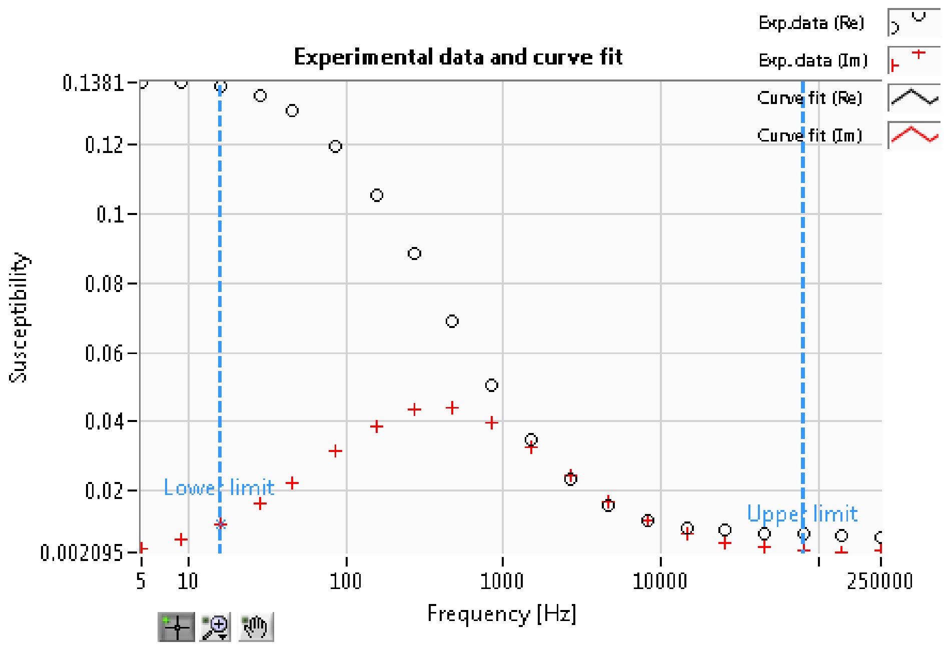


Figure 16. "Crop data" checkbox activates data cropping. Cursors then appear in the graph to allow the user to manually cropping out what measured data that should be included in the curve fit.

2.3.6.3 *Curve fit results*

The “Curve fit results” section is located down to the right in the ”Data analysis” tab. The presented curve fit parameters are a mean value of the separate parameter results from the real and the imaginary part respectively, weighted with the goodness of the fit for each part (R^2-value). Depending on the chosen model the corresponding model parameters will be listed in the tables below.

Table 1. Curve fit parameters : Multi-core model

Median diameter (nm)	Median hydrodynamic particle diameter
sigma, σ	Width of the log-normal particle size distribution (geometrical standard deviation)
χ_{0B}	DC susceptibility for the particles that undergo Brownian relaxation
χ_{high}	AC susceptibility at frequencies much higher than the Brownian relaxation frequency
χ_0	Total DC susceptibility, i.e. $\chi_0 = \chi_{0B} + \chi_{high}$, where χ_{high} is the susceptibility contribution from high frequencies
f_{max} *(kHz)**	Frequency where the imaginary part is at its maximum and is calculated from the median particle diameter.
$\tau_{B\ median}$ *(ms)**	Median Brownian relaxation time for the measured particle distribution (ms). The relaxation time is calculated from the frequency maximum ($\tau_{B\ median} = 1/(2\pi f_{max})$).

* Not included in curve fit model, but calculated from the other parameters.

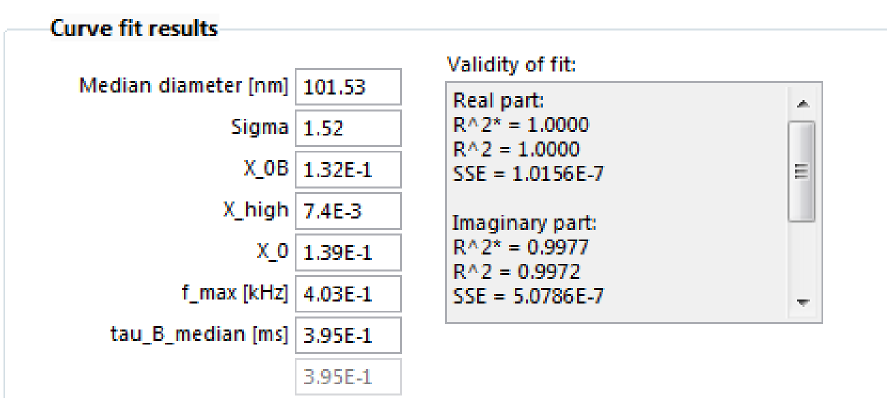


Figure 17. Example of curve fit results using the Multi-core model.

Table 2. Curve fit parameters : Extended multi-core model

Median diameter (nm)	Median hydrodynamic particle diameter (nm)
sigma, σ	Width of the log-normal particle size distribution (geometrical standard deviation)
χ_{0B}	DC susceptibility for the particles that undergo Brownian relaxation
χ_{0N}	DC susceptibility for the particles that undergoes Néel relaxation
τ_N *(ms)*	Néel relaxation time (ms)
α	Distribution parameter, $0 < \alpha < 1$ for the Néel relaxation
f_{max} *(kHz)**	Frequency where the imaginary part is at its maximum
$\tau_{B\ median}$ *(ms)**	Median Brownian relaxation time for the measured particle distribution (ms). The relaxation time parameter is estimated in from the curve fit in the data analyzing part. ($\tau_{B\ median} = 1/(2\pi\ f_{max})$)

* Not included in curve fit model, but calculated from the other parameters.

Table 3. Curve fit parameters : Single-core model

Median diameter (nm)	Median particle magnetic core diameter
sigma, σ	Width of the log-normal particle core size distribution (geometrical standard deviation)
C	Coefficient that includes temperature, particle number density, and intrinsic particle magnetization.
χ_{high}	High frequency AC susceptibility response (real part)

Table 4. Curve fit parameters : Cole-Cole model

τ *(s)*	Relaxation time (s)
α	Width of the relaxation (size) distribution, ($0 < \alpha < 1$)
χ_0	DC susceptibility for the particles that undergo Brownian relaxation
χ_{high}	High frequency AC susceptibility response (real part)
χ''_{peak}*	Peak value of the imaginary AC susceptibility response

* Not included in curve fit model, but calculated from the other parameters.

As a measure of how well the model fits to the experimental data three validity parameters are presented together with the results of the curve fit (Table 5).

Table 5. Validity of the fit (same for all three models)

R^2	The coefficient of determination, which is a statistical measure of the goodness of a fit. ($0 < R^2 < 1$, where a higher value indicates a better fit .
*R^2**	The adjusted R^2 value. This is a modification of R^2 value with respect to the number of fit parameters used in the model.
SSE	The sum of squares of errors (residuals).

2.3.6.4 *Model plot tool*

From software version B1k the data analysis part has been expanded with a *Model Plot Tool*. This feature makes it possible to plot the included susceptibility models by manually enter the input parameters. The susceptibility model data and the experimental data, if there is some, will be plotted together in the left figure in the "Data Analysis" tab window (see Figure 12). The particle size distribution will be plotted in the figure to the right, according to the size parameters entered by the user.

The buttons in the middle called "Curve fit mode" and "Plot tool mode" are used to switch between the two modes. When in the "Plot tool mode" the chosen susceptibility model can be plotted by clicking on the button "Plot model" (Figure 18).

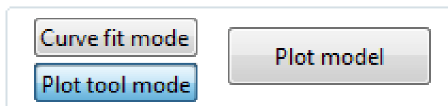


Figure 18. Buttons used for switching between "Curve fit mode" and "Plot tool mode".

Similar to the "Curve fit mode" the "Settings" box down to the left (Figure 19) are used for selecting which susceptibility model to use and to define some of the additional parameters according to:

- *Viscosity [Pa s]*: Viscosity of the liquid surrounding the magnetic particles. (default value = 1E-3, corresponding to water at room temperature)
- *Temperature [K]*: Temperature in sample (default value = 295)
- *Characteristic relaxation time [s]* (τ_0): (default value = 1E-10)
- *Magnetic anisotropy [J / m^3] (K)*: (default value = 1E+4)
- *Surface layer thickness [nm]*: Thickness of the outer surface layer of the particles, in this case the difference between the hydrodynamic radius and the magnetic core radius. (default value = 1)
- *Max plot freq. [Hz]*: Maximum plotting frequency (default = 500 kHz)

Parameters that are not used for a given model are greyed out and disabled.

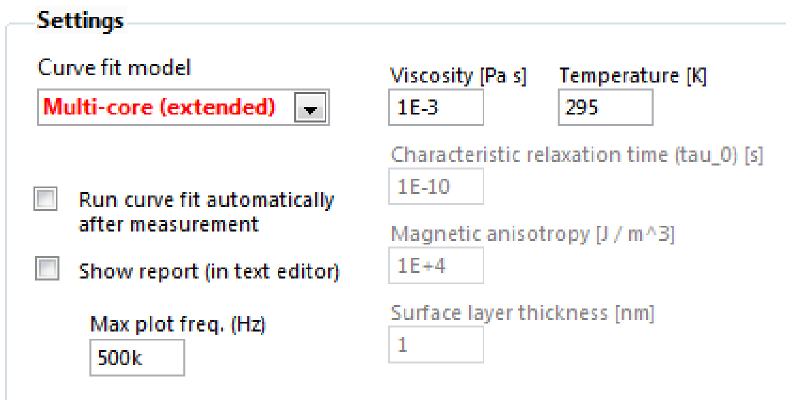


Figure 19. Settings for the "Plot tool mode".

The "Model plot parameters" box located down to the right contains the corresponding curve fit parameters for the chosen model. When in the "Plot tool mode" these parameters can be modified by the user without any limitations. For further information about these parameters, see chapter 2.5.

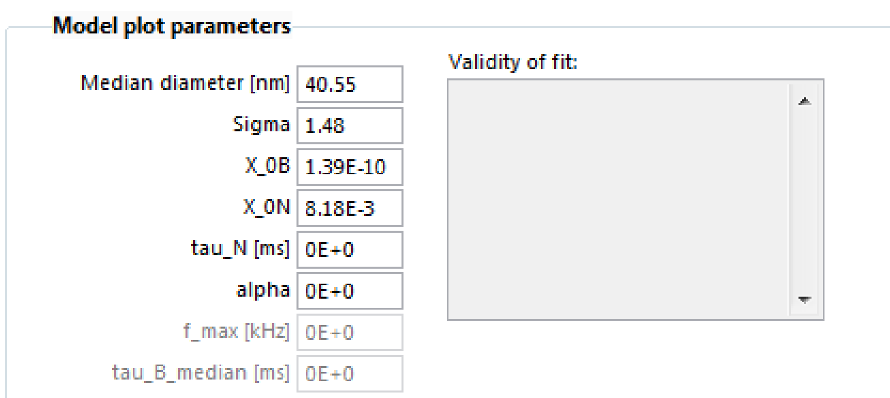


Figure 20. In the "Plot tool mode" the model parameters can be modified by the user.

Please note: When plotting a model the same set of parameters are used for both the real and the imaginary part of the susceptibility. The results of the curve fit shows the fit result for real- and imaginary part separately, but the presented curve fit parameters (showed in Figure 17) are a weighted mean value from the separate results (see chapter 2.3.6.3). As a consequence, plotting a model by using the resulting parameters from a curve fit may result in a slightly different susceptibility curves compared those plotted as a result of the curve fit.

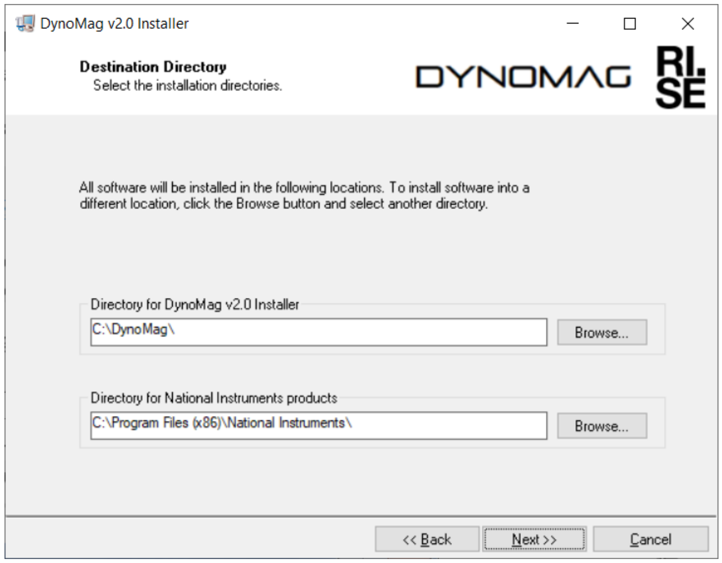
2.4 Installation guide for DynoMag Software

The DynoMag software is normally pre-installed on the supplied PC and should only be reinstalled in the case of a PC hardware failure, or if the DynoMag software gets corrupted for some reason.

The **Installer package** which is provided by RISE AB will install the DynoMag Software together with all necessary supporting software libraries and drivers (*NI LabVIEW Runtime Engine 2022 Q3* and *MATLAB Runtime R2022b*). The package also contains this manual.

To install the DynoMag software on a computer, please follow the steps below:

1. Start the installation by run the Installer file called **"setup.exe"** and follow the instructions. This will install the main program to a user specified directory plus all supporting software and drivers. In order to avoid write-protection issues we recommend to not install the software in Windows "Program Files" directory. The default path is set to "C:\DynoMag\".



2. The **FTDI-drivers** for USB communication should normally be installed automatically by Windows when the instrument is connected (and powered up) to the PC the first time. Otherwise please visit https://ftdichip.com/drivers/vcp-drivers/ and download the VCP (Virtual COM Port) drivers for your operating system.

3. The DynoMag Software should now be ready to be started from the Start menu under “DynoMag” or from the program directory by running the application file **DynoMag.exe**.

2.5 Modeling of the AC susceptibility

2.5.1 Multi-core model

In order to model the AC susceptibility for a magnetic nano-particle system that undergo Brownian relaxation (nano-particles that contain single-domain crystals with slower internal Néel relaxation than the Brownian particle relaxation) with a distribution of Brownian relaxation times (due to a distribution of hydrodynamic sizes of the particles), the Debye model is integrated over the size distribution according to (the AC susceptibility is built from two parts, the Brownian relaxation part and the Néel relaxation part):

$$\chi = \int \frac{\chi_{0B}(r_H)}{(1 + j\omega\tau_B(r_H))} f(r_H) dr_H + \chi_{high} = \chi_{0B} \int \frac{1}{(1 + j\omega\tau_B(r_H))} f(r_H) dr_H + \chi_{high} \quad [4]$$

where χ_{0B} is the DC susceptibility for the particles that undergo Brownian relaxation, χ_{high} is the dynamic susceptibility at frequencies much higher than the Brownian relaxation frequency (due to single-domain crystals with fast Néel relaxation with respect to the Brownian relaxation), ω the angular frequency ($2\pi f$), r_H the hydrodynamic radius of the particles, $f(r_H)$ is the hydrodynamic radius distribution function, and τ_B is the Brownian relaxation time according to:

$$\tau_B = \frac{3V_H\eta}{k_B T} = \frac{4\pi r_H^3 \eta}{k_B T} \quad [5]$$

where V_H is the hydrodynamic volume, η the viscosity of the liquid the magnetic nano-particles are placed in, k_B the Boltzmann constant and T is the temperature.
In the last step in equation 4, the DC magnetic susceptibility of the Brownian part is assumed to be independent of the hydrodynamic volume of the particles. This assumption has been tested in a large number of fittings, and the approximation gives results which are comparable to other methods determining the hydrodynamic size. The total DC susceptibility at zero frequency is $\chi_0 = \chi_{0b} + \chi_{high}$. In the DynoMag software, this model is referred to as the *multi-core model* and it can be used for magnetic multi-core particles (meaning that the magnetic particles containing several numbers of single-domains). This model should also be used when the Néel relaxation part is at much higher frequencies than the Brownian relaxation part.

By fitting experimental data of the dynamic susceptibility to the above described model, equation 4, it is possible to determine the hydrodynamic size distribution of the magnetic nano-particle system provided that the particles contain single-domain

crystals with slow internal Néel relaxations with respect to the particle Brownian relaxation.
The log-normal distribution is used for the hydrodynamic radius distribution function, $f(r_H)$, and is expressed as:

$$f(r_H) \propto \frac{1}{r_H} e^{-\frac{1}{2\ln^2\sigma}\ln^2\left(\frac{r_H}{r_{mH}}\right)} \qquad [6]$$

in which σ sets the width of the distribution (geometrical standard deviation).

The log-normal distribution is seen below for the same median hydrodynamic radius, r_{mH} (5 nm) but with different distribution widths (σ (sigma) values). The sigma values starts at 1 which gives a delta distribution function, meaning a particle system with only one hydrodynamic size.

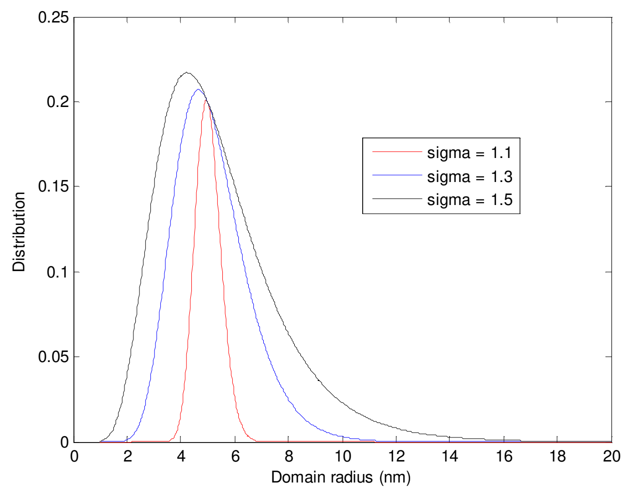


Figure 21. The log-normal distribution versus hydrodynamic particle radius with a median radius of 5 nm and plotted for different widths (the sigma value).

As can be seen in Figure 21, the width of the distribution increases with increasing sigma. The sigma value can also be expressed in size units (radius in this case) according to

$$\sigma_r = r_{mH} e^{\ln^2\sigma/2}\sqrt{e^{\ln^2\sigma} - 1} \qquad [7]$$

where the width, σ_r, is now expressed in the same unit as the particle radius.

2.5.2 Extended multi-core model

In the DynoMag software it is also possible to choose an extended multi-core model where the Cole-Cole expression is used to model the Néel relaxation part. In this case the Néel relaxation part does not have to be at much higher frequencies than the Brownian relaxation part which is the case for the basic multi-core model described earlier. This model can be used when the Néel relaxation is more or less overlapping the Brownian relaxation in frequency. The extended multi-core model is described according to

$$\chi(\omega) = \chi_{0_B} \int \frac{1}{\left(1 + j\omega\tau_B\left(r_H\right)\right)} f\left(r_H\right) dr_H + \frac{\chi_{0_N}}{1 + \left(j\omega\tau_N\right)^{\alpha}} \qquad [8]$$

where the last part in equation 8 is the Cole-Cole expression that describe the AC susceptibility contributions from the Néel relaxation. χ_{0N} is the DC susceptibility due to the Néel relaxation, τ_N is the Néel relaxation time and α describes the degree of distribution of the Néel relaxation times (due to size distribution of the single-domains and/or magnetic interactions between the single-domains) and can have the values $0 \leq \alpha \leq 1$. The result of this modelling gives the size distribution of the particles, the DC susceptibility contributions from the Brownian and Néel relaxation process and the distribution of the Néel relaxation times (through the relaxation time distribution parameter α).

2.5.3 Single-core model

In the case of measuring on single-core particles (magnetic particles containing only one single-domain crystal), the single-core model should be selected. The AC susceptibility can in this case be described by

$$\chi(\omega) = C \int \frac{r_C^6}{\left(1 + j\omega\tau_{eff}(r_C,\delta)\right)} f(r_C) dr_C + \chi_{high} \qquad [9]$$

in which C is a coefficient that includes temperature, particle number density, and intrinsic particle magnetization, r_C the radius of the magnetic single-domain crystal, χ_{high} the AC susceptibility contribution at higher frequencies and τ_{eff} is the effective relaxation time given by:

$$\frac{1}{\tau_{eff}} = \frac{1}{\tau_B} + \frac{1}{\tau_N} \qquad [10]$$

In this expression, τ_B is the Brownian relaxation time defined earlier in the text and τ_N is the Néel relaxation time described by:

$$\tau_N = \tau_0 e^{\frac{KV_C}{k_B T}} \qquad [11]$$

where τ_0 is characteristic relaxation time (in the range of 10^{-10} s), K the magnetic anisotropy of the single-domain crystals (the bulk value of K for magnetite particles at room temperature is in the range of 10^4 J/m^3), V_c the magnetic core volume of the particle, k_B the Boltzmann constant and T the temperature. The relation between the hydrodynamic radius, r_H, and the magnetic single-domain radius, r_C, is:

$$r_H = r_C + \delta \qquad [12]$$

where δ is the thickness of the hydrodynamic surface layer.

The parameters, η, τ_0, K and δ, can be set in the software before the fitting starts. The result from the single-core fitting procedure is the size distribution of single-core particle system, the high frequency contribution, χ_{high}, and the coefficient C.

2.5.4 Cole-Cole model

The Cole-Cole model is a dynamic response model that can be used for both multi-core and single-core particles and it is described by

$$\chi = \frac{\chi_0}{1+(j\omega\tau)^{\alpha}} + \chi_{high} \qquad [13]$$

where χ_0 is the DC value of the frequency dependent part of the susceptibility, τ the relaxation time in (s), α is the Cole-Cole parameter ($0 < \alpha < 1$) defining the width of the relaxation (size) distribution and χ_{high} is the high frequency contribution.

3 Measurement instructions

3.1 Sample preparation and sample mounting

Together with the DynoMag system follows a few glass sample vials. New sample vials can be purchased from VWR international (Cat./Art. No 548-0042).

The DynoMag instrument is designed for sample volumes of 200 µl, but smaller volumes can also be measured if the calibration sample is changed accordingly (see the text in the calibration chapter).

For liquid samples, slowly fill the glass sample vial with 200 µl of the substance to be measured. If some of the liquid have contaminated the upper walls of the sample vial, refill another sample vial with new liquid.

For powder samples, fill the sample vial with enough amounts so that the height of the powder corresponds to the calibration sample (ca. 6 mm). Weigh the sample vial before and after the filling of the powder so you have the mass of the powder. Instead of the sample volume in the setting window, write the mass of the powder sample in milligrams. The determined susceptibility in this case will be the mass susceptibility (in SI units m^3/kg).

The sample is mounted from the top of the instrument and the opening is protected by a sliding lid. To insert the sample vial, first open the lid by slide it backwards. Mount the sample carefully into the vial holder and make sure it has reached the bottom. Close the lid by sliding it back again. The lid must be closed during the whole calibration or measurement procedure.

Figure 22. The sample is mounted from the top and the opening is protected by a sliding lid.

Figure 23. Picture of a sample vial mounted in the instrument. .

3.2 Starting and stopping the measurement

Set the measurement parameters according to the software chapter (chapter 2.3.2) and press the start button on the main program panel to the left. - The data points of the real and imaginary part of the dynamic susceptibility will continuously be displayed on the susceptibility graph (chapter 2.3.5).

The measurement stops automatically when the last frequency point has been measured. When the data is fitted to the susceptibility model (described in chapter2.3.6), the result of the fitting is shown in the graphs. The data is automatically stored after the measurement. If the user does not want to save the data, press the cancel button when the system asks the user for the file name.

If the measurement for some reason must be cancelled, then the stop/save button can be pressed on main panel to the left. The program will then terminate the measurement and proceed to the data fit and after that continue to the save routine.

After the measurement is finished, the sample moves automatically to the top of the coil system and the sample can be dismounted.

3.3 Interpretation of the data

The graph below shows the real and imaginary part of a typical magnetic nano-particle system with a single magnetic relaxation frequency of 1 kHz. It could be from a magnetic nano-particle system with a very sharp size distribution and that the particle contains single-domain crystals that have internal relaxations longer than the Brownian relaxation of 1 kHz (particle diameters of about 75 nm at room temperature).

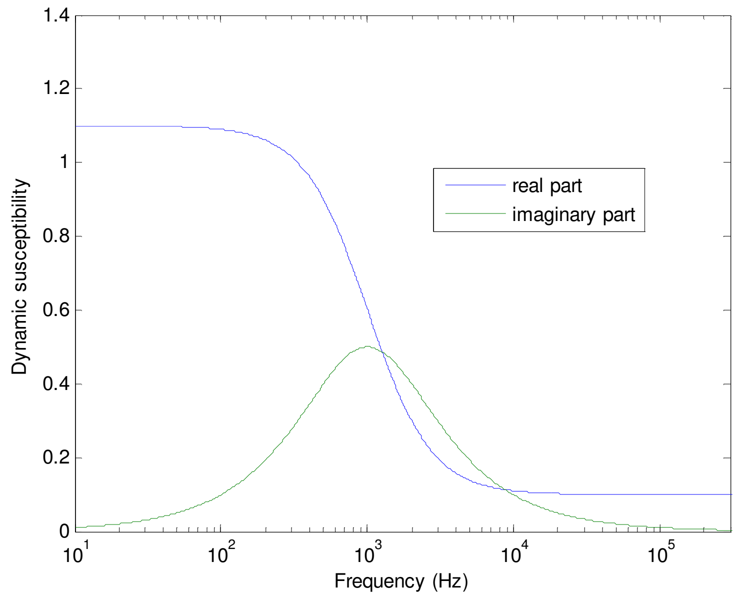


Figure 24. Real and imaginary part of the dynamic susceptibility versus frequency for a magnetic nano-particle system with a single relaxation frequency at 1 kHz.

At low frequencies compared to relaxation frequency the magnetization can follow the excitation field. At these low frequencies the real part of the susceptibility is more or less constant in frequency and the imaginary part is close to zero. At frequencies close to the relaxation frequency the magnetization lags the excitation field and the real part decreases and the imaginary part exhibits a maximum at the relaxation frequency.

The imaginary part corresponds to energy loss of the particle system, and it absorbs maximum energy at the frequency where imaginary part shows a maximum. At higher frequencies compared to the relaxation frequency both the real and the imaginary part decreases to small values. At these frequencies the magnetization due to the relaxation at 1 kHz has difficulties to follow the excitation field.

If the particles contain single-domain crystals with fast internal relaxation compared to the relaxation at 1 kHz there is a non-zero component of the real part of the susceptibility at higher frequencies which is shown in Figure 24. At these frequencies only the magnetization part due to these fast internal relaxations of the single-domains can follow the excitation field.

The real and imaginary values can be visualized in the complex plane (almost the same when studying AC electric circuits).

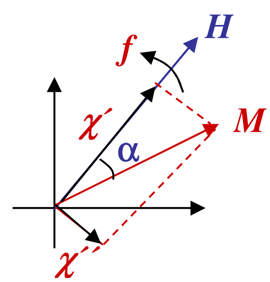


Figure 25. The excitation field and the phase lagged magnetization. The definitions of the real and imaginary part of the dynamic susceptibility are also shown.

The phase angle between the excitation field and the magnetization is given by:

$$\tan\alpha = \frac{\chi''}{\chi'} \qquad [14]$$

In a real magnetic nano-particle system there is always some distribution of relaxation frequencies. This gives a wider AC susceptibility spectrum than shown in Figure 24.

4 Calibration

4.1 Introduction

The DynoMag system is calibrated by a two-step routine. In the first step, the response is measured with an empty sample vial. The effect of this measurement is to detect the difference in signal when the empty sample vial is in the upper coil to when the sample vial is in the lower coil. The difference is due to a variety of phenomena, for example dielectric and diamagnetic properties of the sample vial and the mechanical arm moving the sample. The difference signal is a complex number and it is a background level which is subtracted from the measured signal in the subsequent measurements.

The second step is performed with a sample containing a material with a known and preferably frequency independent magnetic susceptibility. In DynoMag a Dysprosium oxide, Dy_2O_3, a paramagnetic sample is used. The real part of a paramagnetic sample is completely constant in the whole frequency range of the DynoMag system. The dimensions of the calibration sample should be the same as the measurement samples, in order to have the same coupling factor in the detection coil system for calibration sample and measurement samples.

The frequency dependency of the gain and the phase is a major concern in construction of a high bandwidth AC susceptometer. The frequency dependency of the gain and the phase shift becomes strong at high frequencies, especially at frequencies close to the resonance frequency of the detection coil system or the excitation coil system. The gain and phase shift can also become frequency dependent due to the properties of the excitation electronics and/or the detection electronics. The measured sample data will become incorrect, especially at high frequencies, if these effects are not compensated for.

The frequency dependency of the gain and the phase shift is compensated for using a calibration routine at many different frequencies through the whole frequency range of the DynoMag system. The measured complex voltage from the calibration sample, V_{cal}, divided with the frequency subtracted with the background level, V_b, divided with the frequency, gives the frequency dependent complex calibration factor, *C(f)*, according to:

$$C(f) = \frac{m_{cal}}{\left(\frac{V_{cal}}{f_{cal}} - \frac{V_b}{f_{cal}}\right)} \qquad [15]$$

where f_{cal} is the used frequencies for the calibration procedure and m_{cal} is the magnetic moment from the calibration sample given by:

$$m_{Cal} = \chi_{mass} \cdot mass \cdot H_0 \qquad [16]$$

Where χ_{mass} is the mass susceptibility in m^3/kg of the calibration sample, *mass* is the mass of the calibration substance in kg and H_0 is the calibration field amplitude in A/m (the field amplitude in the DynoMag system is 0.5 mT (5 Gauss)= 398 A/m). The reason for dividing with the frequency is due to that the induced voltage is directly related to the frequency (see the introduction chapter). From the above calibration factor, *C(f)*, the frequency dependent gain and phase calibration factors (see chapter 2.3.4) can be obtained.

In the DynoMag system a paramagnetic material (Dy_2O_3) is used in which the real part of the susceptibility is constant and the imaginary part is equal to zero in the entire DynoMag frequency range.

The room temperature value of the [2]mass susceptibility of Dy_2O_3 is $3.020 \cdot 10^{-6}$ m^3/kg (SI units). For each frequency point that is measured, the mass susceptibility is compensated using the current measured temperature in the coils. A new calibration value (valid for Dy_2O_3 powder) is showed to the user in the program front panel, $\chi_{mass} = 9.00 \cdot 10^{-7}$ [m^3 K/ g].

$$\chi_{mass} = \frac{9.00 \cdot 10^{-7} \left[\frac{m^3 K}{g} \right]}{T[K]} \qquad [17]$$

The measured sample temperature will also be logged in the *Background* and *Gain&phase* calibration files.

The complex calibration factor is then used to calibrate the measured sample signal to magnetic moment both in magnitude and phase. The DynoMag system then calculates the volume susceptibility (in SI units, unitless) by dividing the magnetic moment with the sample volume and the used excitation field amplitude at each measurement frequency. If the mass susceptibility is chosen the magnetic moment is divided with the mass of the sample and the field amplitude (SI units of mass susceptibility m^3/kg). If the measurement frequency does not coincide with the chosen calibration frequencies the DynoMag program makes a linear interpolation between the data points to find the correct calibration factors.

[2] *D.R. Lide, Handbook of Chemistry and Physics, 79th Edition, CRC Press LLC, 1998*

4.2 Calibration sample

As calibration sample the paramagnetic material Dy_2O_3 is used. For this material the value of the real susceptibility is constant and the imaginary part is zero with frequency (at constant temperature) in the whole frequency range of DynoMag.

A calibration sample is included in the DynoMag system. The sample is sealed with silicone in order to avoid moisture absorption in the calibration sample. The calibration sample can be stored at room temperature.

If a new calibration shall be prepared it is very important to have the right length of the powder sample (the length should be the same as for the measured sample, about 6 mm). The mass of the powder shall also be documented. The mass shall be set in the calibration window before the calibration is performed. New Dy_2O_3 substance (CAS No 1308-87-8) can be purchased from for instance Sigma-Aldrich (99.9 % purity, No 289 264).

4.3 Calibration instructions

A calibration test routine can be used in order to verify that the last calibration files are valid. It is recommended to perform the calibration test routine once every second month or when the instrument has been moved. If the calibration test shows that the DynoMag needs re-calibration, this should be done.

4.3.1 Background calibration

Mount an empty sample vial onto the sample rod. The program will automatically select the optimized frequency points stored in the file *calibration freq points.txt*. Start the calibration by pressing the **start background calibration** button in the calibration window (chapter 2.3.4). Enter comments in the **Comments** window. After the calibration, enter a descriptive filename in the save dialog.

4.3.2 Gain and phase calibration

Load the last proper background calibration file, by pressing the **Select background calibration file** button (chapter 2.3.4). Mount the calibration sample onto the sample rod. The program will automatically select the optimized frequency points stored in the file *calibration freq points.txt*. Start the calibration by pressing the **start calibration** button in the calibration window (chapter 2.3.4). Enter comments in the **Comments** window. After the calibration, enter a descriptive filename in the save dialog.

4.3.3 Test Calibration

A *Calibration Test* can be performed in order to control if the instrument needs to be recalibrated. In this case a standard measurement will be performed, using a calibration sample but in the end some statistics (*mean, standard deviation (σ), RMSE)* will be calculated for the real and imaginary part of the susceptibility respectively. The *RMSE* (*root mean square error* or *root mean square deviation*) is calculated as:

$$RMSE = \sqrt{MSE} = \sqrt{\frac{1}{n}\sum_{i=0}^{n-1}(x_i - y_i)^2} \qquad [18]$$

The calculated *RMSE* value (for both Re and Im-part) will be compared to a control value, *RMSE_limit*, and if the calculated values exceed the control value, the user will be recommended to re-calibrate the DynoMag instrument.

The value for *RMSE_limit* has been defined as $3*10^{-8}$, which corresponds to ~1% of mass susceptibility of the calibration sample.

The *Calibration Test* is started by pressing the **Test Calibration** button in the "Calibration" page. The software will then automatically choose the frequency points from the file *test calibration freq points.txt*. The data will (also automatically) be measured in mass susceptibility because the calibration sample is a powder.

Measurement data will be saved to file as usually (default name: *"calibration_test [date][time].txt*"). An additional log file, which will include the statistical data, can also optionally be stored (default name: *"calibration test statistics [date][time].txt"*).

4.3.4 Calibration history

It is recommended to monitor and compare the last calibration files to previous ones in order to check that the system is stable (for instance by plotting the files).

If the user observes large variations of the calibration factors or if the calibration factors show a continuous changing trend in time, we recommend you to contact with RISE AB for further support.

5 Specifications

Table 6. System specifications.

Property	Value	Comments
Frequency interval	1 Hz – 500 kHz	Measurement accuracy is lower below 5 Hz and above 100 kHz.
Amplitude of excitation field	0.5 mT = 5 G	The magnetic field strength is constant below 1 kHz, falling off at higher frequencies.
Volume susceptibility resolution	$2 \cdot 10^{-5}$	The resolution in magnetic response is determined from the standard deviation of the magnetic signal of the Dy_2O_3 calibration sample at excitation frequency 1 kHz and 0.5 mT field amplitude using 20 data points.
Sample size	Cylindrical sample holder with volume 0.2 cm^3	The sample volume can be customized to smaller volumes than 0.2 cm^3.
Measurement time	Typically around 15 minutes	Depends on the number of data points chosen, 15 minutes is for 20 points
Operating temperature	Normal lab temperatures	It is recommended to use the DynoMag system at normal lab temperatures.