Mageleka

A Quick Start Guide to Making Measurements with the MAGELEKA *Magno*Meter XRS™ Relaxometer

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Measurements using the *M*agno*M*eter XRS are straightforward and fast, and are based are on a simple GUI concept described below. Operation requires minimal training and expertise. This quick start guide is intended for simple training purposes and to allow a novice user to be able to make measurements. For more detailed and in-depth explanations of all the features of the *M*agno*M*eter, the reader should consult the instrument's full manual.

To begin, start the software by double clicking on the "Mageleka icon" on your computer.

Home Screen

The program starts in a default configuration – the "Home" page (Fig. 1).

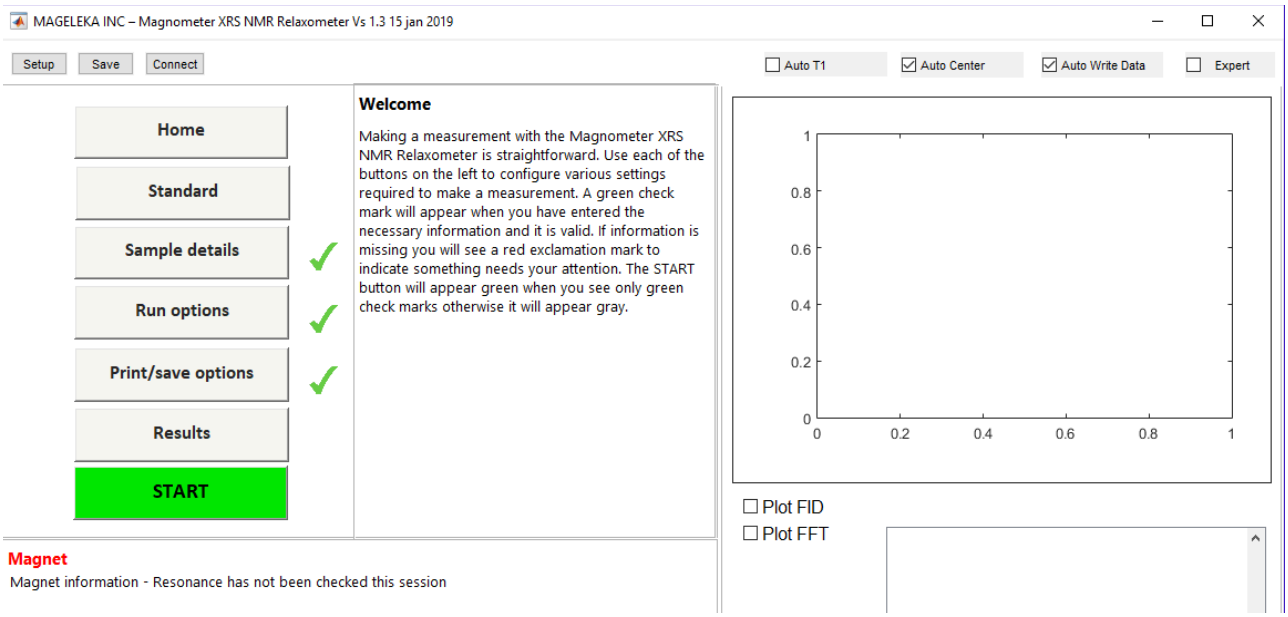

FIGURE 1.

NOTE: The most current version of the software will be displayed at the very top left of the page. In this example it is Version 1.3.

There are four small grey checkboxes at the top right-hand corner:

Auto T1

In normal operation this is unchecked. If checked, the instrument performs an automatic measurement of a T_1 relaxation time – using a Progressive Saturation pulse sequence – prior to a user making a T_2 relaxation time measurement. A more complete explanation of its use is given in the *M*agno*M*eter manual.

Auto Center

In normal operation this should be automatically checked. If checked, the device runs a short frequency sweep before the next scan to make sure the instrument is always on resonance.

Auto Write Data

In normal operation this should be automatically checked. If checked, every set of data, including the short frequency sweep, will be automatically saved. The default filename where the data is saved can be seen by clicking the Print/save options command button.

Expert

This option is not activated (and thus its box not checked), under normal operation. It is used when the instrument is initially installed and, thereafter, either for diagnostic purposes or for those with expertise in running an advanced NMR device. It gives access to many extra parameters – for example, when running samples with very short relaxation times (<10 ms).

NOTE: If there is no spectrometer connected, then the large button located at the bottom left will show the warning message Not Connected in place of Start.

To connect to the spectrometer, press the small Connect button (upper left-hand side).

If/when the spectrometer is connected, then the three large boxes (on the left-hand side) Sample details Run options Print/save options will each have a green check mark ✓ to their right (Fig. 1).

Basic Setup

Before any measurements are made on “unknown” samples, a setup routine should be conducted to ensure that the *M*agno*M*eter XRS is configured and operating correctly. The complete basic set-up operation takes less than 5 minutes.

The first measurement to run is the standard 50 mM copper sulphate ($CuSO_4$) sample that is supplied with the instrument. Click the small Setup button (upper left-hand side). A dialog box will appear prompting you to use the sample (Fig. 2).

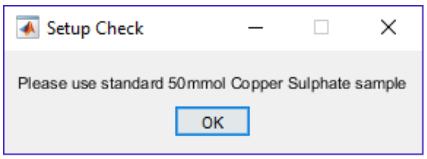

FIGURE 2.

Insert a standard NMR tube containing an aliquot of the $CuSO_4$ solution (see **Note** below) into the *M*agno*Pod* assembly. Then click “OK”.

NOTE: The volume of the $CuSO_4$ solution in the NMR tube is important. Check that the sample is no more than 1 cm in height from the bottom of the NMR tube – approximately 0.5 mL (see Fig. 3 for an example). Use the 1.0 mL syringe supplied.

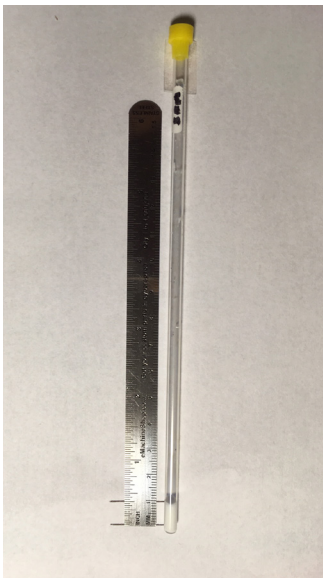

FIGURE 3.

Then click “OK”. The following screen should appear (Fig. 4):

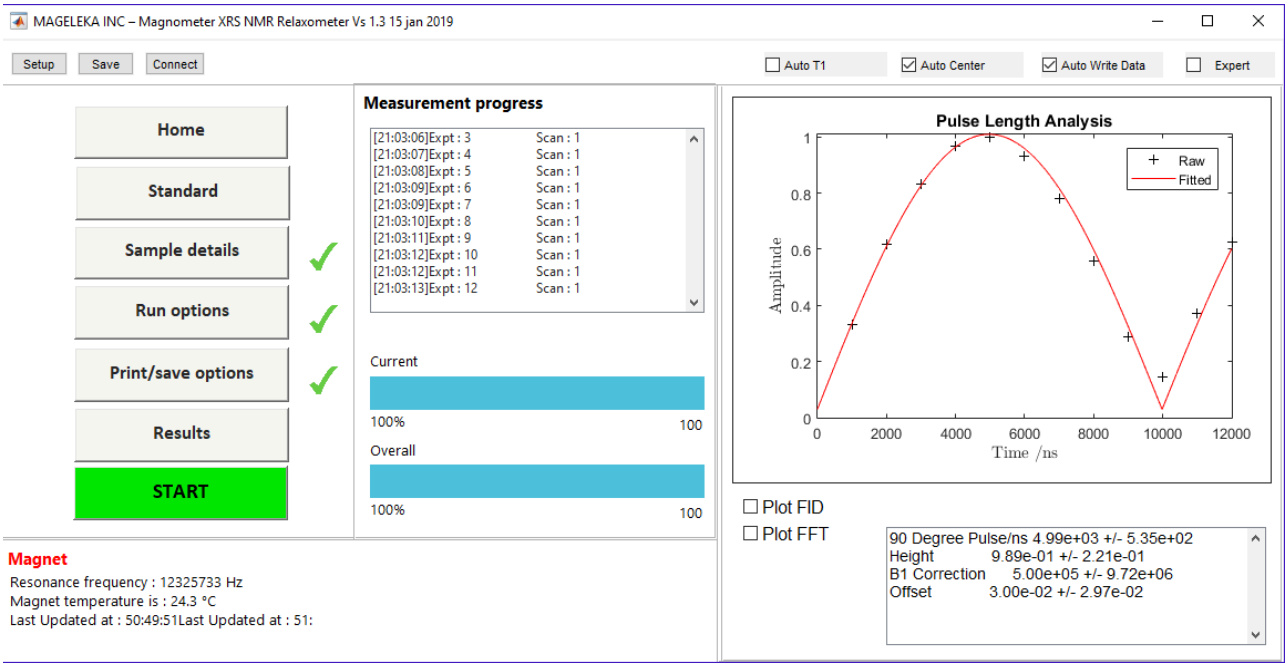

FIGURE 4.

The device now should track through the frequency in 1 KHz increments and set the frequency (shown in the box at bottom left of screen) and, similarly, it sets the pulse length by incrementing it in 1 µs steps and storing the result. Once the scan is complete, a dialog box will appear (Fig. 5) indicating the frequency and pulse length. In this example, the frequency was 12.32 MHz and the 90° pulse length was 4.99 µs.

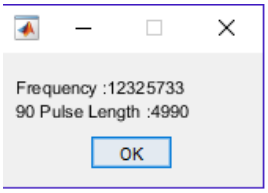

FIGURE 5.

NOTE: Typically the resonance frequency should be ~12.4 MHz and the pulse length 4-6 µs.

Click “OK” to close the dialog box.

Next, click on the Run options command button. The following page will be displayed (Fig. 6):

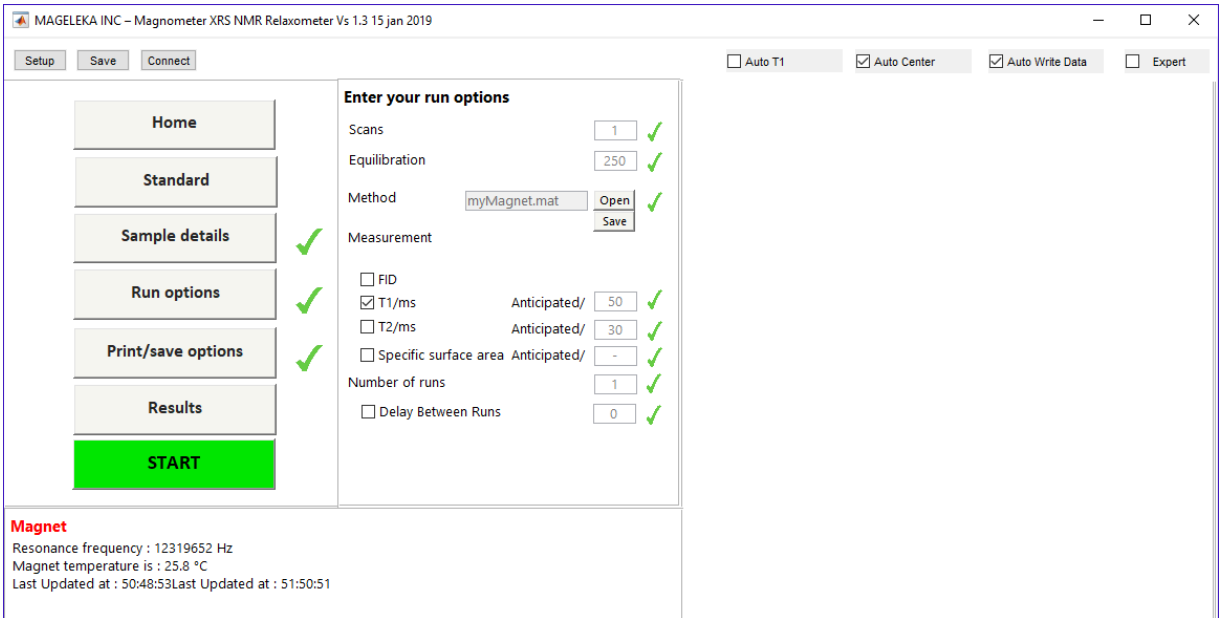

FIGURE 6.

NOTE: The default Anticipated value for T_1 will be 50 (ms) and the "T1/ms" option should be *checked*.

NOTE: Make sure that both the "T2/ms" and "Specific surface area" options are *unchecked*.

Now click the Start button.

On completion of the measurement, the following Result screen should be displayed (Fig. 7):

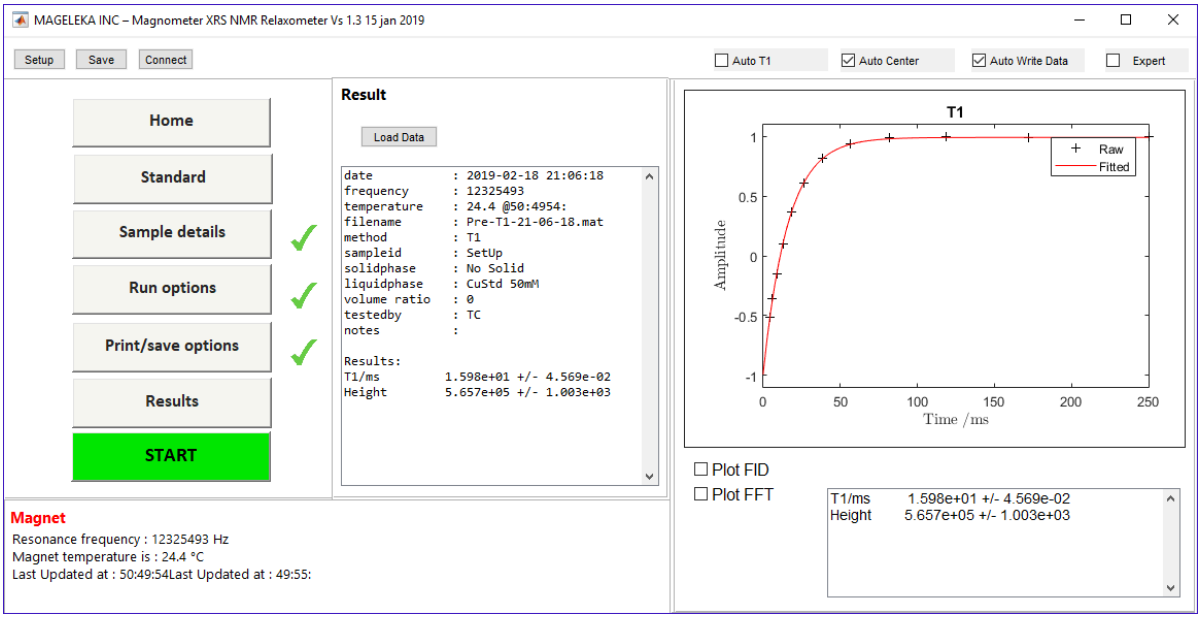

FIGURE 7.

NOTE: A typical value for the T_1 relaxation time of the $CuSO_4$ solution will be within the range 16 ± 1.5 ms (as determined from multiple measurements). In this example it was **15.98 ms** at a magnet temperature of 24.4°C (the error shown in Fig. 6 is the error on the fit to the experimental data points). The value will change with temperature by about 1% per °C.

Clicking the Run options button again, you can now choose to do a T_2 relaxation time measurement by now checking the "T2/ms", as seen in Figure 4.

NOTE: The default Anticipated value for T_2 will be 30 (ms) and the box should have a green check mark to the right.

Press the Start button to initiate the T_2 measurement.

On completion, the following screen should be displayed (Fig. 8):

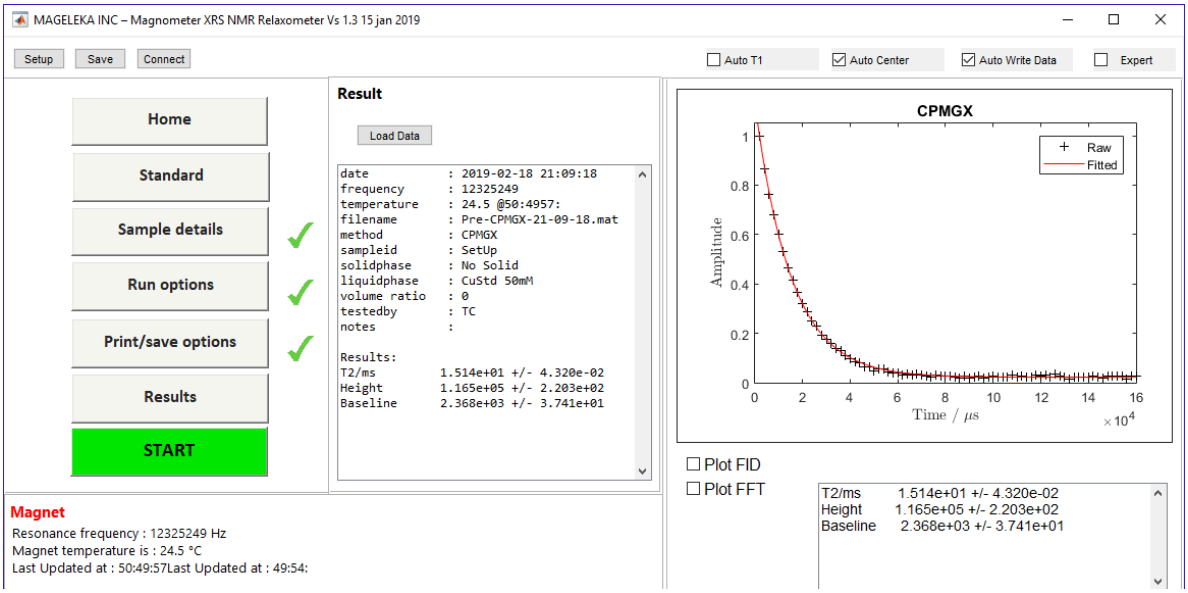

FIGURE 8.

NOTE: A typical value for the T_2 relaxation time of the $CuSO_4$ should be within the range 15 ± 1.5 ms (as determined from multiple measurements). In this example it was **15.14 ms** at a magnet temperature of 24.5°C (the error shown in Fig. 7 is the error on the fit to the experimental data points). The value will change with temperature by about 1% per °C.

The basic setup checklist is now complete.

The instrument is ready and functioning correctly.

A measurement is now made using the supplied Silica Reference sample.

Running the Silica Reference Sample

The supplied Silica Reference sample is a suspension of colloidal-size silica (SiO_2) particles in water, whose wetted surface area is known.

Press the Sample details command button. The screen should display as below (Fig. 9):

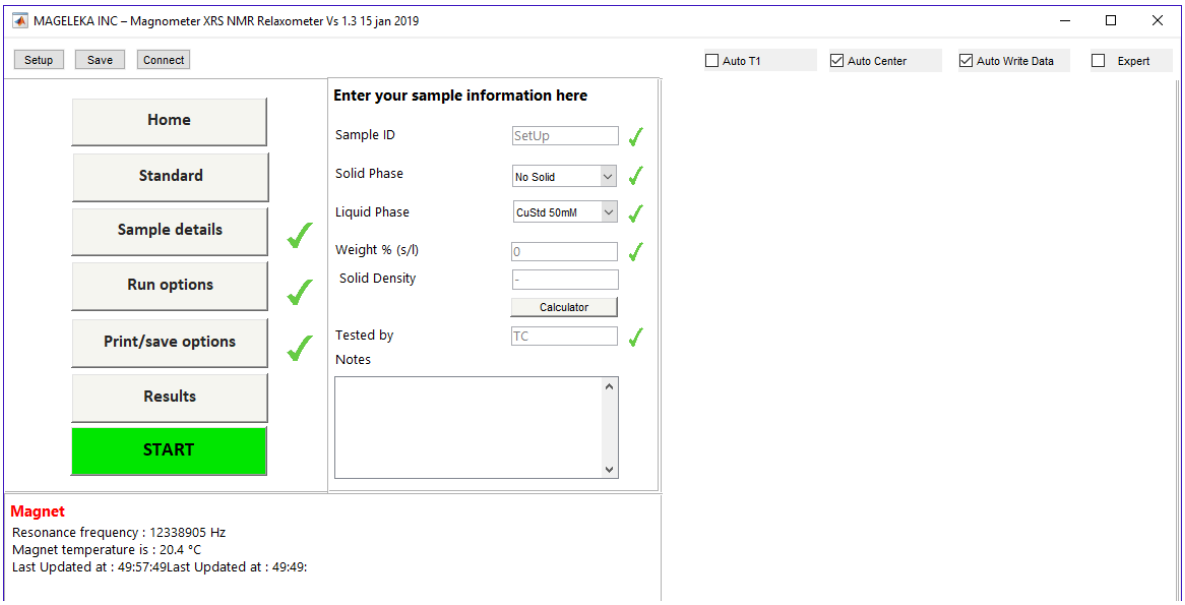

FIGURE 9.

First choose "**Water**" from the pull-down menu found to the right of "Liquid Phase", and then choose "**Silica Std II**" from the pull-down menu found to the right of "Solid Phase". The "Wt% (s/l)" should be entered as "50". The "Solid Density" is automatically set as "2.2".

The screen should now display as (Fig. 10):

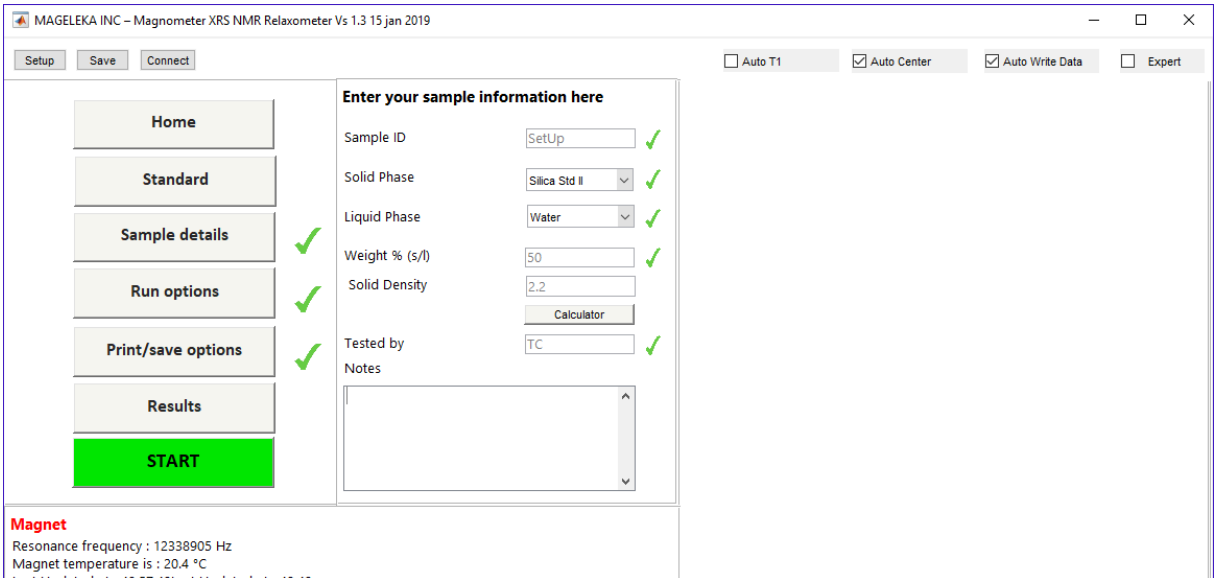

FIGURE 10.

Insert a standard NMR tube containing an aliquot of the SiO_2 reference suspension solution into the *M*agno*P*od assembly.

NOTE: Again, ensure that the volume of the suspension in the NMR tube is no more than 1 cm in height from the bottom of the NMR tube (see Fig. 3 for an example).

Now press the Run options command button. Ensure that both the "T2/ms" and "Specific surface area" options are checked.

NOTE: The anticipated values for both these parameters are already listed.

The screen should now display as (Fig. 11):

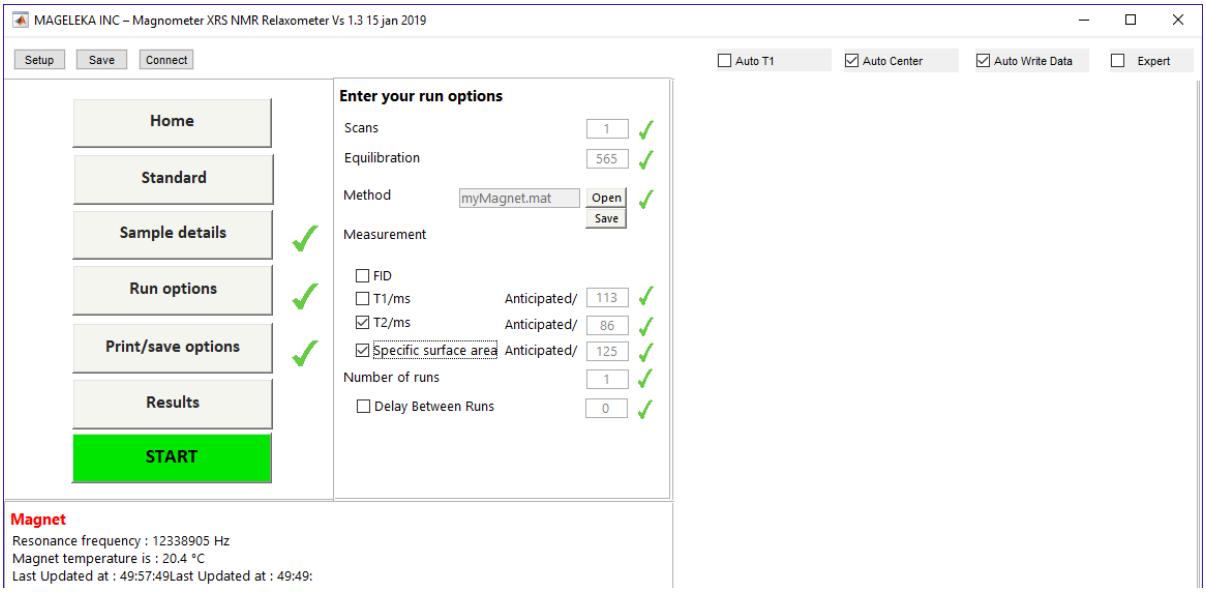

FIGURE 11.

Then press the Start button to initiate the measurement.

A typical (T_2) surface area experimental result for the silica reference sample should look like this (Fig. 10):

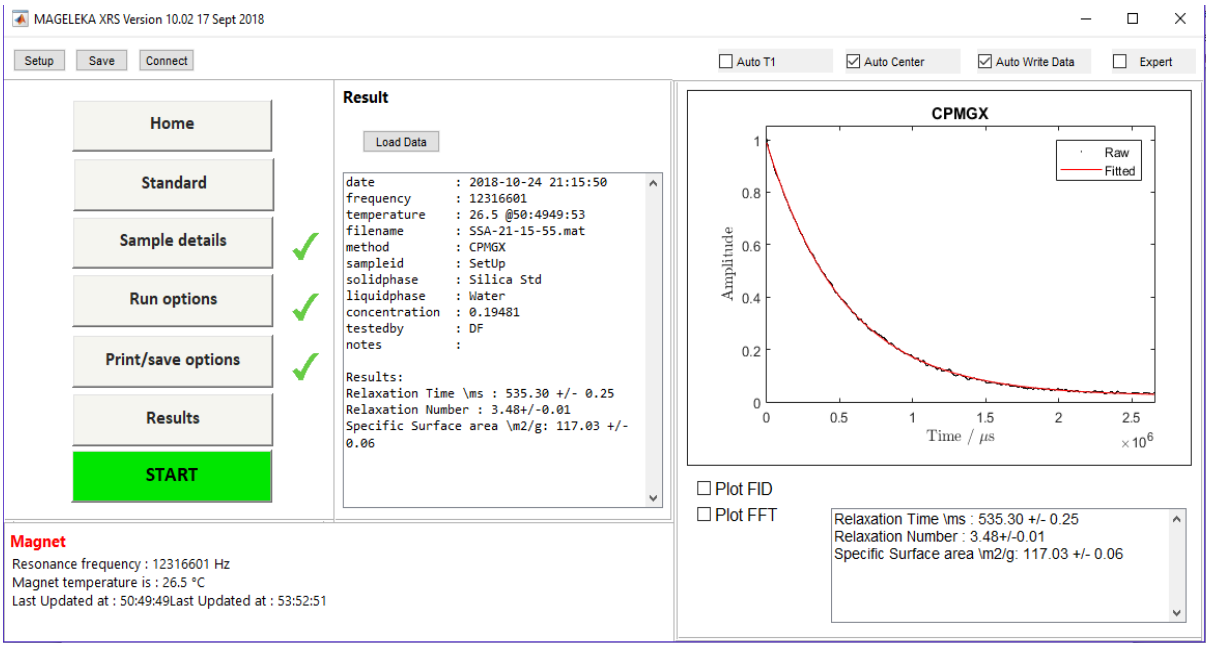

FIGURE 10.

In the example shown in Figure 10, the T_2 relaxation time was determined to be **535 ms** at a temperature of 26.5 °C. The measured surface area was **117 m^2g^{-1}**.

NOTE: The certified value of surface area for the silica reference suspension sample is 120 m^2g^{-1} ± 8m^2g^{-1}.

This result confirms that the methodology for determining the wetted surface area of suspensions is working correctly. The instrument is now ready for measurements on unknown samples.

Running the Zinc Oxide Reference Sample

The Zinc Oxide Reference sample is an alternative (supplied on request) suspension of colloidal-size zinc oxide (ZnO) particles in water, whose wetted surface area is also known. Optionally, this reference material can be measured. The procedure is identical to that for the standard Silica Reference material.

In this case, click the Sample details command button. Again, first choose "**Water**" as the from the pull-down menu located to the right of "Liquid Phase", but now choose "**Zinc Oxide Std**" from the pull-down menu located to the right of "Solid Phase". The "Wt% (s/l)" should be entered as "50". The "Solid Density" is automatically set as "5.6".

Insert a standard NMR tube containing an aliquot of the ZnO reference suspension solution into the *M*agno*P*od assembly.

NOTE: Again, ensure that the volume of the suspension in the NMR tube is no more than 1 cm in height from the bottom of the NMR tube (see Fig. 3 for an example).

Now press the Run options command button. Again, ensure that both the "T2/ms" and "Specific surface area" options are checked

NOTE: The anticipated values for both these parameters are already listed.

Then press the Start button to initiate the experiment.

A typical (T_2) surface area experimental result for the ZnO reference sample should look like this (Fig. 13):

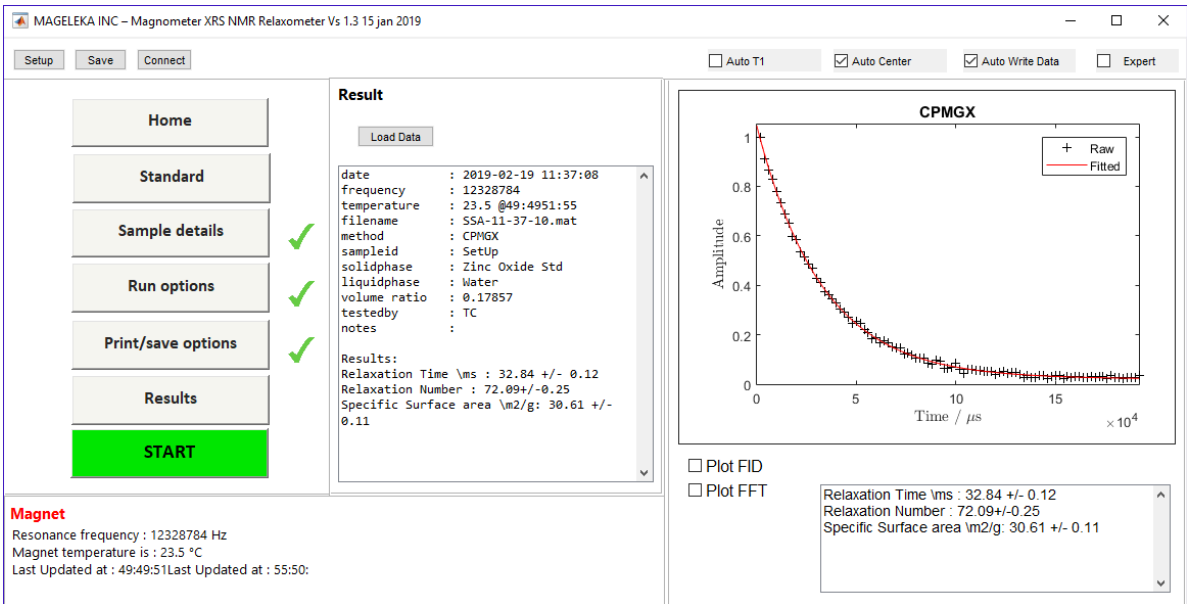

FIGURE 13.

In this example, the T_2 relaxation time was determined to be **32.8 ms** at a temperature of 23.5°C. The measured surface area was **30.6 m^2g^{-1}**.

NOTE: The certified value of surface area for the ZnO reference suspension sample is 30 m^2g^{-1} ± 4 m^2g^{-1}.

Making Measurements on an Unknown Sample

The procedure for making measurements on an unknown sample is similar to those described above.

For pure liquids and solutions it is recommended that they be filtered before taking an aliquot for measurement. Before sampling from suspensions, mix thoroughly to ensure complete homogeneity.

NOTE: The volume of the sample in the NMR tube is important. Check that the sample is no more than 1 cm in height from the bottom of the NMR tube (see Fig. 3 for an example).

A file folder should first be created on the computer where the measurement data will be stored. Then click the Print/save options command button. The screen that appears should look like this (Fig. 14):

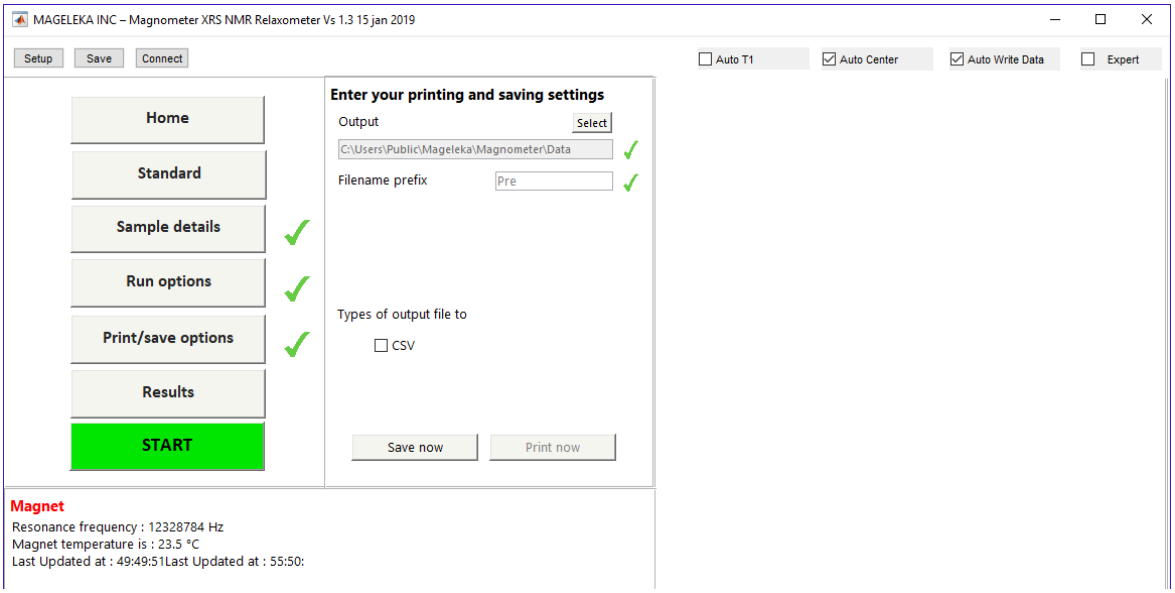

FIGURE 14.

NOTE: If you want to export the results into Excel, highlight the "☐ CSV" box.

Click on the Select button on the upper right of the centre pane and select the folder from the desktop. The filename prefix is an option. The screen should now look like this (Fig. 15):

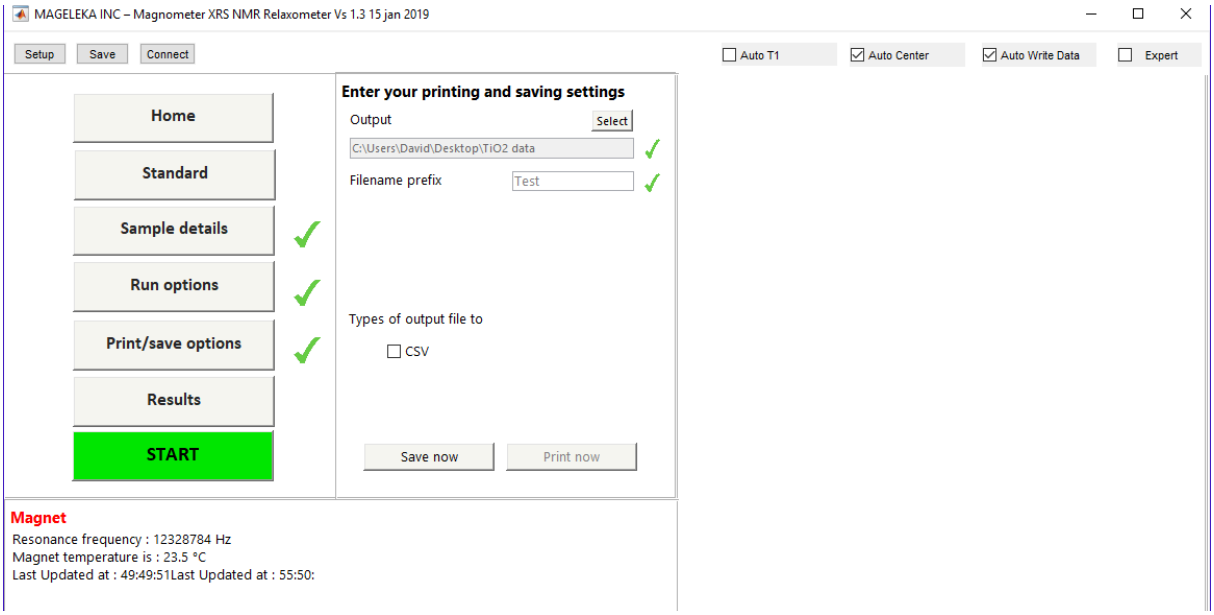

FIGURE 15.

In this example, the folder is “TiO2 data” and the filename prefix is “Test”.

Determination of the Relaxation Value

If all that is needed is a value for relaxation time, then first click the Sample details button and enter details for the “Sample ID”, “Weight % (s/l)”, “Solid Density”, “Tested by”, and any “Notes” as seen here (Fig. 16):

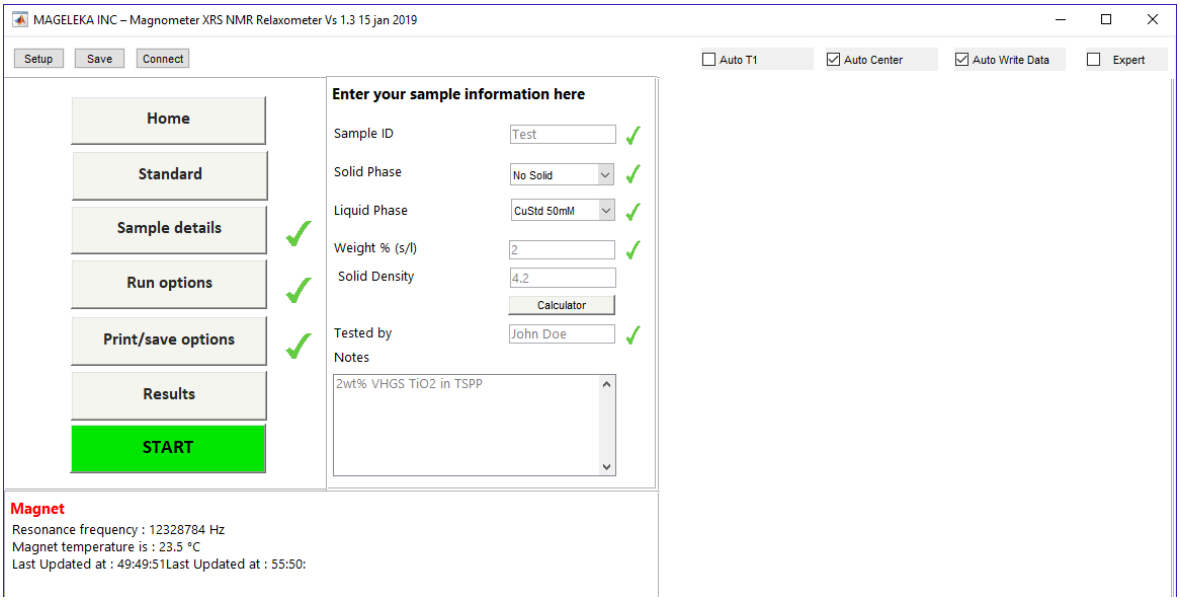

FIGURE 16.

NOTE: Ignore both the “Solid Phase” and “Liquid Phase” boxes. Also, inputting values for “Weight %” and “Solid Density” are optional.

Next, click the Run options button and check the “T1/ms” option.

NOTE: Make sure that the “T2/ms” and “Specific surface area” boxes are *unchecked*.

STEP (1). Set the "Anticipated" time for "T1/ms" to "1250". Leave the "Number of scans" and "Number of runs" each as "1", as can be seen here (Fig. 17):

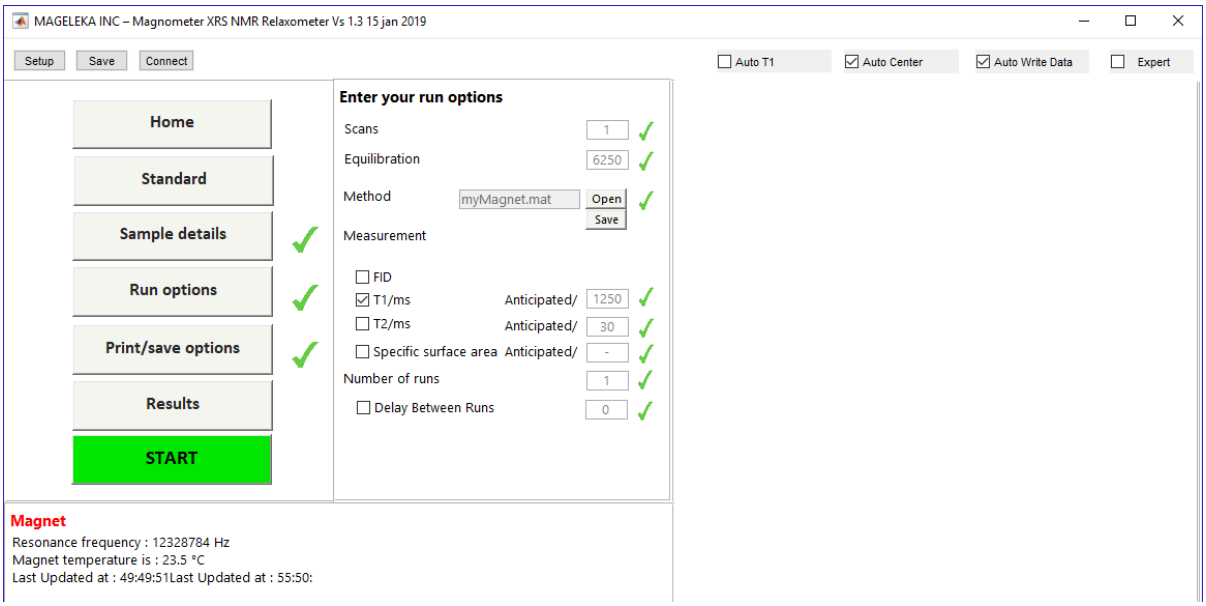

FIGURE 17.

NOTE: The "1250" entered as the "Anticipated" value for "T1/ms" is arbitrary and can be any number. However, for all practical purposes in virtually all fields and applications, the measured relaxation time will generally be between 1 ms and 2500 ms. "1250" was selected because it is the midpoint of this range.

NOTE: The "Equilibration" value is automatically set depending on the "Anticipated" time entered. This is a system parameter – the thermal equilibrium of the spin system – and is set as $5\times T_1$.

NOTE: Check that the sample temperature does not differ significantly from that shown by the *M*agno*P*od LED read-out. If so, insert the NMR tube containing the sample into the *M*agno*P*od and wait at least 2 mins before time starting the first measurement.

NOTE: Important! The small box at the bottom left labelled "Delay Between Runs" should be *unchecked* and left as "0".

Press the Start command button. At the conclusion of the measurement record the T_1 value obtained. The screen will look like this (Fig. 18):

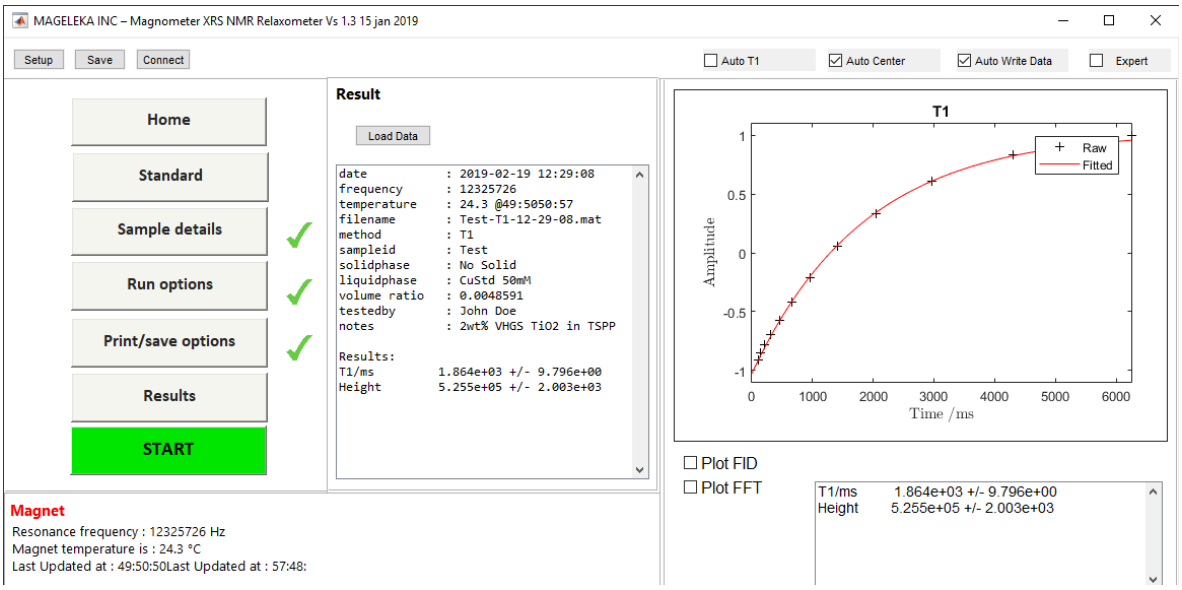

FIGURE 18.

In this example, the T_1 relaxation time was **1864 ms** at 24.3°C.

NOTE: By inputting values for "Wt%" and "Solid Density", the software automatically calculates a Volume Ratio of 0.0048591.

STEP (2). Now, check the “T2/ms” option from the “Run Options” menu. Set the “Anticipated” value for “T1/ms” to the measured value (1810 ms) and the “Anticipated” value for “T2/ms” to 1250 ms.

Again, leave the “Number of scans” and “Number of runs” each as “1” . The screen should look like this (Fig. 19):

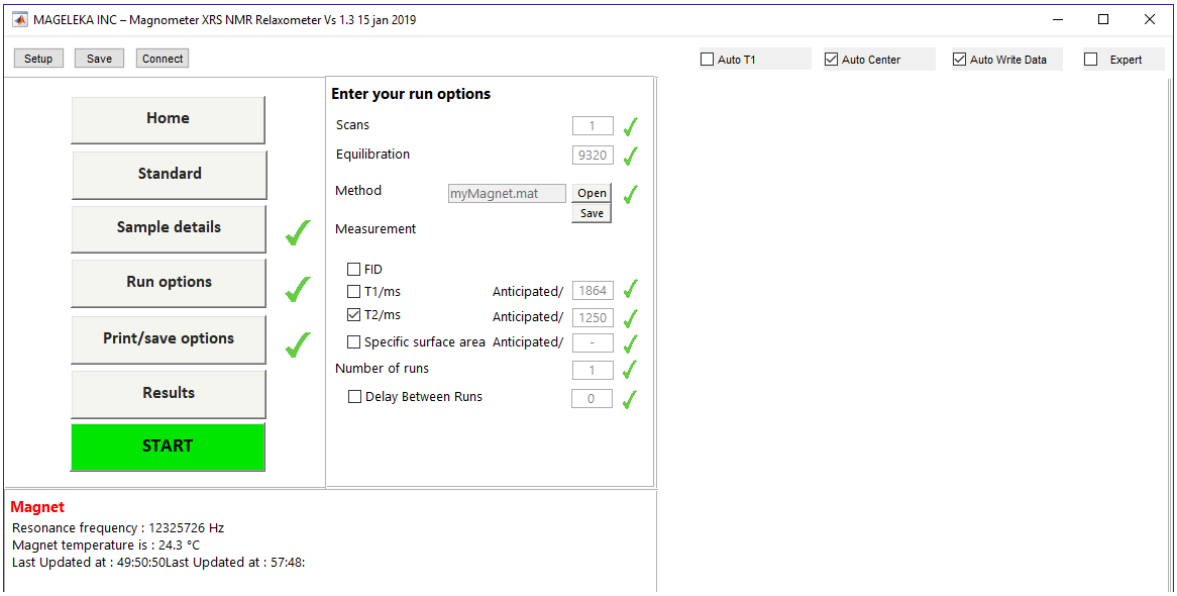

FIGURE 19.

NOTE: Important! The small box at the bottom left labelled “Delay Between Runs” should again be should be *unchecked* and left as “0”.

Press the Start command button.

At the conclusion of the measurement record the T_2 value from the screen that looks like this (Fig. 20):

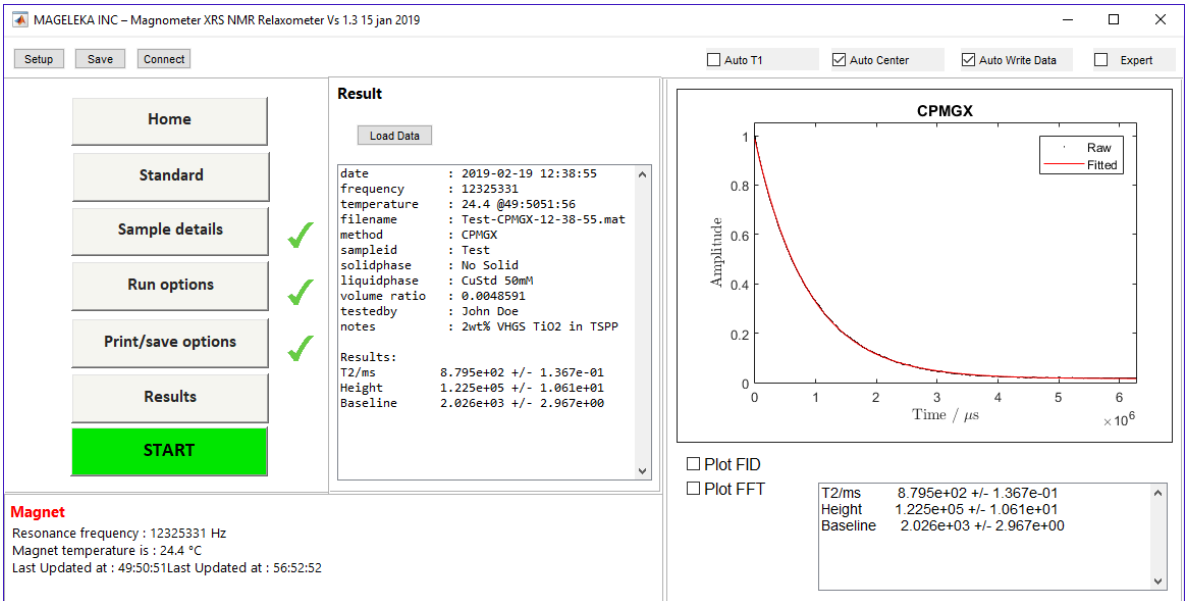

FIGURE 20.

The T_2 relaxation time is **879.5 ms** at 24.4°C.

STEP (3). Repeat the T_2 measurement by pressing the Start command button.

NOTE: Before hitting START, if you check the Run options button you will see that "Anticipated" value for "T2/ms" has automatically been entered (as 879) on the screen below (Fig. 21):

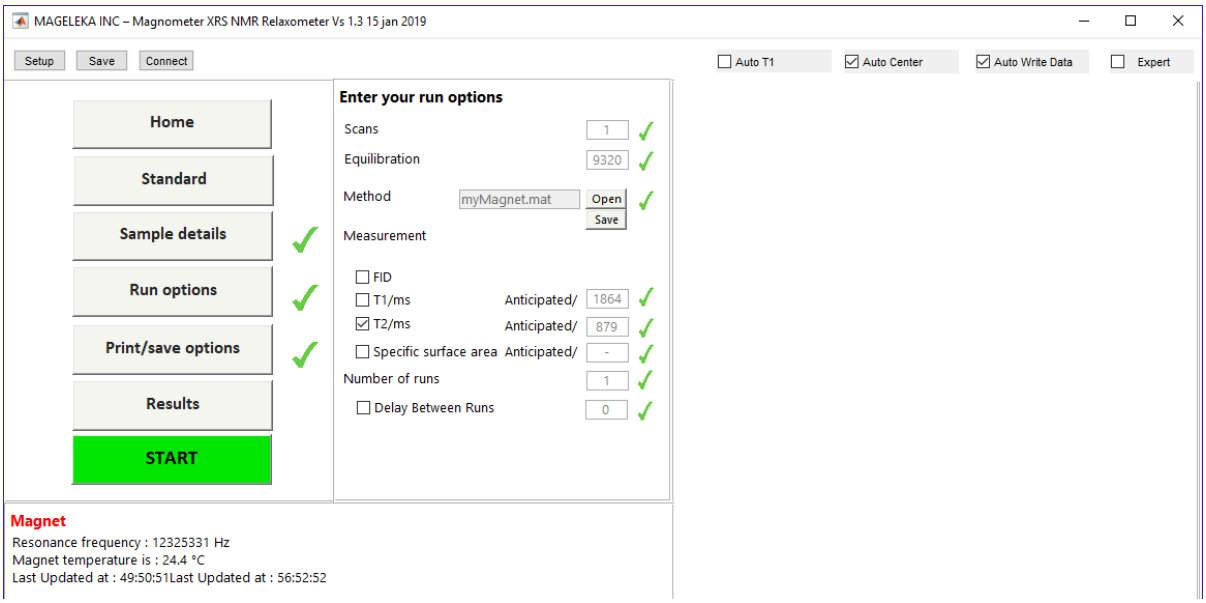

FIGURE 21.

When the measurement is finished record the T_2 value obtained - *which is the Relaxation Value for that sample at the given solids concentration*. In this example it is **890.1 ms** at 24.5°C (Fig. 22):

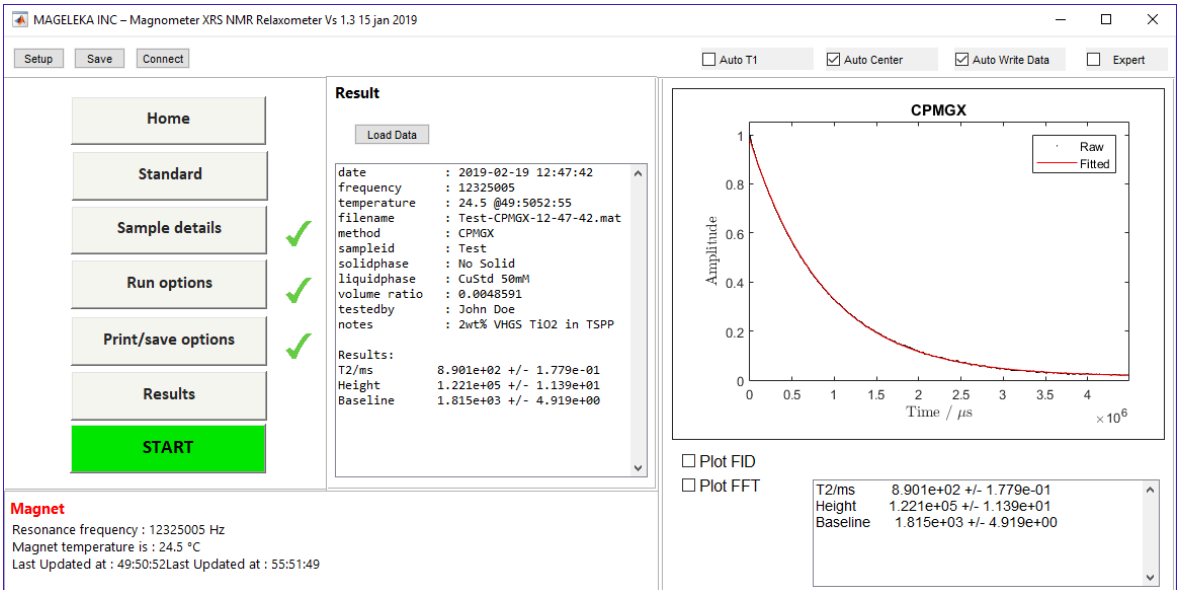

FIGURE 22.

NOTE: (i) The error on the graphic fit – in this last result (above) it was 0.18 ms – to the experimental data can be improved by increasing the number of scans; increasing the scans proportionally increases the time taken to make a measurement.

(ii) For maximum precision, the sample should be measured a minimum of 5 times (i.e., set the number of runs to 5 after the initial Relaxation Value has been determined) and the data averaged.

(iii) We recommend that 3 samplings be taken from a well-mixed bulk sample to reduce the potential for sampling errors.

Determination of the Specific Surface Area

In the vast majority of cases, the determination of specific surface area will use the T_2 methodology because it is the fastest measurement.

The first step is to determine the T_2 relaxation time for the suspension under investigation. This is the same procedure as described in Steps 1, 2 and 3 in the "**Determination of the Relaxation Value**" section, above. A single measurement will suffice for each.

NOTE: This still necessitates knowing a value for the T_1 relaxation time. The values for both should be noted.

In the following example (Figs. 23-29), the values were found to be approximately 1900 ms (T_1) and 890 ms (T_2), respectively.

Then click the Sample details command button. The screen should look like this (Fig. 23):

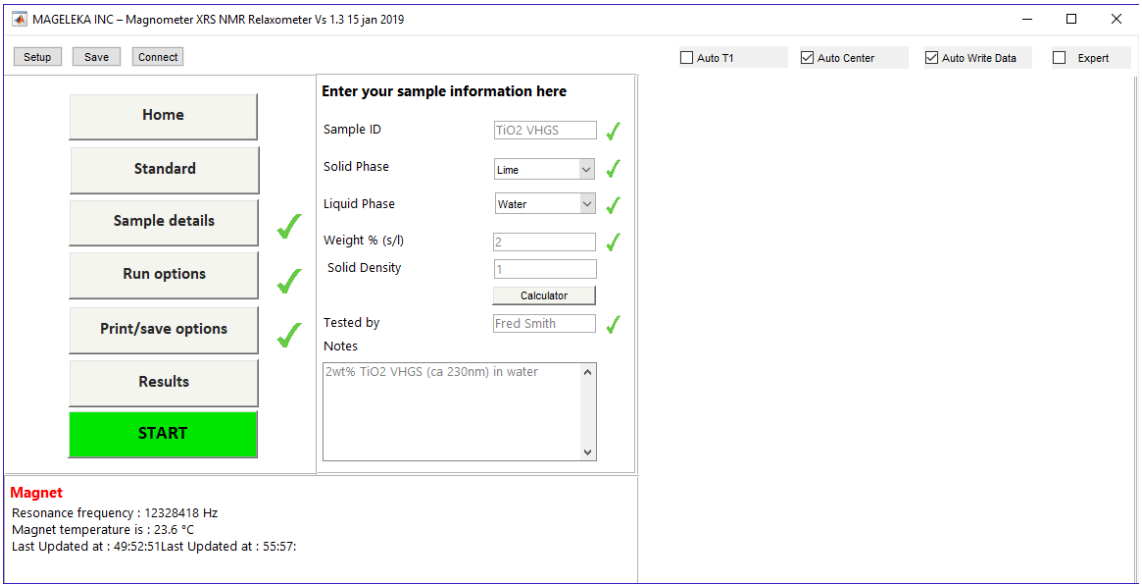

FIGURE 23.

NOTE: Enter any relevant information but, for the time being, ignore the entry for "Solid Phase".

In the example shown above (Fig. 23), the Sample ID is "TiO2 VHGS", the "Wt% (s/l)" is "2", the "Solid

density" (of this titania) is "1", it was tested by "Fred Smith" and in the "Notes" box it indicates that the material is ca. 230 nm in size.

Now, click on the Calculator command button. The following "calc" sub-screen should appear (Fig. 24):

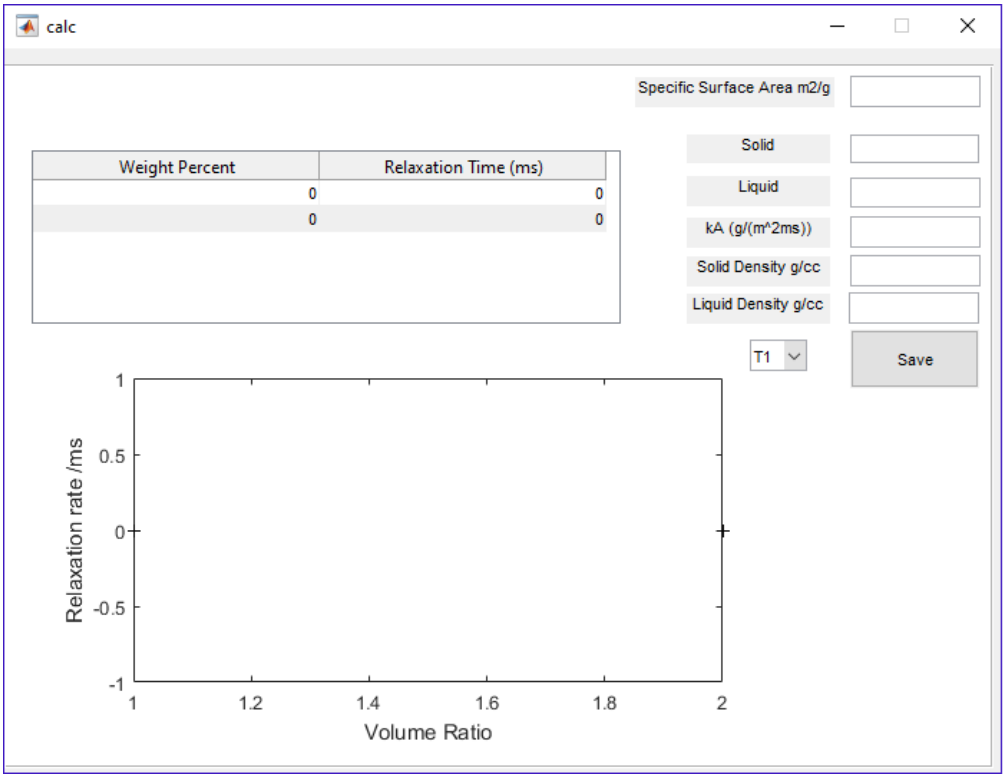

FIGURE 24.

Click on the "T1" pull-down menu (next to the Save button) and change it to "T2". Then, in the "Weight Percent I Relaxation Time (ms)" panel, enter the T_2 relaxation value for water and the concentration and T_2 relaxation time for the suspension.

NOTE: The T_2 default value for water is **2350 ms**. However, wherever possible, you should use a T_2 relaxation time for the actual water used in preparation of the suspension being measured. And, in this example, the T_2 time for the titania is **890 ms**.

Enter an estimated value for the surface area of the suspension. For this (rutile) titania material it was estimated to be ca. 6 m^2g^{-1}.

NOTE: A reasonable estimate can be calculated (using the macro supplied) from a particle size for the material – usually measured by a light scattering device. While the mean particle size can be used, the D_{10} size (from the particle size distribution) is a much better indicator of surface area.

In the "Solid" field, enter a name that will be stored in the "Solid Phase" pull-down menu found under Sample details.

NOTE: A value for the "kA" parameter will be automatically calculated from the data entered. More details about the kA parameter are given in the MAGELEKA White Paper 1 – a discourse on NMR relaxation and surface area.

The "calc" sub-screen should now look like this (Fig. 25):

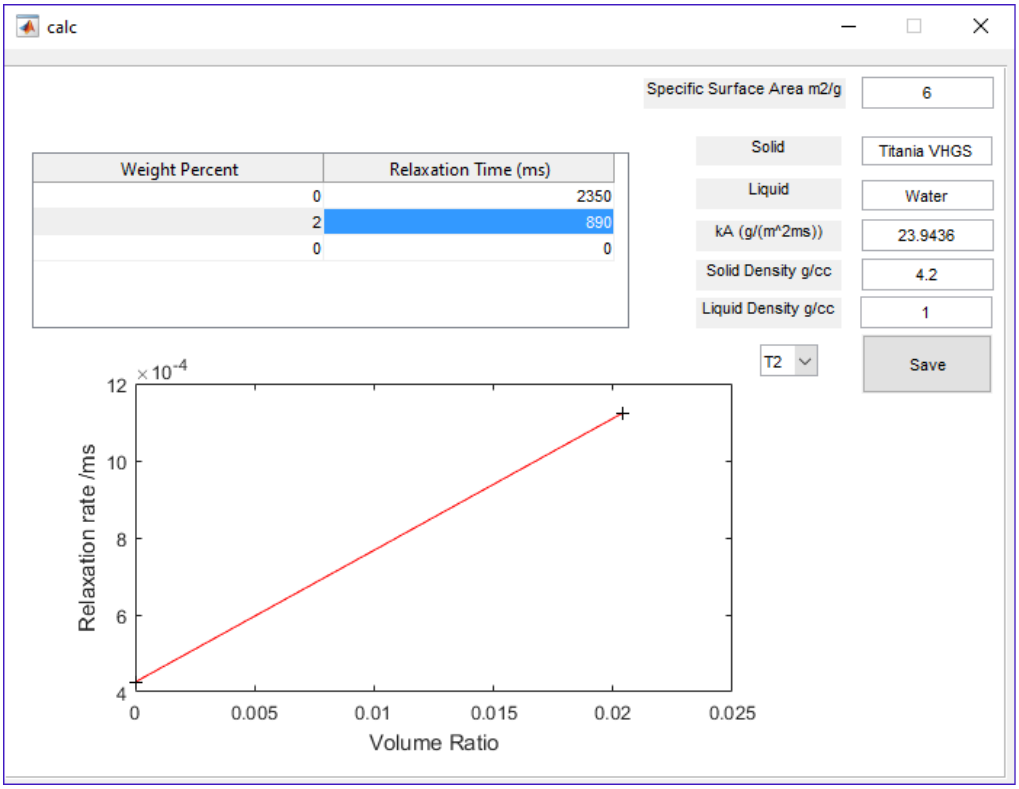

FIGURE 25.

NOTE: Make certain that the small pull-down menu to the left of the Save button is changed to "T2", and then click the Save button. Another dialogue box will open (Fig. 26):

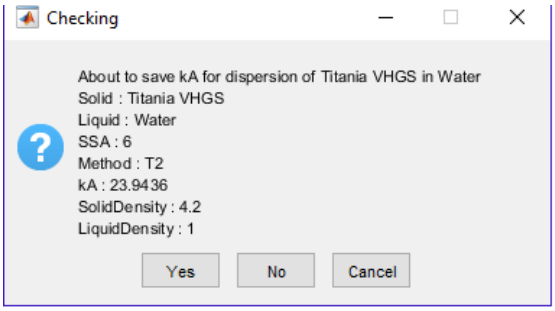

FIGURE 26.

Click the "Yes" button.

Now, return to the Sample details menu and check in the "Solid Phase" pull-down menu that the "Solid Phase" name, (in this example, "Titania VHGS") has been saved (Fig. 27):

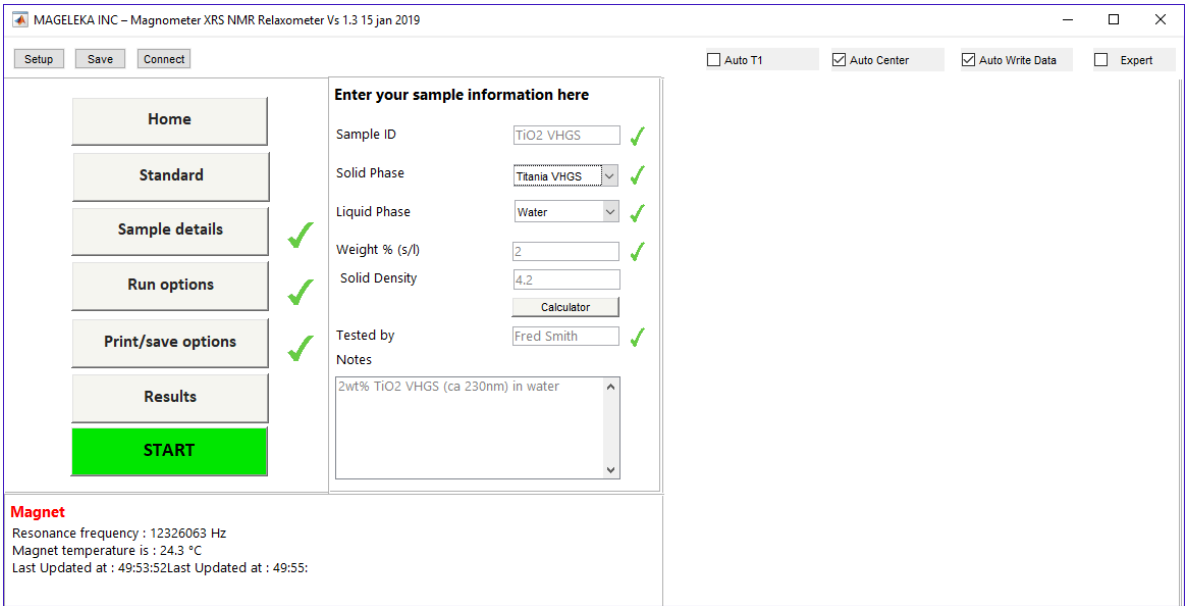

FIGURE 27.

NOTE: If it has not been saved, exit the program, reload the *M*agno*M*eter software and re-check.

Next, from the Run options menu select “Specific surface area”. The screen should now look like this (Fig. 28):

NOTE: The “T2/ms” option box should already be checked and all the “Anticipated” values for “T1/ms”, “T2/ms”, and “Specific surface area” should already be entered. In this current example, the values are 1938, 897, and 6, respectively (Fig. 28).

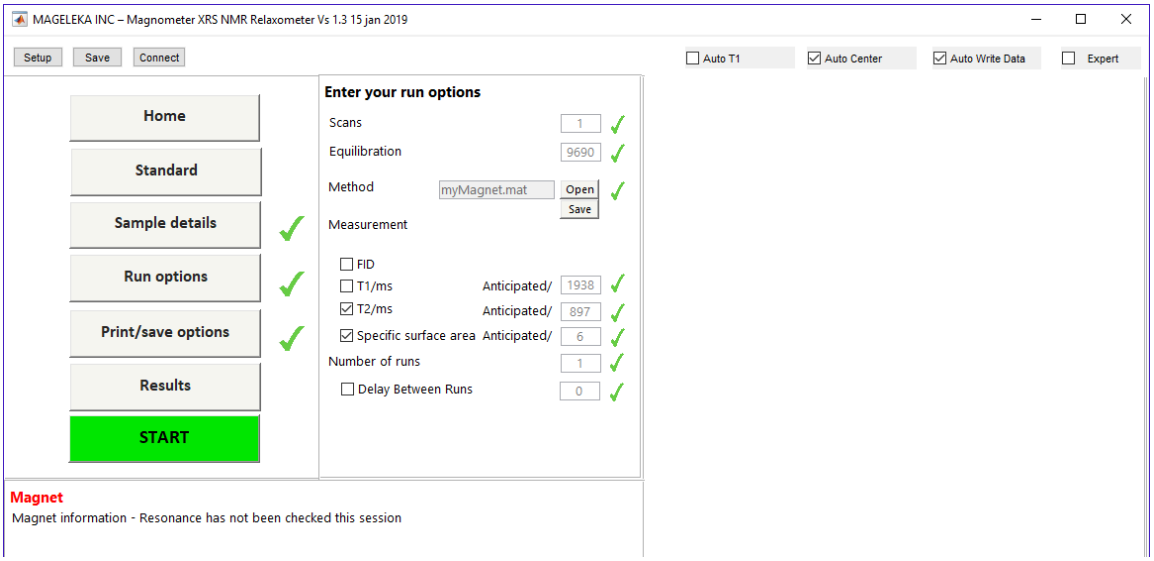

FIGURE 28.

Now, hit the Start command button to commence a measurement.

At the conclusion, the following screen will appear (Fig. 29):

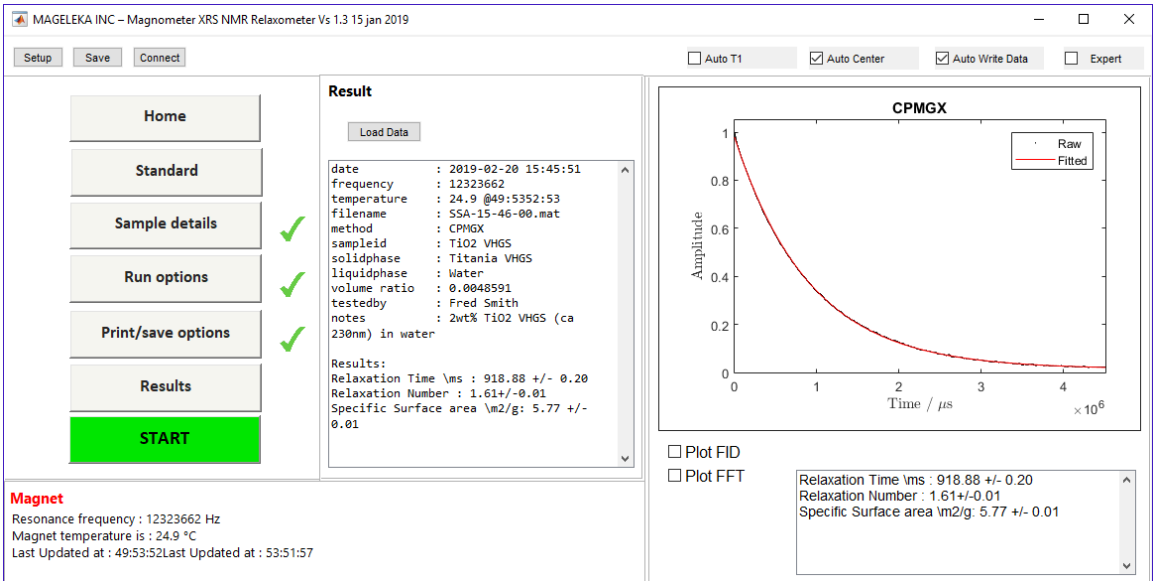

FIGURE 29.

For this measurement, we see that the T_2 relaxation time **918.9 ms** and the measured wetted surface area was **5.77 m^2g^{-1}** at 24.9°C.

If desired, the measurement can now be repeated by, again, simply hitting the Start command button.

NOTE: A value for the "Relaxation Number" is also calculated. A detailed explanation of this useful dimensionless parameter can be found in the full *M*agno*M*eter manual and the Mageleka Technical Note #4.