Mageleka

A Manual for Making Measurements with the MAGELEKA *M*agno*M*eter XRS™ *R*elaxo*M*eter

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Table of Contents

Name of Manufacturer: Mageleka Inc

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In accordance with the following directive(s):

2014/30/EU The Electromagnetic Compatibility Directive

2. Equipment identified within this Declaration of Conformity

Product Code	Product Name
MAG-001	Magnometer

3. Titles and Numbers of Relevant standards

Number	Title
BS EN 61326-1: 2013	Electrical equipment for measurement, control and laboratory use. EMC requirements. General requirements
Sub Sections	
BS EN55011:2009	Conducted Emissions .15-30MHz
BS EN55011:2009	Radiated Emissions 30-1000MHz
BS EN55011:2009	Magnetic Emissions .15-30MHz

Signed on behalf of Mageleka Inc:

Roger Pettman
Signature Date of Issue: 10th October 2018
President

Measurements using the *M*agno*M*eter XRS *R*elaxo*M*eter are straightforward and fast, and are based on a simple GUI concept described below. Operation requires minimal training and expertise. This manual is intended for simple training purposes and to allow a novice user to be able to quickly make measurements.

To begin, activate the software by double clicking on the “Mageleka” icon on your computer. The following box briefly appears, **then disappears** (Fig. 1).

Home Screen

Please Wait

FIGURE 1.

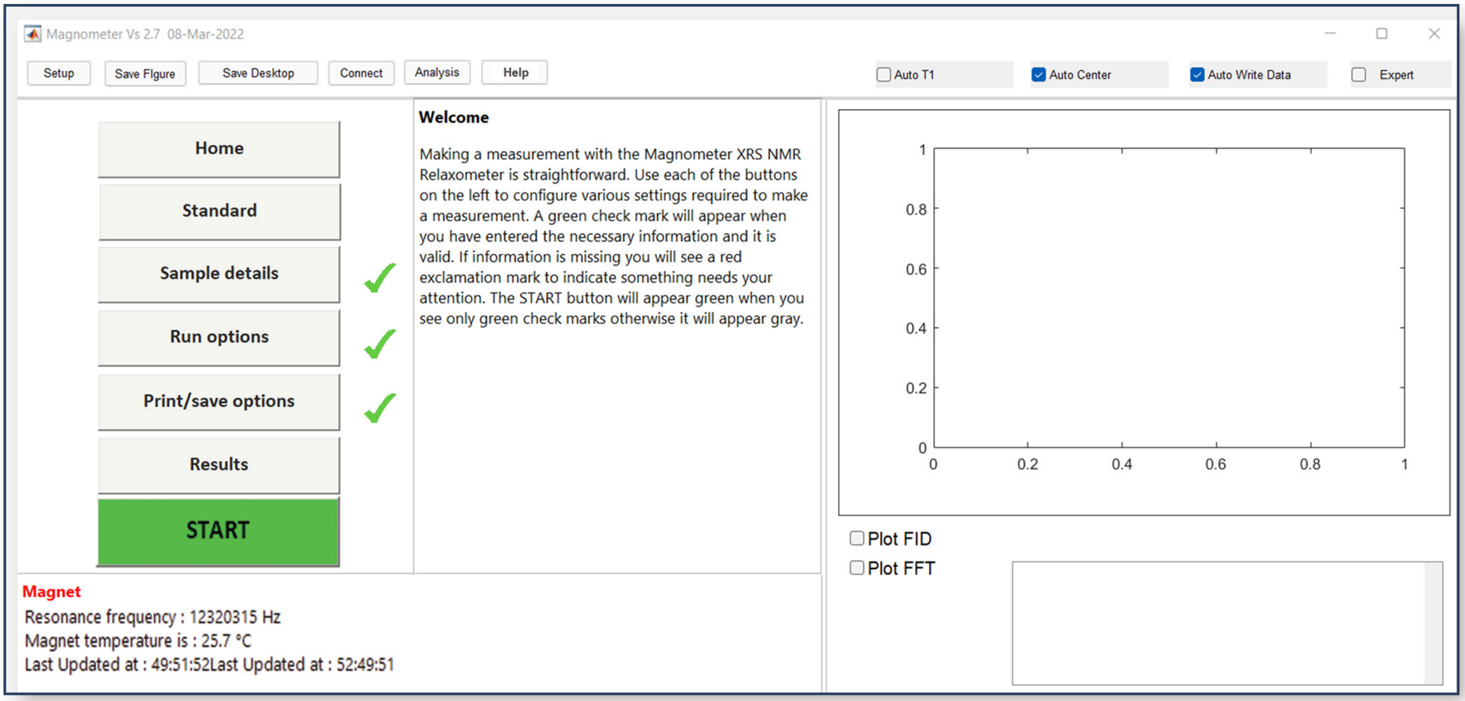
NOTE: It may take a few minutes for the program to load. PLEASE WAIT - Do not try to restart or run again!

The following “splash screen”, which shows the most current version, will appear (Fig. 2).

Version 2.6 31-Dec-2021

FIGURE 2.

The program starts in a default configuration - the “Home” page (Fig. 3).



Welcome

Making a measurement with the Magnometer XRS NMR Relaxometer is straightforward. Use each of the buttons on the left to configure various settings required to make a measurement. A green check mark will appear when you have entered the necessary information and it is valid. If information is missing you will see a red exclamation mark to indicate something needs your attention. The START button will appear green when you see only green check marks otherwise it will appear gray.

Magnet
Resonance frequency : 12320315 Hz
Magnet temperature is : 25.7 °C
Last Updated at : 49:51:52Last Updated at : 52:49:51

FIGURE 3.

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 2.7.

There are five grey buttons at the top left-hand corner.

The functions of the two Save Figure and Save Desktop buttons are self-explanatory. The functions for the others are described later in this guide.

In addition, 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_1 or T_2 relaxation time measurement.

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.

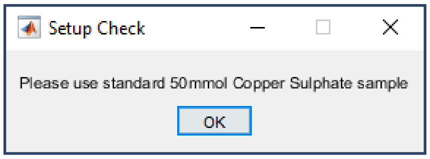
To connect to the spectrometer, press the small Connect button (upper left-hand side).

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. 3).

Basic Setup

Before any measurements are made on “unknown” samples, a setup routine should be conducted to ensure that the *R*elaxo*M*eter is configured and operating correctly. The complete basic set-up operation takes less than five 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. 4).



Please use standard 50mmol Copper Sulphate sample

FIGURE 4.

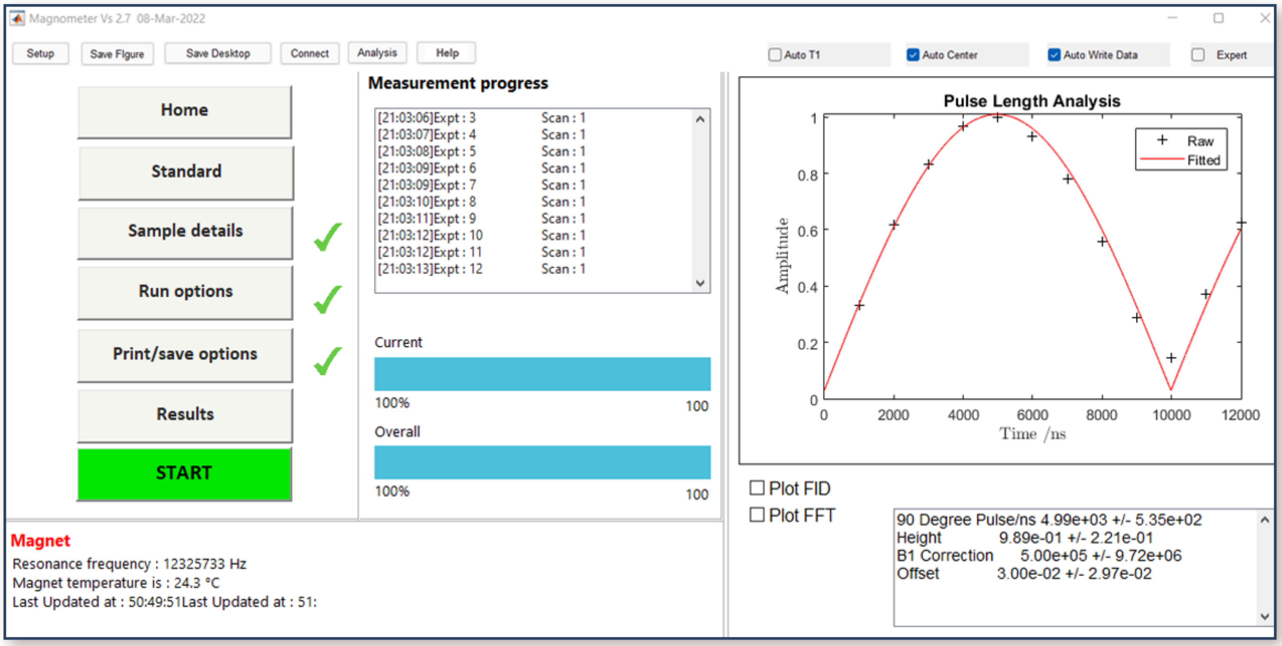
NOTE: Another useful test material is Glycerol which has a T_1 and T_2 relaxation time of *ca* 30 ms.

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 any test 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 – approximately 0.5 mL (see Fig. 5 for an example). An optimum height is 0.5 cm (0.25mL). Use the 1.0 mL syringe supplied.

FIGURE 5.

Then click OK. The following screen should appear (Fig. 6):



```
[21:03:06]Expt : 3        Scan : 1
[21:03:07]Expt : 4        Scan : 1
[21:03:08]Expt : 5        Scan : 1
[21:03:09]Expt : 6        Scan : 1
[21:03:09]Expt : 7        Scan : 1
[21:03:10]Expt : 8        Scan : 1
[21:03:11]Expt : 9        Scan : 1
[21:03:12]Expt : 10       Scan : 1
[21:03:12]Expt : 11       Scan : 1
[21:03:13]Expt : 12       Scan : 1
```

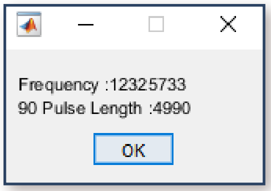
Magnet

Resonance frequency : 12325733 Hz
Magnet temperature is : 24.3 °C
Last Updated at : 50:49:51Last Updated at : 51:

```
90 Degree Pulse/ns 4.99e+03 +/- 5.35e+02
Height            9.89e-01 +/- 2.21e-01
B1 Correction       5.00e+05 +/- 9.72e+06
Offset            3.00e-02 +/- 2.97e-02
```

FIGURE 6.

The device should now track through the frequency in 1 KHz increments and set the frequency (shown in the box at bottom left of screen). 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. 7) indicating the frequency and pulse length. In this example, the frequency was 12.32 MHz and the 90° pulse length was 4.99 µs.



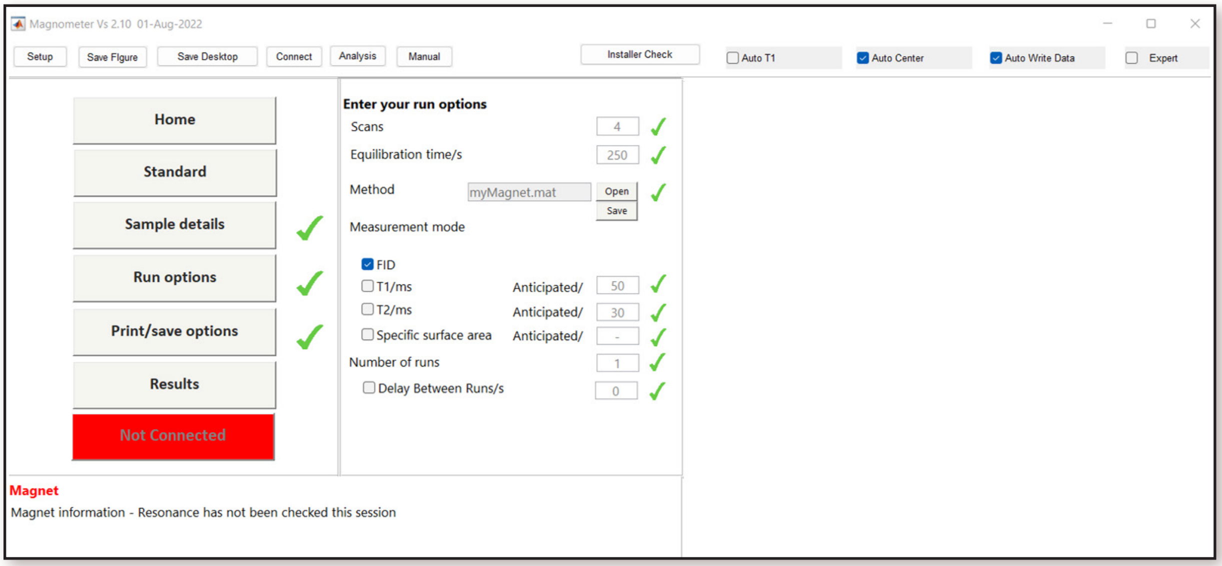
Frequency :12325733
90 Pulse Length :4990

FIGURE 7.

> **NOTE**: Typically, the resonance frequency should be ~12.4 MHz and the pulse length 4-6 µs. The frequency value depends on the exact specification of the magnet.

Click OK to close the dialog box.

Next, click on the Run options command button. The following page will be displayed (Fig. 8):



☐ Auto T1
☑ Auto Center
☑ Auto Write Data
☐ Expert

Enter your run options

Scans 4 ✓
Equilibration time/s 250 ✓
Method myMagnet.mat ✓
Measurement mode
☑ FID
☐ T1/ms Anticipated/ 50 ✓
☐ T2/ms Anticipated/ 30 ✓
☐ Specific surface area Anticipated/ - ✓
Number of runs 1 ✓
☐ Delay Between Runs/s 0 ✓

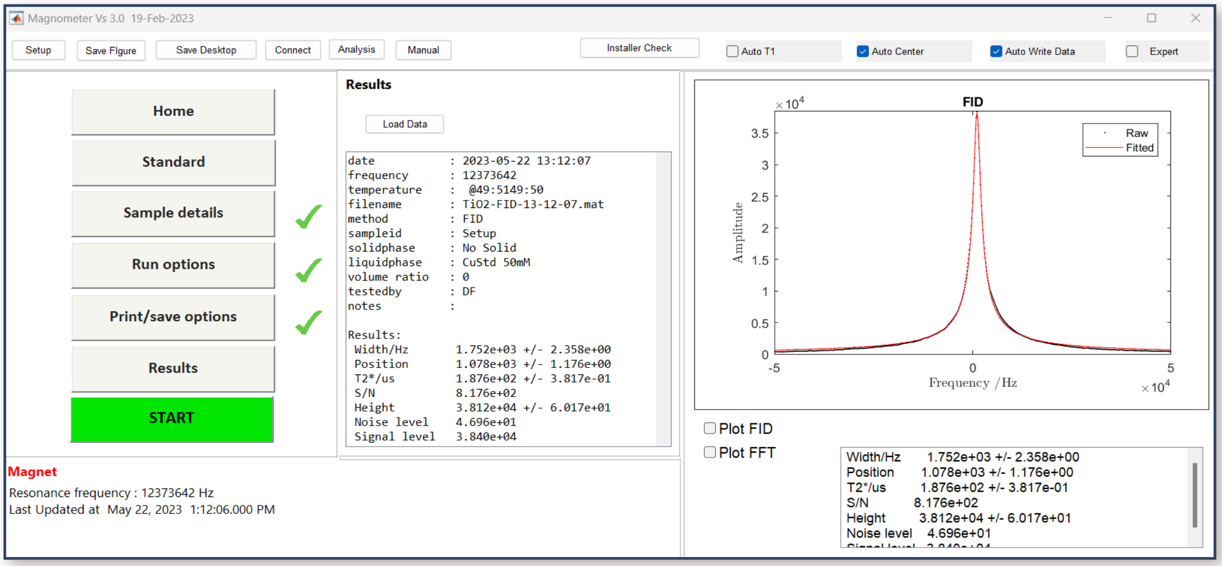
Magnet
Magnet information - Resonance has not been checked this session

FIGURE 8.

Check the FID button.

NOTE: Make sure that the “T1/ms”, “T2/ms”, and “Specific surface area” options are *unchecked*.

Now click the START button to initiate FID measurement. The following Figure 9 should result:



```
date           : 2023-05-22 13:12:07
frequency      : 12373642
temperature    :  @49:5149:50
filename       : TiO2-FID-13-12-07.mat
method         : FID
sampleid       : Setup
solidphase     : No Solid
liquidphase    : CuStd 50mM
volume ratio   : 0
testedby       : DF
notes          :

Results:
 Width/Hz       1.752e+03 +/- 2.358e+00
 Position       1.078e+03 +/- 1.176e+00
 T2*/us         1.876e+02 +/- 3.817e-01
 S/N            8.176e+02
 Height         3.812e+04 +/- 6.017e+01
 Noise level    4.696e+01
 Signal level   3.840e+04
```

```
Width/Hz      1.752e+03 +/- 2.358e+00
Position     1.078e+03 +/- 1.176e+00
T2*/us       1.876e+02 +/- 3.817e-01
S/N         8.176e+02
Height       3.812e+04 +/- 6.017e+01
Noise level   4.696e+01
```

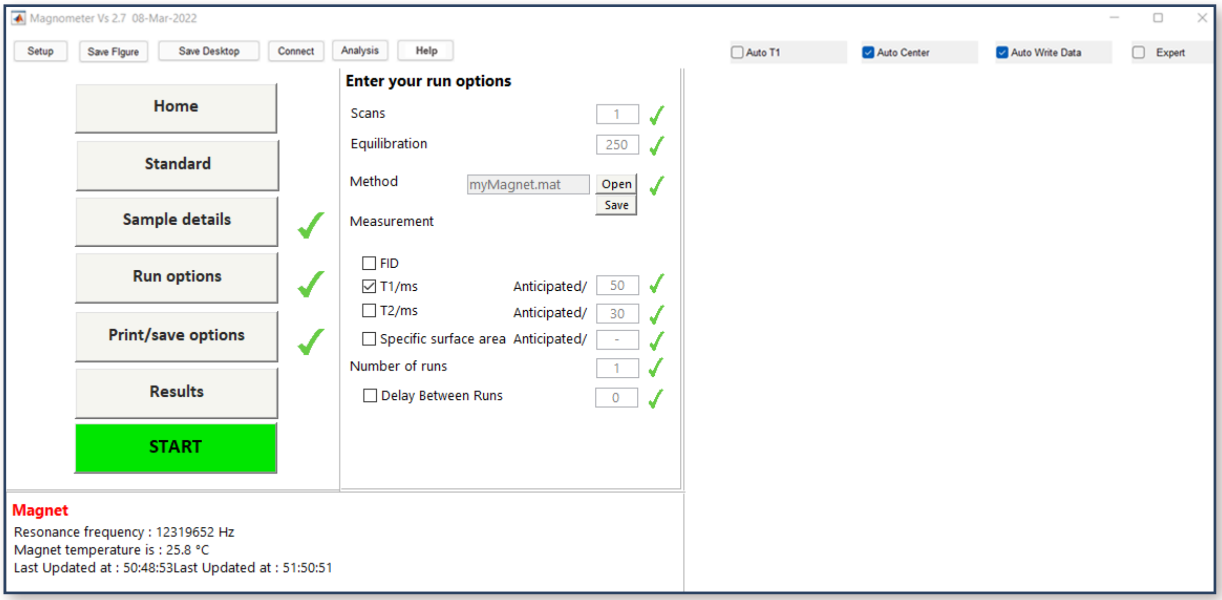
Magnet

Resonance frequency : 12373642 Hz
Last Updated at May 22, 2023 1:12:06.000 PM

FIGURE 9

NOTE: The value for S/N should be within the range of 500-1000, and T2* should be *ca* 200 us.

Next, click on the Run options command button. Then check the T1 button as shown in Fig. 10.



Enter your run options

Scans 1 ✓
Equilibration 250 ✓
Method myMagnet.mat ✓
Measurement
☐ FID
☑ T1/ms Anticipated/ 50 ✓
☐ T2/ms Anticipated/ 30 ✓
☐ Specific surface area Anticipated/ - ✓
Number of runs 1 ✓
☐ Delay Between Runs 0 ✓

Magnet

Resonance frequency : 12319652 Hz
Magnet temperature is : 25.8 °C
Last Updated at : 50:48:53Last Updated at : 51:50:51

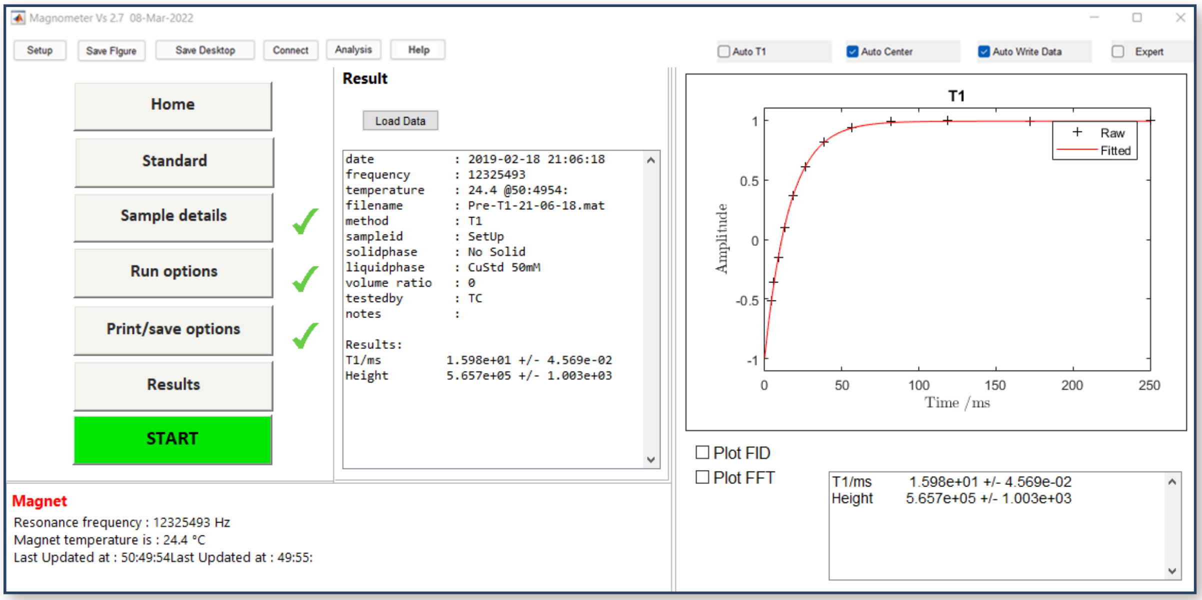
FIGURE 10.

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

NOTE: Make sure that the “FID”, “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. 11):



```
date          : 2019-02-18 21:06:18
frequency     : 12325493
temperature   : 24.4 @50:4954:
filename      : Pre-T1-21-06-18.mat
method        : T1
sampleid      : SetUp
solidphase    : No Solid
liquidphase   : CuStd 50mM
volume ratio  : 0
testedby      : TC
notes         :

Results:
T1/ms         1.598e+01 +/- 4.569e-02
Height        5.657e+05 +/- 1.003e+03
```

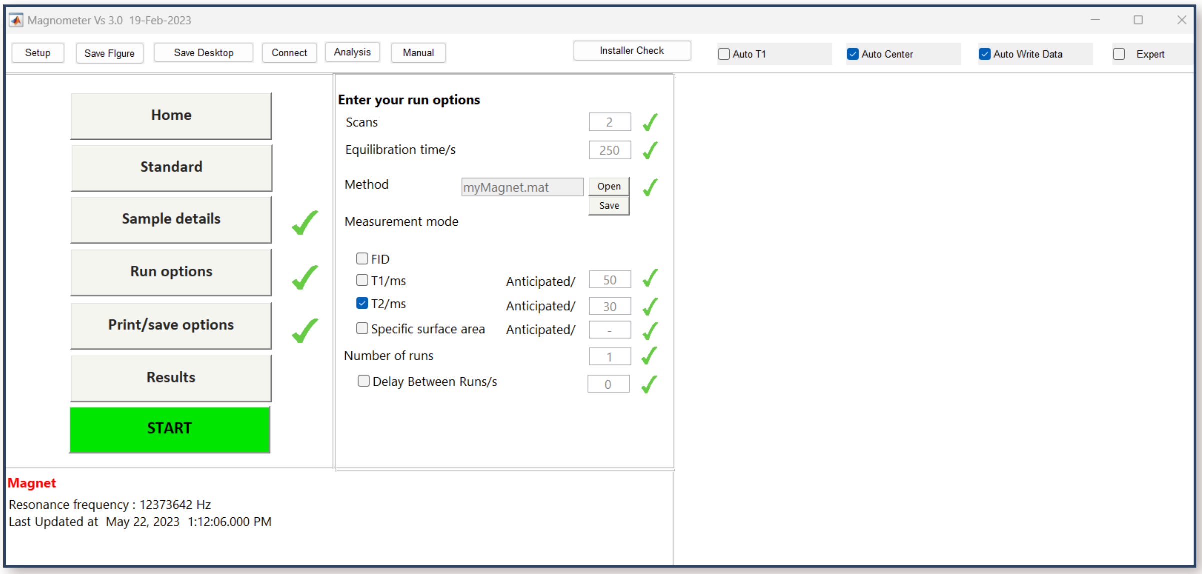
```
T1/ms      1.598e+01 +/- 4.569e-02
Height     5.657e+05 +/- 1.003e+03
```

Magnet
Resonance frequency : 12325493 Hz
Magnet temperature is : 24.4 °C
Last Updated at : 50:49:54Last Updated at : 49:55:

FIGURE 11.

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. 11 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 12:



☐ Auto T1
☑ Auto Center
☑ Auto Write Data
☐ Expert

Enter your run options

Scans	2	✓
Equilibration time/s	250	✓
Method	myMagnet.mat	✓

Measurement mode

☐ FID			
☐ T1/ms	Anticipated/	50	✓
☑ T2/ms	Anticipated/	30	✓
☐ Specific surface area	Anticipated/	-	✓
Number of runs		1	✓
☐ Delay Between Runs/s		0	✓

Magnet
Resonance frequency : 12373642 Hz
Last Updated at May 22, 2023 1:12:06.000 PM

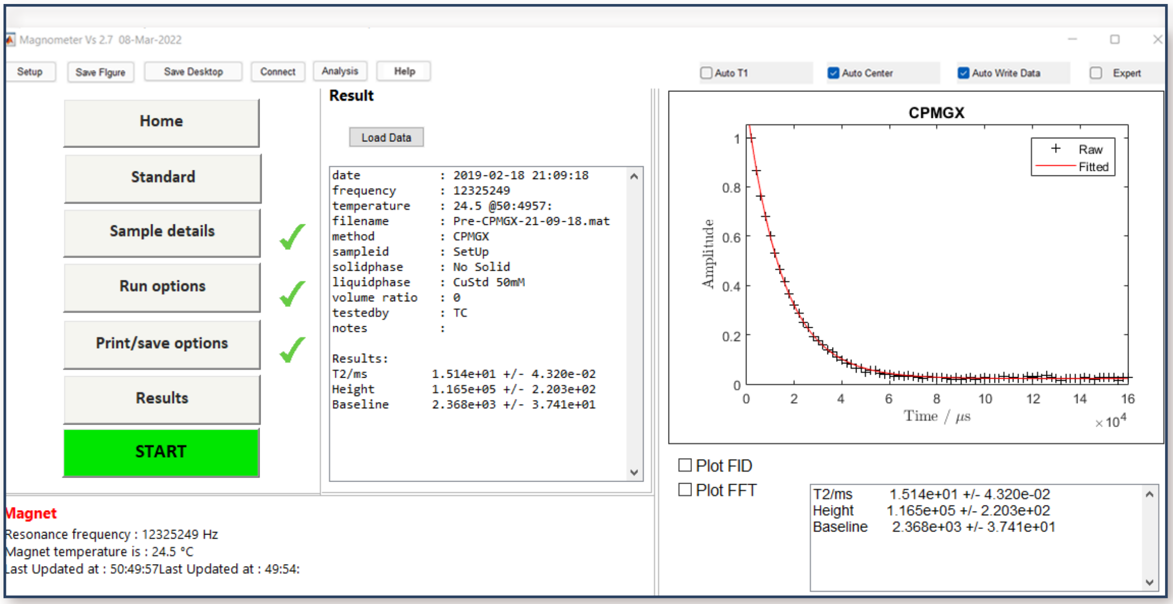
FIGURE 12

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

NOTE: Make sure that the “FID”, “T1/ms”, and “Specific surface area options” are *unchecked.*

Press the START button to initiate the T_2 measurement.

On completion, the following screen should be displayed (Fig. 13):



☐ Auto T1 ☑ Auto Center ☑ Auto Write Data ☐ Expert

```
date          : 2019-02-18 21:09:18
frequency     : 12325249
temperature   : 24.5 @50:4957:
filename      : Pre-CPMGX-21-09-18.mat
method        : CPMGX
sampleid      : SetUp
solidphase    : No Solid
liquidphase   : CuStd 50mM
volume ratio  : 0
testedby      : TC
notes         :

Results:
T2/ms         1.514e+01 +/- 4.320e-02
Height        1.165e+05 +/- 2.203e+02
Baseline      2.368e+03 +/- 3.741e+01
```

☐ Plot FID
☐ Plot FFT

```
T2/ms       1.514e+01 +/- 4.320e-02
Height      1.165e+05 +/- 2.203e+02
Baseline     2.368e+03 +/- 3.741e+01
```

Magnet
Resonance frequency : 12325249 Hz
Magnet temperature is : 24.5 °C
Last Updated at : 50:49:57Last Updated at : 49:54:

FIGURE 13.

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. 13 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.

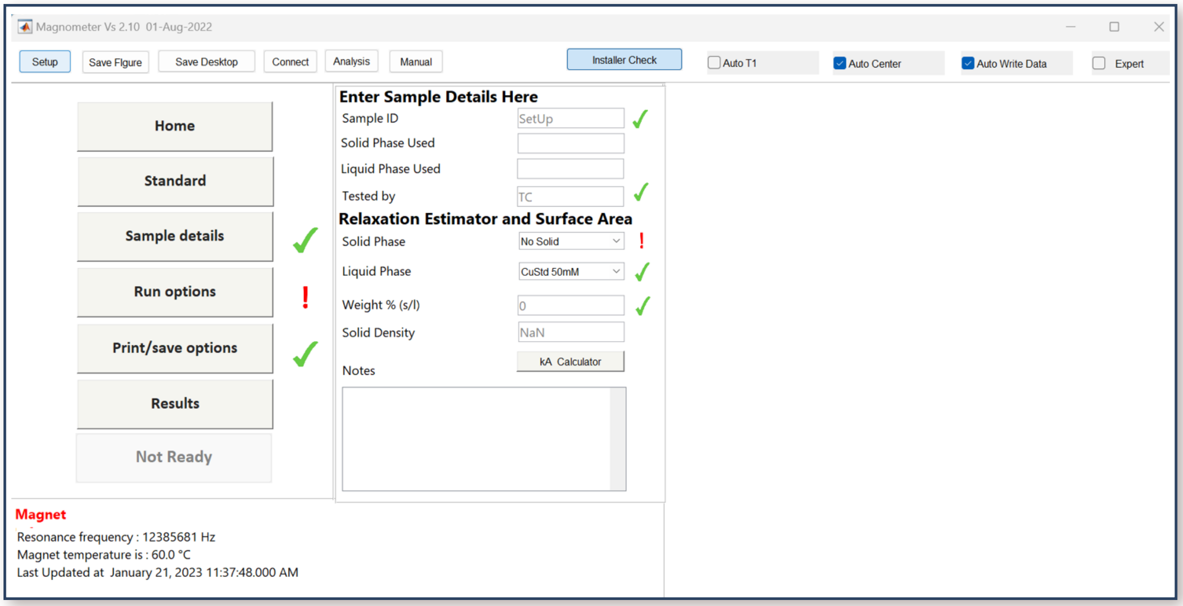
The instrument is ready and functioning correctly.

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. 14):



Enter Sample Details Here

Sample ID: SetUp ✓

Solid Phase Used:

Liquid Phase Used:

Tested by: TC ✓

Relaxation Estimator and Surface Area

Solid Phase: No Solid !

Liquid Phase: CuStd 50mM ✓

Weight % (s/l): 0 ✓

Solid Density: NaN

Notes:

Magnet

Resonance frequency : 12385681 Hz

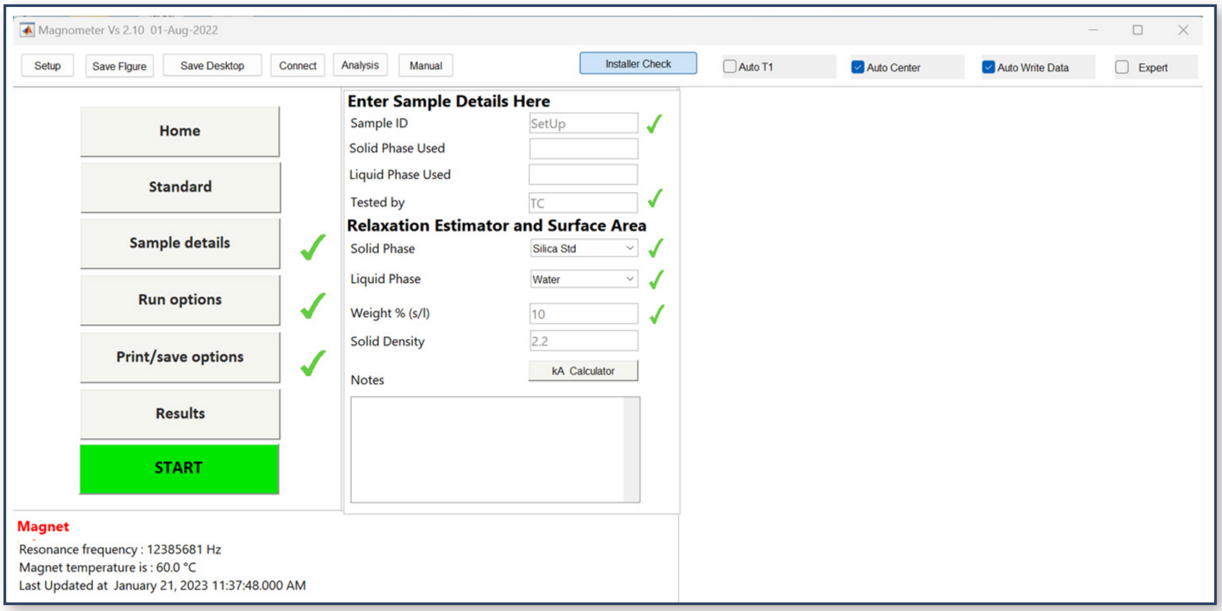
Magnet temperature is : 60.0 °C

Last Updated at January 21, 2023 11:37:48.000 AM

FIGURE 14.

First choose “**Water**” using the pull-down menu chevron found to the right of “Liquid Phase”, and then choose “**Silica Std**” using the pull-down menu chevron found to the right of “Solid Phase”. The “Wt% (s/l)” should be entered as “10”. The “Solid Density” is automatically set as “2.2”.

The screen should now display as (Fig. 15):



☐ Auto T1 ☑ Auto Center ☑ Auto Write Data ☐ Expert

Enter Sample Details Here

Sample ID: SetUp ✓
Solid Phase Used:
Liquid Phase Used:
Tested by: TC ✓

Relaxation Estimator and Surface Area

Solid Phase: Silica Std ✓
Liquid Phase: Water ✓
Weight % (s/l): 10 ✓
Solid Density: 2.2
Notes:

Magnet
Resonance frequency : 12385681 Hz
Magnet temperature is : 60.0 °C
Last Updated at January 21, 2023 11:37:48.000 AM

FIGURE 15.

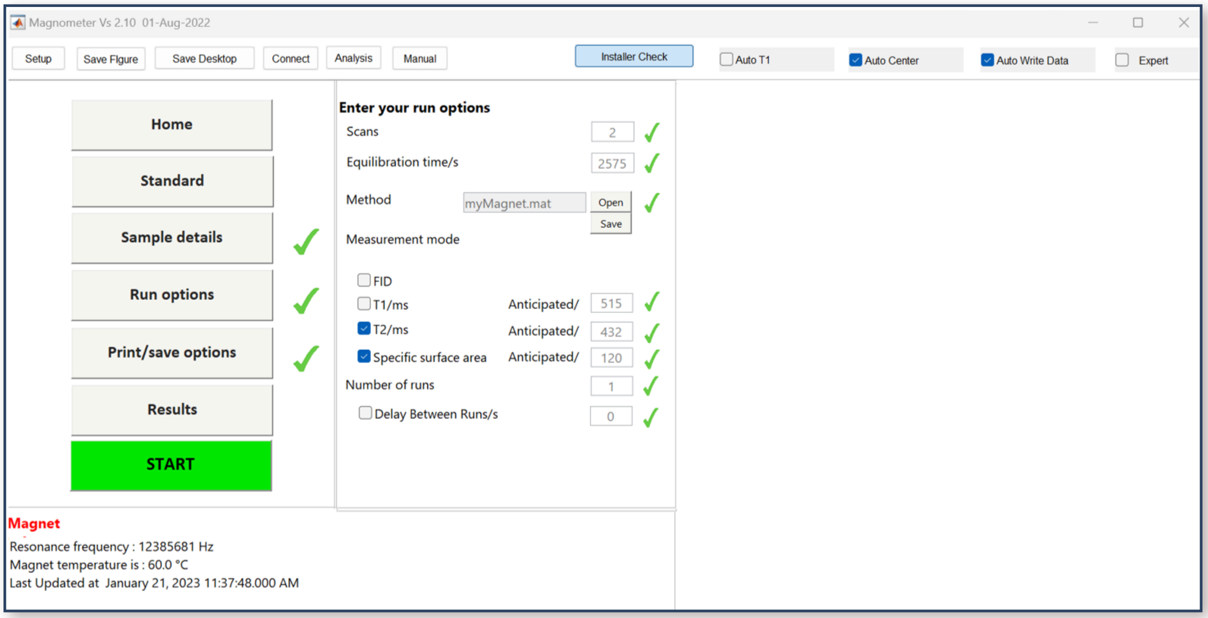
Insert a standard NMR tube containing an aliquot of the silica reference suspension solution into the *M*agno*P*od assembly.

> **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. 5 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 Figure 16):



☐ Auto T1
☑ Auto Center
☑ Auto Write Data
☐ Expert

Enter your run options

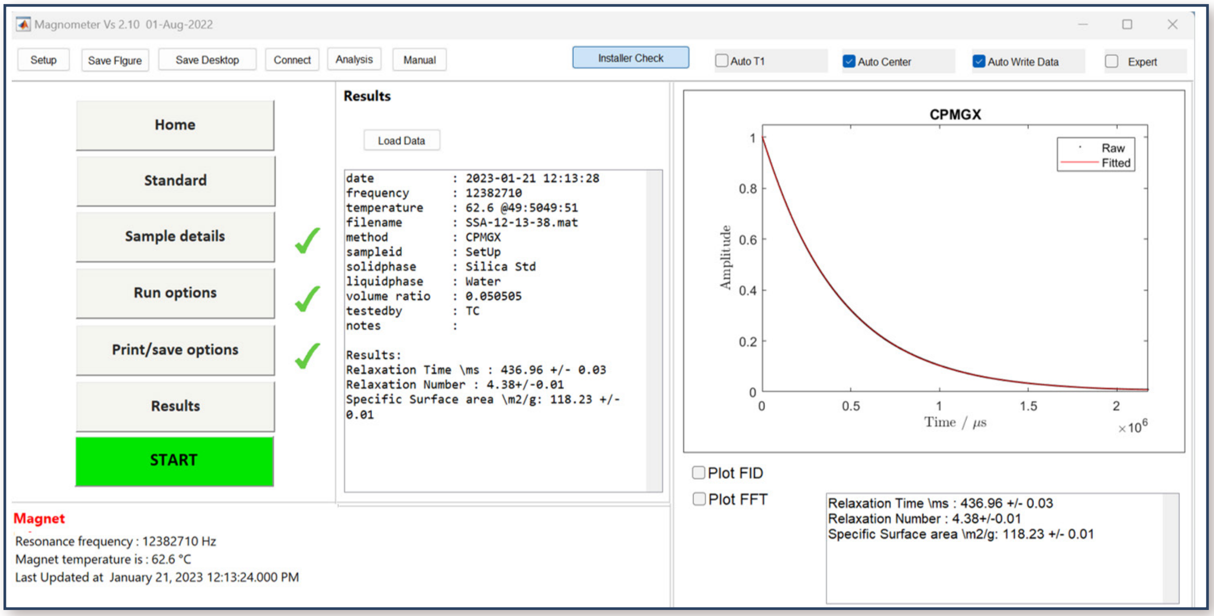
Scans: 2
Equilibration time/s: 2575
Method: myMagnet.mat
Measurement mode
☐ FID
☐ T1/ms Anticipated/ 515
☑ T2/ms Anticipated/ 432
☑ Specific surface area Anticipated/ 120
Number of runs: 1
☐ Delay Between Runs/s: 0

Magnet
Resonance frequency : 12385681 Hz
Magnet temperature is : 60.0 °C
Last Updated at January 21, 2023 11:37:48.000 AM

FIGURE 16.

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. 17):



☐ Auto T1
☑ Auto Center
☑ Auto Write Data
☐ Expert

```
date          : 2023-01-21 12:13:28
frequency     : 12382710
temperature   : 62.6 @49:5049:51
filename      : SSA-12-13-38.mat
method        : CPMGX
sampleid      : SetUp
solidphase    : Silica Std
liquidphase   : Water
volume ratio  : 0.050505
testedby      : TC
notes         :

Results:
Relaxation Time \ms : 436.96 +/- 0.03
Relaxation Number : 4.38+/-0.01
Specific Surface area \m2/g: 118.23 +/-
0.01
```

☐ Plot FID
☐ Plot FFT

```
Relaxation Time \ms : 436.96 +/- 0.03
Relaxation Number : 4.38+/-0.01
Specific Surface area \m2/g: 118.23 +/- 0.01
```

Magnet
Resonance frequency : 12382710 Hz
Magnet temperature is : 62.6 °C
Last Updated at January 21, 2023 12:13:24.000 PM

FIGURE 17.

In the example shown in Figure 17, the T_2 relaxation time was determined to be **437 ms** at a temperature of 23.5 °C. The measured surface area was **118 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.

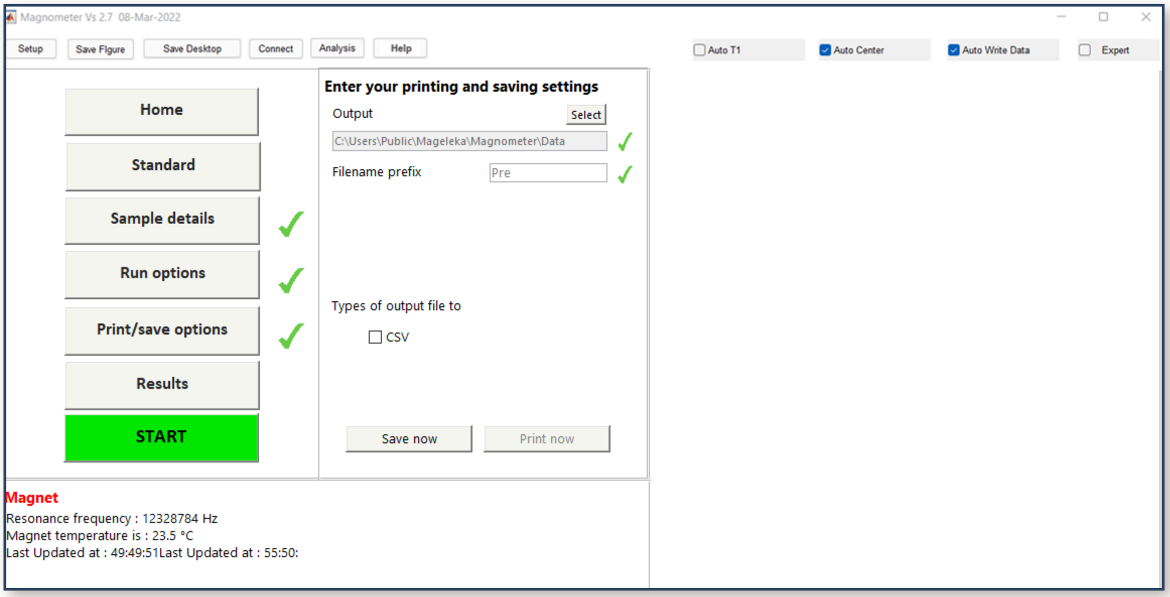
Making Measurements on an Unknown Sample

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. 5 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. 18):



☐ Auto T1 ☑ Auto Center ☑ Auto Write Data ☐ Expert

Enter your printing and saving settings

Output: C:\Users\Public\Mageleka\Magnometer\Data ✓

Filename prefix: Pre ✓

Types of output file to

☐ CSV

Magnet

Resonance frequency : 12328784 Hz

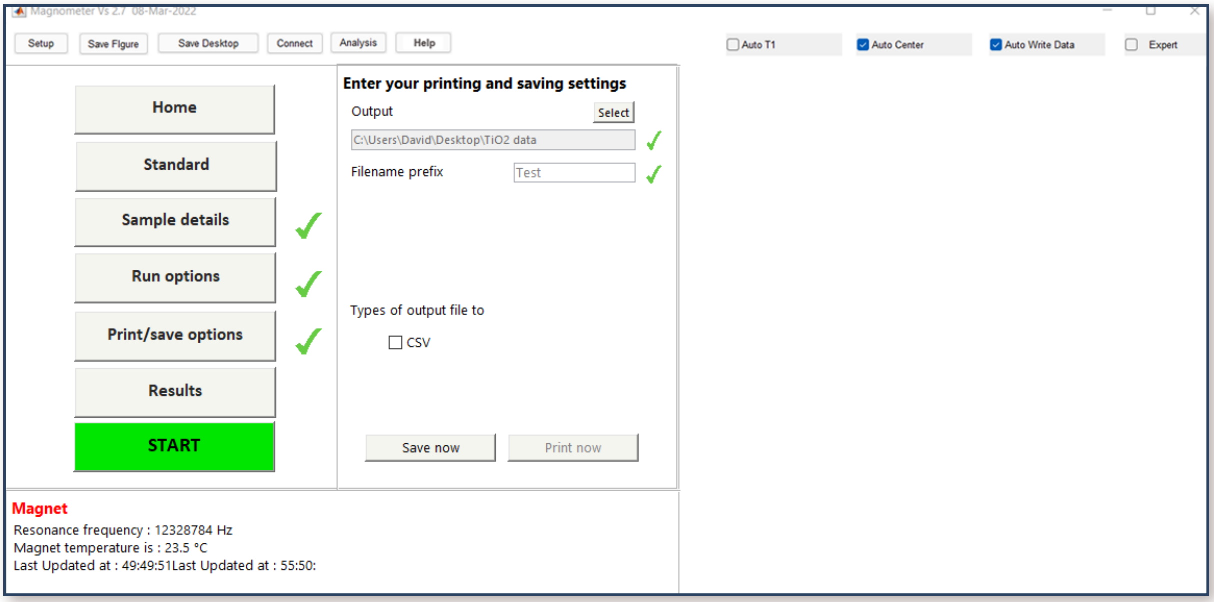
Magnet temperature is : 23.5 °C

Last Updated at : 49:49:51Last Updated at : 55:50:

FIGURE 18.

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 center pane and select the folder from the desktop. The filename prefix is an option. The screen should now look like this (Fig. 19):



Enter your printing and saving settings

Output: C:\Users\David\Desktop\TiO2 data ✓

Filename prefix: Test ✓

Types of output file to

☐ CSV

Magnet

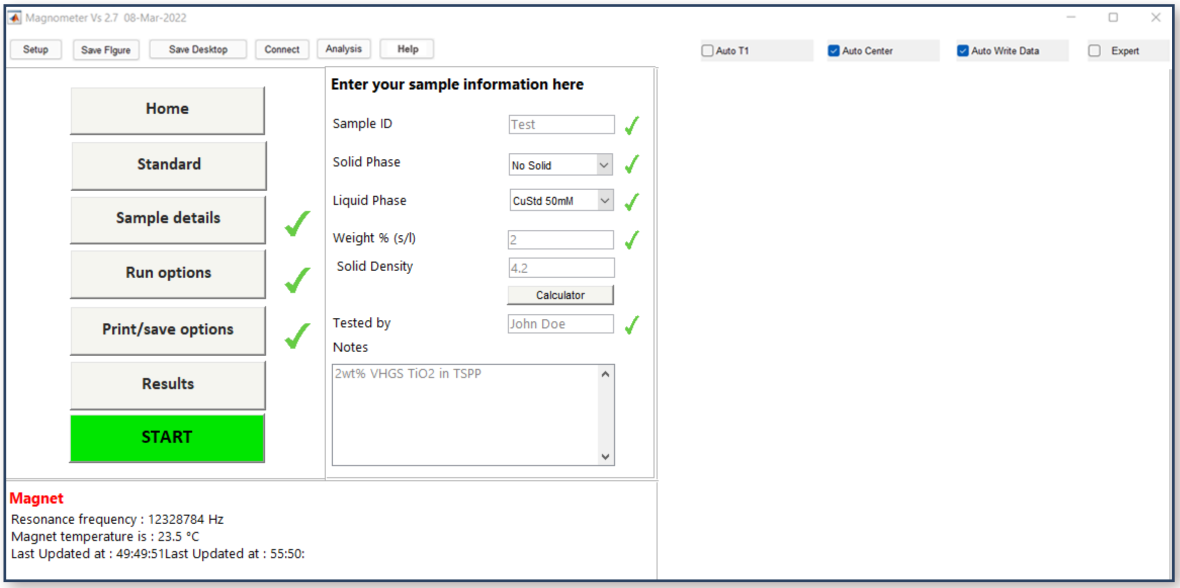
Resonance frequency : 12328784 Hz
Magnet temperature is : 23.5 °C
Last Updated at : 49:49:51Last Updated at : 55:50:

FIGURE 19.

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 the basic 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. 20):



☐ Auto T1 ☑ Auto Center ☑ Auto Write Data ☐ Expert

Enter your sample information here

Sample ID: Test ✓
Solid Phase: No Solid ✓
Liquid Phase: CuStd 50mM ✓
Weight % (s/l): 2 ✓
Solid Density: 4.2
Tested by: John Doe ✓
Notes: 2wt% VHGS TiO2 in TSPP

Magnet
Resonance frequency : 12328784 Hz
Magnet temperature is : 23.5 °C
Last Updated at : 49:49:51Last Updated at : 55:50:

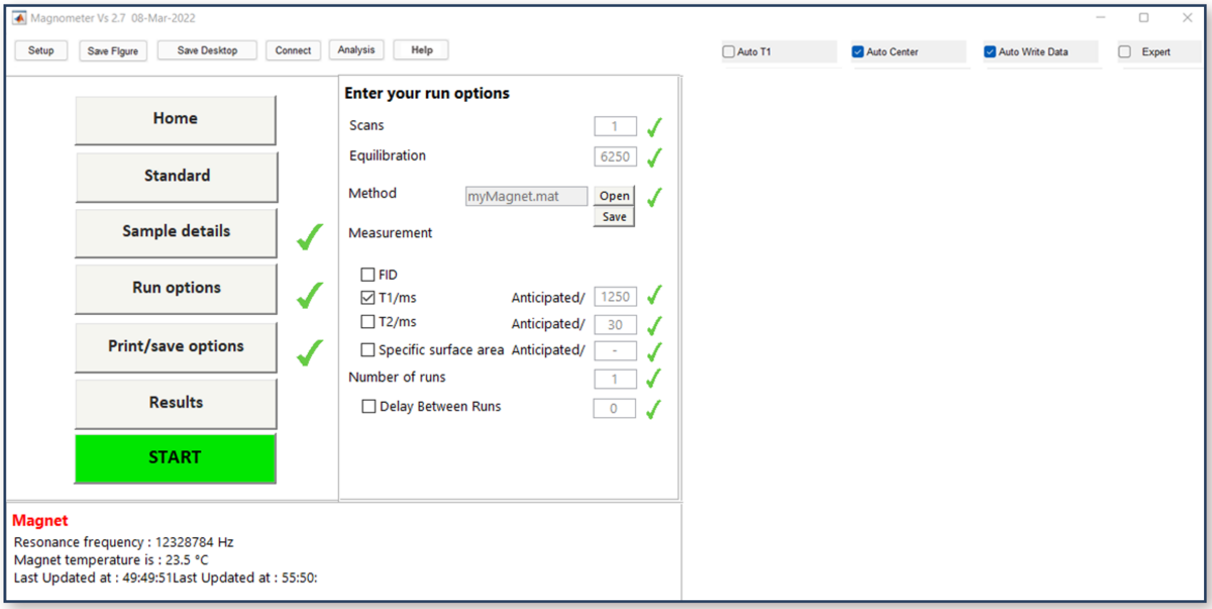
FIGURE 20.

> **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. 21):



Enter your run options

Scans 1 ✓
Equilibration 6250 ✓
Method myMagnet.mat ✓
Measurement
☐ FID
☑ T1/ms Anticipated/ 1250 ✓
☐ T2/ms Anticipated/ 30 ✓
☐ Specific surface area Anticipated/ - ✓
Number of runs 1 ✓
☐ Delay Between Runs 0 ✓

Magnet
Resonance frequency : 12328784 Hz
Magnet temperature is : 23.5 °C
Last Updated at : 49:49:51Last Updated at : 55:50:

FIGURE 21.

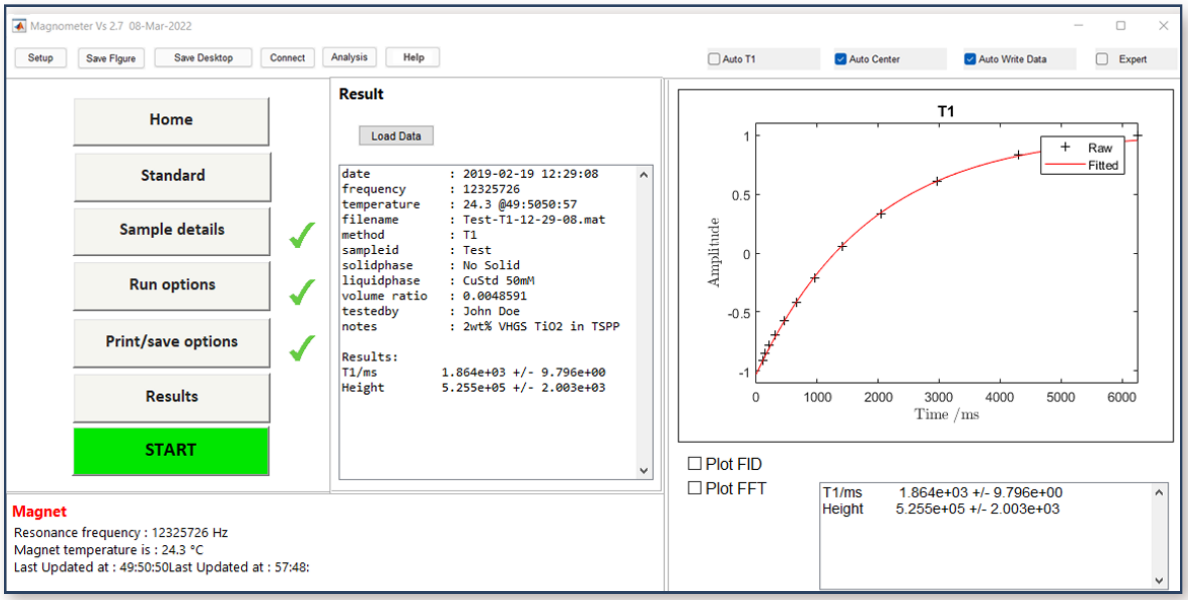
NOTE: The “1250” entered as the “Anticipated” value for “T1/ms” is arbitrary and can be any number. However, for all practical purposes in virtually all fields and applications, the measured relaxation time will generally be between 1 ms and 2500 ms. “1250” was selected because it is the midpoint of 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 *Magno**P**od* LED read-out. If so, insert the NMR tube containing the sample into the *Magno**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 button. At the conclusion of the measurement record the T_1 value obtained. The screen will look like this (Fig. 22):



```
date           : 2019-02-19 12:29:08
frequency      : 12325726
temperature    : 24.3 @49:5050:57
filename       : Test-T1-12-29-08.mat
method         : T1
sampleid       : Test
solidphase     : No Solid
liquidphase    : CuStd 50mM
volume ratio   : 0.0048591
testedby       : John Doe
notes          : 2wt% VHGS TiO2 in TSPP

Results:
T1/ms         1.864e+03 +/- 9.796e+00
Height        5.255e+05 +/- 2.003e+03
```

Magnet
Resonance frequency : 12325726 Hz
Magnet temperature is : 24.3 °C
Last Updated at : 49:50:50Last Updated at : 57:48:

```
T1/ms      1.864e+03 +/- 9.796e+00
Height     5.255e+05 +/- 2.003e+03
```

FIGURE 22.

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

NOTE: For an unknown sample, if this is the first time that a relaxation time has been measured, it needs to be repeated since the first value obtained is approximate. If the "Anticipated" value (of 1250 ms) is too short a guess, then the relaxation plot will look like that shown in Fig 20(a); if it is too long then the relaxation plot will be like Fig 20(b).

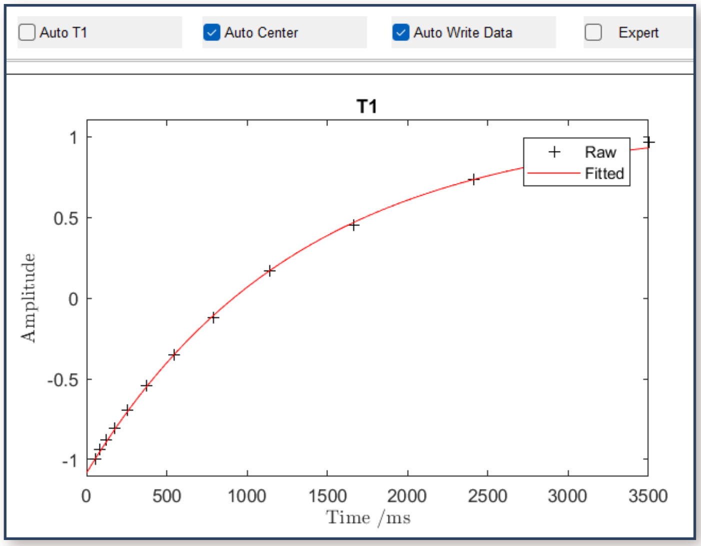

☐ Auto T1 ☑ Auto Center ☑ Auto Write Data ☐ Expert

FIGURE 20(a).

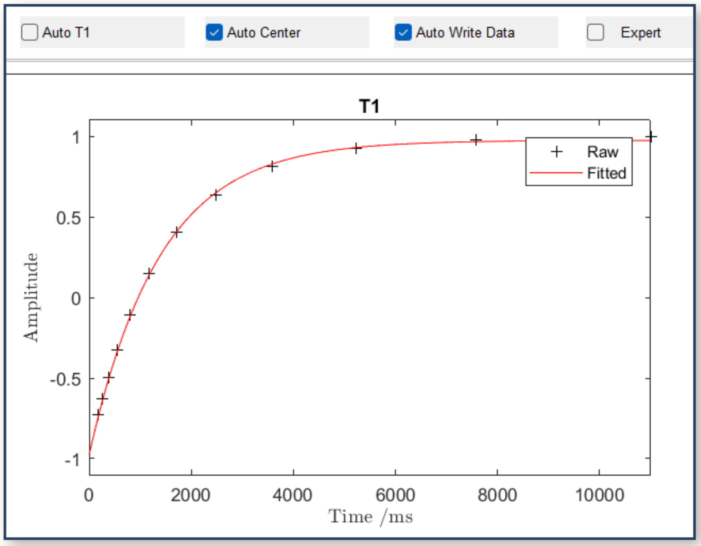

☐ Auto T1 ☑ Auto Center ☑ Auto Write Data ☐ Expert

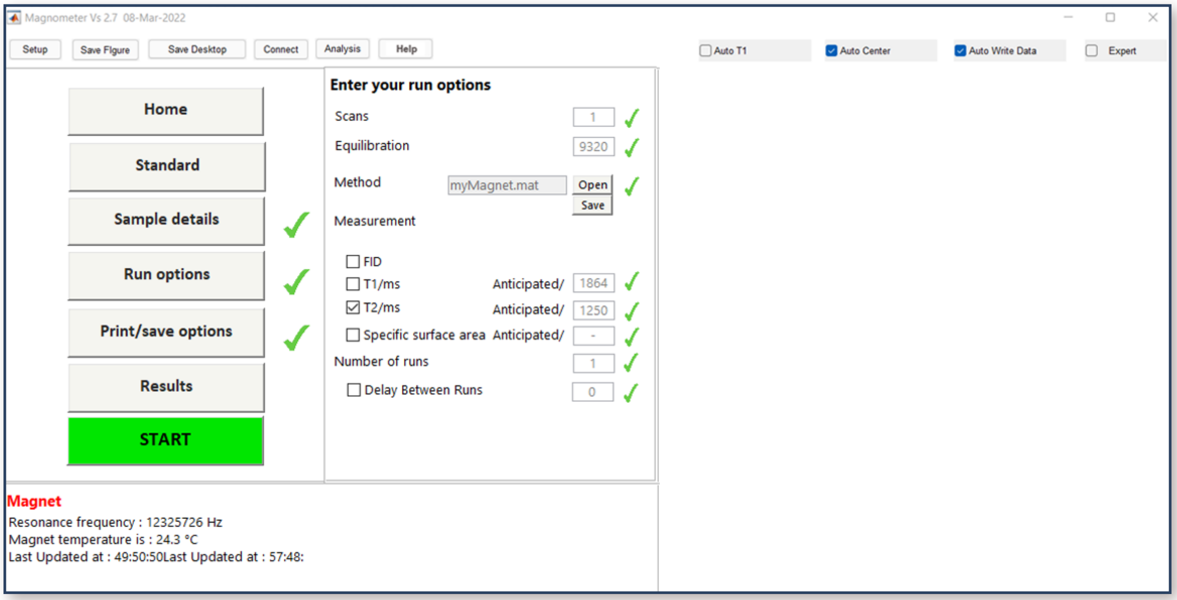
FIGURE 20(b).

NOTE: If the guess is too short, there will be too much baseline; if too long, there will be insufficient baseline.

NOTE: By inputting values for “Wt%” and “Solid Density”, the software automatically calculates a value for the Volume Ratio - seen here as 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 (1864 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. 23):



Enter your run options

Scans 1 ✓

Equilibration 9320 ✓

Method myMagnet.mat ✓

Measurement

☐ FID

☐ T1/ms Anticipated/ 1864 ✓

☑ T2/ms Anticipated/ 1250 ✓

☐ Specific surface area Anticipated/ - ✓

Number of runs 1 ✓

☐ Delay Between Runs 0 ✓

Magnet

Resonance frequency : 12325726 Hz

Magnet temperature is : 24.3 °C

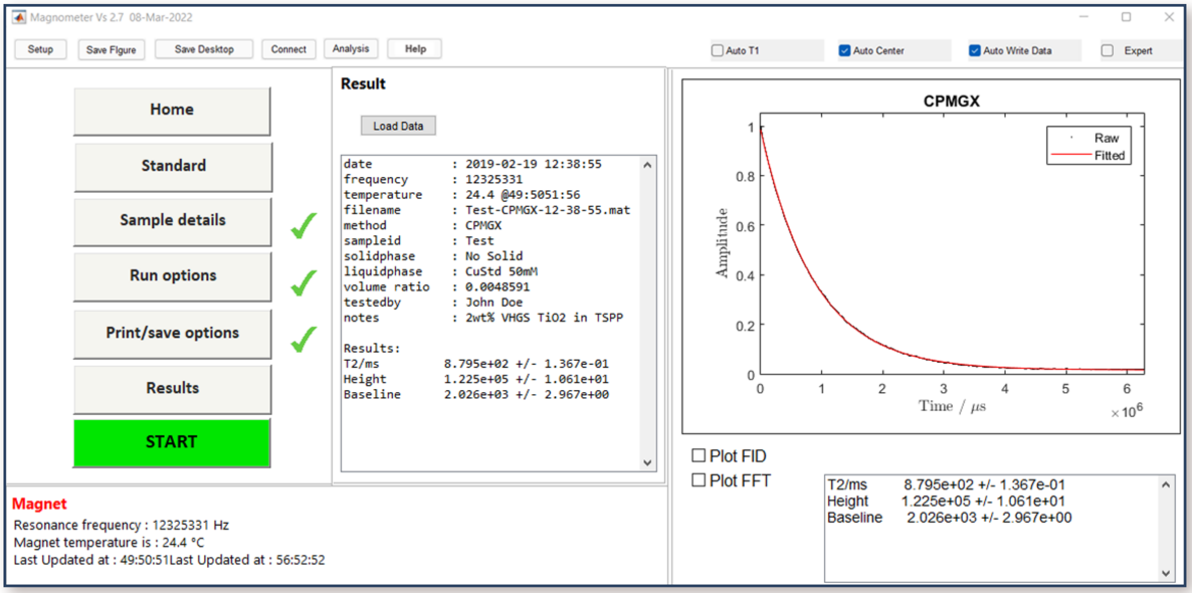
Last Updated at : 49:50:50Last Updated at : 57:48:

FIGURE 23.

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

Press the START button.

At the conclusion of the measurement record the T_2 value from the screen that looks like this (Fig. 24):



☐ Auto T1 ☑ Auto Center ☑ Auto Write Data ☐ Expert

```
date           : 2019-02-19 12:38:55
frequency      : 12325331
temperature    : 24.4 @49:5051:56
filename       : Test-CPMGX-12-38-55.mat
method         : CPMGX
sampleid       : Test
solidphase     : No Solid
liquidphase    : CuStd 50mM
volume ratio   : 0.0048591
testedby       : John Doe
notes          : 2wt% VHGS TiO2 in TSPP

Results:
T2/ms         8.795e+02 +/- 1.367e-01
Height        1.225e+05 +/- 1.061e+01
Baseline      2.026e+03 +/- 2.967e+00
```

Magnet
Resonance frequency : 12325331 Hz
Magnet temperature is : 24.4 °C
Last Updated at : 49:50:51Last Updated at : 56:52:52

☐ Plot FID
☐ Plot FFT

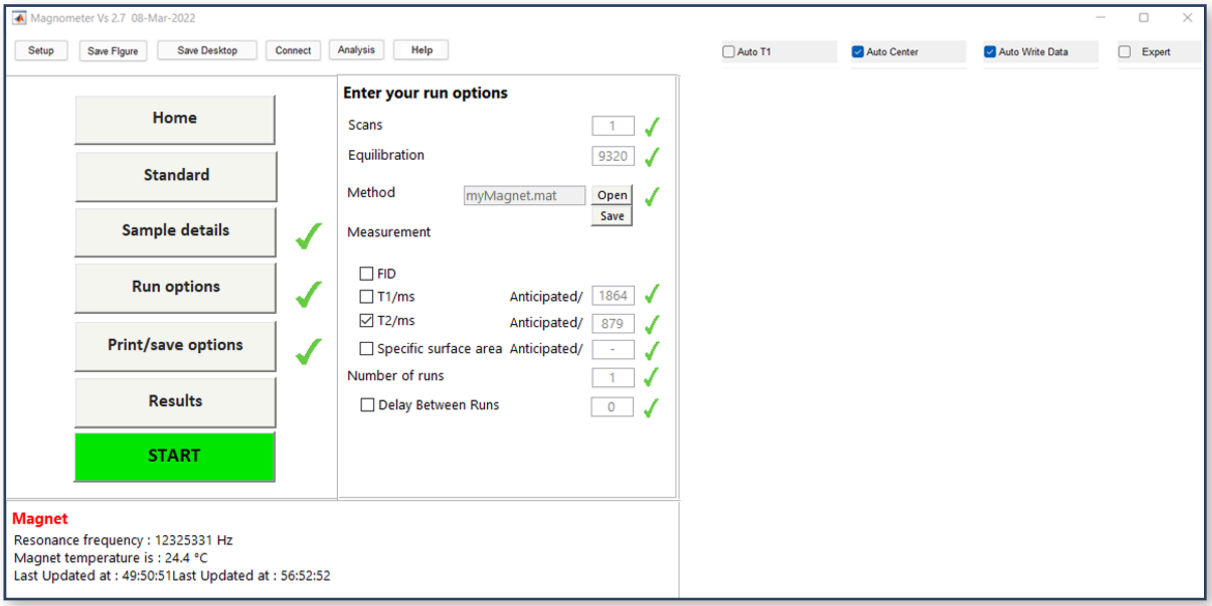
```
T2/ms       8.795e+02 +/- 1.367e-01
Height      1.225e+05 +/- 1.061e+01
Baseline     2.026e+03 +/- 2.967e+00
```

FIGURE 24.

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

STEP (3). Repeat the T_2 measurement by pressing the START 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. 25):



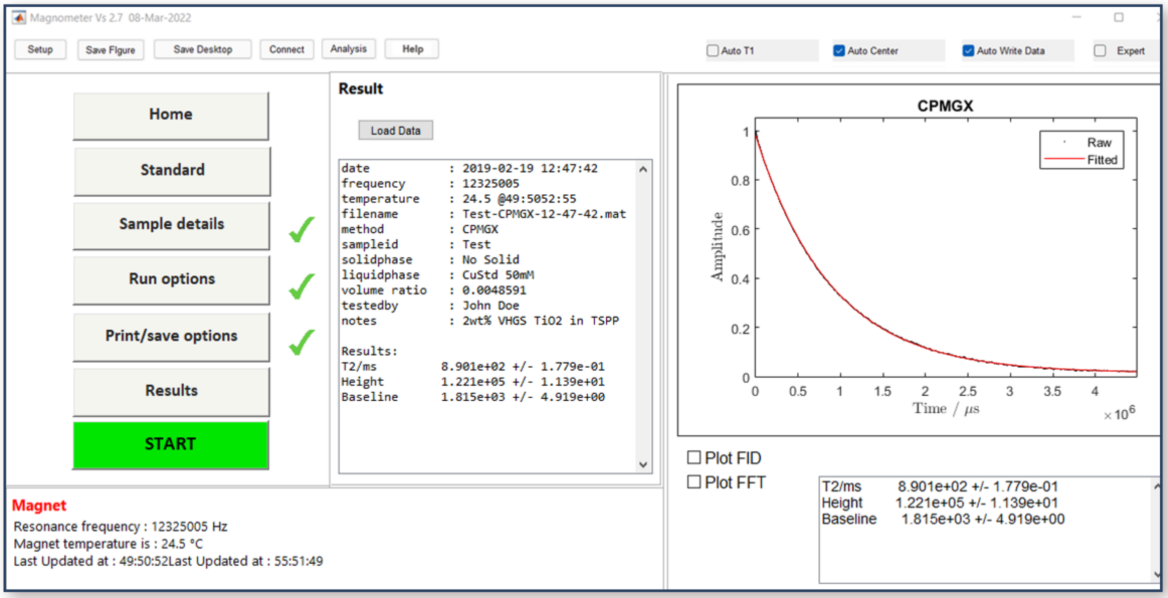
Enter your run options

Scans 1 ✓
Equilibration 9320 ✓
Method myMagnet.mat ✓
Measurement
☐ FID
☐ T1/ms Anticipated/ 1864 ✓
☑ T2/ms Anticipated/ 879 ✓
☐ Specific surface area Anticipated/ - ✓
Number of runs 1 ✓
☐ Delay Between Runs 0 ✓

Magnet
Resonance frequency : 12325331 Hz
Magnet temperature is : 24.4 °C
Last Updated at : 49:50:51Last Updated at : 56:52:52

FIGURE 25.

When the measurement is finished record the T_2 value obtained - *and this 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. 26):



Result

```
date           : 2019-02-19 12:47:42
frequency      : 12325005
temperature    : 24.5 @49:5052:55
filename       : Test-CPMGX-12-47-42.mat
method         : CPMGX
sampleid       : Test
solidphase     : No Solid
liquidphase    : CuStd 50mM
volume ratio   : 0.0048591
testedby       : John Doe
notes          : 2wt% VHGS TiO2 in TSPP

Results:
T2/ms         8.901e+02 +/- 1.779e-01
Height        1.221e+05 +/- 1.139e+01
Baseline      1.815e+03 +/- 4.919e+00
```

```
T2/ms      8.901e+02 +/- 1.779e-01
Height     1.221e+05 +/- 1.139e+01
Baseline    1.815e+03 +/- 4.919e+00
```

Magnet
Resonance frequency : 12325005 Hz
Magnet temperature is : 24.5 °C
Last Updated at : 49:50:52Last Updated at : 55:51:49

FIGURE 26.

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 saved) and the data averaged.

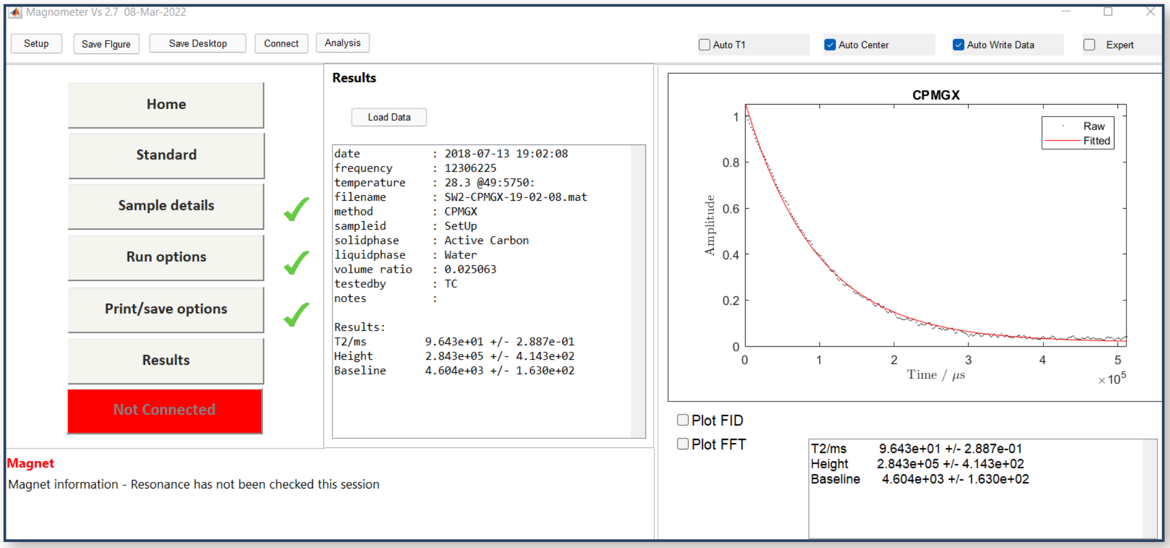
(iii) When making multiple runs the Auto Center button must be unchecked.

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

Reanalysis of Stored Data

The results of any measurement that have been made (and stored) can be reanalyzed at any time by using the Analysis button at the top right of the Home page screen (Fig. 3).

An example is shown in Figure 27.



☐ Auto T1 ☑ Auto Center ☑ Auto Write Data ☐ Expert

```
date           : 2018-07-13 19:02:08
frequency      : 12306225
temperature    : 28.3 @49:5750:
filename       : SW2-CPMGX-19-02-08.mat
method         : CPMGX
sampleid       : SetUp
solidphase     : Active Carbon
liquidphase    : Water
volume ratio   : 0.025063
testedby       : TC
notes          :

Results:
T2/ms         9.643e+01 +/- 2.887e-01
Height        2.843e+05 +/- 4.143e+02
Baseline      4.604e+03 +/- 1.630e+02
```

Magnet

Magnet information - Resonance has not been checked this session

☐ Plot FID

☐ Plot FFT

```
T2/ms      9.643e+01 +/- 2.887e-01
Height     2.843e+05 +/- 4.143e+02
Baseline    4.604e+03 +/- 1.630e+02
```

FIGURE 27.

Here we see the results of a measurement in which the T_2 relaxation time was found to be 96.43ms. The suspension was a sample of an Active Carbon dispersed in water.

The filename (where the data has been stored) is “SW2-CPMGX-19-02-08”.

NOTE: It can be seen that there isn’t a good single exponential fit (black line) to the raw data (red line). Such results are typically found in suspensions where there is a fraction of larger (aggregated) particles, or if the material is porous. It also occurs with emulsions where there are two distinct liquid phases. For more detailed information please visit the Mageleka website (www.mageleka.com).

On activating the Analysis button, the following screen should appear (Fig. 28).

NOTE: This may take a few seconds. Please be patient!

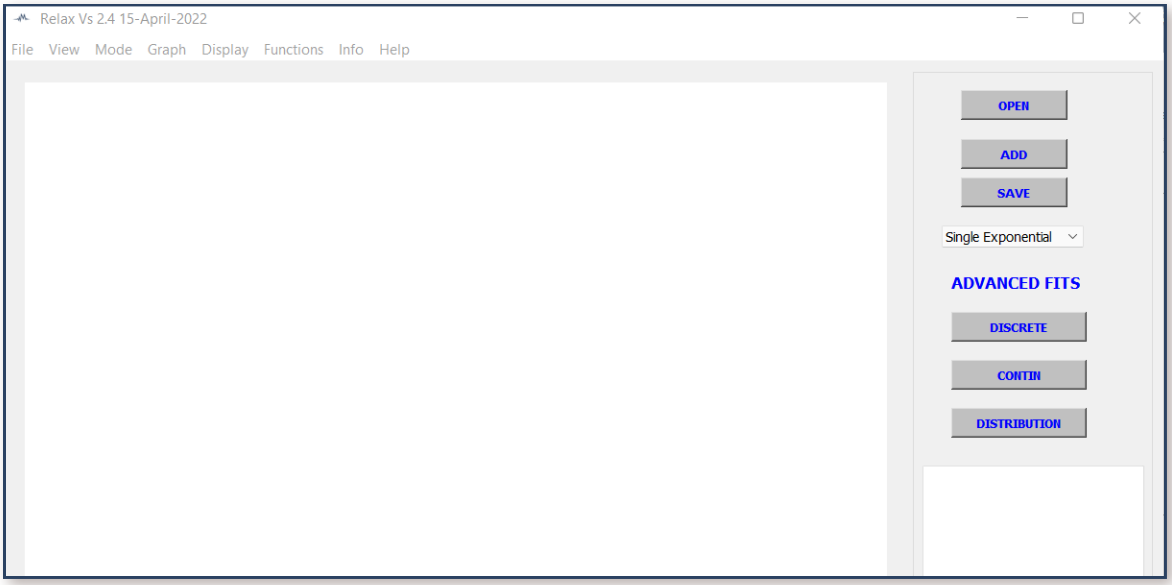
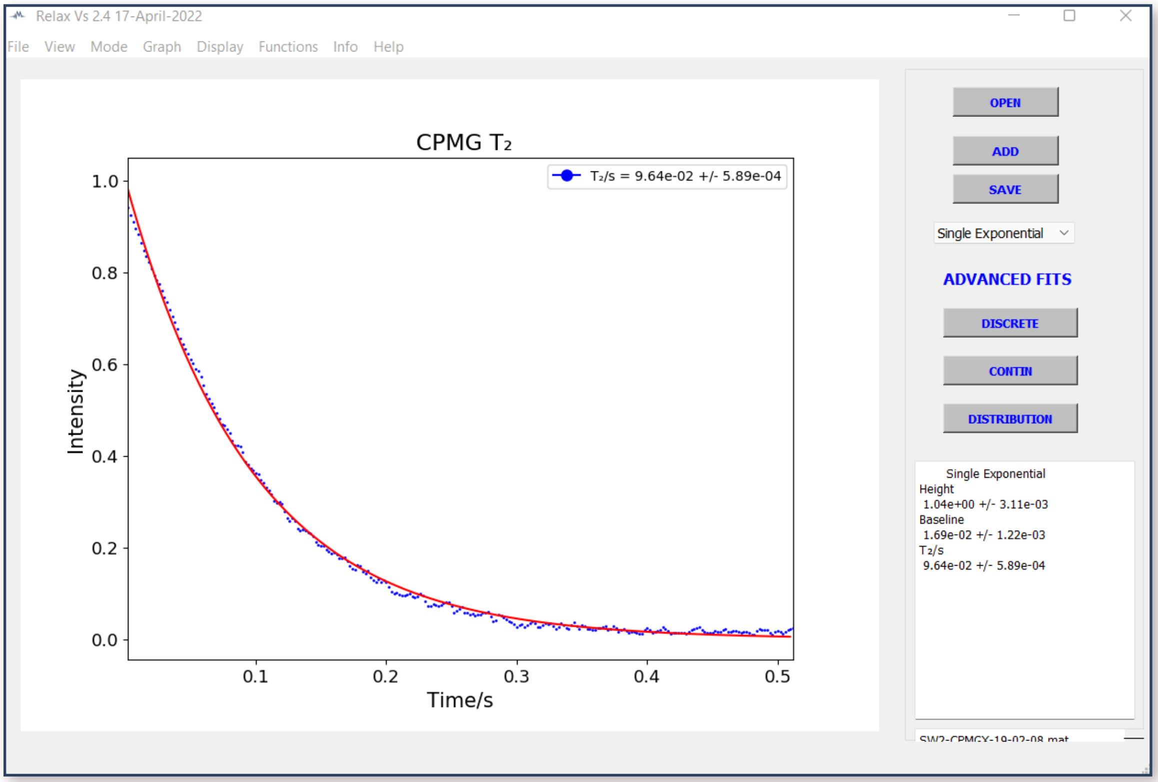


FIGURE 28.

NOTE: The most current version of the Relax software will be displayed at the very top left of the page. In this example it is Version 2.4.

Now click the Open button and download the data (filename SW2-CPMGX-14-44-04).

The following screen should appear (Fig. 29).



Single Exponential
Height
1.04e+00 +/- 3.11e-03
Baseline
1.69e-02 +/- 1.22e-03
T_2/s
9.64e-02 +/- 5.89e-04

FIGURE 29.

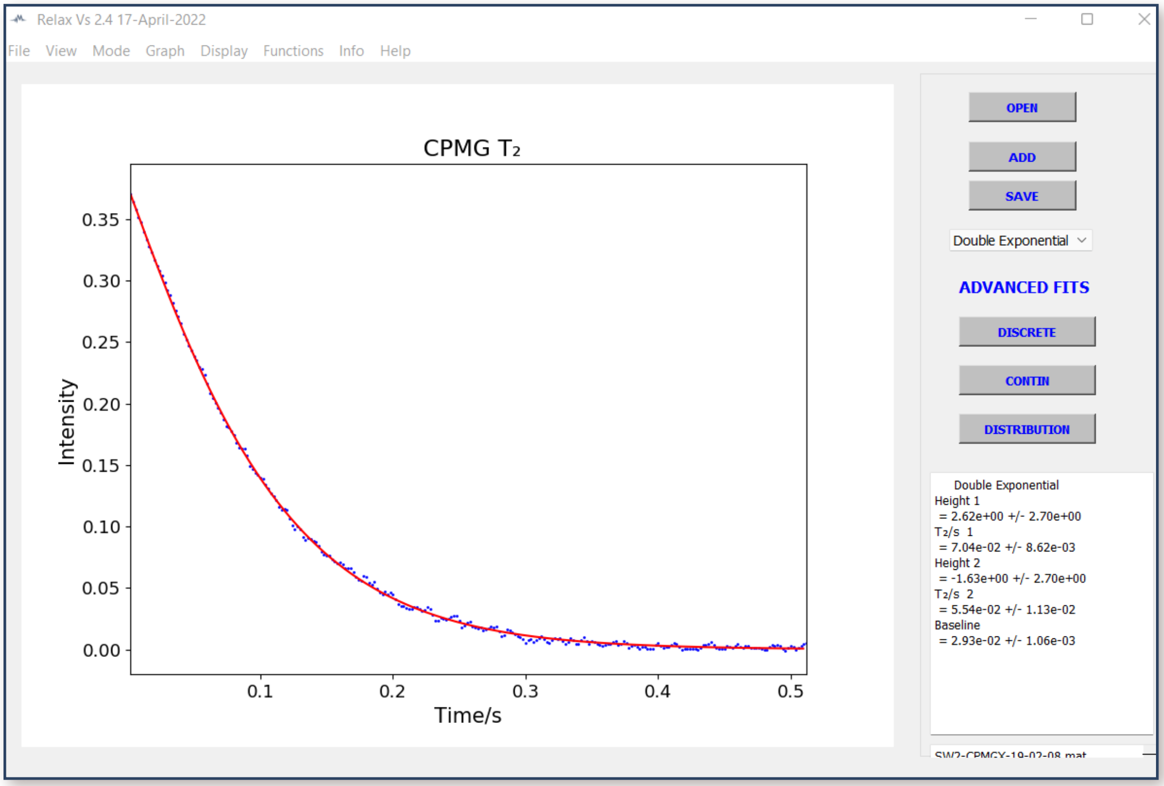
NOTE: Here, the T_2 value is shown in “seconds” as 9.64e-02 -- i.e., 96.4ms

Now click on the small box labeled “Single Exponential”. A draw-down menu will appear (Fig. 30).

Single Exponential
Double Exponential
Single Diffusion
Double Diffusion
Lorentzian
Double Lorentzian
Voigt
Frequency Sweep
Pulse Sweep

FIGURE 30.

Now click on “Double Exponential” and the following screen should appear (Fig. 31):



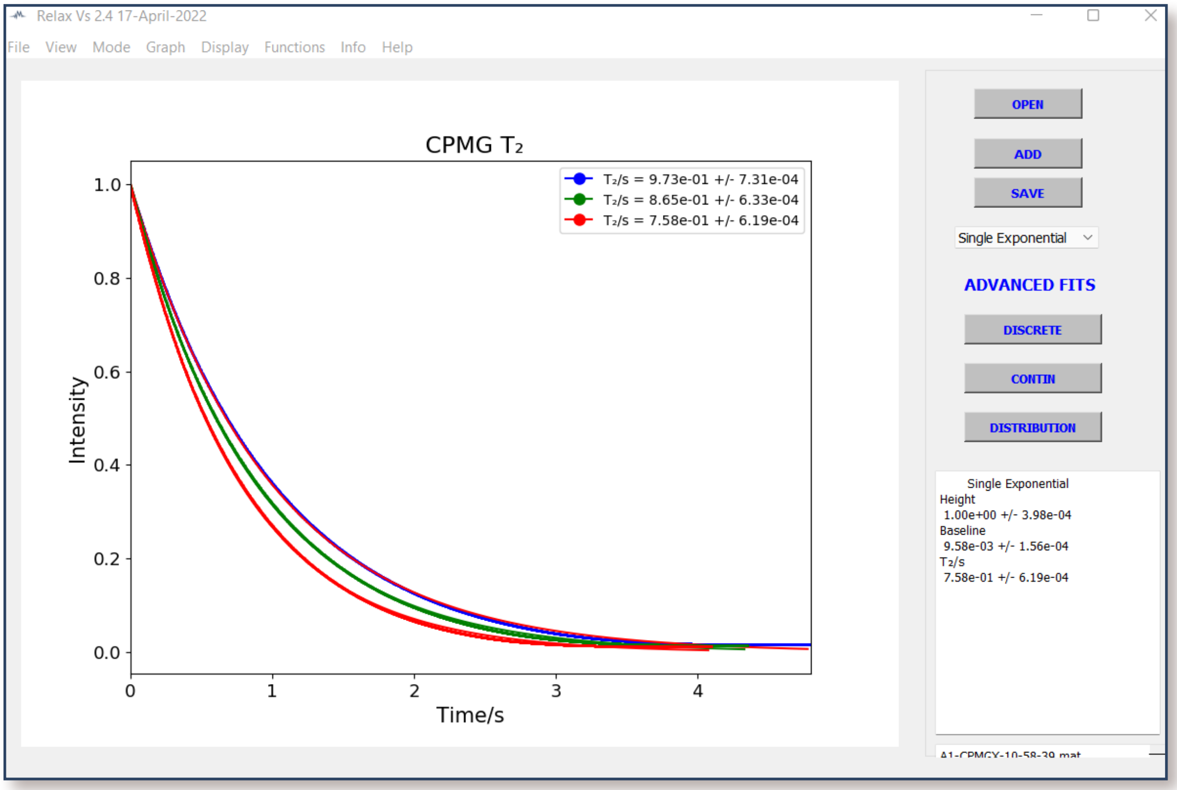
```
    Double Exponential
Height 1
 = 2.62e+00 +/- 2.70e+00
T₂/s  1
 = 7.04e-02 +/- 8.62e-03
Height 2
 = -1.63e+00 +/- 2.70e+00
T₂/s  2
 = 5.54e-02 +/- 1.13e-02
Baseline
 = 2.93e-02 +/- 1.06e-03
```

FIGURE 31.

The fit to the raw relaxation data is now good, and it shows that there are now two distinct relaxation times for this suspension – a short T_2 time (55.4ms) and a long T_2 time (70.4ms) – reflecting the presence of two different dispersed size fractions of the active carbon.

NOTE: All the other functions in the draw-down menu are not used with the *R*elaxo*M*eter.

Multiple plots (up to five) of individual relaxation data can be overlaid for comparison purposes by using the ADD button. An example is shown for three plots (in Fig. 32).



T_2/s = 9.73e-01 +/- 7.31e-04
T_2/s = 8.65e-01 +/- 6.33e-04
T_2/s = 7.58e-01 +/- 6.19e-04

```
    Single Exponential
Height
 1.00e+00 +/- 3.98e-04
Baseline
 9.58e-03 +/- 1.56e-04
T₂/s
 7.58e-01 +/- 6.19e-04
```

A1-CPMGX-10-58-39.mat

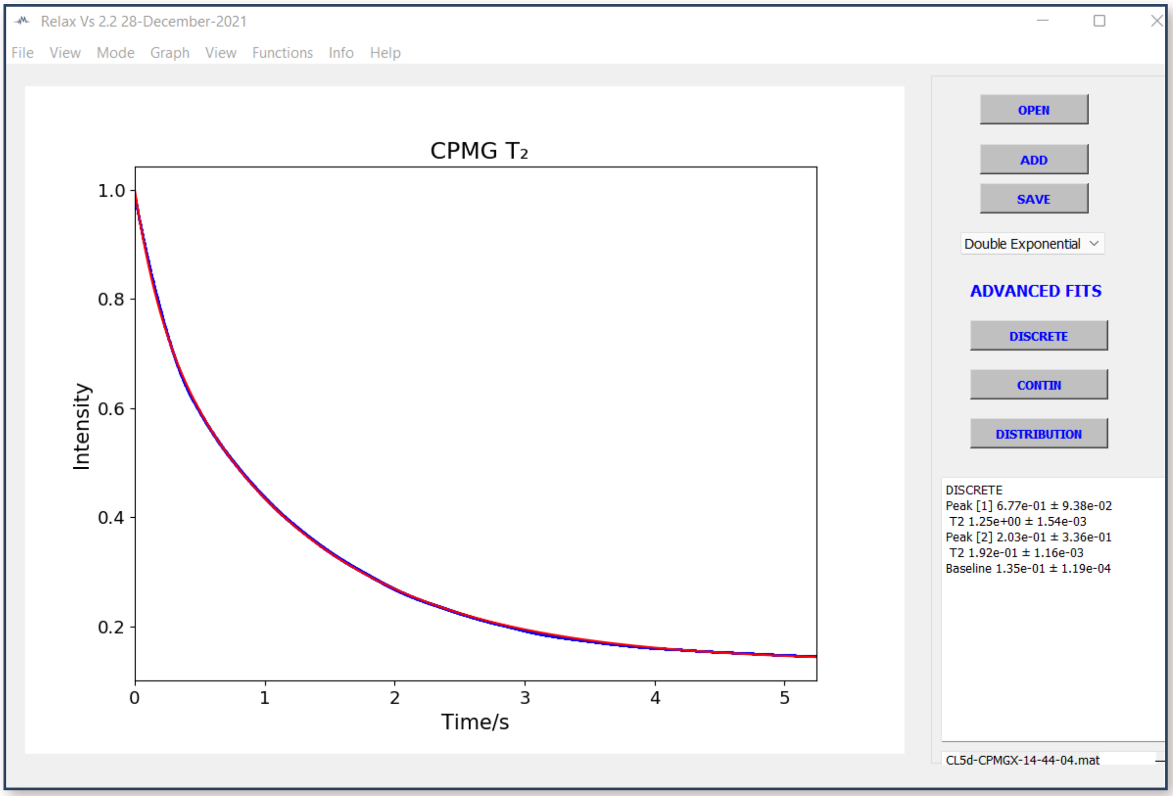
FIGURE 32.

Advanced Fitting

The ADVANCED FITS option – on the right-hand side - provides three buttons for more sophisticated analysis of the raw data.

The DISCRETE button activates a higher order fitting routine that can deconvolute complex relaxation data comprising up to five exponentials (Fig. 33). It is a simplified version of CONTIN.

The example shown is for a blue pigment dispersed in butyl cellosolve. The short T_2 (192ms) and long T_2 (1250ms) relaxation times are, respectively, contributions of the pigment and solvent.



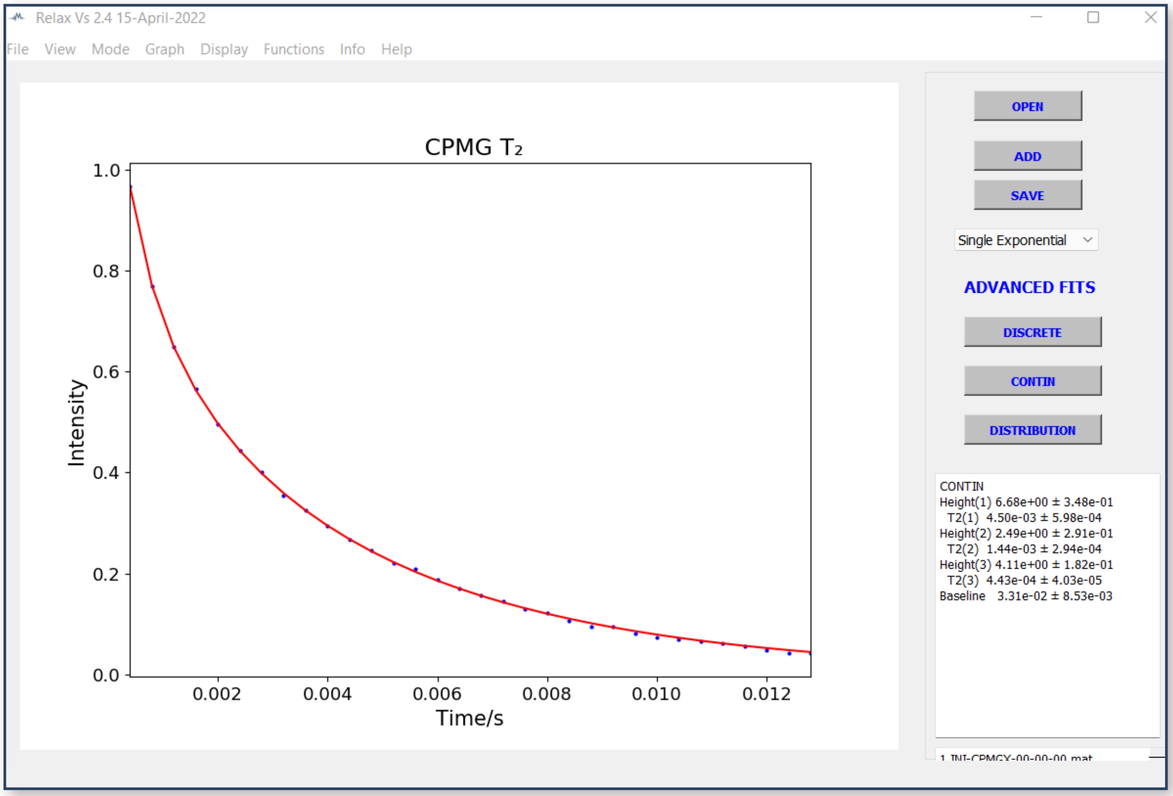
```
DISCRETE
Peak [1] 6.77e-01 ± 9.38e-02
 T2 1.25e+00 ± 1.54e-03
Peak [2] 2.03e-01 ± 3.36e-01
 T2 1.92e-01 ± 1.16e-03
Baseline 1.35e-01 ± 1.19e-04
```

CL5d-CPMGX-14-44-04.mat

FIGURE 33.

CONTIN is a well-established, powerful regularization method frequently used, for example, to obtain the size distribution of colloidal suspensions from dynamic light scattering data. It is typically not needed for most routine relaxation measurements.

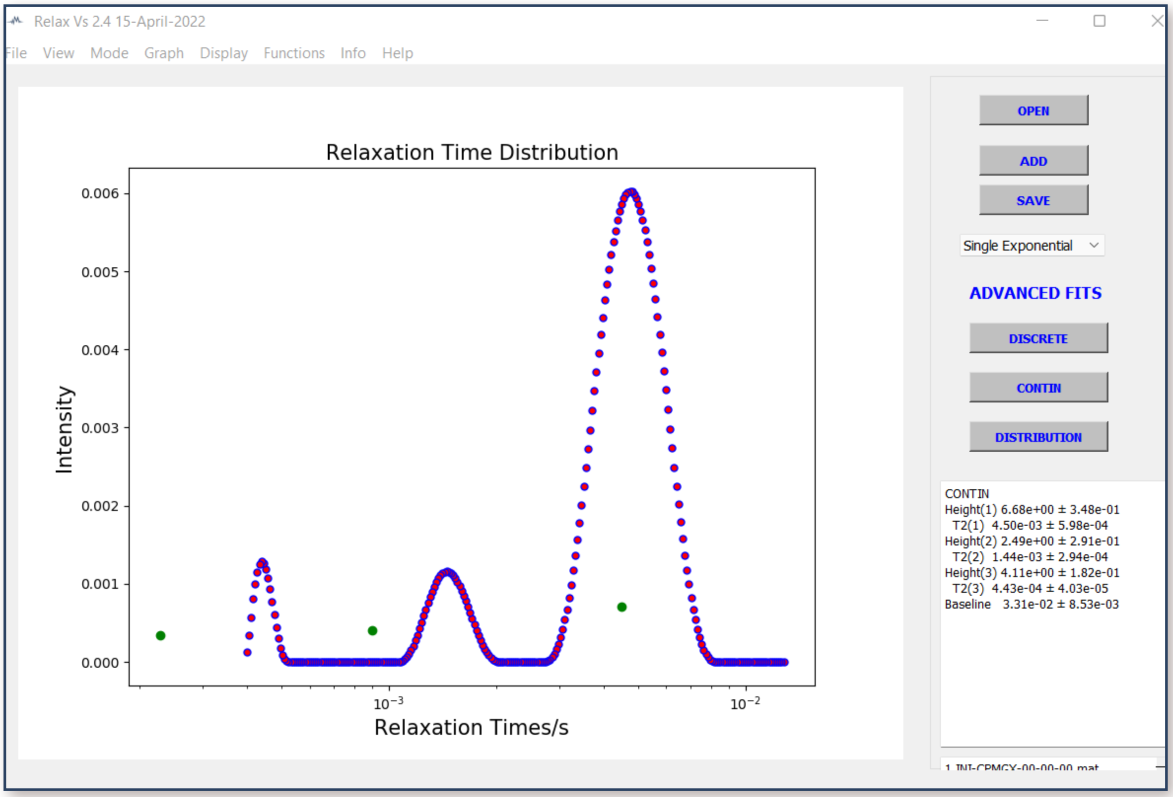
However, it can be useful for obtaining pore distribution data for porous materials. The example shown (Fig. 34) is data for a porous graphene in which there are three distinct relaxation times.



```
CONTIN
Height(1) 6.68e+00 ± 3.48e-01
  T2(1)  4.50e-03 ± 5.98e-04
Height(2) 2.49e+00 ± 2.91e-01
  T2(2)  1.44e-03 ± 2.94e-04
Height(3) 4.11e+00 ± 1.82e-01
  T2(3)  4.43e-04 ± 4.03e-05
Baseline   3.31e-02 ± 8.53e-03
```

FIGURE 34.

Clicking on the DISTRIBUTION button results in the following graph (Fig. 35).



Single Exponential

```
CONTIN
Height(1) 6.68e+00 ± 3.48e-01
  T2(1)  4.50e-03 ± 5.98e-04
Height(2) 2.49e+00 ± 2.91e-01
  T2(2)  1.44e-03 ± 2.94e-04
Height(3) 4.11e+00 ± 1.82e-01
  T2(3)  4.43e-04 ± 4.03e-05
Baseline   3.31e-02 ± 8.53e-03
```

FIGURE 35.

NOTE: To obtain actual pore size distribution data requires calibration. A separate device – the *Magno**M**eter* *Porosi**M**eter* – is available for this purpose. It employs intrusion porosimetry but without mercury. For more detailed information please visit the Mageleka website (www.mageleka.com).

There are a further eight small buttons at the top left-hand side. Most will not be needed for normal, routine measurements.

The File button provides a drop-down menu that allows you the following options:

- “Save Figure” - saves the image as a JPG or PNG file
- “Save Data” - makes a CSV file of the data for other analysis
- “Save Fit” - saves the fit as a CSV file
- “Save Distribution” - saves a CONTIN fit as a CSV file
- “Save Report” - saves a copy as a PDF file
- “Save as Excel” - saves the data in Excel format
- “Copy Result” - copies the results
- “Exit” - exits the program

The Info button provides complete, detailed data contained within the current file.

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.

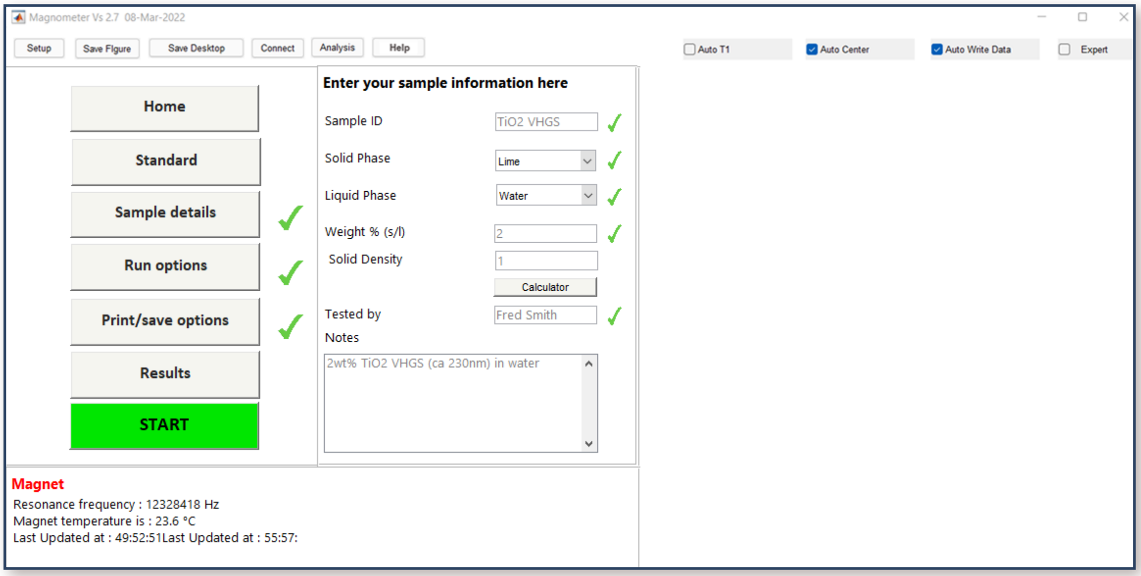
NOTE: The T_1 method is more advantageous if the initial relaxation time is found to be less than 10ms.

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. 36-42), 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. 36):



Enter your sample information here

Sample ID: TiO2 VHGS ✓
Solid Phase: Lime ✓
Liquid Phase: Water ✓
Weight % (s/l): 2 ✓
Solid Density: 1
Tested by: Fred Smith ✓
Notes: 2wt% TiO2 VHGS (ca 230nm) in water

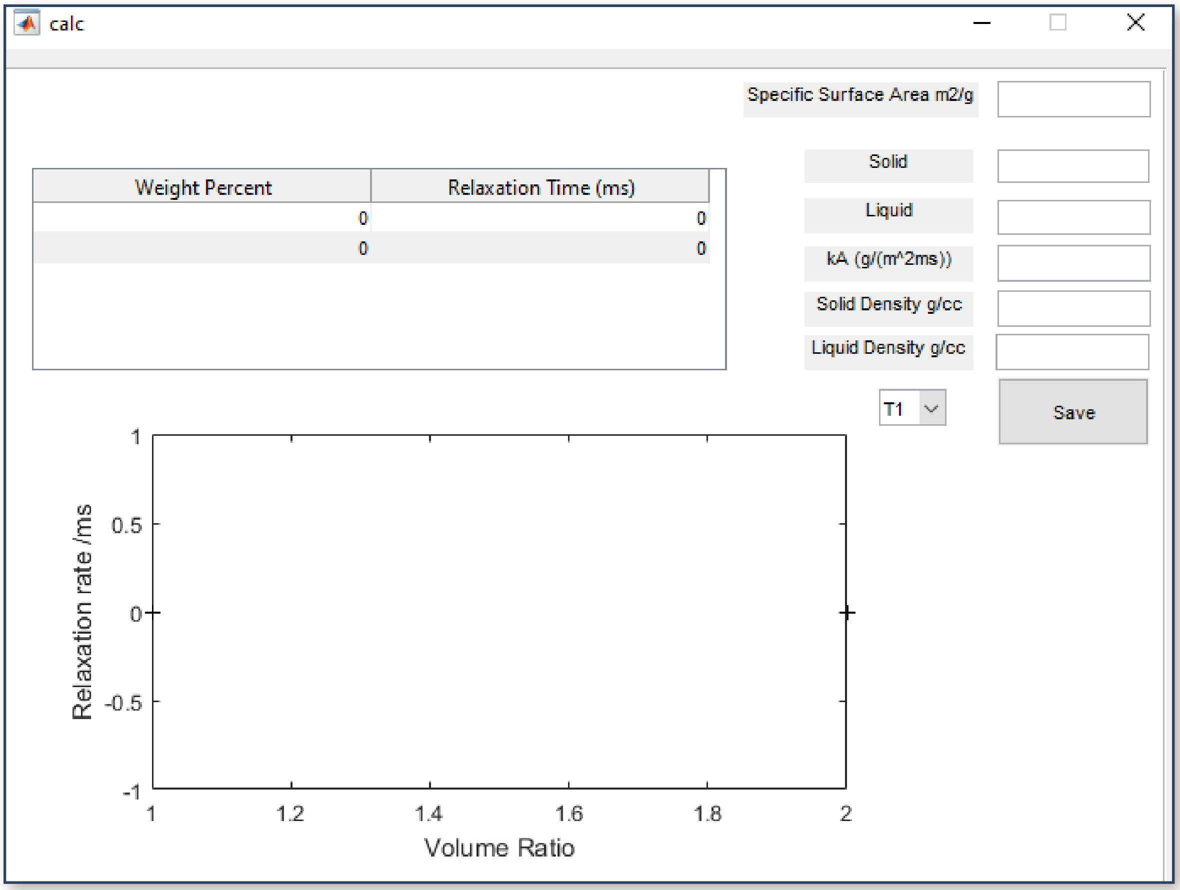
Magnet
Resonance frequency : 12328418 Hz
Magnet temperature is : 23.6 °C
Last Updated at : 49:52:51Last Updated at : 55:57:

FIGURE 36.

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

In the example shown above (Fig. 36), 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. 37):



Weight Percent	Relaxation Time (ms)
0	0
0	0

FIGURE 37.

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. Also, in this example, the T_2 time for the titania is **890 ms**.

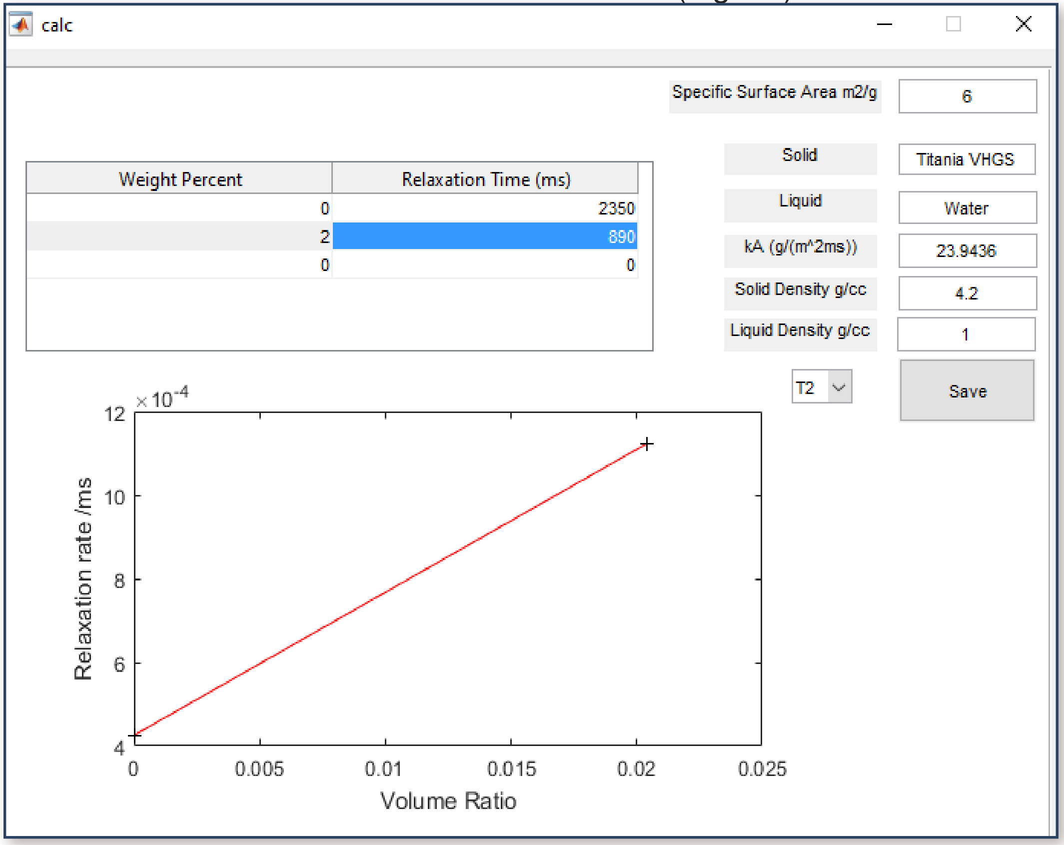
Enter an estimated value for the surface area of the suspension. For this (rutile) titania material it was estimated to be *ca.* 6 m^2g^{-1}.

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 “k_A” parameter will be automatically calculated from the data entered. More details about the k_A parameter are given in the Mageleka Technical Note 10 (see Appendix). A detailed discourse on NMR relaxation and its relevance to the measurement of the wetted surface area of particulate suspensions can be found in the Mageleka White Paper 1.

The “calc” sub-screen should now look like this (Fig. 38):

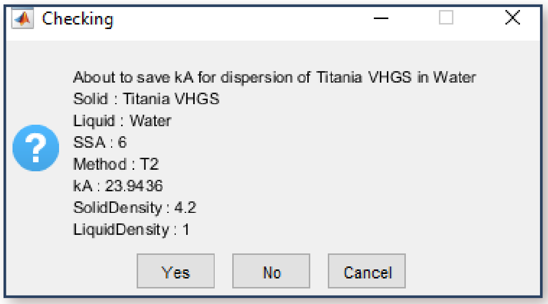


Weight Percent	Relaxation Time (ms)
0	2350
2	890
0	0

Specific Surface Area m2/g: 6
Solid: Titania VHGS
Liquid: Water
kA (g/(m^2ms)): 23.9436
Solid Density g/cc: 4.2
Liquid Density g/cc: 1

FIGURE 38.

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. 39):

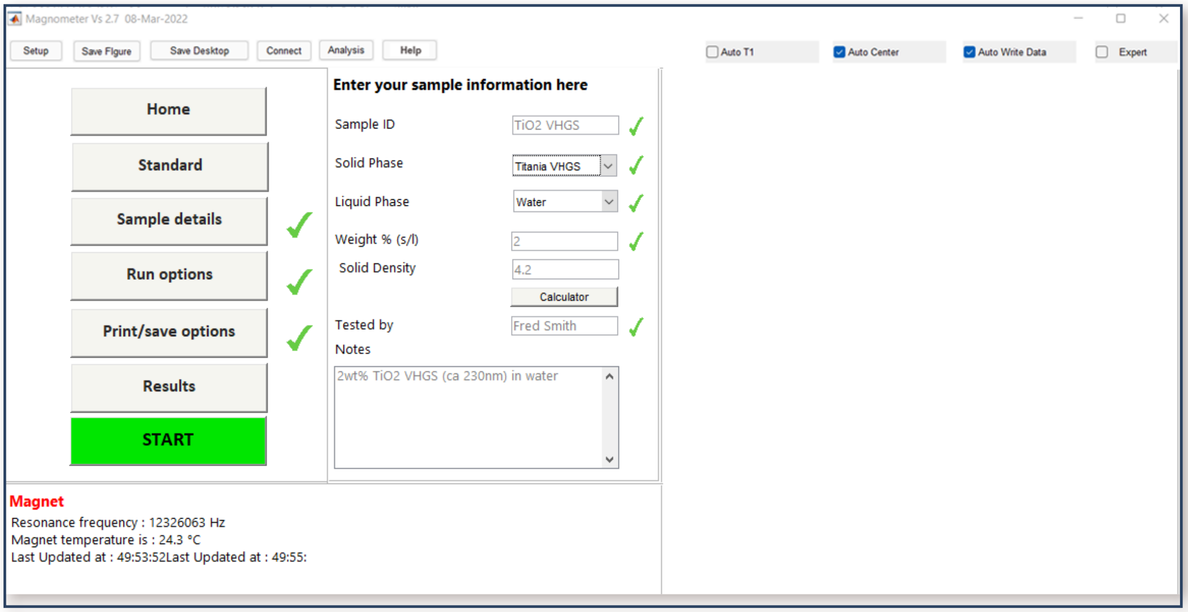


About to save kA for dispersion of Titania VHGS in Water
Solid : Titania VHGS
Liquid : Water
SSA : 6
Method : T2
kA : 23.9436
SolidDensity : 4.2
LiquidDensity : 1

FIGURE 39.

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. 40):



☐ Auto T1
☑ Auto Center
☑ Auto Write Data
☐ Expert

Enter your sample information here

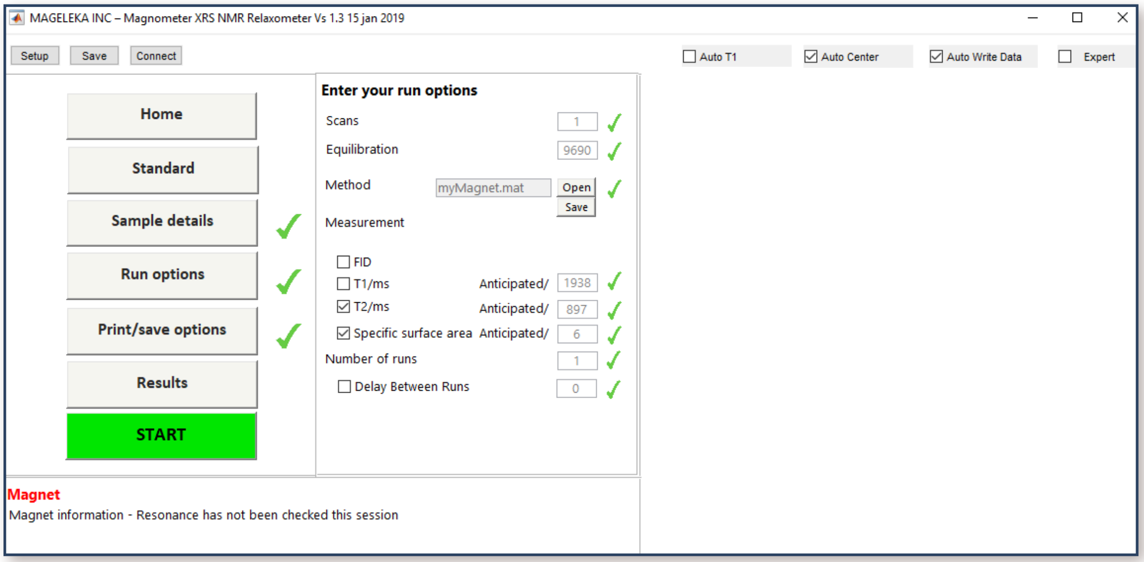
Sample ID: TiO2 VHGS ✓
Solid Phase: Titania VHGS ✓
Liquid Phase: Water ✓
Weight % (s/l): 2 ✓
Solid Density: 4.2
Tested by: Fred Smith ✓
Notes: 2wt% TiO2 VHGS (ca 230nm) in water

Magnet
Resonance frequency : 12326063 Hz
Magnet temperature is : 24.3 °C
Last Updated at : 49:53:52Last Updated at : 49:55:

FIGURE 40.

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

Next, from the Run options menu select “Specific surface area”. The screen should now look like this (Fig. 41):



MAGELEKA INC – Magnometer XRS NMR Relaxometer Vs 1.3 15 jan 2019

Setup Save Connect

☐ Auto T1 ☑ Auto Center ☑ Auto Write Data ☐ Expert

Home

Standard

Sample details ✓

Run options ✓

Print/save options ✓

Results

START

Enter your run options

Scans 1 ✓

Equilibration 9690 ✓

Method myMagnet.mat Open Save ✓

Measurement

☐ FID

☐ T1/ms Anticipated/ 1938 ✓

☑ T2/ms Anticipated/ 897 ✓

☑ Specific surface area Anticipated/ 6 ✓

Number of runs 1 ✓

☐ Delay Between Runs 0 ✓

Magnet

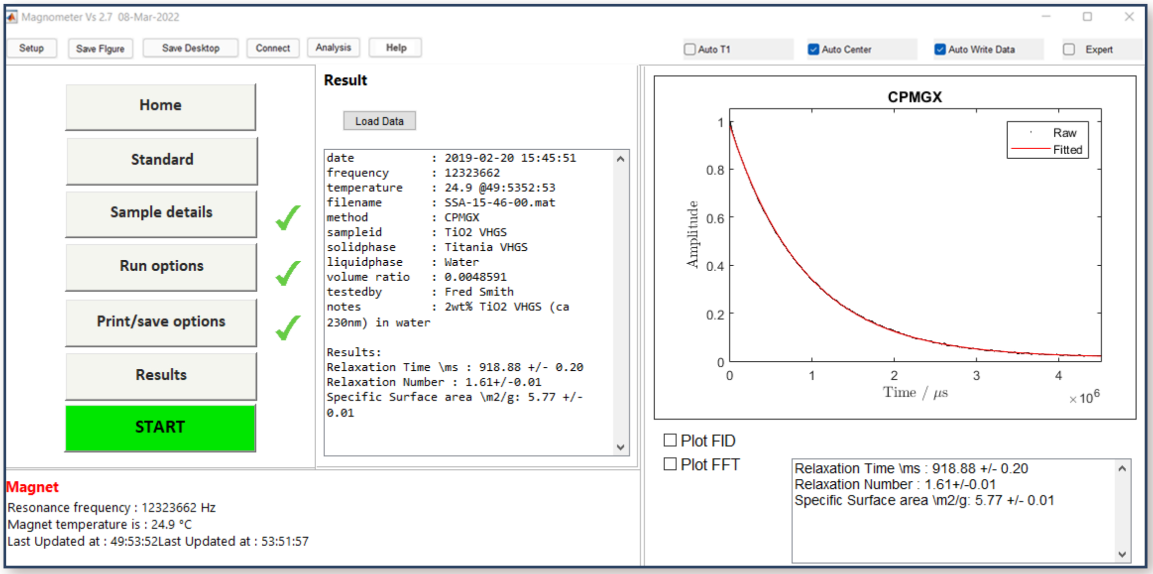
Magnet information - Resonance has not been checked this session

FIGURE 41.

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. 41).

Then hit the START button to begin a measurement.

At the conclusion, the following screen will appear (Fig. 42):



```
date           : 2019-02-20 15:45:51
frequency      : 12323662
temperature    : 24.9 @49:5352:53
filename       : SSA-15-46-00.mat
method         : CPMGX
sampleid       : TiO2 VHGS
solidphase     : Titania VHGS
liquidphase    : Water
volume ratio   : 0.0048591
testedby       : Fred Smith
notes          : 2wt% TiO2 VHGS (ca
230nm) in water

Results:
Relaxation Time \ms : 918.88 +/- 0.20
Relaxation Number : 1.61+/-0.01
Specific Surface area \m2/g: 5.77 +/-
0.01
```

```
Magnet
Resonance frequency : 12323662 Hz
Magnet temperature is : 24.9 °C
Last Updated at : 49:53:52Last Updated at : 53:51:57
```

```
Relaxation Time \ms : 918.88 +/- 0.20
Relaxation Number : 1.61+/-0.01
Specific Surface area \m2/g: 5.77 +/- 0.01
```

FIGURE 42.

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

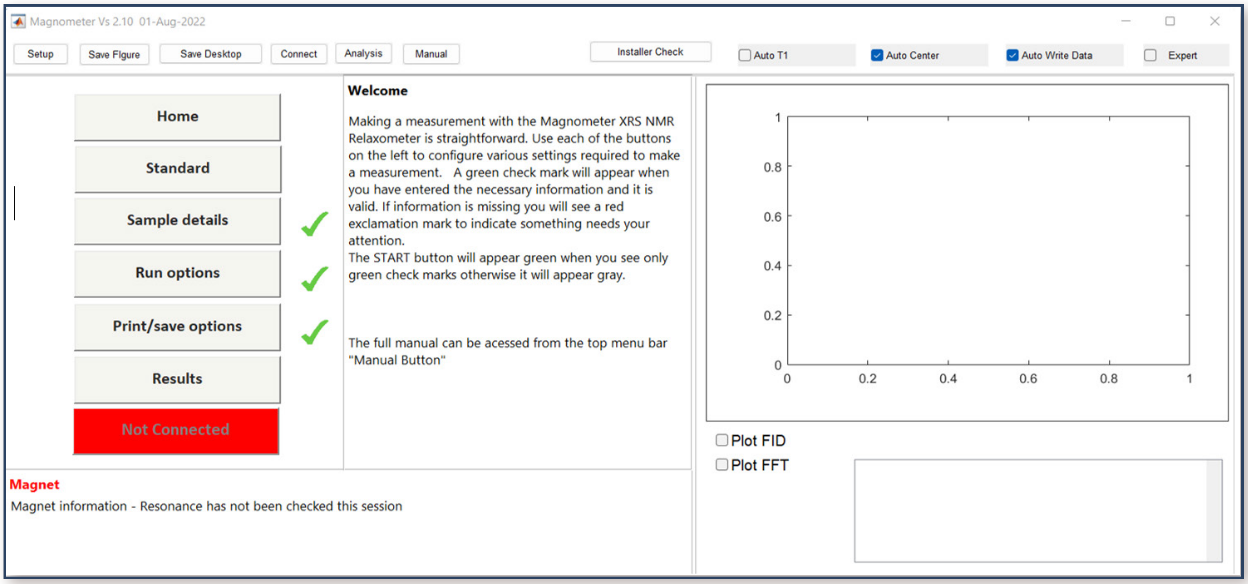
If desired, the measurement can now be repeated by, again, simply hitting the START button.

NOTE: A value for the “Relaxation Number” is also calculated. A detailed explanation of this useful dimensionless parameter can be found in the Mageleka Technical Note #4.

New or updated software versions, specific documents, or instructions will be added here.

Appendix A

Figure A1 shows a screen shot of software Version 3.00.



Welcome

Making a measurement with the Magnometer XRS NMR Relaxometer is straightforward. Use each of the buttons on the left to configure various settings required to make a measurement. A green check mark will appear when you have entered the necessary information and it is valid. If information is missing you will see a red exclamation mark to indicate something needs your attention.
The START button will appear green when you see only green check marks otherwise it will appear gray.

The full manual can be acessed from the top menu bar "Manual Button"

Magnet

Magnet information - Resonance has not been checked this session

FIGURE A1.

A new button Installer Check has been added. When clicked, the following box appears (Fig. A2):

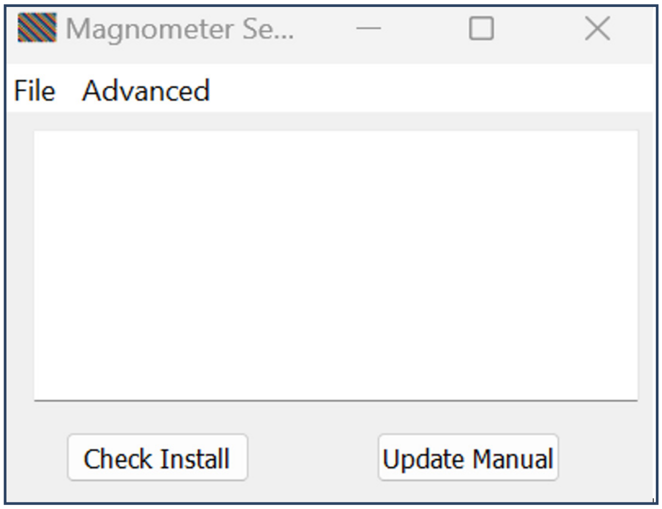
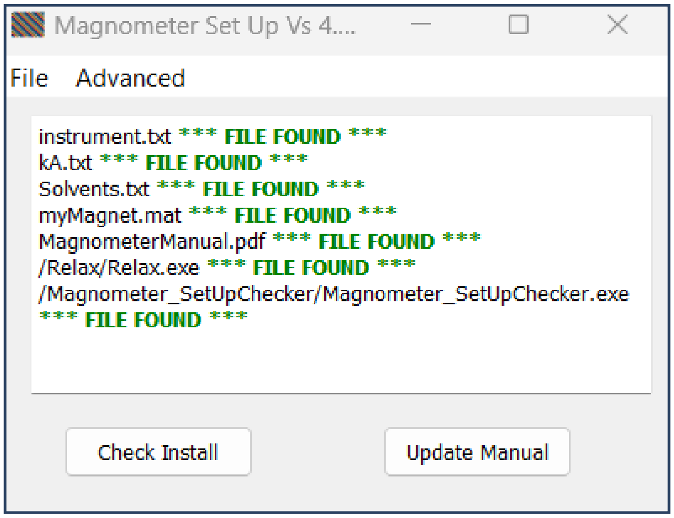


FIGURE A2.

There are two buttons, Check Install and Update Manual.

On clicking Check Install , the following dialog box appears (Fig. A3):

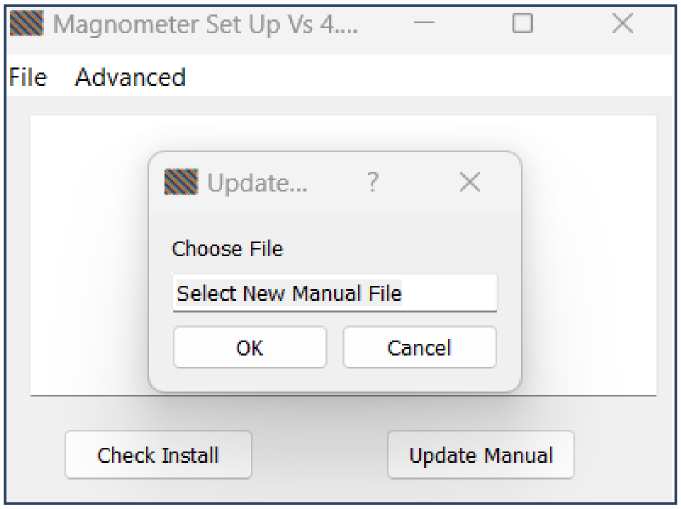


```
instrument.txt *** FILE FOUND ***
kA.txt *** FILE FOUND ***
Solvents.txt *** FILE FOUND ***
myMagnet.mat *** FILE FOUND ***
MagnometerManual.pdf *** FILE FOUND ***
/Relax/Relax.exe *** FILE FOUND ***
/Magnometer_SetUpChecker/Magnometer_SetUpChecker.exe
*** FILE FOUND ***
```

FIGURE A3.

This function is used to see if there are any installation issues and it should be run after the main installation. It checks that all the various files are present in the Users/Public folder including the manual and if any are missing, it will write them in here.

On clicking Update Manual , the following dialog box appears (Fig. A4):



Choose File: Select New Manual File

FIGURE A4.

This function enables the user to update the manual and then puts it into the correct directory.

Mageleka **Technical Note 10**

Calculating a Wetted Surface Area Value from NMR Relaxation Data: The Specific Surface Relaxivity Parameter, k_A

A detailed discourse on nuclear magnetic resonance (NMR) relaxation and its relevance to the measurement of the wetted surface area of particulate suspensions can be found in the Mageleka White Paper 1 (at www.mageleka,com). See also reference [1]. Although the NMR relaxation method is independent of the particle size distribution and shape of a material, in the calculation of an absolute value for the wetted surface area from relaxation data it is necessary to input values for two parameters.

The first is the "volume ratio" of solid-to-liquid*,* ψ. This parameter requires knowing not only the particle concentration but also the density for both the dispersion fluid and the particles. For convenience, the Mageleka *R*elaxo*M*eter *M*agno*S*oft™ software will automatically calculate ψ from the weight percentage of solid.

Note that the precision of the density value of both the solid material and the suspension (from supplied MSDS, manufacturer's technical data, or from the literature) is important, as it directly affects the calculation of the solid-liquid *volume ratio* which is used in the determination of the surface area.

The second parameter is the specific surface relaxivity, or k_A. The k_A value characterizes the relaxation time shift per unit surface area for a given particle-liquid combination and is typically determined through a reference material (i.e., a particle of known surface area dispersed in the test liquid). The selection of a reference material is important. The essential component of a good reference material is that, ideally, its surface chemistry should be as close as possible to that of the material whose surface area is to be measured.

The calculation of "Surface Area" (*SA*) from relaxation measurements is then straightforward:

$$SA = [R_{av} - R_{solv}] / [k_A.\ \psi]$$

Where:

ψ is the volume ratio of solid-to-liquid

R_{av} is the spin relaxation rate constant for a suspension (i.e., R_{susp}) and it is the reciprocal of the spin relaxation time (T)

R_{solv} is the relaxation rate for the "solvent" (or suspension fluid).

The k_A parameter is, essentially, a simple multiplication factor in the same manner that a zeta potential value is obtained from the measured electrophoretic mobility value (by multiplying it by, for example, the Smoluchowski or Hückel factor). Alternatively, the k_A parameter can be considered similar to the refractive index, *RI,* required in a laser light scattering device to calculate the correct particle size distribution (PSD), in the sense that it depends on the combination of the particle and liquid. So, a k_A value for silica in water will be different from the k_A for the same silica in ethanol.

For user convenience, a list of k_A values, obtained for a variety of common materials (carbon black,

> *" A knowledge of the wetted surface area is critical in the formulation of particulate suspensions, and NMR relaxation measurements are a convenient means of directly determining this metric. "*

silica, etc.) in water and some solvents, is stored as an instrument file of the *R*elaxo*M*eter. These values are guidelines only. Users should always determine the k_A for their individual application and that value can be added to the list and then recalled for any future measurements.

We can normalize out the effect of solvent (which could include dispersants, additives, etc.):

$$R_{sp} = [R_{susp} - R_{solv}] / R_{solv}$$

R_{sp} is termed the specific relaxation rate enhancement and is dimensionless.

Thus,

$$SA = [R_{sp} . R_{solv}] / [k_A . \psi]$$

R_{sp} is often called the Relaxation Number, R_{no}, and it provides a direct rank of the strength of solvent-surface interaction. For a given PSD, higher values of R_{no} indicate a stronger net surface interaction.

Note that any surface area result can be recalculated (using the saved measurement raw data) at any future time if/when a more appropriate k_A value is available. However, it will not affect any relative differences found between samples since, in general, k_A is a constant for a given system (i.e., solvent, particle, etc.).

When monitoring changes in particle surface area for a given particle-liquid combination (e.g., in milling), the differences in relaxation rate are absolute and so determination of a specific value for k_A is not really necessary. This is somewhat analogous to the situation with the measurement of the zeta potential, where only relative changes in the value of the zeta potential of a suspension (as a function of some chemical additive) are absolute.

Factors to consider

A definitive k_A value for a given system will depend upon the chemical nature of the suspension and of the dispersing fluid, as well as the particle size and porosity of the material under investigation. Suspension fluids used in industrial/commercial formulations are not necessarily the pure liquid but may contain additives such as dispersants or surfactants. For example, there will be a difference between values obtained for a colloidal silica (SiO_2) depending on whether the dispersion has been highly dialyzed or not. Note also that we have found that, typically, a 10% variation in the relaxation time for a bulk liquid translates to a maximum change of *ca* 5% in a calculated surface area.

Further, industrial/commercial materials will differ owing to presence of impurities in the solid. The k_A for alumina is much different from that of silica. It is known that commercial silica materials can contain alumina (and also iron). Hence, a small amount of alumina (or iron) impurity in a silica will manifest a difference in relaxation time that is not necessarily related to a difference in total surface area but to a variation in the alumina (or iron) content. For example, both *pure* silica and *pure* alumina at *ca* 40% produce similar relaxation rate (R_1) values (between 4 and 8). However, for T_2 measurements, the situation is much different, as the relaxation rate (R_2) value for alumina is far greater than that for silica (*ca* 50 compared to *ca* 8). This is also important when analyzing alumino-silicate materials such as clays.

Determination of a k_A value

Ideally, a k_A value should be an average determined from one or more samples of the same material from different suppliers/manufacturers. To determine the k_A for a *specific* particle-liquid combination, you first need to have a single value of the *wetted* surface area determined from some other means. For example, for silica, you can obtain a value using the Sears titration method. However, this is not always possible. In such cases, an estimate can be obtained from empirical calculations based on particle size measurements using dynamic light scattering, centrifugal sedimentation, laser diffraction, or microscopy. Each of these methods has limitations associated with it.

When using particle size, we have found that, typically, the D_{10} value from a volume-weighted size distribution is the best metric from which to estimate the wetted surface area, in large part because NMR relaxation measurements are strongly sensitive to the smallest particles in a size distribution.

The surface area value from a BET/N_2 gas adsorption measurement can also be used but, because NMR measures the *wetted* surface area of suspensions, a BET value should only be used as a rough guide. This is because a wetted surface area depends critically on how the suspension has been prepared.

An additional way to interpret relaxation data sets is through the slopes of plots of R_{sp} *vs* ψ. This slope, λ, is a dimensionless quantity that describes surface wetting and is easily determined by measuring relaxation rates over a range of particle concentrations:

$$\lambda = k_A \, [SA] \, / \, R_{solv}$$

Hence, for a given surface chemistry, higher values of λ means that more water (solvent) is adsorbed (since the available surface area is larger).

The last equation can be re-written [2] as:

$$\lambda = [R_{susp} - R_{solv}] \, / \, [R_{solv} \cdot \psi]$$

In that paper, k_A values for a series of colloidal silicas were computed initially using surface area values derived from BET/N_2 gas adsorption measurements [2]. However, the paper shows that the slope, λ, can be calculated *directly* from the NMR parameters and ψ *independently* of BET. Hence, using the λ parameter, or R_{no}, eliminates the need for a reference surface area. Note that because no assumptions are made about particle shape or size, it is possible to utilize measurements of λ or R_{no} to study complicated morphologies such as highly aggregated particles, gels and fractal networks.

Measuring nanoparticulates

For coarse suspensions and colloidal dispersions where the particle size is >100nm, the k_A parameter is essentially constant. However, when NMR relaxation is used to measure *nanoparticulate* dispersions (<100nm), two effects must be considered that impact the value assumed for k_A: (i) the relaxation and translation/rotation of the particles and (ii) the dependence of effective volume of the surface zone upon particle radius.

The volume, V_{curve}, of a monolayer shell with thickness, δ, on a sphere of radius, a, is given by:

$$V_{curve} = 4/3 \, \pi \, [(a + \delta)^3 - a^3]$$

Hence the relative volume of the surface film will depend on curvature unless $\delta \ll a$.

The following experimental data [3] illustrates the effect of curvature (particle size) on the derivative of R_{sp} with surface area plotted against particle size (Fig. 1).

As can be seen, the effects become especially important when the particle radius is less than about 80 nm. The form of the plot is similar for all materials,

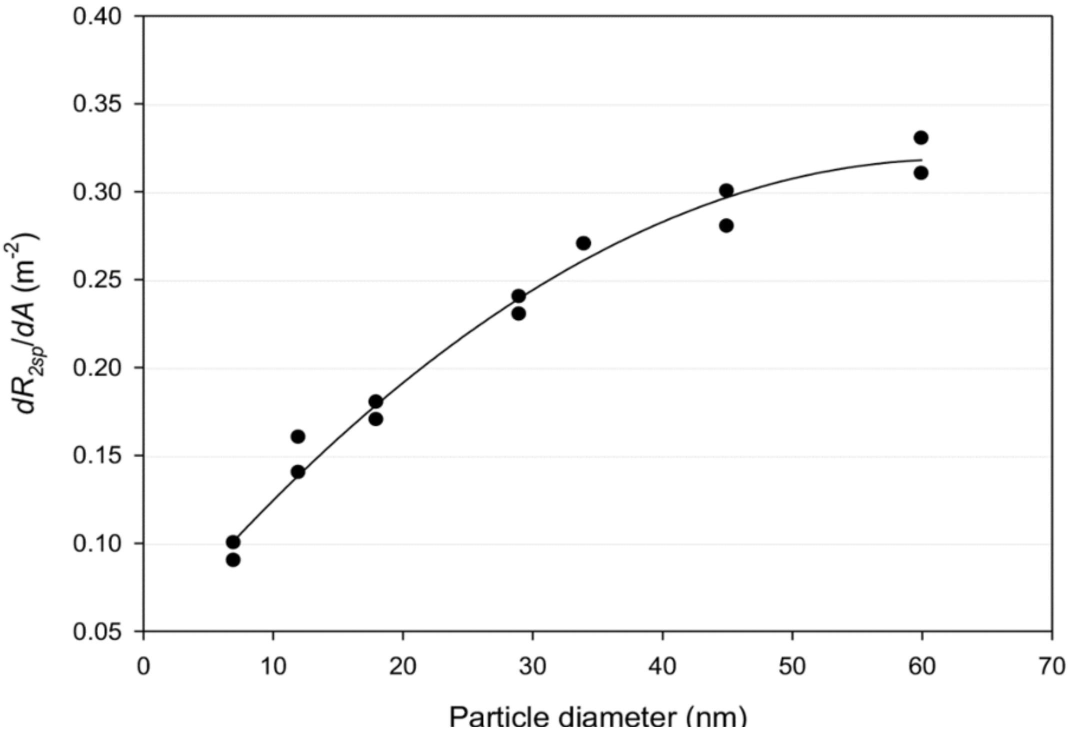


Figure 1: Effective curvature on the derivative of specific relaxation rate enhancement.

however, the scale for k_A will vary. For a given PSD, k_A is, however, still a constant.

In conclusion, a knowledge of the wetted surface area is critical in the formulation of particulate suspensions, and NMR relaxation measurements are a convenient means of directly determining this metric. The affordable benchtop Mageleka *R*elaxo*M*eter is ideal because relaxation measurements can be made at any industrially-relevant concentration without the need for dilution, and with little sample preparation. As this Technical Note has explained, ψ and k_A are important determinants in calculating a value from relaxation data, and the latter parameter particularly so for nanoparticulates. The *R*elaxo*M*eter's *M*agno*S*oft™ software makes it easy to compute a value for the wetted surface area.

References

[1]. C. L. Cooper *et al*, “The use of solvent relaxation NMR to study colloidal suspensions”. *Soft Matter*, 9(30): 7211–7228 (2013).
[2]. T. Cosgrove *et al*, “Using Low-field NMR Relaxation to optimise Particulate Dispersions”, *Powder Technology*, 414 118065 (2023)
[3]. C. L. Cooper, “Competitive Adsorption in Complex Aqueous Systems of Polymers and Particles”, Ph.D. Thesis, *University of Bristol* (2013).