microESR

Electron Spin Resonance Spectrometer

User Manual

Version 002

Innovation with Integrity

EPR

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Contents

1 Introduction ... 7
1.1 Policy Statement ... 7
1.2 References ... 7

2 Safety ... 9
2.1 General ... 9
2.2 System Owner's Responsibility ... 9
2.3 Personnel Requirements ... 10
2.3.1 Qualifications ... 10
2.3.2 Unauthorized Persons ... 11
2.4 Personal Protective Equipment ... 11
2.5 Location of the Safety Label ... 11
2.6 Basic Dangers ... 11
2.6.1 Electrical Safety ... 11
2.6.2 Microwave Safety ... 12
2.6.3 Dangers from Magnetic Fields ... 12
2.6.4 Lifting Hazard ... 12
2.7 Danger from Chemical Substances ... 13

3 Transport, Packaging and Storage ... 15
3.1 Symbols on the Packaging ... 15
3.2 Inspection at Delivery ... 16
3.3 Packaging ... 16
3.4 Storage ... 17

4 Design and Function ... 19

5 Installation ... 21
5.1 Unpacking the Device ... 21
5.2 Electrical Connections ... 21
5.3 Powering Up ... 23

6 Operation ... 25
6.1 Start the Software ... 25
6.2 ESR Samples ... 25
6.3 ESR Tubes ... 25
6.4 Loading a Sample ... 26
6.5 Run a Scan ... 27
6.6 Viewing the Spectrum ... 31
6.7 Changing the Default Data Directory ... 32

7 Sample Tubes and Sample Preparation ... 33
7.1 Types of Sample Tubes ... 33
7.1.1 5 mm Quartz ESR Sample Tubes ... 33
7.1.2 Capillaries and 2 mm Sample Tubes ... 34
7.1.3 Rectangular Sample Tubes ... 35
7.1.4 4 mm Quartz Sample Tubes ... 36

Contents

7.2 Liquid Samples ... 36
7.3 Viscous Samples ... 37
7.4 Solid Samples ... 37

8 Description of Settings ... 39
8.1 Spectrometer Tab ... 39
8.1.1 Sweep Settings ... 40
8.1.2 Parameter Arrays ... 45
8.1.3 Steady State Settings ... 52
8.1.4 Magnetic Field ... 53
8.1.5 Power Settings ... 54
8.1.6 Baseline Correction ... 54
8.2 Tuning Control ... 55
8.2.1 Automation Controls ... 56
8.2.2 Saved Settings ... 57
8.2.3 Auto Tune Routine ... 57
8.2.4 Auto Tune Failure ... 60
8.2.5 Tuner Settings ... 60
8.2.5.1 Phase Settings ... 61
8.2.6 Other Options ... 61
8.2.7 Tuner Controls ... 62
8.2.8 RF Phase ... 62
8.2.9 Noise Floor ... 62
8.2.10 Mod Coil Settings ... 63
8.3 Data Plot Tab ... 65
8.4 Settings Tab ... 78
8.4.1 Preset Settings ... 78
8.4.2 Error Recovery ... 79
8.4.3 Use Least Squares ... 79
8.4.4 File Locations ... 79
8.4.5 Sensor Info ... 80
8.4.6 Data Entry Method ... 80
8.5 Sweep Control Buttons ... 84
8.6 System Status ... 84
8.7 Status Window and Output Data ... 86
8.7.1 Sweep Progress ... 86

9 Cavity Dimensions ... 89

10 Optional Accessories ... 91
10.1 ESR at 77K Using the Finger Dewar ... 91
10.2 ESR with Ultra Violet Irradiation ... 91
10.3 microESR VT Control Using the ThermoCube ... 93
10.3.1 Sample Preparation and Handling ... 93
10.3.2 Start-up ... 93
10.3.3 Setting Up the ThermoCube ... 94
10.3.4 Starting Up the ThermoCube ... 96
10.3.5 ThermoCube Operation ... 96
10.3.6 References ... 97

10.4 Directions for Testing the ThermoCube ... 97
10.4.1 Factory Set-up ... 97
10.4.2 ThermoCube Set-up ... 97
10.4.3 Testing the ThermodCube ... 98
10.4.4 ThermoCube End Test Protocol ... 98

11 Maintenance ... 101
11.1 Cleaning ... 101
11.1.1 Cleaning the Outside of the Device Chassis and Units ... 101
11.1.2 Other Cleaning Operations ... 101
11.2 Preventative Maintenance ... 101

12 Troubleshooting ... 103
12.1 Basic ... 103
12.2 Tuning ... 103
12.3 Locking ... 103
12.4 Signal to Noise ... 104
12.5 Output Distorted ... 104

13 Replacement of Parts ... 105
13.1 Returning the Unit for Repair ... 105

14 Dismantling and Disposal ... 107
14.1 Dismantling ... 107
14.2 Disposal Europe ... 107
14.3 Disposal USA and Other Countries ... 108

15 Technical Data ... 109
15.1 Power Supply ... 109
15.2 Instrument Dimensions and Operating Conditions ... 109
15.3 Operating Conditions ... 109
15.4 Rating Plate ... 109

16 Appendix ... 111
16.1 Filtering ... 111
16.2 Display Modes ... 112
16.3 Tuning and Signal to Noise ... 113
16.4 Standard Samples ... 114
16.5 Mod Coil Settings ... 120
16.6 The Configuration Settings File ... 121
16.7 Changing the Configuration File ... 123
16.8 Calibrating the g-factor ... 127
16.9 ESR Settings File ... 127
16.10 Data File Contents ... 128
16.11 File Name Format ... 129

17 Contact ... 131

List of Figures ... 133

List of Tables ... 137

1 Introduction

The aim of the microESR User Manual is to describe the basic processes involved in setting up and running the spectrometer. The microESR comes calibrated and ready to use out of the box. This manual provides directions on tuning, data acquisition, basic processing, saving data, and basic troubleshooting.

This manual does not attempt to cover any theory involved in electron spin/paramagnetic resonance. Below are a few references which cover ESR theory and applications.

Please note that this manual is for the microESR, a separate manual is available for the microESR-XR with Extended Magnet Range.

This manual enables safe and efficient handling of the device.

This manual is an integral part of the device, and must be kept in close proximity to the device where it is permanently accessible to personnel. In addition, instructions concerning labor protection laws, operator regulations tools and supplies must be available and adhered to.

Before starting any work, personnel must read the manual thoroughly and understand its contents. Compliance with all specified safety and operating instructions, as well as local work safety regulations, are vital to ensure safe operation.

The figures shown in this manual are designed to be general and informative and may not represent the specific Bruker model, component or software/firmware version you are working with. Options and accessories may or may not be illustrated in each figure.

1.1 Policy Statement

It is Bruker's policy to improve products as new techniques and components become available. Bruker reserves the right to change specifications at any time.

Every effort has been made to avoid errors in text and figure presentation in this publication. In order to produce useful and appropriate documentation, we welcome your comments on this publication. Field Service Engineers are advised to check regularly with Bruker for updated information.

Bruker is committed to providing customers with inventive, high-quality, environmentally-sound products and services.

1.2 References

- Weil, John A., Bolton, James R., Wertz, John E., *Electron Paramagnetic Resonance: Elementary Theory and Practical Applications*, John Wiley & Sons, Inc, 1994.
- Giamello, Elio, Brustolon, Marina, *Electron Paramagnetic Resonance: A Practitioner's Tool Kit*, John Wiley & Sons, Inc., Hoboken, NJ, 2009.
- Poole, Charles P., *Electron Spin Resonance: A Comprehensive Treatise on Experimental Techniques*, Dover Publications, Inc., Mineola, New York, 1983.
- Eaton, Gareth R., Eaton, Sandra S., Barr, David P., Weber, Ralph T., *Quantitative EPR*, Springer Wien New York, 2010.
- Drago, Russell S., *Physical Methods for Chemists*, Surfside cientific Publishers, Gainsville, FL, 1992.

2 Safety

This section provides an overview of all the main safety aspects involved in ensuring optimal personnel protection, as well as safe and smooth operation.

Non-compliance with the action guidelines and safety instructions contained in this manual may result in serious hazards.

2.1 General

All work on the unit must be performed by Bruker trained personnel. Opening of unit without the supervision of a Bruker representative will void any and all warranty.

2.2 System Owner's Responsibility

System Owner

The term *system owner* refers to the person who operates the device for trade or commercial purposes, or who surrenders the device to a third party for use/application, and who bears the legal product liability for protecting the user, the personnel or third parties during the operation.

System Owner‘s Obligations

The device is used in the industrial sector, universities and research laboratories. The system owner of the device must therefore comply with statutory occupational safety requirements.

In addition to the safety instructions in this manual, the safety, accident prevention and environmental protection regulations governing the operating area of the device must be observed.

In this regard, the following requirements should be particularly observed:

- The system owner must obtain information about the applicable occupational safety regulations, and - in the context of a risk assessment - must determine any additional dangers resulting from the specific working conditions at the usage location of the device. The system owner must then implement this information in a set of operating instructions governing operation of the device.
- During the complete operating time of the device, the system owner must assess whether the operating instructions issued comply with the current status of regulations, and must update the operating instructions if necessary.
- The system owner must clearly lay down and specify responsibilities with respect to installation, operation, troubleshooting, maintenance and cleaning.
- The system owner must ensure that all personnel dealing with the device have read and understood this manual. In addition, the system owner must provide personnel with training and hazards information at regular intervals.
- The system owner must provide the personnel with the necessary protective equipment.
- The system owner must warrant that the device is operated by trained and authorized personnel as well as all other work, such as transportation, mounting, start-up, the installation, maintenance, cleaning, service, repair and shutdown, that is carried out on the device.
- All personnel who work with, or in the close proximity of the device, need to be informed of all safety issues and emergency procedures as outlined in this user manual.

- The system owner must document the information about all safety issues and emergency procedures in a laboratory SOP (Standard Operating Procedure). Routine briefings and briefings for new personnel must take place.
- The system owner must ensure that new personnel are supervised by experienced personnel. It is highly recommended to implement a company training program for new personnel on all aspects of product safety and operation.
- The system owner must ensure that personnel are regularly informed of the potential hazards within the laboratory. This is all personnel that work in the area, but in particular laboratory personnel and external personnel such as cleaning and service personnel.
- The system owner is responsible for taking measures to avoid inherent risks in the handling of dangerous substances, preventing industrial disease, and providing medical first aid in emergencies.
- The system owner is responsible for providing facilities according to the local regulations for the prevention of industrial accidents and generally accepted safety regulations according to the rules of occupational medicine.
- All substances needed for operating and cleaning the device samples, solvents, cleaning agents, gases, etc. have to be handled with care and disposed of appropriately. All hints and warnings on storage containers must be read and adhered to.
- The system owner must ensure that the work area is sufficiently illuminated to avoid reading errors and faulty operation.
- The system owner must ensure that the laboratory is equipped with an oxygen warning device, in case the device is operated with nitrogen.

Furthermore, the system owner is responsible for ensuring that the device is always in a technically faultless condition. Therefore, the following applies:

- The system owner must ensure that the maintenance intervals described in this manual are observed.
- The system owner must ensure that all (electrical, mechanical, etc.) safety devices are regularly checked to ensure full safety functionality and completeness.

2.3 Personnel Requirements

Retrofitting, repairs, adjustments or dismantling of the device must only be carried out by Bruker Service or personnel authorized by Bruker. Damage due to servicing that is not authorized by Bruker is not covered by your warranty.

In special cases, for example to operate the spectrometer in flow mode, or to retrieve a sample that has dropped inside, Bruker may authorize removal of the base plate. However, to avoid violation of the warranty, it is necessary to contact Bruker beforehand for authorization.

2.3.1 Qualifications

Make sure that only authorized personally are within the operating area of the device. The authorized personnel involved in device operation must have access to this manual at all times. Keep this manual near the device.

2.3.2 Unauthorized Persons

⚠ WARNING

Risk to life for unauthorized personnel due to hazards in the danger and working zone!

Unauthorized personnel who do not meet the requirements described in this manual will not be familiar with the dangers in the working zone. Therefore, unauthorized persons face the risk of serious injury or death.

- Unauthorized persons must be kept away from the danger and working zone.
- If in doubt, address the persons in question and ask them to leave the danger and working zone.
- Cease work while unauthorized persons are in the danger and working zone.

2.4 Personal Protective Equipment

Personal protective equipment is used to protect the personnel from dangers which could affect their safety or health while working.

Personnel must wear personal protective equipment while carrying out the different operations at and with the device.

This equipment will be defined by the head of the laboratory. Always comply with the instructions governing personal protective equipment posted in the work area.

2.5 Location of the Safety Label

The laboratory supervisor is responsible for ensuring that all the warning labels are maintained in their proper place any time that the device is used.

2.6 Basic Dangers

The following section specifies residual risks which may result from using the device and have been established by means of a risk assessment. In order to minimize health hazards and avoid dangerous situations, follow the safety instructions specified here as well as in the following chapters of this manual.

2.6.1 Electrical Safety

There is no danger of electrical shock during routine operation of the device, however precautions must be followed to prevent injuries or property damage during service.

All work on the unit must be performed by Bruker trained personnel. Opening the unit without the supervision of a Bruker representative will void any and all warranty. Safety precautions applicable to any electronic device connected to AC power must be observed. Damaged wires or cables must be replaced immediately.

⚠ WARNING

Hazard from electrical shock

A life threatening shock may result when the housing is open during operation.

- Only qualified personnel should open the housing.
- Disconnect the device from the electrical power supply before opening the device. Use a voltmeter to verify that the device is not under power!
- Be sure that the power supply cannot be reconnected without notice.

2.6.2 Microwave Safety

The device is a microwave producing device. Microwaves are safe as long as they are contained in metal structures.

2.6.3 Dangers from Magnetic Fields

People with pacemakers or medical implants are not allowed in the vicinity of the magnet box. The 5 Gauss line is inside the box.

⚠ WARNING

Risk to life due to strong magnetic fields

Strong magnetic fields may cause serious injuries or death and significant damage to property.

- People with heart pacemakers must be kept away from the device. The functionality of the heart pacemaker could be compromised.
- Persons with metal implants must be kept away from the device. Implants may heat up or be subject to magnetic attraction.
- Do not use ferromagnetic tools or items within the vicinity of the device.

2.6.4 Lifting Hazard

⚠ CAUTION

Risk of injury or damage from heavy objects

The unit packing crate is quite heavy:

- To prevent injury to your back, be very careful when lifting the box in or out of the packaging crate.
- Take care to avoid trapping fingers under the unit box as it is placed.
- Be sure that any surface on which the unit is temporarily or permanently placed is capable of supporting its weight and will not allow the unit to tip or slide off.

2.7 Danger from Chemical Substances

⚠ WARNING

Danger of injury from glassware or ceramics breakage!

Broken glassware or ceramics may cause minor injuries or material damage, but may also result in a life threatening situation if hazardous substances are used.

- If glassware or ceramics breaks, refer to the corresponding precautions and cleaning/ disinfection instructions.
- Wear protective equipment.
- Perform all tasks with the glassware or ceramics carefully.
- Before carrying out any maintenance work, remove the samples and use dummy samples if necessary.
- Strictly observe the correct sample adjustment, e.g. the maximum sample height.
- Always transport the glassware or ceramics with the cover, if applicable. Never turn the glassware or ceramics upside down or on it's side.
- **The laboratory supervisor is responsible for:**
  - ⇨ Establishing and enforcing standard sample handling and cleaning procedures.
  - ⇨ Establishing and enforcing the use of protective clothing and equipment.
  - ⇨ Training laboratory personnel.
  - ⇨ Preparing an emergency plan.

⚠ WARNING

Danger of injury from vapor formation!

During the work process, vapors may form which cause serious injury if inhaled.

- If there is any chance hazardous vapors could form, the device should be operated in a fume hood.

⚠ WARNING

Exposure and other health hazards to maintenance personnel.

If contaminated, the device must be cleaned before any maintenance work can be performed.

- Register all substances with which the device has come into contact.
- Sign a certification form verifying that the device has been properly cleaned if contaminated to protect maintenance personnel.
- Obviously contaminated, insufficiently cleaned units, as well as units without a signed cleaning certification will not be repaired and will be returned to the sender.

NOTICE

Material damage hazard from material contact with solvents.

Material damage may result when the device comes in contact with solvents.

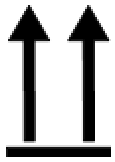
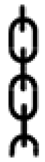
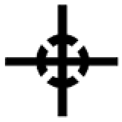
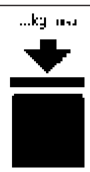
- Do not fill sample tubes while inserted in the spectrometer.
- If surface damage should occur, contact Bruker for repair of damaged parts.

3 Transport, Packaging and Storage

Retrofitting, repairs, adjustments or dismantling of the device must only be carried out by Bruker Service or personnel authorized by Bruker. Damage due to servicing that is not authorized by Bruker is not covered by your warranty.

3.1 Symbols on the Packaging

The following symbols are affixed to the packaging material. Always observe the symbols during transport and handling.

Top		The arrow tips on the sign mark the top of the package. They must always point upwards; otherwise the content may be damaged.
Fragile		Marks packages with fragile or sensitive contents. Handle the package with care; do not allow the package to fall and do not allow it to be impacted.
Protect Against Moisture		Protect packages against moisture and keep dry.
Attach Here		Lifting gear (lifting chain, lifting strap) must only be attached to points bearing this symbol.
Center of Gravity		Marks the center of gravity of packages. Note the location of the center of gravity when lifting and transporting.
Weight, Attached Load		Indicates the weight of packages. Handle the marked package in accordance with its weight.
Permitted Stacking Load		Indicates packages which are partially stackable. Do not exceed the maximum load-bearing capacity specified on the symbol in order to avoid damaging or destroying the content.

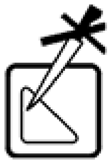
Do not Damage Air-tight Packaging		The packaging is air-tight. Damage to the barrier layer may render the contents unusable. Do not pierce. Do not use sharp objects to open.
Component Sensitive to Electrostatic Discharge		The packaging contains components which are sensitive to an electrostatic discharge. Only allow packaging to be opened by trained personnel. Establish potential equalization before opening.
Protect from Heat		Protect packages against heat and direct sunlight.
Protect from Radioactive Sources		Protect packages against radioactive sources.

Table 3.1: Symbols on the Packaging

3.2 Inspection at Delivery

Upon receipt, immediately inspect the delivery for completeness and transport damage.

Proceed as follows in the event of externally apparent transport damage:

- Do not accept the delivery, or only accept it subject to reservation.
- Note the extent of the damage on the transport documentation or the shipper's delivery note.
- Initiate complaint procedures.

Issue a complaint in respect to each defect immediately following detection. Damage compensation claims can only be asserted within the applicable complaint deadlines.

3.3 Packaging

About Packaging

The individual packages are packaged in accordance with anticipated transport conditions. Only environmentally friendly materials have been used in the packaging.

The packaging is intended to protect the individual components from transport damage, corrosion and other damage prior to assembly. Therefore do not destroy the packaging and only remove it shortly before assembly.

Handling Packaging Materials

Keep the original container and packing assembly, at least as long the warranty is valid, in case the unit has to be returned to the factory. When the packaging material is no longer needed dispose of in accordance with the relevant applicable legal requirements and local regulations.

3.4 Storage

Storage of the Packages

Store the packages under the following conditions:

- Do not store outdoors.
- Store in dry and dust-free conditions.
- Do not expose to aggressive media.
- Protect against direct sunlight.
- Avoid mechanical shocks.
- Storage temperature: 15 to 35 °C.
- Relative humidity: max. 60%.
- If stored for longer than 3 months, regularly check the general condition of all parts and the packaging. If necessary, top-up or replace preservatives.

Under certain circumstances, storage instructions may be affixed to packages which expand the requirements specified here. Comply with these accordingly.

4 Design and Function

The microESR uses a small, 0.348 Tesla, rare-earth magnet. The magnet assembly contains a low power electromagnet coil used to vary the field. The microESR is a strictly CW instrument with a sweep range of over 500 Gauss. The field is centered near the free electron spin g-factor. The spectrometer uses a linear voltage controlled oscillator as a microwave source, and can generate between 0.5 and 70 mW RF power at 9.7 GHz. The microESR uses quadrature lock-in detection, and the lock-in amplifier is internal to the system.

Figure 4.1: The Benchtop microESR

The resonator is a cylindrical dielectric TM_{011} cavity with a maximum volume of ~ 400 µL (5 mm diameter by 20 mm length). The field is homogeneous across the resonator within 0.1 Gauss. The entire microESR has a small footprint (30.5 x 30.5 x 30.5 cm^3) and a mass of only 10 kg, so can be easily moved. The microESR requires no special installation and no special maintenance making it accessible to any undergraduate laboratory.

5 Installation

No special installation is required, but the three standard samples that ship with the system should be run as soon as the spectrometer has been received and unpacked. The data should be compared to the data shipped with the spectrometer.

This rest of this chapter describes how to prepare the benchtop microESR spectrometer for operation.

5.1 Unpacking the Device

The device is shipped in a Pelican case to protect it during shipping, but occasionally damage does occur. The device should be inspected for damage upon receipt (broken screen, cracked case).

People with pacemakers should not pick up the device. The 5 Gauss line is inside the box.

The device is only 10 kg, but be careful to use proper technique when lifting it out of the Pelican case as to not injure your back. The device can be placed on a table, lab bench, or even in a hood or glove box. Do not place where heating or air conditioning blows on the spectrometer.

Keep ferromagnetic objects away from the device.

5.2 Electrical Connections

The microESR unit contains a built-in computer system. The box has the following electrical interfaces:

- Touchscreen Monitor
- Master Power Switch
- Power Cable Connector
- Ethernet Cable Connector
- 2 x USB Connectors
- DVI Connector
- AUX

The figure below shows the back panel ports of the spectrometer. From left to right: DVI connector, Ethernet cable connector, auxiliary (empty) port, power cable connector, and master power switch.

Figure 5.1: Back Panel Ports

The 2 USB ports are in the front lower right:

Figure 5.2: Front USB Ports

5.3 Powering Up

To activate the unit, plug the supplied power supply into a standard wall socket and turn on the power switch. The computer will boot into Windows.

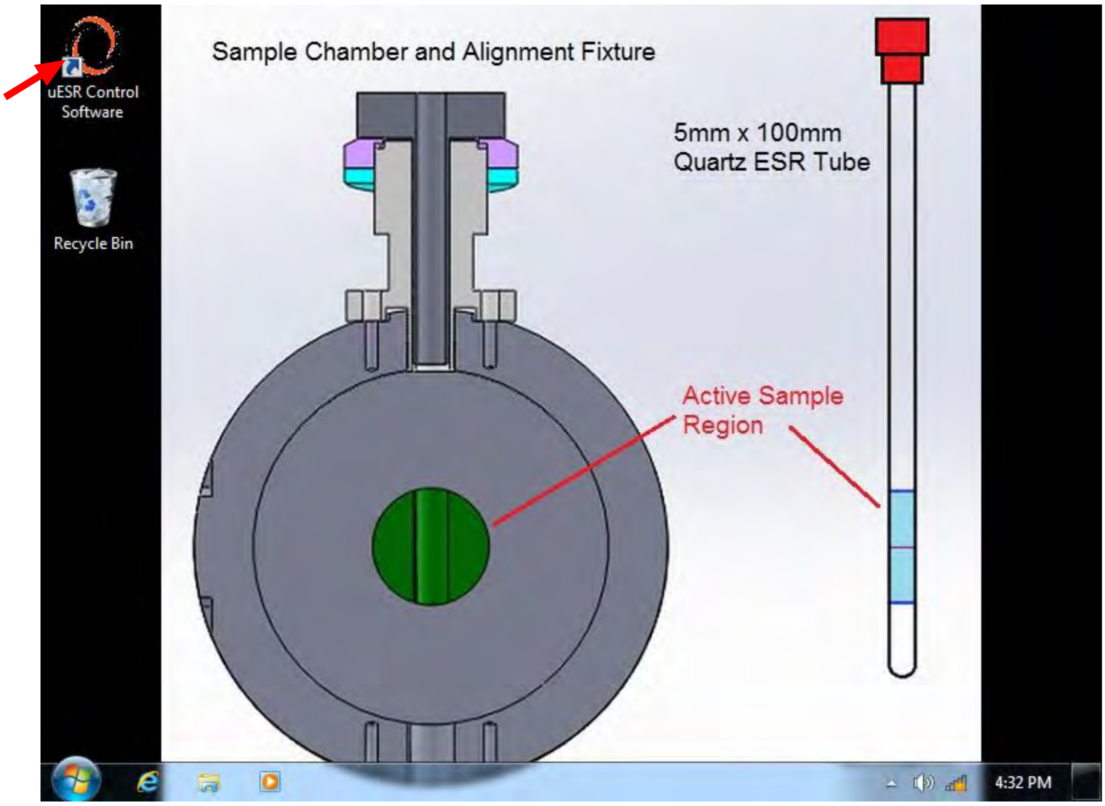


Figure 5.3: Windows Desktop and uESR Software Icon [microESR]

Once Windows has booted, double-click on the uESR Control Software icon on the desktop to start the program. If the icon does not appear on the desktop, the software can be started through the Start menu. The computer can be interfaced solely through the touchscreen; additionally, a mouse and/or keyboard can be plugged into the USB ports. The software is in *C:\ProgramFiles\uESR Control Software*.

6 Operation

6.1 Start the Software

To start the software, double-click the **uESR Control Software** icon on the desktop, or select it from the **Start** menu.

The software will load and initialize. When the initialization is complete, the status window will say ‘Ready’, and the user interface will become available.

6.2 ESR Samples

In order to see an ESR signal, the sample must contain some molecules with at least one unpaired electron.

Radicals are common ESR samples, especially long-lived radicals such as TEMPO and trityl (there are many other examples).

Transition metals with at least one unpaired electron; although some of these have T1s that are so short that they cannot be observed at room temperature.

Radicals, including very short lived radicals, can also be observed indirectly by using a spin trap.

Samples in polar solvents such as water, ethanol, and acetonitrile will need to be in small capillary tubes, or the system will not lock. The spectrometer must be locked to see a signal.

6.3 ESR Tubes

For best results, use quartz tubes. For quantitative work requires precision quartz tubes.

Varying sizes of capillary tubes can be used inside larger quartz ESR tubes. Quartz capillary tubes can be purchased, but may not be necessary depending on the application.

Non-paramagnetic clay can be purchased to seal capillary tubes.

Figure 6.1: Various sized ESR tubes and Sample Holders

6.4 Loading a Sample

NOTE: Ensure that the sample tube is clean before inserting into the chamber! A dirty tube can contaminate the resonator cavity. Clean the outside of the tubes with a Kimwipe before use. Load the sample in a tube or capillary. For best results use quartz tubes.

- Refer to the figure in *Cavity Dimensions [▸ 89]* to make certain your sample is centered in the resonator. O-rings can be used to position the sample.
- Carefully insert the tube into the aperture on top of the device:

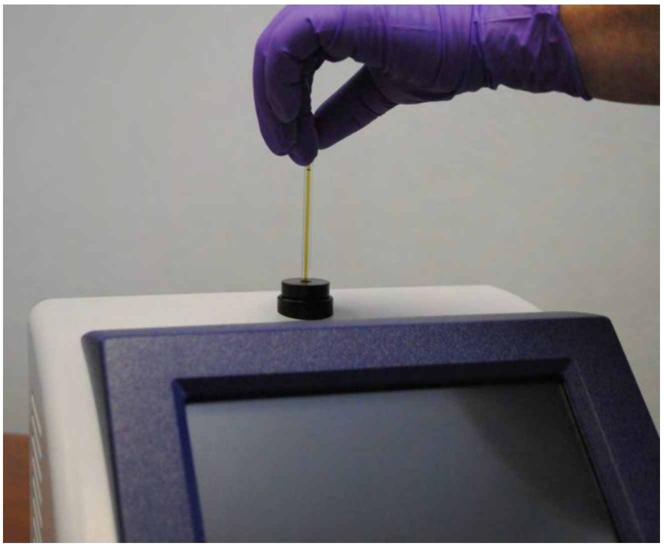


Figure 6.2: Loading a Sample

6.5 Run a Scan

- Select the **Spectrometer** tab in the uESR Control Software. Note also the **Tuning**, **Data Plot**, and **Settings** tabs which are referred to later in the text.

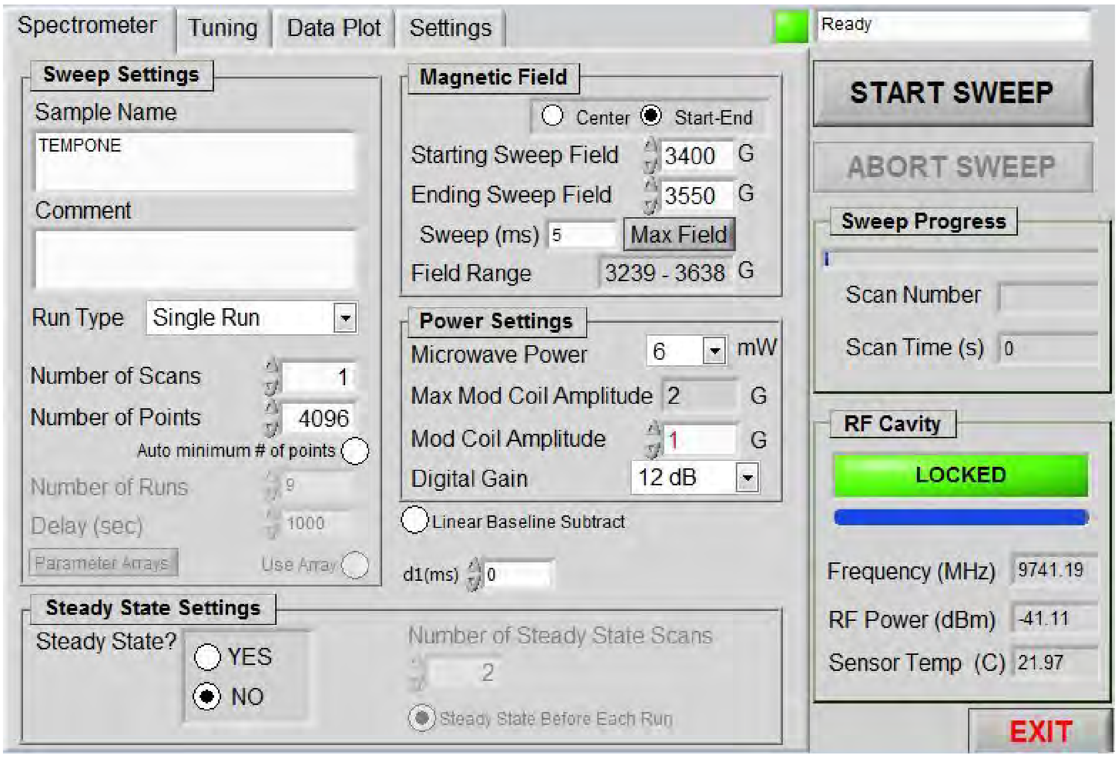


Figure 6.3: Spectrometer Tab in the uESR Control Software

Refer to the above figure for the following:

- Input the **Sample Name**. You may also add a **Comment**.
- In the **Run Type** pull-down menu, select **Single Run**:

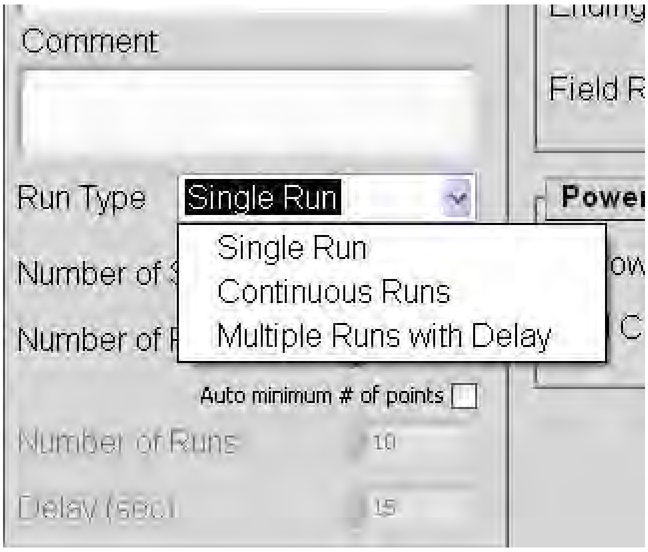


Figure 6.4: Run Type Option

- Enter **Number of Scans** =1, **Number of Points** =2048. Check that the **Microwave Power** is 15 mW, and the coil amplitude is 100%.
- Check that the **Starting Sweep Field** is set to the minimum and the **Ending Sweep Field** is set to the maximum The **Field Range** box under the spectrometer tab displays the minimum to maximum range.

A detailed description of these parameters is given in the section *Spectrometer Tab [▸ 39]* of this manual. Parameters are meant to be changed depending on the sample and conditions. The above parameters will work for any reasonably concentrated sample, although, they may not be optimum.

The data file will be saved in the designated directory with the extension ESR. The data are saved as SampleName_date_time.ESR. Files will not be overwritten.

Once the sample has been placed in the cavity, there are two settings that will change the coupling: RF phase and tuner position. Both will be adjusted by the auto tune routine to obtain an optimal signal.

- Select the **Tuning Control** tab (see *Figure 6.3 [▸ 27]*).
- Press the **AUTO TUNE** button. This will bring up a separate pop-up window:

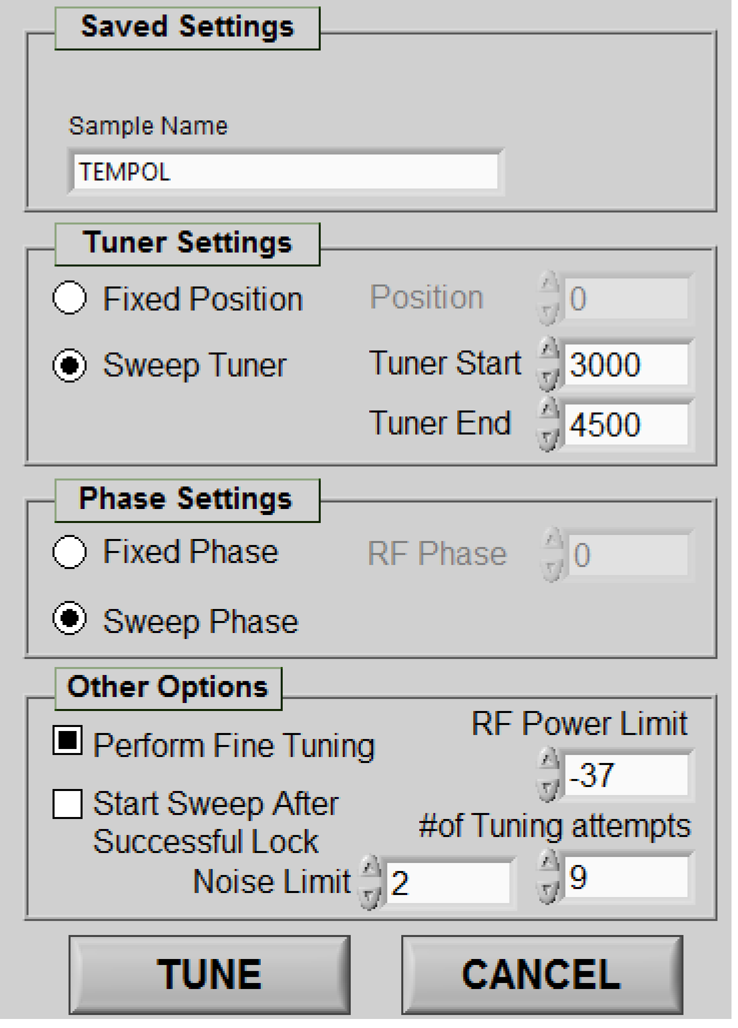


Figure 6.5: Auto Tune Window

You can change the **Sample Name** here as well as from the spectrometer tab. By default, Sweep Tuner, Sweep Phase, and Perform Fine Tuning will be selected.

Select **Start Sweep After Successful Lock** to have the spectrometer start the experiment as soon as the tuning has finished.

RF Power Limit

The RF Power Limit is the maximum reflected power that the spectrometer will be allowed to tune with. This number is set in the factory to a number that is 2 dBm higher than the system's best tune on an aqueous sample. The values are given in the paper work sent with the spectrometer.

Noise Limit

The Noise Limit is the greatest noise limit allowed. In most cases, a number between one and two is best. Note that if the gain is turned up, this noise limit will most likely need to be increased.

# of Tuning Attempts

The number of times the spectrometer tries to tune to the specifications before giving up. Nine is the default number; however, the spectrometer usually tunes on the first or second try unless there is something wrong.

Press the **TUNE** button to start the tuning.

The spectrometer will display the tune progress as shown in the following figures.

The tuning starts by Initializing the Tuner, Stepping the Tuner, and then Moving Tuner to Critical Coupling. The blue bar displays the progress.

Figure 6.6: Initializing the Tuner, Stepping the Tuner, and Moving Tuner to Critical Coupling.

The next step is Locking the AFC to the Resonator, Fine Tuning, and Sweeping for Optimal Phase. The blue bar continues to display the tune progress.

Figure 6.7: Locking the AFC to the Resonator, Fine Tuning, and Sweeping for Optimal Phase.

The spectrometer is locked, and starts sweeping the field.

Figure 6.8: Spectrometer Locked, Sweeping the Field has Started.

If sample is too lossy (an aqueous sample, or any polar solvent) the cavity will be unable to obtain lock. In this case, reload sample in a smaller tube or flat cell cavity. Aqueous solutions require < 1.3 mm ID capillary tubes to lock (section *Liquid Samples [▶ 36]*).

6.6 Viewing the Spectrum

The results for a sweep will be displayed in the Data Plot tab.

The section *Data Plot Tab [▶ 65]* contains detailed explanations of everything in the Data Plot Tab.

6.7 Changing the Default Data Directory

- Select the **Settings** tab (section *Settings Tab [▸ 78]*).
- The **Save Directory** indicates where the recorded data files will be saved. If you want to change this, type in or browse to a new location.

Figure 6.9: Saved File Location

- Type the path directly into the Save Directory window. In the above example, all data will be saved in a directory called My_Samples in C:\Data. If the directory My_Samples does not already exist, it will be created.
- Clicking on the directory icon to the right will bring up a window that will allow you to browse for a location to save your data.

IMPORTANT

- Be careful when loading and unloading the sample tubes. If they break inside the unit, it can be difficult to extract them.
- The spectrometer contains an internal cooling device designed to hold the sensor at a fixed temperature, ensuring uniformity of results. Running the sensor in a warm environment or restricting the output airflow can impede the cooling unit and cause the device to heat up and malfunction.
- Also keep unit out of drafts, and avoid having cooling or heating air from an HVAC blowing on the instrument.

7 Sample Tubes and Sample Preparation

Materials that can be prepared for electron magnetic resonance come in a myriad of shapes, sizes, and properties. This section covers some of the most basic procedures for the typical kinds of samples you might encounter.

7.1 Types of Sample Tubes

The ESR tubes need to be at least 100 mm long; however, capillaries which are inserted into another tube can still be any length.

The figure in the chapter *Cavity Dimensions [▸ 89]* shows the sample alignment and resonator cavity in detail. Note this diagram is not to scale; however, the laminated copy included with the spectrometer is to scale.

7.1.1 5 mm Quartz ESR Sample Tubes

5 mm quartz ESR sample tubes, such as the one pictured below, are probably the most commonly used. These tubes are appropriate for most samples, the exception being samples in polar solvents such as water.

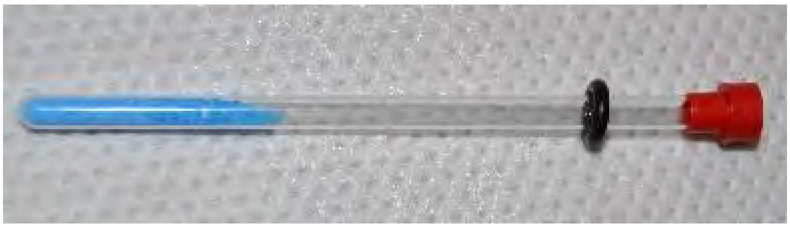


Figure 7.1: Standard 5 mm Quartz ESR Sample Tube

O-rings are used to adjust the sample height such that the resonator cavity is full.

If the sample is shorter than 20 mm (the length of the resonator cavity), it is important to center the sample in the resonator cavity. Adjust the O-ring on the tube such that the sample is centered in the resonator:

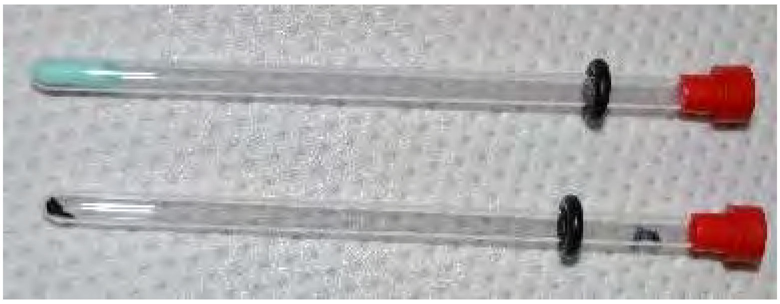


Figure 7.2: Adjustment O-Ring on the Tube

Sample tubes can be cleaned, rinsed with acetone, left upside-down to dry, and reused many times.

Precision quartz ESR tubes come with varying wall thickness, and cost more than the standard 5 mm quartz tubes. The precision tubes may give better results for quantitative work.

7.1.2 Capillaries and 2 mm Sample Tubes

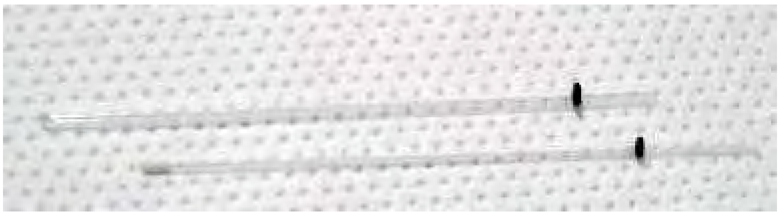


Figure 7.3: 1.7 mm Capillaries

1.7 mm capillaries are necessary for aqueous samples. The spectrometer will not be able to lock on lossy samples if the volume is too large.

Open ended 1.7 mm capillaries can be filled by capillary action, and one or both ends sealed with non-paramagnetic clay.

1.7 mm capillaries that are sealed on one end can be filled with a pipetor using gel tips. The open end can be sealed with clay or a blow torch if necessary. It is best to use clay with flammable solvents.

The 1.7 mm tubes can be used for solids as well. Samples with large signals work well with smaller tubes.

Most capillaries are borosilicate. Placing the smaller tube inside the 5 mm quartz tube will give best results. A 1 x 1.5 mm O-ring will fit on both tubes, and inside the 5 mm tube. The O-ring keeps the smaller tube aligned inside the 5 mm Tube:

Figure 7.4: O-Ring Used to Keep the Smaller Tube Aligned Inside the 5 mm Tube

2 mm quartz or suprasil tubes are also available. These tubes do not have to be placed in a 5 mm quartz tube.

Special sample holders for 2 mm sample tubes are available upon request.

Capillaries (available from Bruker) are generally not reused.

7.1.3 Rectangular Sample Tubes

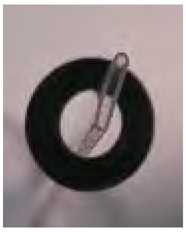


Figure 7.5: Rectangular Tube

Rectangular sample tubes (available from Bruker):

- Are specifically designed to use with aqueous solutions
- Are more than doubles the signal to noise over 1.7 mm capillaries
- Use a pipetor with gel tips to fill the tube.
- Use special sample holders to ensure the sample is perfectly aligned.

Figure 7.6: Tube Alignment Holder

The rectangular tube holder (available from Bruker) is held tightly in place by the ring of the Tube Alignment Holder.

The bottom of the rectangular tube holder has two pins:

Figure 7.7: Pins on the Bottom of the Rectangular Tube Holder

These pins fit into the groove of the alignment socket on top of the spectrometer:

Figure 7.8: Groove of the Alignment Socket

7.1.4 4 mm Quartz Sample Tubes

Figure 7.9: 4 mm Quartz ESR Sample Tube

4 mm quartz sample tubes can be used with both solids and liquids.

Some solvents, such as methylene chloride are too polar to use with 5 mm tubes, but the spectrometer will not have problem locking with 4 mm tubes.

Special 4 mm sample holders are available on request.

7.2 Liquid Samples

Aqueous and other polar samples need to be placed in either 1.7 mm capillaries or rectangular tubes.

Less polar solvents can be placed in 4 mm tubes.

Non-polar solvents can be used with 5 mm tubes.

Larger volume tubes can be filled using a Pasteur pipette. A pipetor fitted with a gel tip is ideal for filling smaller volume tubes. Open-ended capillaries will fill by capillary action.

The larger the diameter tube, the better the signal to noise. Unless the sample being analyzed is concentrated, use the largest diameter tube that the solvent polarity will allow.

If too large a volume of polar solvent is placed in the resonator cavity, the system will be unable to lock. There will be no harm done to the spectrometer. Simply remove the sample, and put in a smaller diameter tube.

7.3 Viscous Samples

Viscous samples, like other liquid samples, should be placed in the largest diameter tube sample polarity will allow.

Using blunt needles attached to a luer-lock syringe greatly aids in loading viscous solutions such as oils.

Blunt needles are available from Bruker in both 12 and 20 gauge sizes.

7.4 Solid Samples

For coarse solids or dried materials, such as tea leaves:

- Take a piece of Glassine weighing paper, and fold it in half.
- Place the sample on the paper, and carefully funnel into 5 mm tube.

For powders:

- Take a piece of Glassine weighing paper, and fold it in half.
- Place the sample on the center of the paper. With the tube parallel to the paper, use a spatula to push some solid into the tube.
- Gently tap the bottom of the tube on a hard surface, such as a lab bench, until the solid moves to the bottom of the tube.
- Repeat until the tube has the desired amount of solid. Two centimeters will fill the resonator.

8 Description of Settings

8.1 Spectrometer Tab

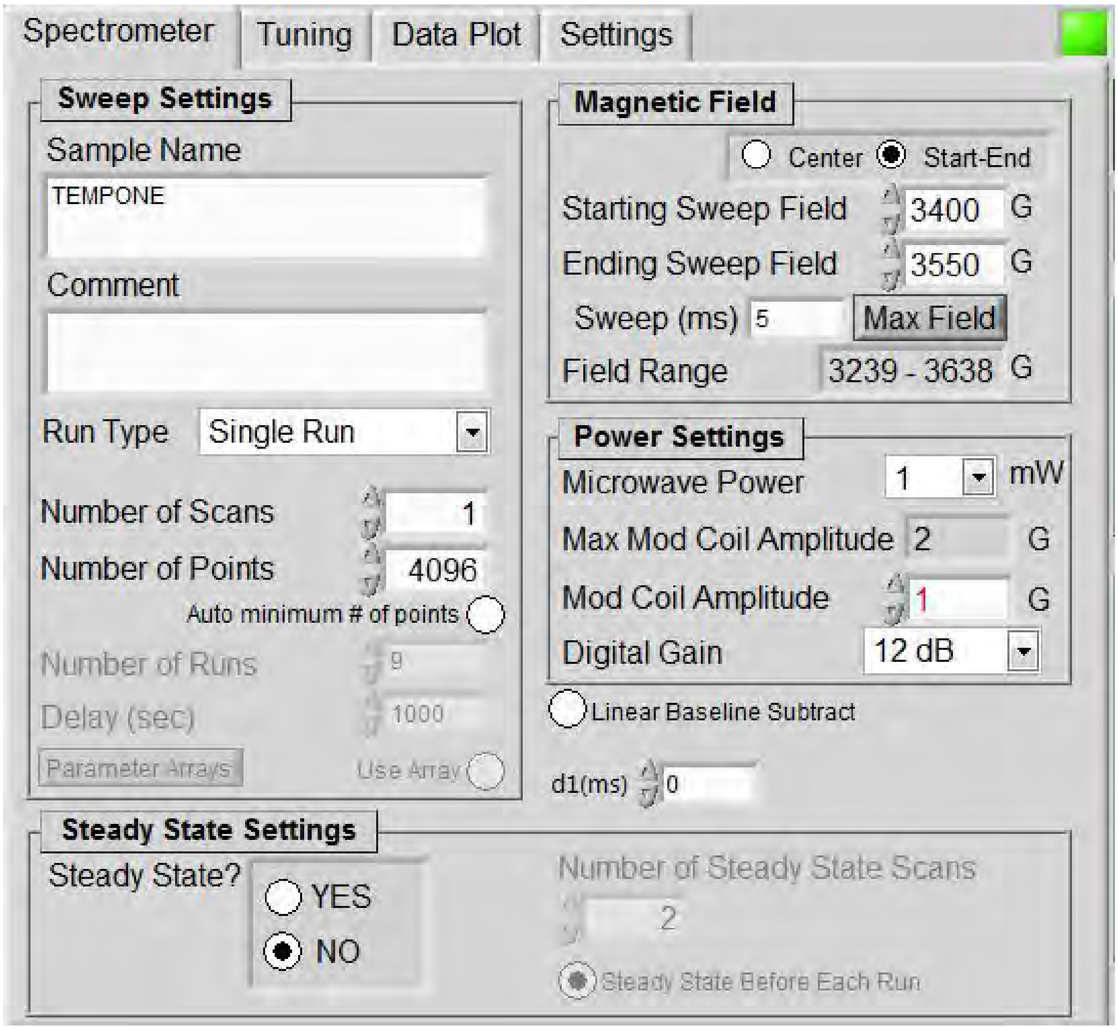


Figure 8.1: Spectrometer Tab

8.1.1 Sweep Settings

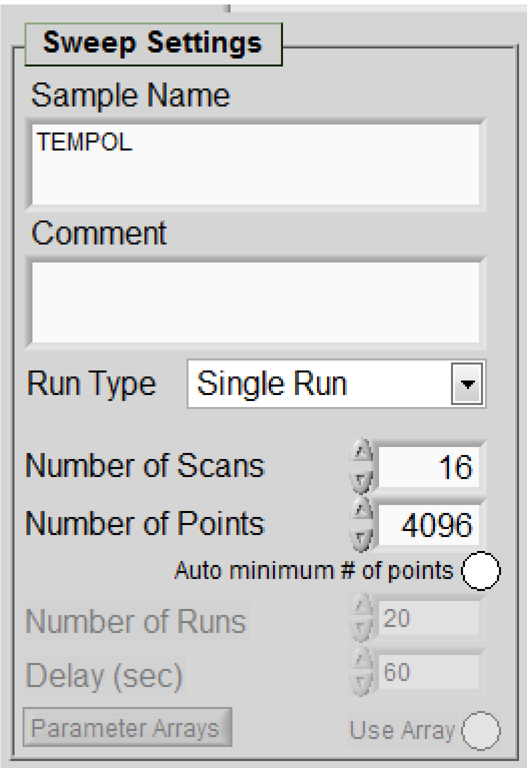


Figure 8.2: Sweep Settings – Single Run Selected

Sample Name

Name of the sample. This will be in the saved file name. All files are saved as Sample_Name_Date_Time.

Comment

Optional comment that will be saved in the data file.

Run Type

Select among Single, Continuous, or Multiple Run options.

Single Run

Acquires one spectrum with the specified number of scans and points.

Note that when Single Run is Selected, the Number of Runs and Delay inputs are grayed out.

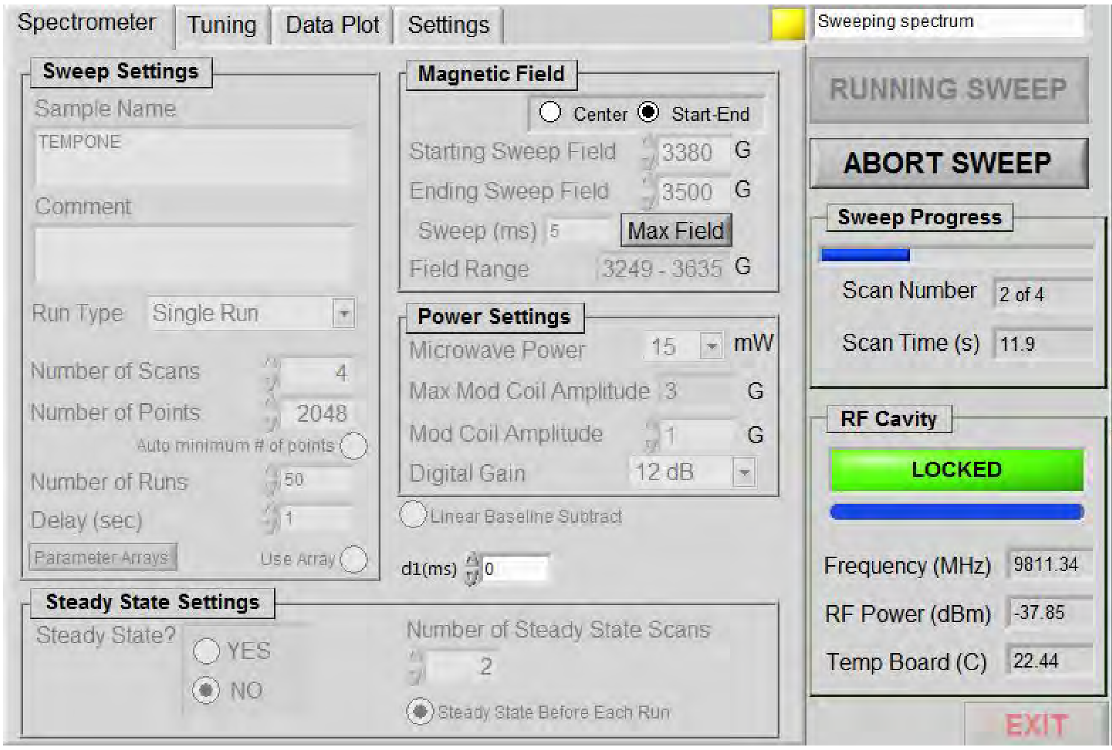


Figure 8.3: Single Run Sweep in Progress

Once the spectrometer has started sweeping the spectrum, most of the window will be grayed out. The **Sweep Progress** menu shows which scan is currently being run, and the blue slider displays the progress of the scan.

Continuous Run

Continually acquires spectra with the specified number of scans and points until system is stopped by clicking **ABORT SWEEP**. Each spectrum that is acquired will be saved in the standard format of sample_name_date_time.

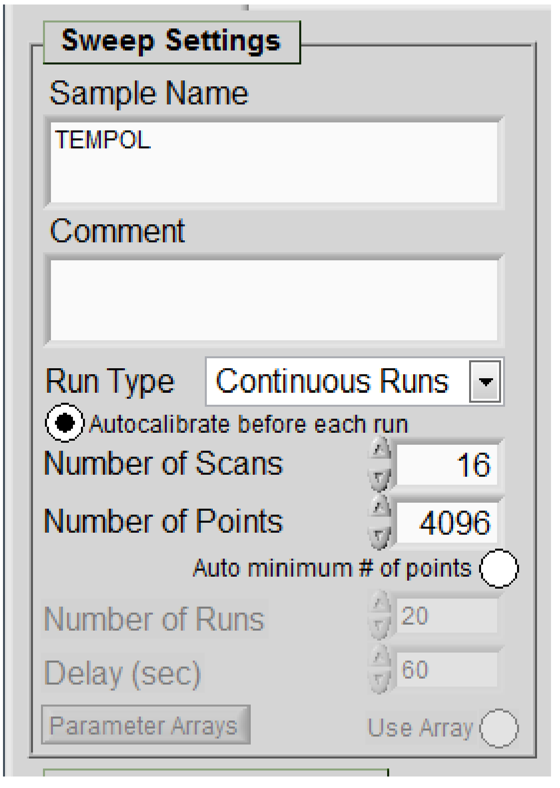


Figure 8.4: Continuous Runs Selected

The sweep progress window will be identical to the single scan window.

Autocalibrate before each run

When this box is selected, the spectrometer will automatically retune the sample before each run. When Autocalibrate before each run is selected, start the acquisition by clicking the **START SWEEP** button. The spectrometer will automatically tune before the first run.

Multiple Runs

When this option is selected, the **Number of Runs** and **Delay** fields become active. This allows the user to run multiple experiments with a specified delay between experiments. This is very useful for kinetics experiments. The specified number of scans and points will be used.

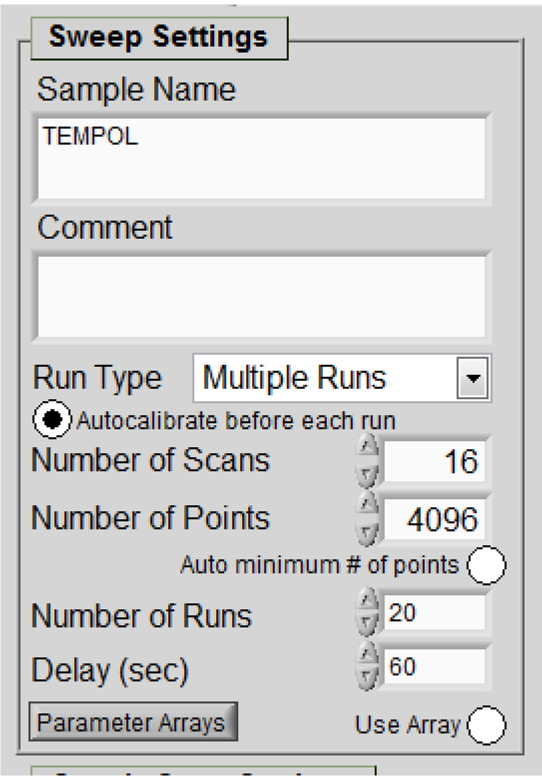


Figure 8.5: Multiple Runs Selected

For example, if number of scans=16, number of points=4096, number of runs=20, and delay=60, the spectrometer will acquire and save 20 spectra each acquired with a 60 second delay between them. Each spectrum will have 16 scans and 4096 points.

Autocalibrate before each run

When this box is selected, the spectrometer will automatically retune the sample before each run. When Autocalibrate before each run is selected, start the acquisition by clicking the **START SWEEP** button. The spectrometer will automatically tune before the first run.

If the number of runs is set to zero, the system will behave in the same way as if Continuous Runs had been selected.

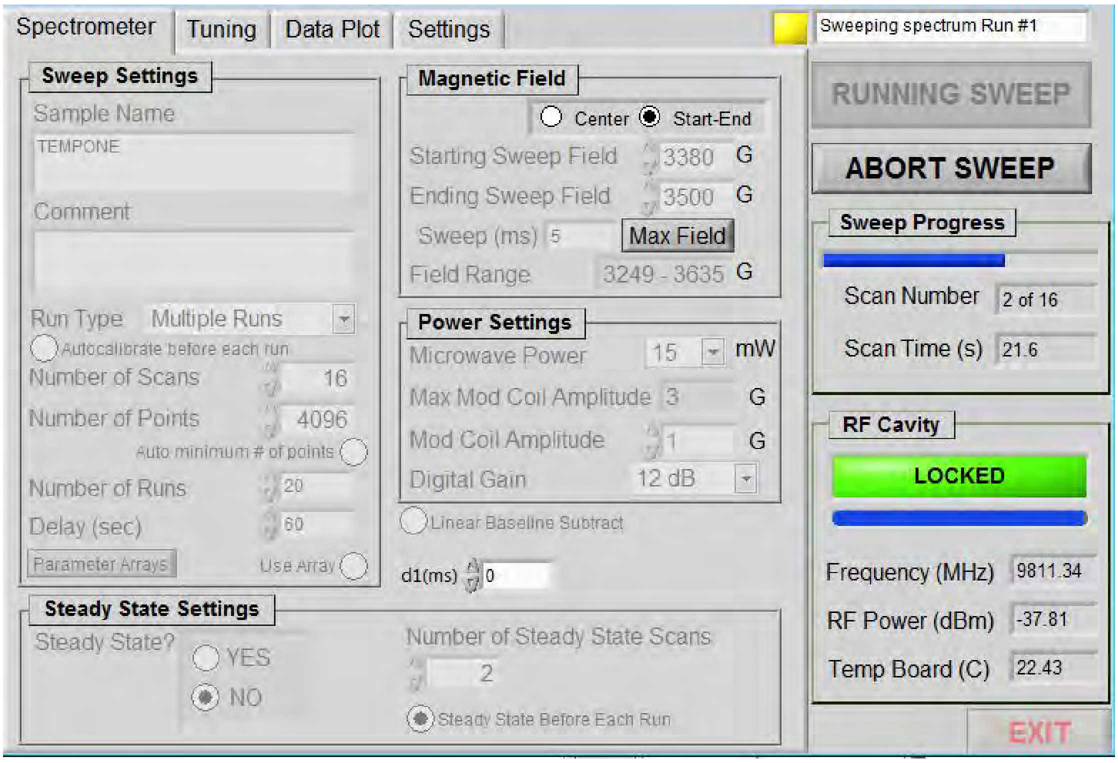


Figure 8.6: Multiple Run Sweep in Progress

Note that as in the single and continuous runs, the scan number is displayed, but also in addition to sweeping spectrum, the run number is also displayed.

Number of Scans

Total number of scans to be acquired.

Number of Points

Number of points sampled per scan.

Auto Minimum # of points

Automatically selects the minimum number of points that can be used to acquire a spectrum with the selected field sweep. The fewer points that are acquired, the less time the sweep takes; however, resolution will be better when more points are acquired. Uncheck the box to manually specify the number of points to acquire.

The minimum number of points that can be acquired is 8 points/Gauss. The maximum number of points that can be acquired in a scan is 8192; however, the maximum number of points per Gauss is 131. This is limited by the 16 bit ADC. The default acquisition time is about 5 ms per point.

8.1.2 Parameter Arrays

There are times, especially when running new sample types or new experiments, when the user wants to run the experiment under different conditions to pick the optimum parameters. The parameter array feature allows the automated running of an experiment under different conditions.

- Make sure that Run Type is set to Multiple Runs
- Click on the **Parameter Array button** (*Figure 8.5 [▸ 43]*).

The following GUI appears, and allows the user to array some parameters. The user may array one or more of the above parameters at a time.

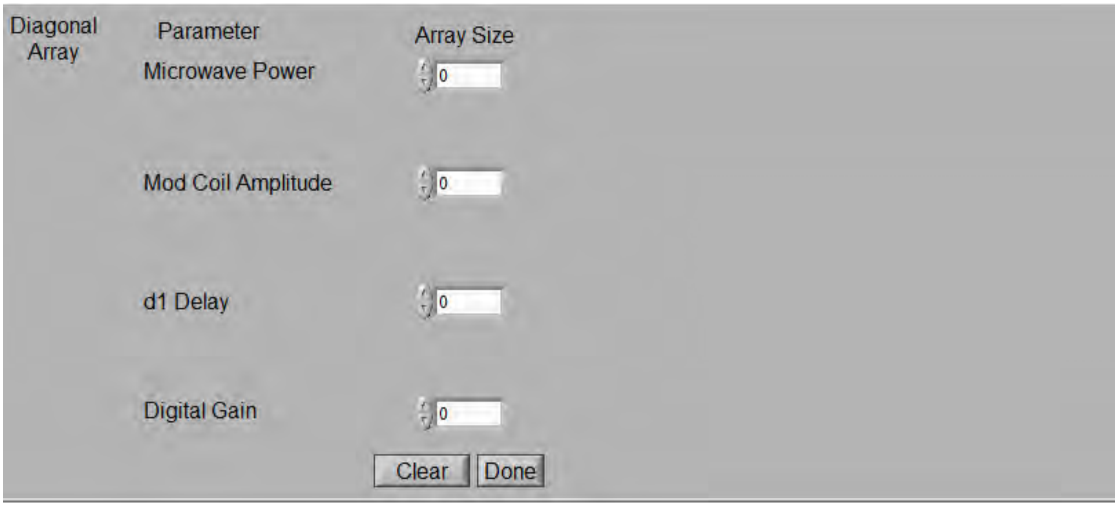


Figure 8.7: Parameter Array Pop-up

Microwave Power

Knowing the power at which a signal becomes saturated is very important. The power array feature allows the user to automatically array the microwave power, and then look at the data to determine the best power to use for the experiment. The microwave power can be set to the following values: 0.5, 1, 3, 6, 10, 15, 40, 50, 60, and 70 dBm.

Mod Coil Amplitude

The sample and nature of the experiment will determine the best modulation amplitude to use. The array parameter allows the user to automatically set up a series of experiments in which the mod coil amplitude is automatically changed to user defined values between experiments. The mod coil amplitude can accept values from the maximum value to 1 percent of the maximum value.

d1 Delay

The d1 delay is a delay before the start of each acquisition in an experiment. This parameter is a bit unusual for a CW instrument. The delay value is in msec.

Digital Gain

This is the gain added before the ADC, it can take values of 0, 12, 18, and 24 dB.

To array microwave power, enter a number in the **Array Size** box next to **Microwave Power**. A pull down menu for each array element will appear to the right:

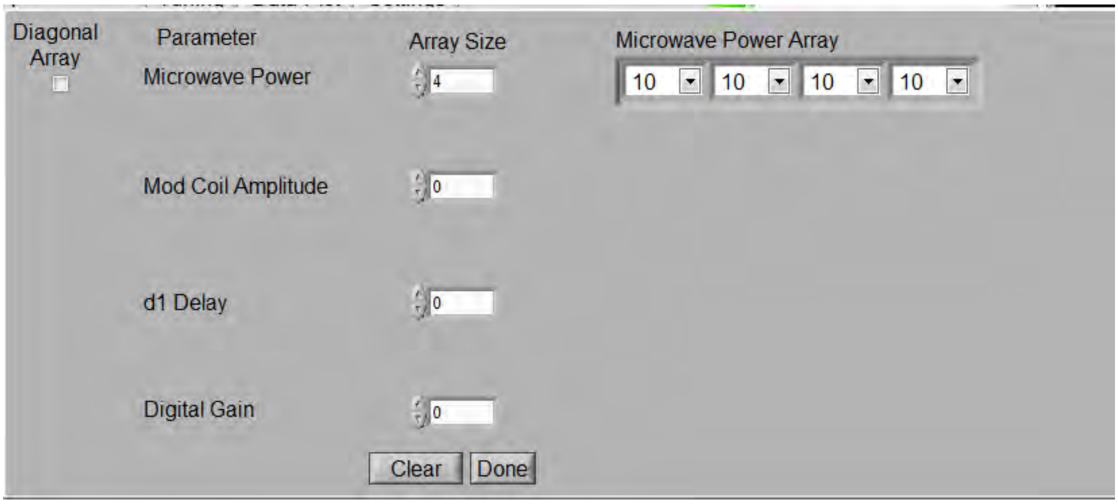


Figure 8.8: Parameter Array Pop-Up with a Non-Zero Array Size for Microwave Power

- Click on the arrow to select the microwave powers you wish to use in the array. The powers available are 0.5, 1, 3, 6, 10, 15, 25, 40 ,50 ,60, and 70 mW
- Then click on **Done**. The array pop-up will disappear.

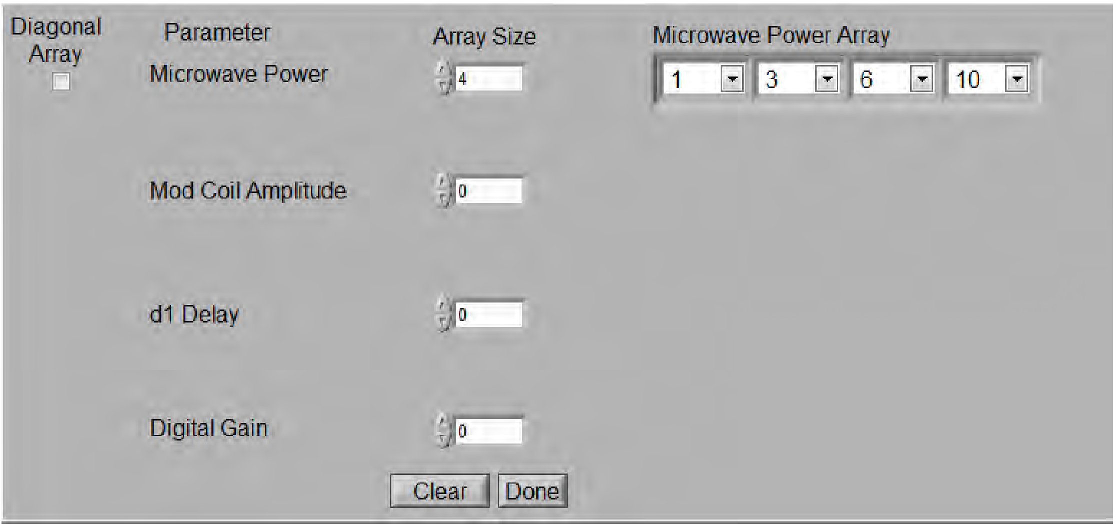


Figure 8.9: Microwave Power Array Set Up

The number of runs must be greater than or equal to the array size. If the number of runs is greater than the array size, the program will keep running the array until the number of acquired spectra equals the number of runs.

- Select the number of scans, number of points, and the delay you wish to use.
- Make sure that the **Use Array** checkbox is selected. If the **Use Array** checkbox is not selected, the array will not run:

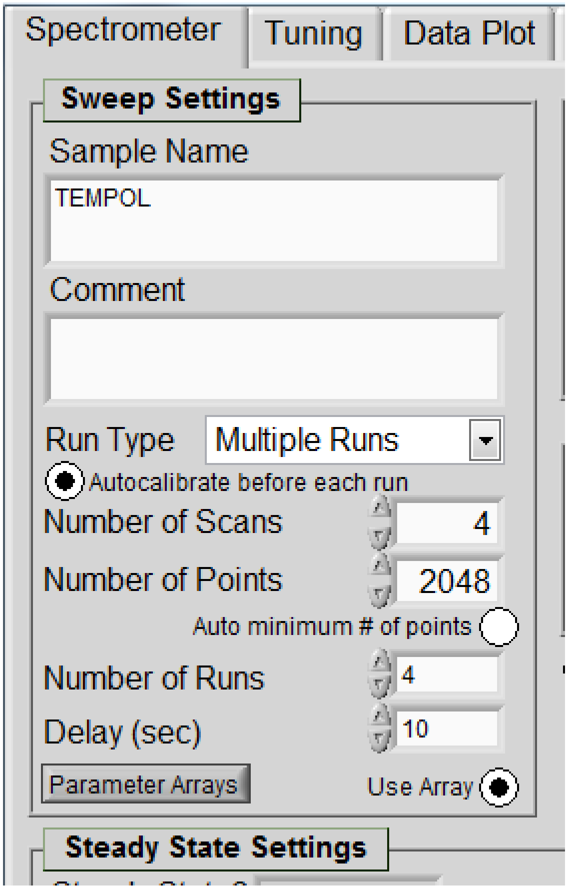


Figure 8.10: The Sweep Settings Displaying the Minimum Number of Scans for an Array with Four Values.

- For best results, click **autocalibrate** before each run under Run Type. Changing the power can change the tuning.
- Click on **START SWEEP** to start the acquisition, and the spectrometer will automatically tune, acquire, and save the data as long as the autocalibrate before each run is selected.
- Clicking on the **Parameter Array** button will bring up the parameter array pop-up which will display any arrayed parameters.
- Clicking on **Clear** will clear out all arrays (*Figure 8.9 [▸ 46]*).
- To array the **Mod Coil Amplitude**, change the array size to a non-zero number in the box next to Mod Coil Amplitude.

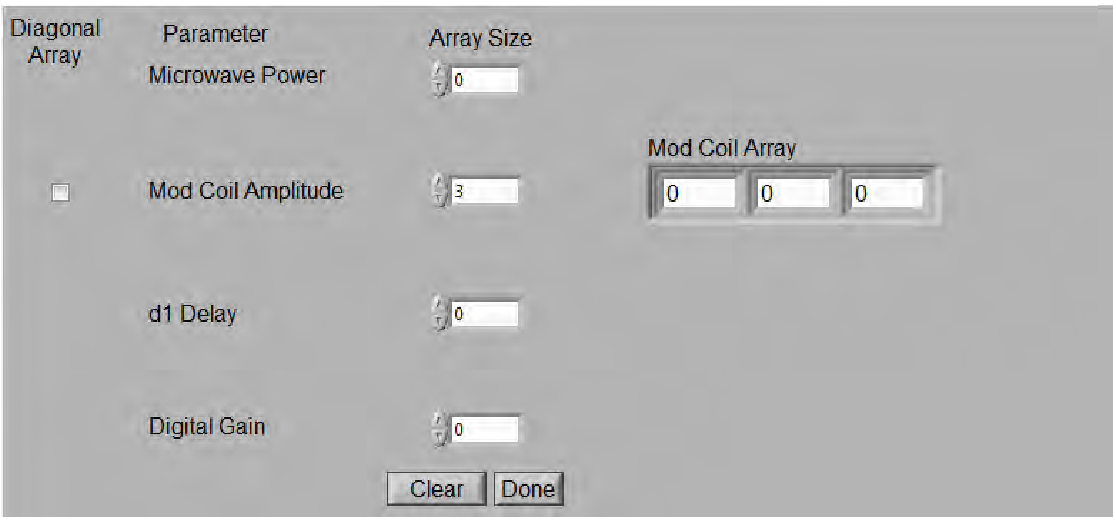


Figure 8.11: Set up to Array the Mod Coil Amplitude

Boxes to enter the array values for the mod coil amplitude will appear to the right. The number of boxes will equal the array size. Clicking in the box will bring up a keypad. Use the keypad to enter the values you wish to use:

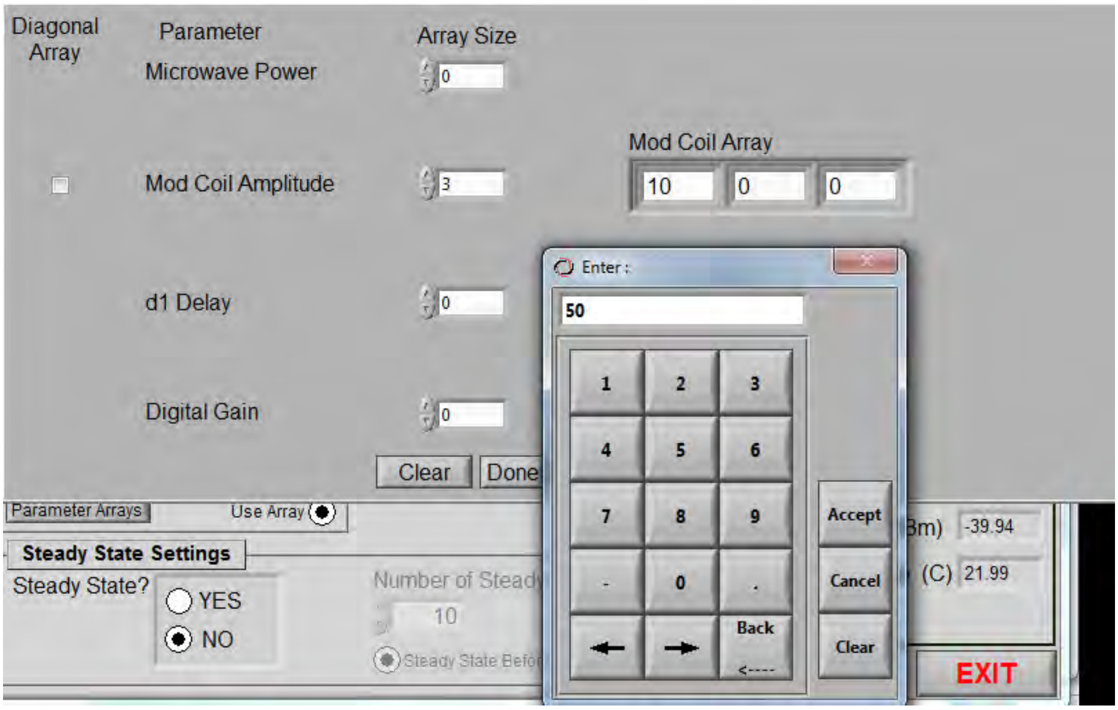


Figure 8.12: Use Keypad to Enter Mod Coil Amplitude Values

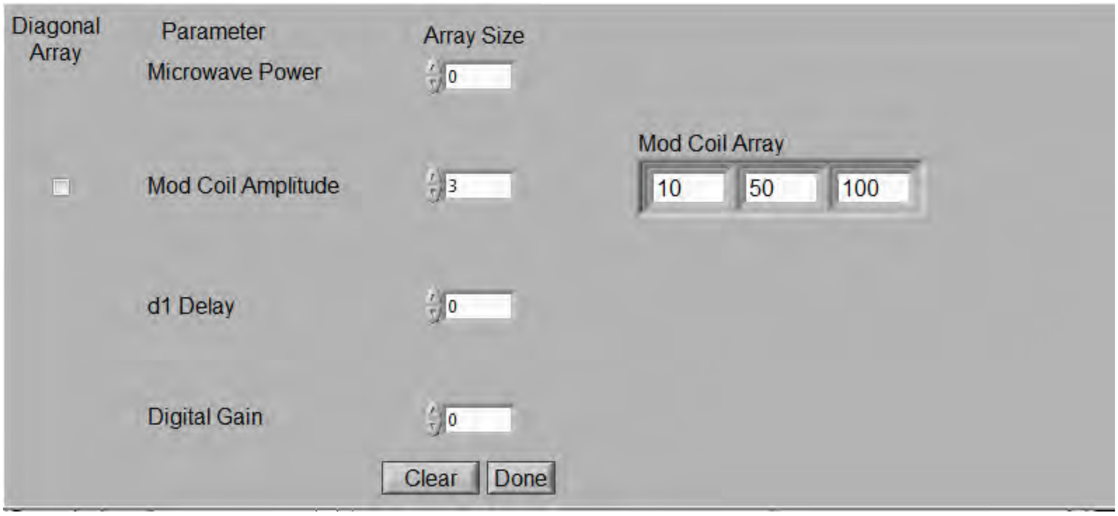


Figure 8.13: Parameter Array Pop-Up Set Up to Array the Mod Coil Amplitude

- The mod coil amplitude values are a percent of the total values. Any integer value from zero to one hundred can be entered. At 100 percent, the mod coil amplitude is about 2 Gauss.
- Click on **Done**, and the parameter array pop-up will disappear.

In the example above, three values for mod coil amplitude have been selected, so there will need to be a minimum of three runs.

- Make sure to check the **Use Array** box (*Figure 8.10 [▸ 47]*).
- For best results, select **autocalibrate** before each run.
- Click **START SWEEP** to start the acquisition if autocalibrate before each run is selected.
- To array the **d1 Delay**, enter a non-zero number in the array size box next to d1 Delay.
- Boxes to enter the array values for the d1 Delay will appear to the right. The number of boxes will equal the array size. Clicking in the box will bring up a keypad. Use the keypad to enter the values you wish to use. See *Figure 8.12 [▸ 48]*.

The values for the d1 Delay are in msec. There is no need to tune between runs for the d1 Delay, unless the acquisitions are very long.

To array the **Digital Gain** value, enter a non-zero number in the array size box next to digital gain. A pull down menu for each array element will appear.

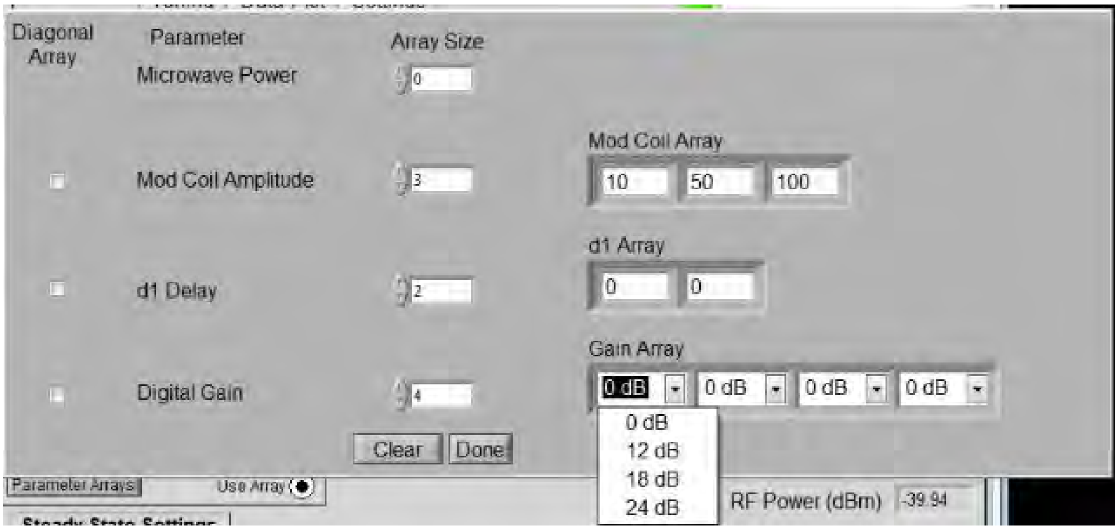


Figure 8.14: Pull Down Menu for each Element

- Select the values you wish to use, and select **Done**.
- You do not need to tune (autocalibrate) for each run if only the digital gain is changed; however, you may need to increase the noise limit in the tune interface if you are running the digital gain above 12 dBm. See *Figure 8.22 [56]*.
- To array more than one parameter at a time, click on the **Parameter Array** button. Enter the values for the parameters you would like to array. For example, arraying both mod coil amplitude and microwave power together can quickly give the user the best combination to use to optimize the experiment.

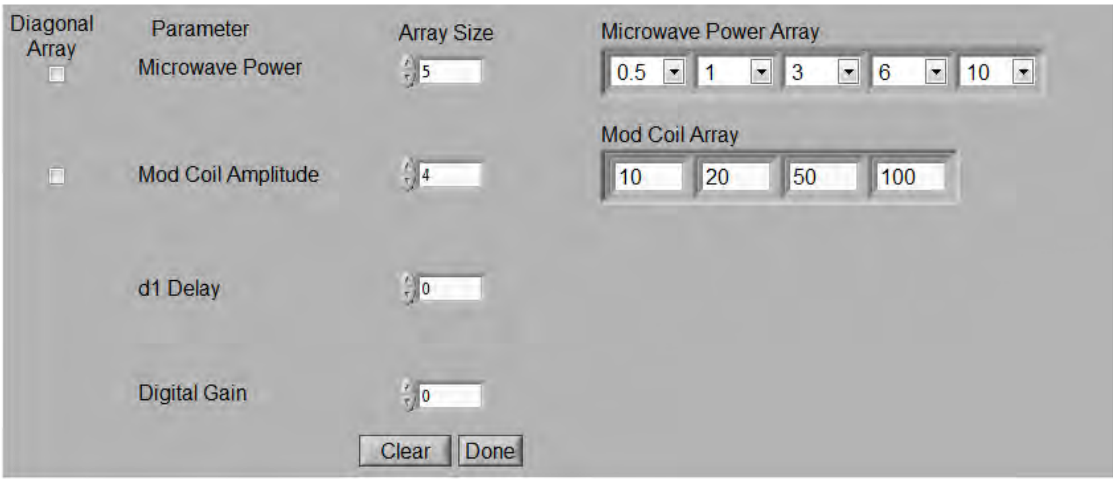


Figure 8.15: Example of an Array of both Microwave Power and Mod Coil Amplitude.

This array will have twenty elements in it. The minimum number of runs in this case will be twenty. A spectrum will be run for every value of microwave power with each value of mod coil amplitude. For best results, select **autocalibrate** before each run.

A **Diagonal Array** in which two parameters are arrayed, but you only wish to run certain pairs of values can also be run.

Both array sizes must be the same. The number of spectra run will equal the number of elements in the array.

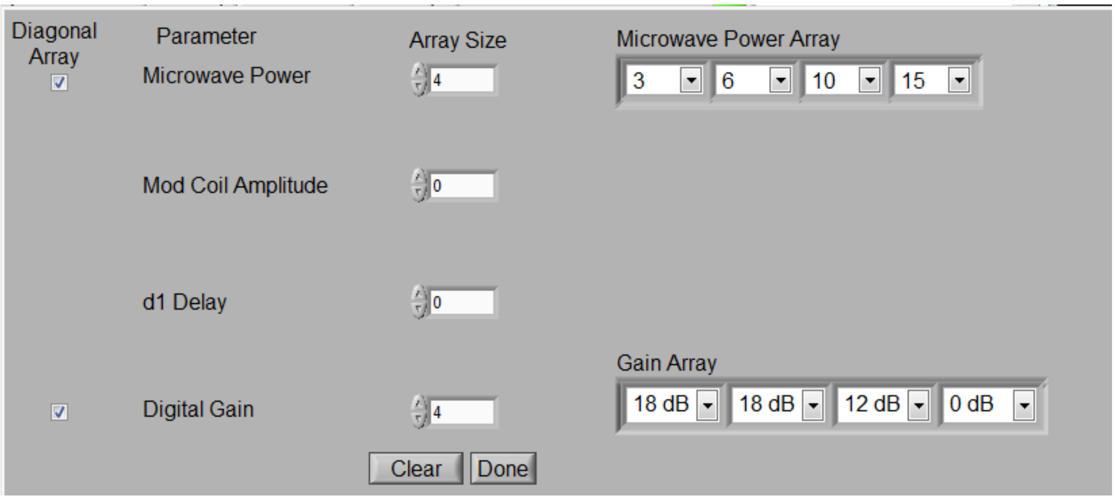


Figure 8.16: An Example of a Diagonal Array.

Note that both parameters have the same array size, and the **Diagonal Array** box is checked for both parameters.

The spectrometer will run four spectra in the above example. The first spectrum will have power 3mW and gain 18 dB, the second power 6 mW and gain 18 dB, the third power 10 mW and gain 12 dB, and the fourth 15 mW and gain 0 dB.

If the **Diagonal Array** buttons are unchecked, the system will run 16 spectra using each microwave power with every gain value. In this case, you would have two sets of identical spectra with gain set to 18 dB.

8.1.3 Steady State Settings

Steady State?

Steady state scans, sometimes referred to as dummy scans, are used to bring a sample into thermal equilibrium before data is acquired. Essentially, the spectrometer runs a number of scans without writing the data to disk before the experiment starts. If the **YES** button is checked under **Steady State Settings**, once the acquisition starts, the square will turn from green to yellow, and the message will say, **Please wait….. Warming Up**. The scan number will display the current scan number out of the total requested steady-state scans.

Figure 8.17: Steady State Warm-Up

The figure above shows the progress of the steady-state scans. In the above example, 4 steady-state scans were requested, and the spectrometer is running the first steady-state scan. The spectrometer will immediately start acquiring data when the steady-state scans are finished. The message will change from Warming Up to Sweeping Spectrum.

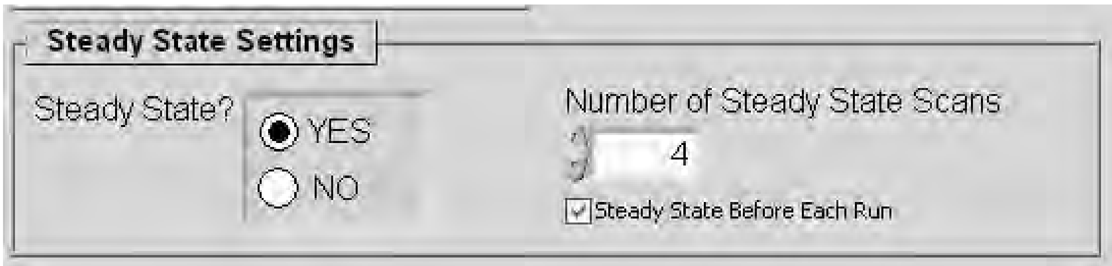


Figure 8.18: Steady State Settings

Number of Steady State Scans

Number of steady state scans to run. The number of steady-state scans will depend on the sample and the experiment.

Steady State Before Each Run

This option only works with Continuous and Multiple Runs (see description given above). If the box is checked, the requested number of steady state scans will be executed before each Run. If the box is not checked, the steady state scans will only be executed once at the start of the first run.

8.1.4 Magnetic Field

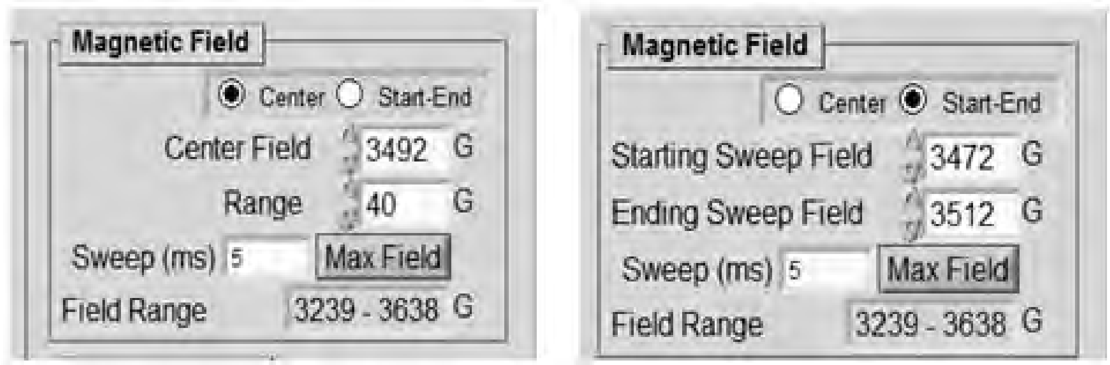


Figure 8.19: Magnetic Field Settings

Center

Allows the user to set the sweep parameters using the desired spectral center and field range.

Start-End

Allows the user to set the sweep parameters by specifying the starting and ending field.

Center Field

The center frequency.

Range

The width of the spectrum.

Sweep (ms)

Controls the sweep rate. The default value is 5 ms per point. The minimum sweep rate is 0.2 ms per point, and the maximum is 100 ms per point. This value works well for most experiments. This parameter will not be visible by default. See the *ESR Settings File [▸ 127]* section in the appendix of this manual.

Max Field

The Max Field button will automatically enter the spectrometer's maximum sweep range.

Field Range

Displays the limits of the magnetic sweep coil's range. This is the maximum sweep range available to the spectrometer. These values cannot be changed.

Starting Sweep Field

Starting value of the electromagnetic sweep coil. This value cannot be lower than the minimum value displayed in the Field Range box.

Ending Sweep Field

Ending value of electromagnetic sweep coil. This value cannot be larger than the maximum value displayed in the Field Range box.

8.1.5 Power Settings

Microwave Power

The microwave power to the cavity. The values for microwave power, in mW, are 0.5, 1, 3, 6, 10, 15, 25, 40, 50, 60, and 70. The values can be selected from the pull-down menu. The best power to use will depend on the specific sample. The power array feature is designed to help determine the highest power that can be used without saturating the sample.

Max Mod Coil Amplitude

The maximum modulation amplitude. This value is calibrated in the factory, and set in the firmware.

Mod Coil Amplitude

Intensity of the modulation signal (gain). The value can be set from the maximum value to 1 percent of the maximum value.

Digital Gain

A gain stage before the ADC. Can be set to values 0, 12, 18, and 24 dB. The default value is 12. If the digital gain is not visible, see the *ESR Settings File [▸ 127]* section in the appendix of this manual.

8.1.6 Baseline Correction

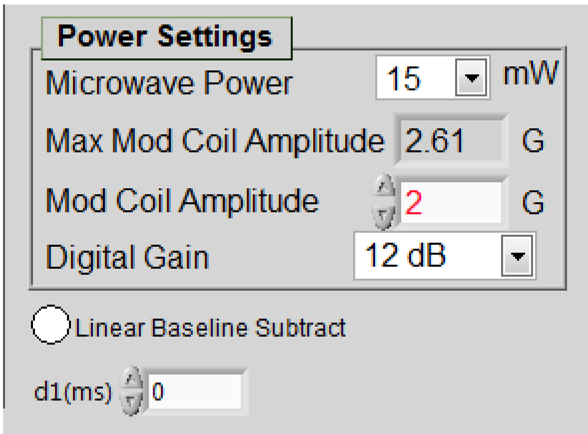


Figure 8.20: Power Settings, Baseline Correction and Preacquisition Delay.

Linear Baseline Subtract

If the box next to linear baseline subtract is checked, the software will display a spectrum with a first order baseline correction. The uncorrected data is displayed if the box is unchecked. The raw data is not altered in any way. Linear and other baseline correction algorithms are available in the Processing and Analysis software package.

File subtraction can be also done in the Processing, Analysis, and Simulation software package.

d1 Delay

Preacquisition delay. A delay, entered in msec, inserted before each scan. This is not commonly used in CW ESR. This delay might not be visible. See the *ESR Settings File [▸ 127]* section of the appendix in this manual.

8.2 Tuning Control

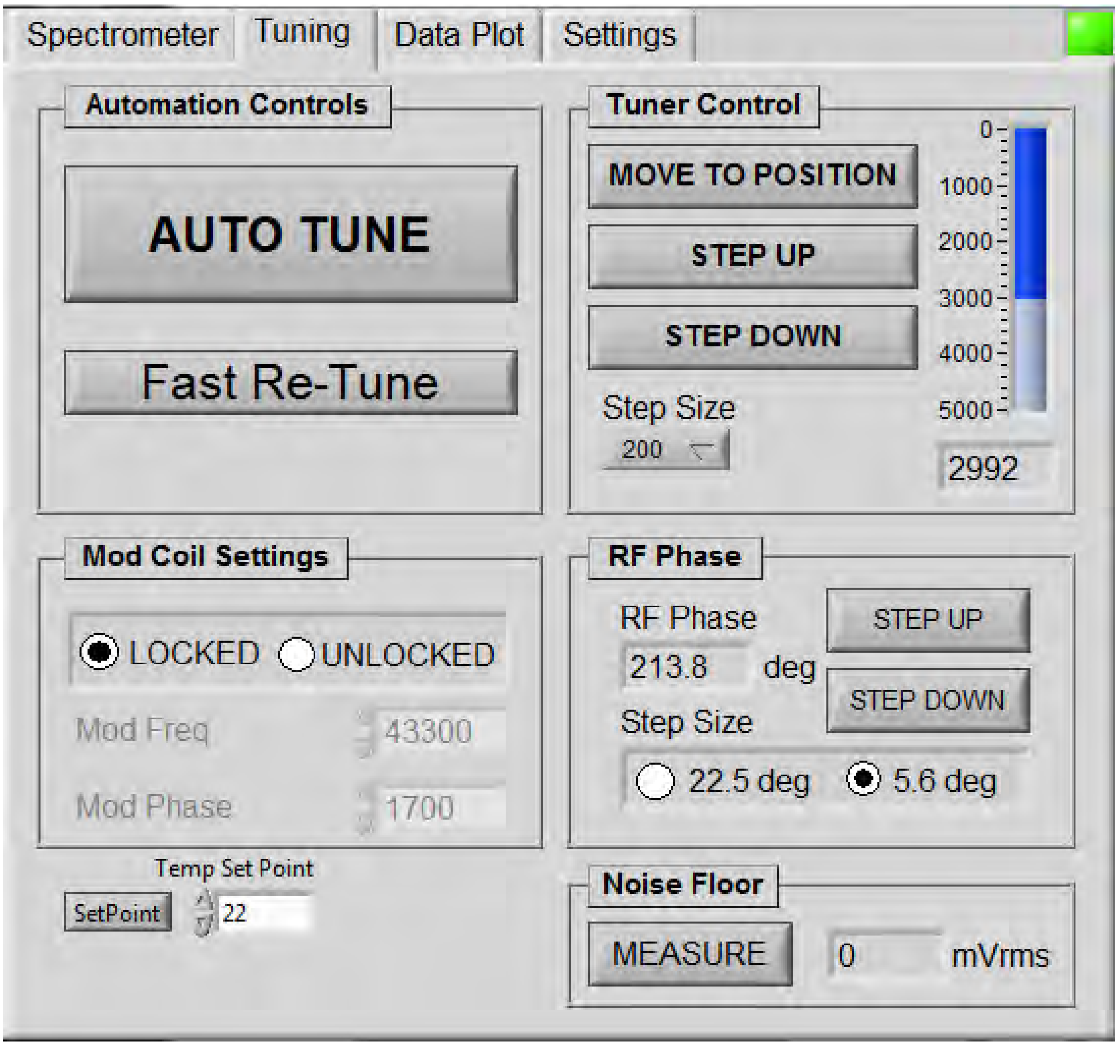


Figure 8.21: Tuning Tab

8.2.1 Automation Controls

AUTO TUNE

Full automatic tuning of the sensor. Brings up pop-up window shown below. As soon as the **AUTO TUNE** button is clicked, the status changes from Ready to Tuning Instrument. The actual tuning process does not start until the **TUNE** button is clicked in the AUTO TUNE pop-up.

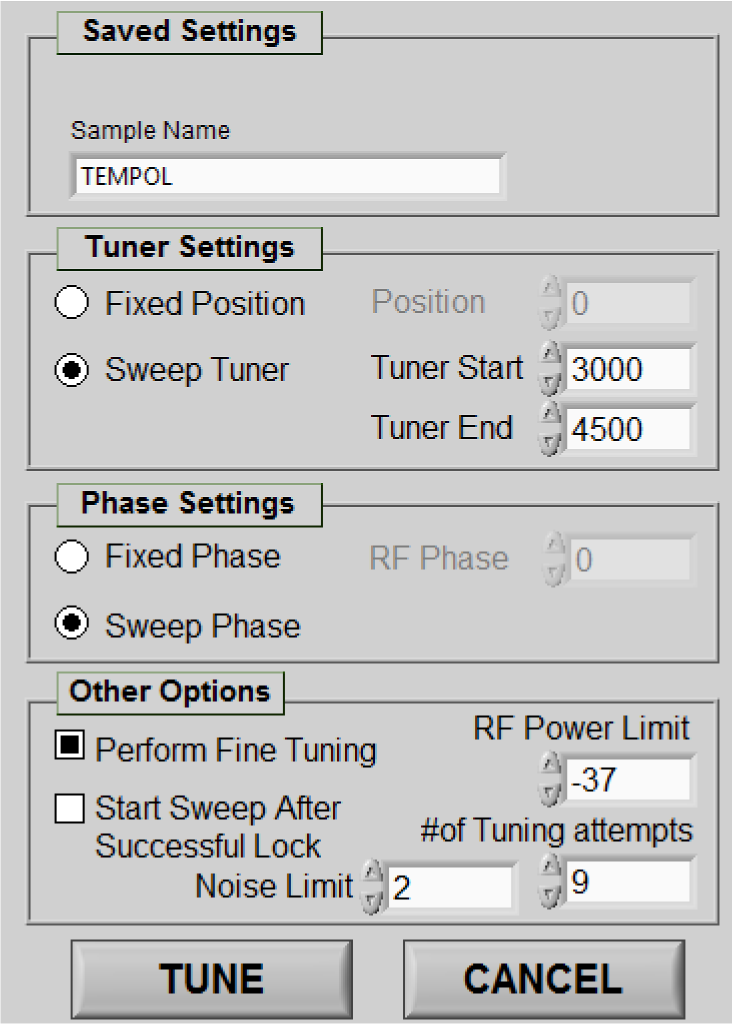


Figure 8.22: Auto Tune Pop-Up

The green light and the Ready message in the upper right corner of the software, should change to an orange light with the message tuning instrument:

Figure 8.23: Ready Light and Message

If the pop-up window does not appear, minimize the uESR window. The pop-up window could be behind the main uESR window.

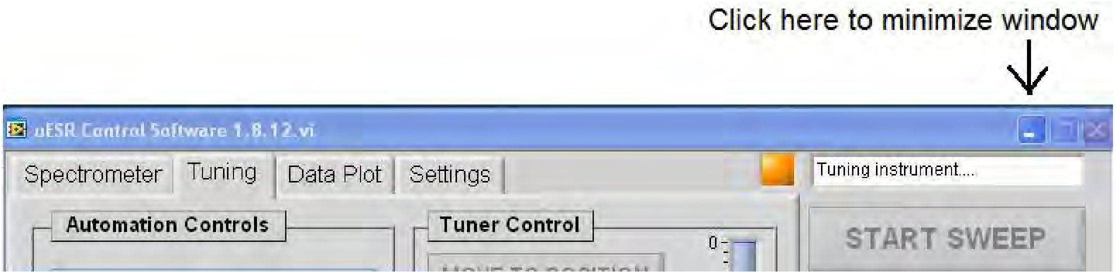


Figure 8.24: Minimize Button

8.2.2 Saved Settings

Sample Name

Can change or enter sample name from the auto tune pop-up if you select.

8.2.3 Auto Tune Routine

The fully automatic tune routine sweeps the specified frequency range, which is set in the factory, to look for the minimum reflected power. Once the frequency with the minimum reflected power is found, the AFC is then locked to the resonator by sweeping the RF phase. Once the AFC is locked to the resonator, the RF phase is again swept to look for the minimum noise floor (fine tuning).

Once the **TUNE** button is clicked, the routine will proceed as described in the section *Run a Scan [▸ 27]*.

If the Show Tune Window is selected under the Settings Tab, the autotune routine will be displayed in a pop-up window:

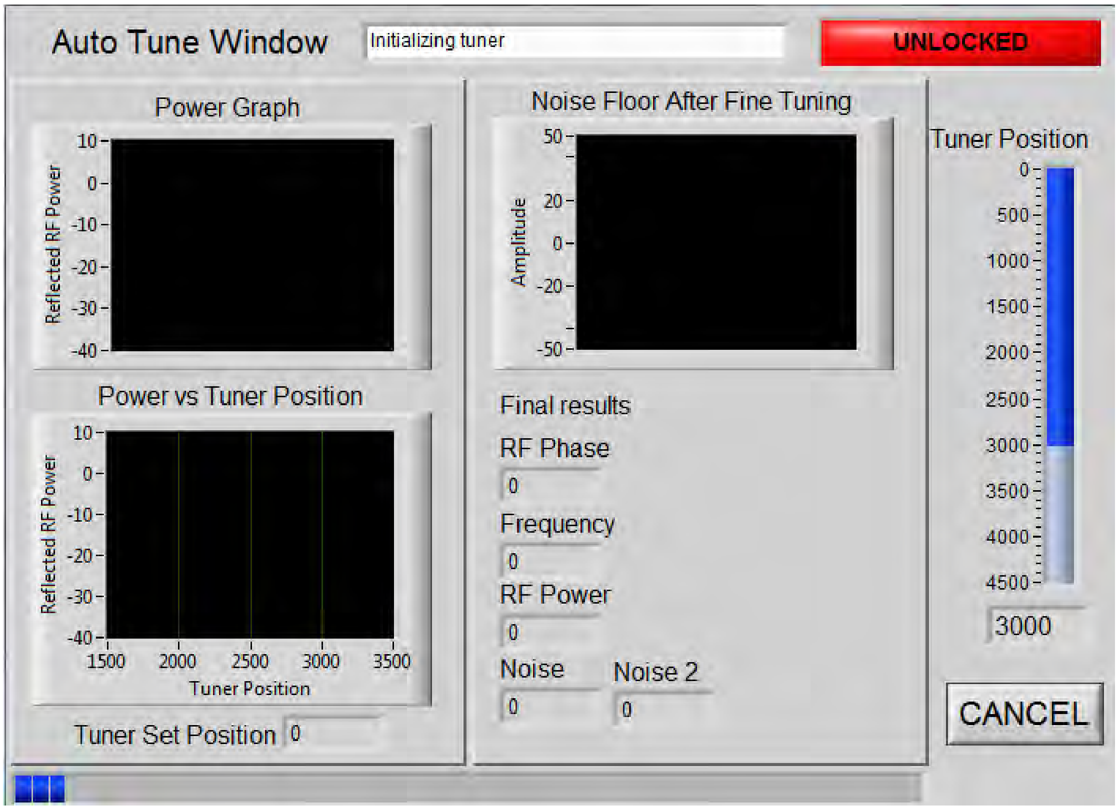


Figure 8.25: Auto Tune Display

Next, the Initializing tuner message will change to Stepping Tuner, as the auto tune routine sweeps the frequency range for the minimum reflected power.

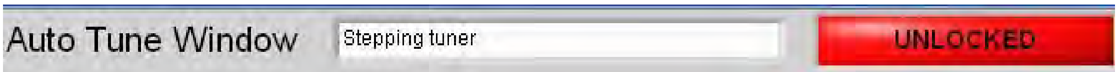


Figure 8.26: Stepping Tuner

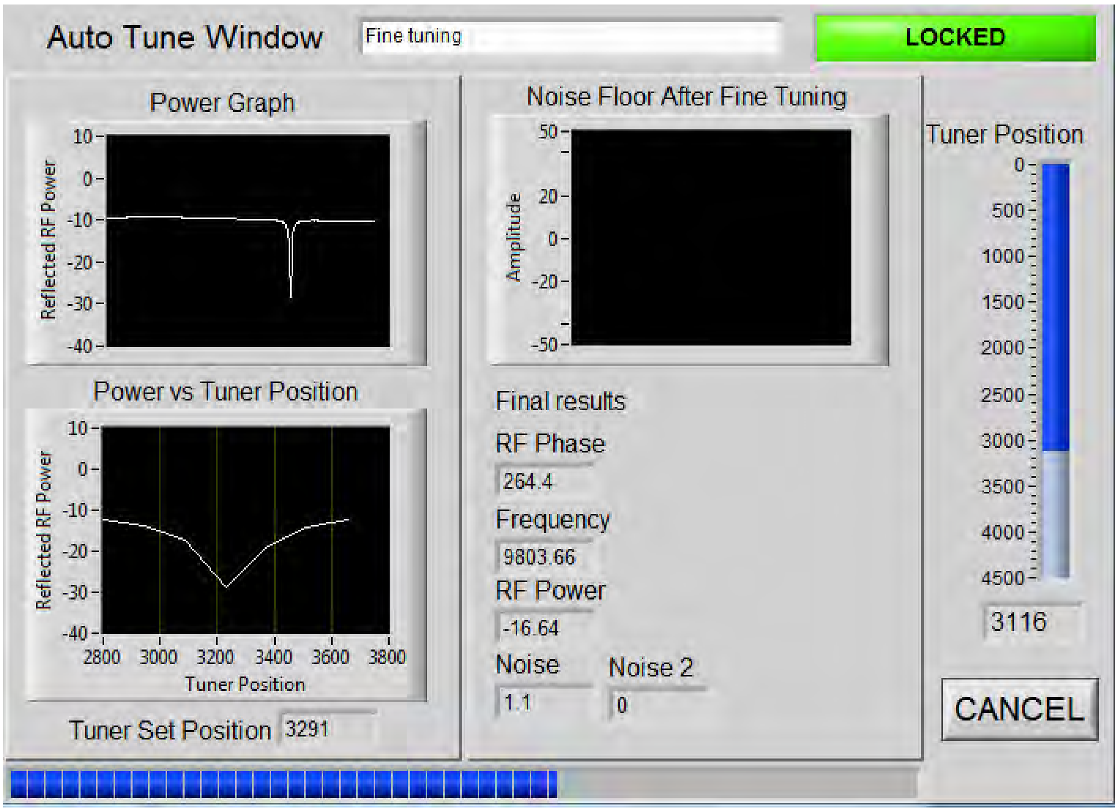


Figure 8.27: Moving to Critical Coupling

Note the blue tracking line at the bottom of the graphic.

Power Graph

The Power Graph window displays the minimum reflected power as the field is swept.

Power vs Tuner Position

The Power vs Tuner Position displays the frequency vs reflected power.

The current tuner position is displayed by the slider.

The tuner is moved to the frequency with minimum reflected power, or critical coupling position. This frequency is displayed as Tuner Set Position. The message in the auto tune window will change to Moving tuner to critical coupling.

The message then changes to Locking AFC to resonator.

Figure 8.28: Locking to Resonator

The auto tune routine will be sweeping the RF phase to find the best frequency. Once the AFC is locked to the resonator, the UNLOCKED message changes to LOCKED.

Figure 8.29: Sweeping for Optimal Phase

If the **Perform Fine Tuning** box in the Auto Tune pop-up is checked, an even finer phase adjustment is done.

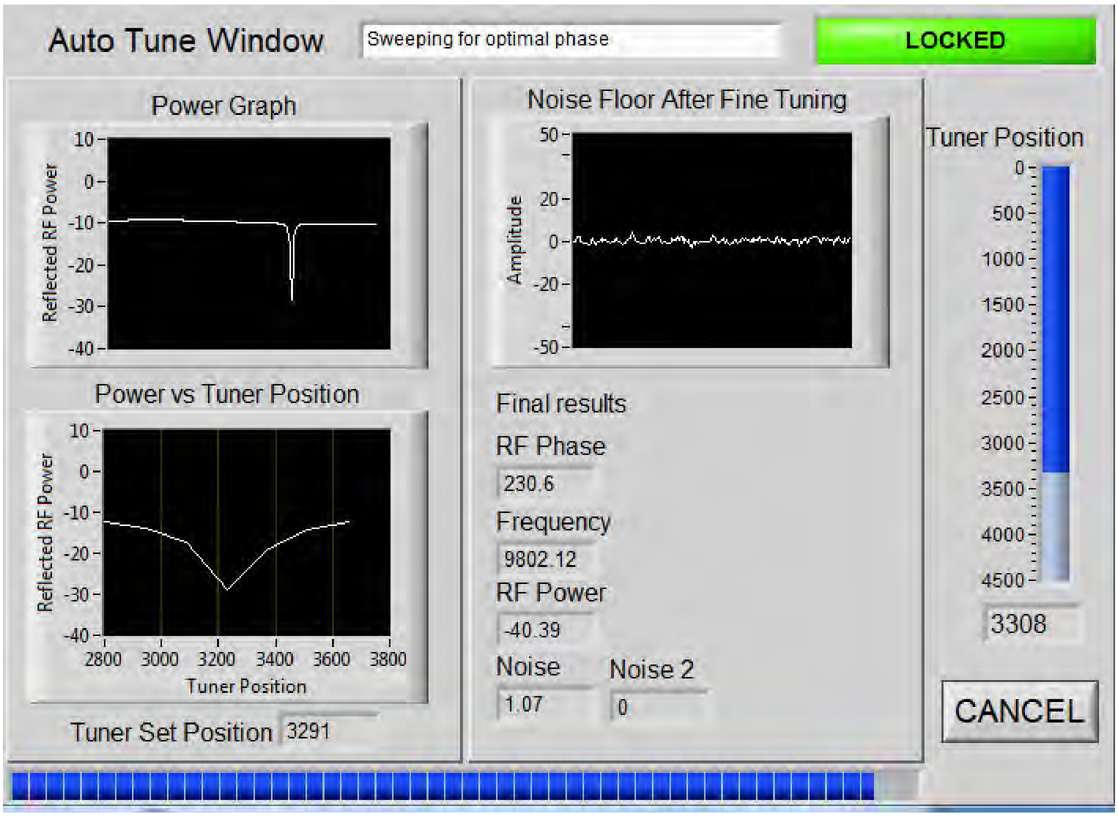


Figure 8.30: Successful Auto Tune

The final RF phase, frequency, and power will be displayed.

When the Auto Tune routine is done, both the Auto Tune window and pop-up will automatically close, and the Tune window will be in the foreground.

8.2.4 Auto Tune Failure

The most likely cause of auto tune failure is that the sample is too lossy. If the system fails to tune, try to run the Auto Tune routine with an empty cavity. If the system tunes with an empty cavity, use a smaller diameter tube for your sample. If this does not resolve the issue, see the troubleshooting section in this manual.

8.2.5 Tuner Settings

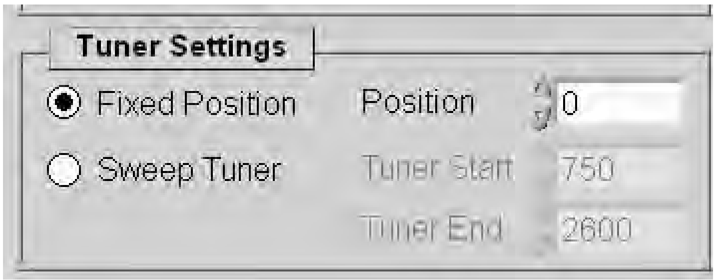


Figure 8.31: Tuner Settings

Fixed Position

Specify the position of the tuner. If this option is selected, the Auto Tune routine will start the routine with the requested tuner position. If it cannot tune at the requested position, it will then sweep the tuner range.

Sweep Frequency

Sweeps the frequency between the **Tuner Start** and **Tuner End** limits to find the position with the minimum reflected power. The **Tuner Start** and **Tuner End** limits are set during factory calibration, but can be manually changed.

8.2.5.1 Phase Settings

Fixed Phase

Specify RF phase to be used during tuning. The settings are 0 to 359 in 5.6 degree increments.

Sweep Phase (recommended)

Sweep the phase during tuning to find the minimum reflected power and then the minimum noise floor.

8.2.6 Other Options

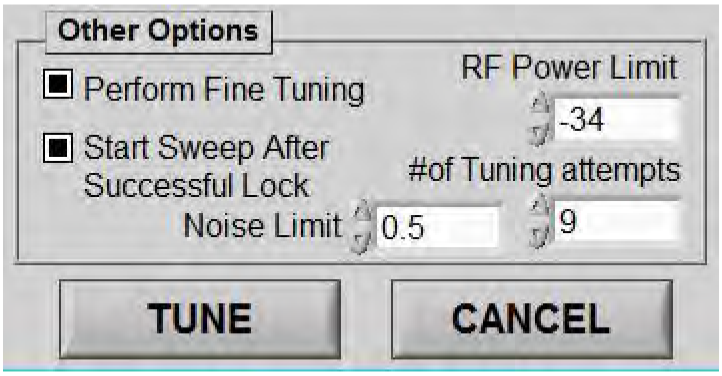


Figure 8.32: Other Tuning Options

Perform Fine Tuning

Run a refined sweep around final phase to ensure minimum noise floor. For best results, this option should be used as it will result in the lowest noise floor.

Start Sweep After Successful Lock

Automatically starts running the experiment after the system is tuned and locked.

RF Power Limit

Specifies the upper limit of the allowed RF power for successful tuning. This number will be system specific.

# of Tuning Attempts

The number of attempts the software will make to tune to below the specified RF power limit.

Noise Limit

Defines the maximum noise floor acceptable.

TUNE

Starts the autotune routine.

CANCEL

Stops autotune routine.

In general, it is best to run the Auto Tune routine on all samples. The Auto Tune routine moves the field such that the reflected power is minimized, and then adjusts the RF phase such that the noise floor is minimized. The rest of this section explains manual tuning. **Manual tuning** can be used to make minor adjustments to the tuning between samples when the samples are very similar, but the autotune is quite quick.

Fast Re-Tune

Makes minor adjustments to tuning. Should not be used for new samples unless the sample is very similar to the previously tuned sample. If microwave power or mod coil amplitude has been changed, Fast Re-Tune is a good option.

8.2.7 Tuner Controls

Move to Position

Moves tuner to specified position. This option should only be used if the resonant frequency is already known. If this is set incorrectly, the spectrometer will fail to lock the sample.

Step Up and Step Down

Steps the tuner up or down by the specified step size. The step-size can be 200, 25, 5 and 1. Select from the pull down menu.

The reflected power measurement should be at a minimum. See *Sweep Progress [▶ 86]*.

8.2.8 RF Phase

RF Phase

Relative phase of the RF carrier signal (0-360 degrees).

Step Up and Step Down

Steps the RF phase up or down by the selected step size. The step size increments are 5.6 and 22.5 degrees.

Both the phase of the spectra and the signal to noise are influenced by the RF phase setting. The correct setting is determined by minimizing the noise floor.

If the RF phase is incorrectly set, the phase of the spectrum may be incorrect, and the signal to noise will be adversely affected.

8.2.9 Noise Floor

Clicking on the **Measure** button returns a RMS voltage reading of the noise floor. The noise floor is minimized by the Auto Tune routine by changing the RF phase. To manually check for the best RF phase setting, click on the **Measure** button under noise floor, and note the reading. Click the RF phase step up or step down button once, and then recheck the noise floor. If the noise floor is lower, step the phase in the same direction, and reread the noise floor. Repeat until the RF phase setting is such that the noise floor is minimized.

8.2.10 Mod Coil Settings

The modulation coil phase and frequency are set in the factory during calibration. By default, these settings are locked. These should not need to be manually adjusted.

The unlocked button must be selected in order to change the value of either the phase or frequency of the mod coil.

Mod Phase

Relative phase of the audio coil (0-3600 which represents 0 to 360°, for example, 1800 is 180 degrees.) The correct phase will depend on the spectrometer. The modulation phase is also function of the modulation amplitude. This dependence is calibrated in the factory, and corrected for in the software. The parameter **Mod Phase Slope** is the parameter that determines the correction. The next two figures illustrate a correctly and incorrectly set Mod Phase

The following figure has the Correct Mod Coil Setting.

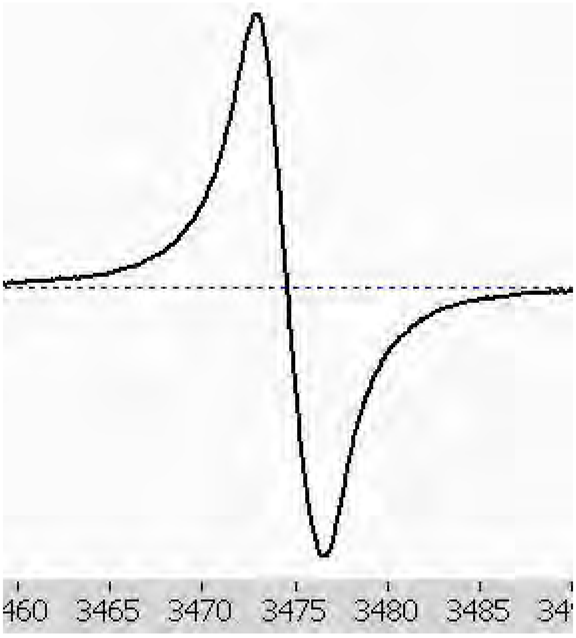


Figure 8.33: Signal with Correct Mod Phase

The next has the mod coil phase set incorrectly. The mod coil setting is different by 1800. Note that the spectrum is 180 degrees out of phase.

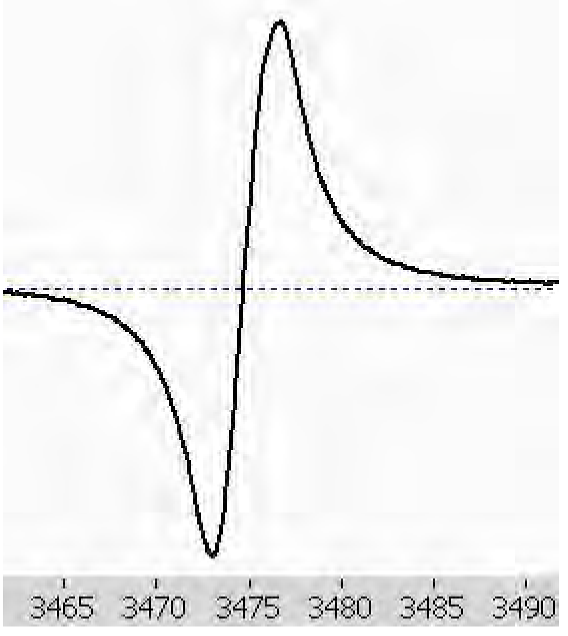


Figure 8.34: Signal with Incorrect Mod Phase

IMPORTANT: If you decide to change either the mod phase and or mod frequency of the spectrometer, write down the original settings before making any changes. These parameters should not need to be adjusted.

Mod Freq

Modulation frequency of the audio coil. The range is 40,000 to 45,000. This value is normally set to 43300.

Temp Set Point

Changes the set point of the TEC temperature controller that regulates the magnet temperature. The TEC temperature has limited heating and cooling abilities, so can only control temperature over a limited range. It will regulate the magnet temperature between twenty to thirty degrees Celsius assuming an ambient temperature of around twenty-four degrees.

This parameter may not be visible. See the *ESR Settings File [▸ 127]* s section to make the Temp Set Point parameter visible. Older spectrometers will not have the internal USB cable and drivers for this feature. If the feature is enabled, and still does not appear, the hardware is not installed. Contact Bruker for more information.

- Enter the desired set point in the box.
- Click on **the Set Point** button, and wait for the temperature to regulate.

The magnet temperature is displayed under the RF Cavity settings in the lower right corner. See the figure in *Sweep Progress [▸ 86]*.

8.3 Data Plot Tab

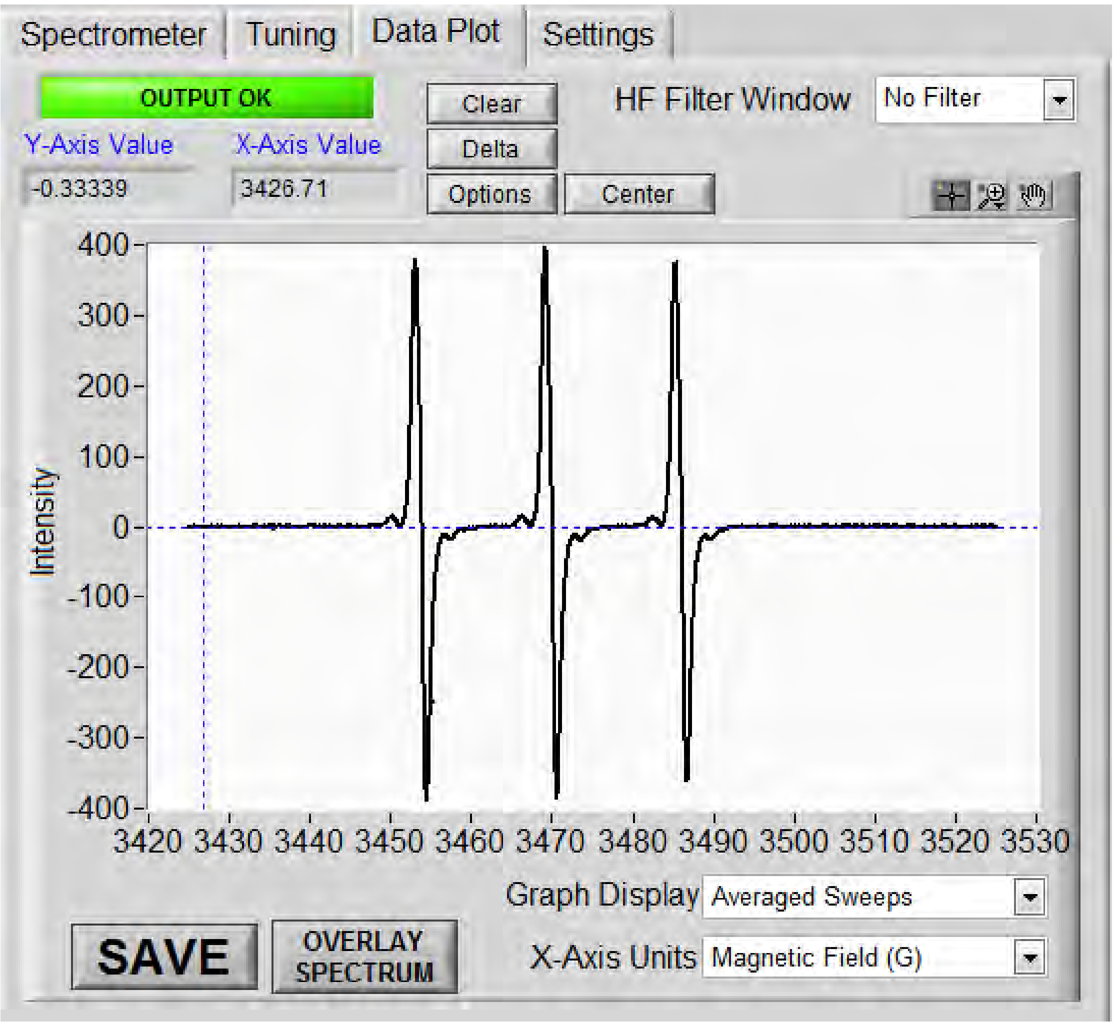


Figure 8.35: Data Plot Tab

OUTPUT OK

The figure above shows the normal green OUTPUT OK message. Too much signal will overload the ADC/receiver, and cause the signal to be distorted. The button will turn red and display an error message in the case of ADC/receiver overflow: OUTPUT DISTORTED:

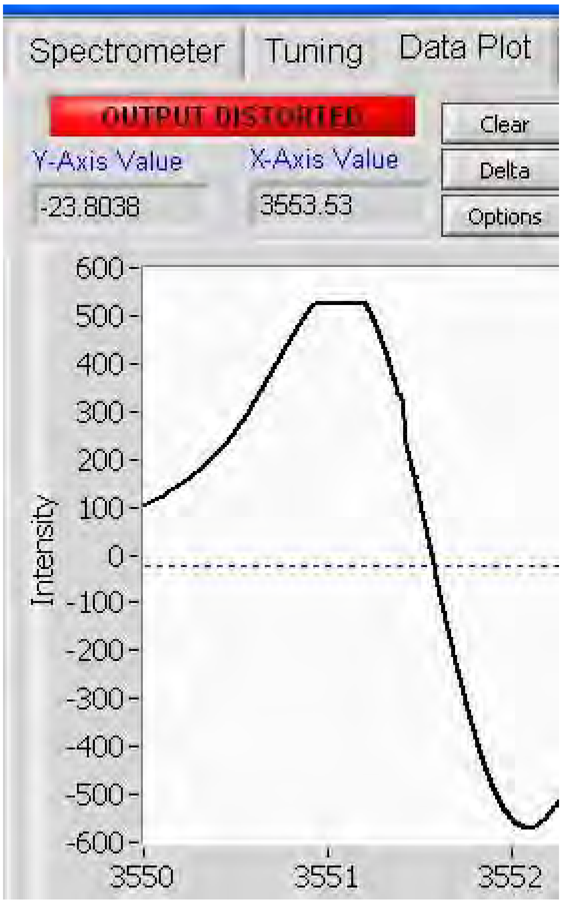


Figure 8.36: Output Distorted

Note that the top of the spectrum is clipped.

Y-Axis Value

Marker position of the Y-axis.

X-Axis Value

Marker position of the X-axis.

The crossed blue dashed lines are the X and Y axis markers.

Clear

Clears the spectrum from screen.

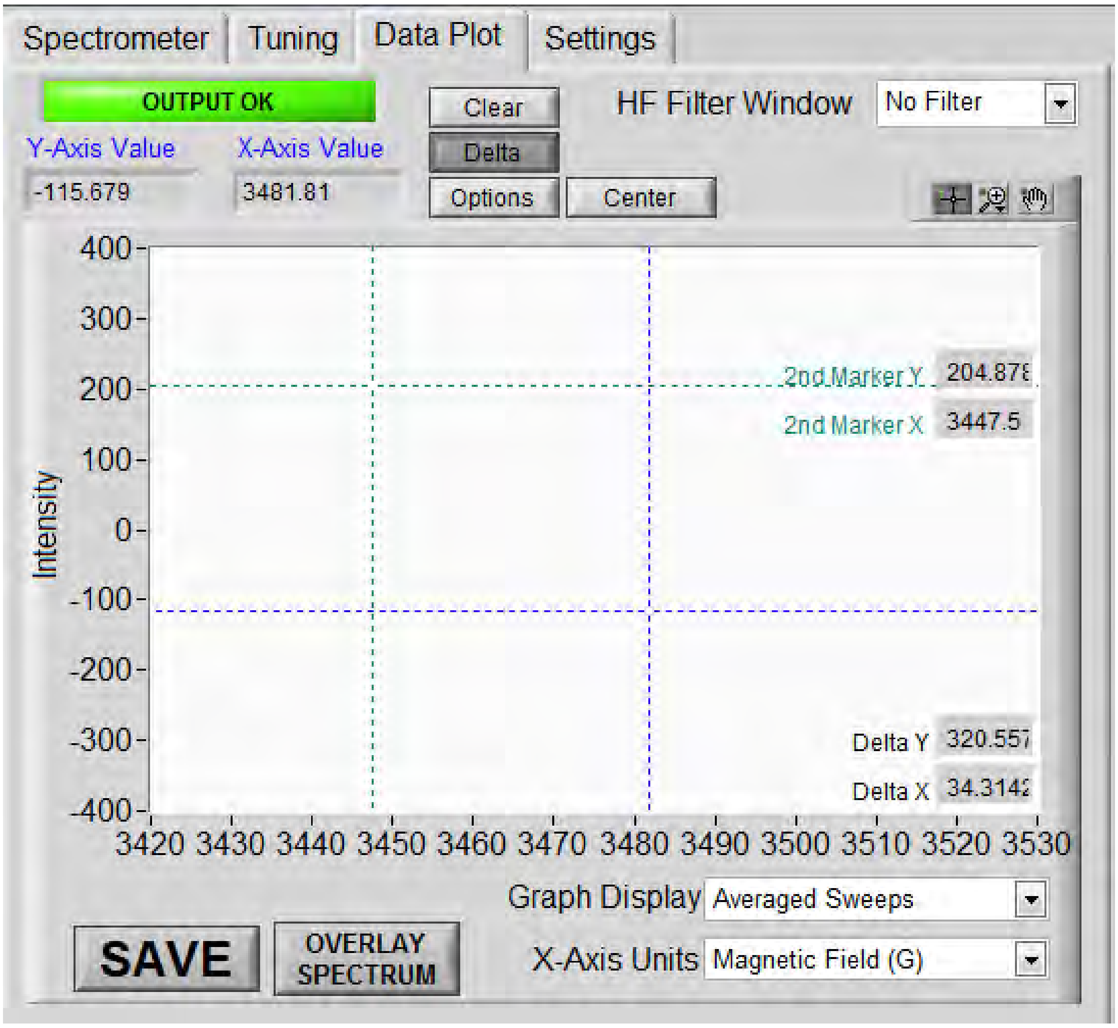


Figure 8.37: Delta Option

Delta

Puts two X and two Y cursors (blue dashed and dark magenta dashed crossed lines) on the screen to give the distance between cursors. The Delta X and Y values appear at the bottom right of the spectral window. Drag cursors in either direction to obtain the value(s) of interest.

Options

Allows the operator to either specify X and Y dimensions, or have the X and Y dimensions autoscaled. The default is autoscale.

Figure 8.38: Graph Options

HF Filter Window

Allows the use of a Savitzky-Golay filter. There is an example in the appendix.

Center

Allows user to use the cursor to select the center of the spectrum, and specify the range to acquire.

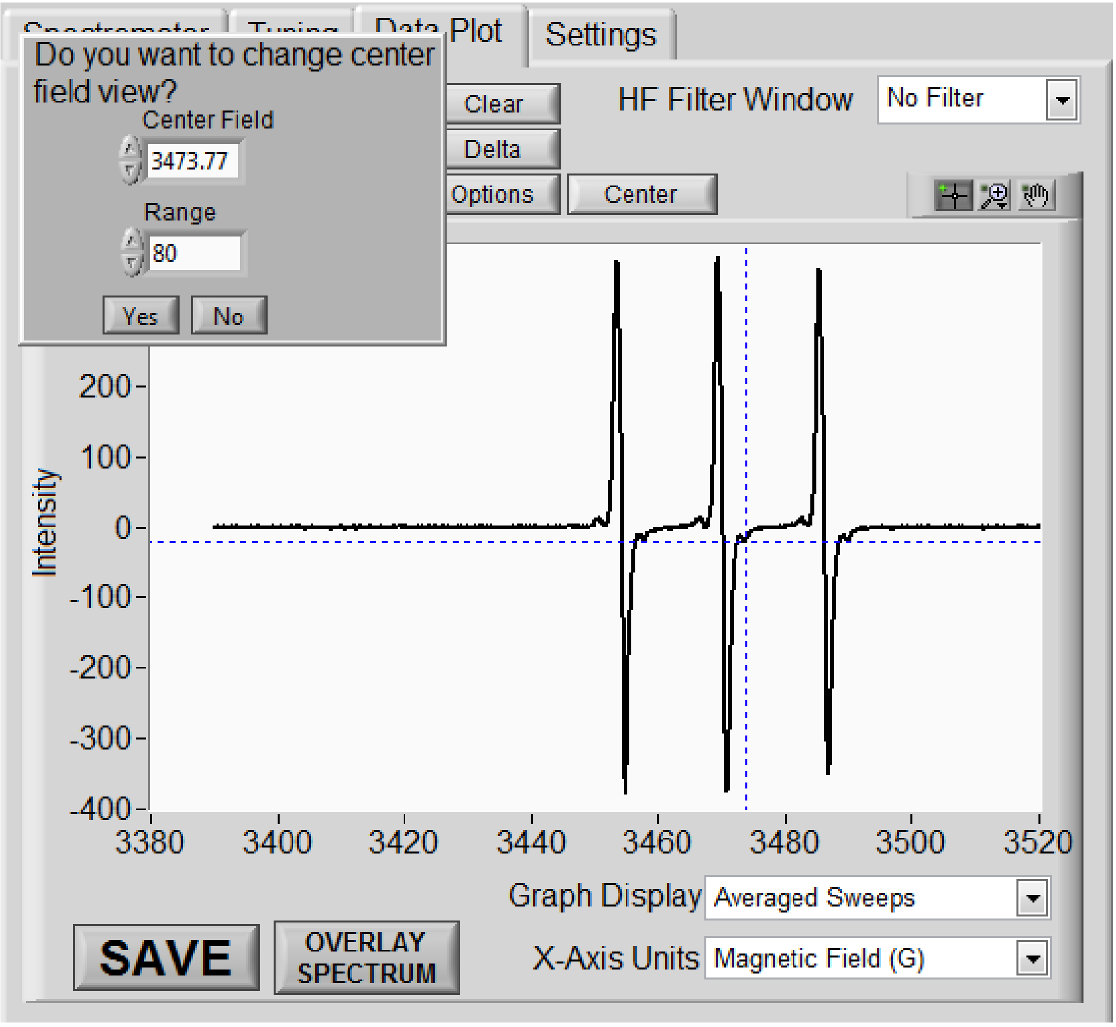


Figure 8.39: Cursor Selects the Desired Center, and Center Pop-Up Allows User to Set Range

- Use the cursor to select the desired center of the spectrum, and then click on the **Center** button. A pop-up will appear with the cursor selected center.

- Enter the range to set the desired spectral width, and click **Yes.** The new spectral width will be displayed:

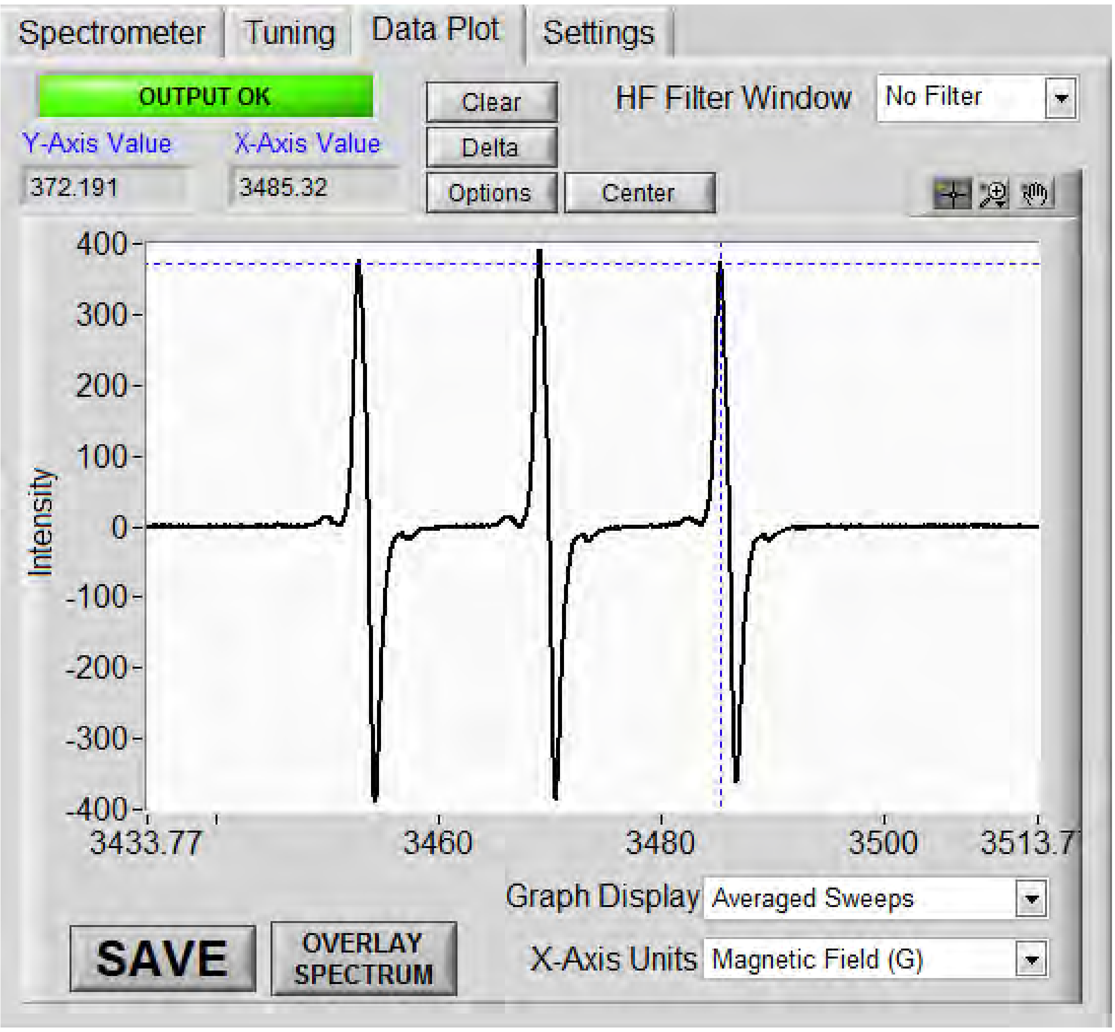


Figure 8.40: New Spectral Width Displayed after Center

The center can also be entered manually into the pop-up window.

The sweep width is displayed as center field and range (left), and as starting field and ending field (right) under the Spectrometer Tab:

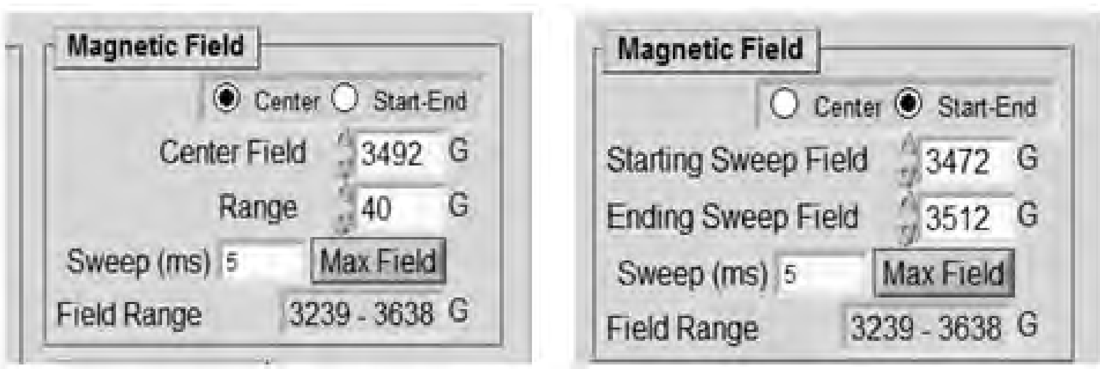


Figure 8.41: Magnetic Field Settings

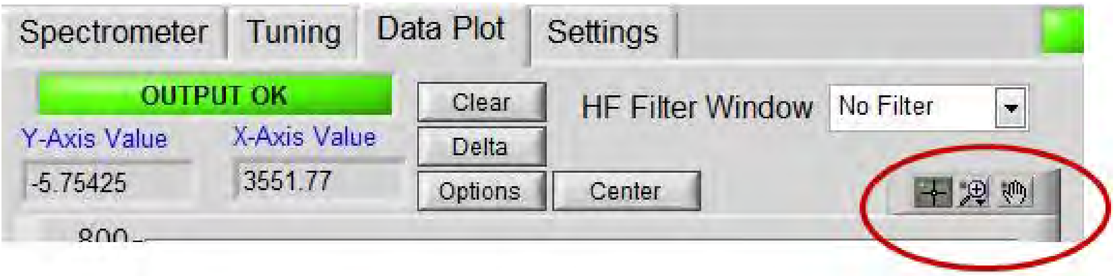


Figure 8.42: Tools to Allow the User to Expand and Move the Spectrum

Expand Spectrum

Go under **Options** and uncheck X and Y Autoscale. The interactive expand function does not work with X and Y autoscale checked:

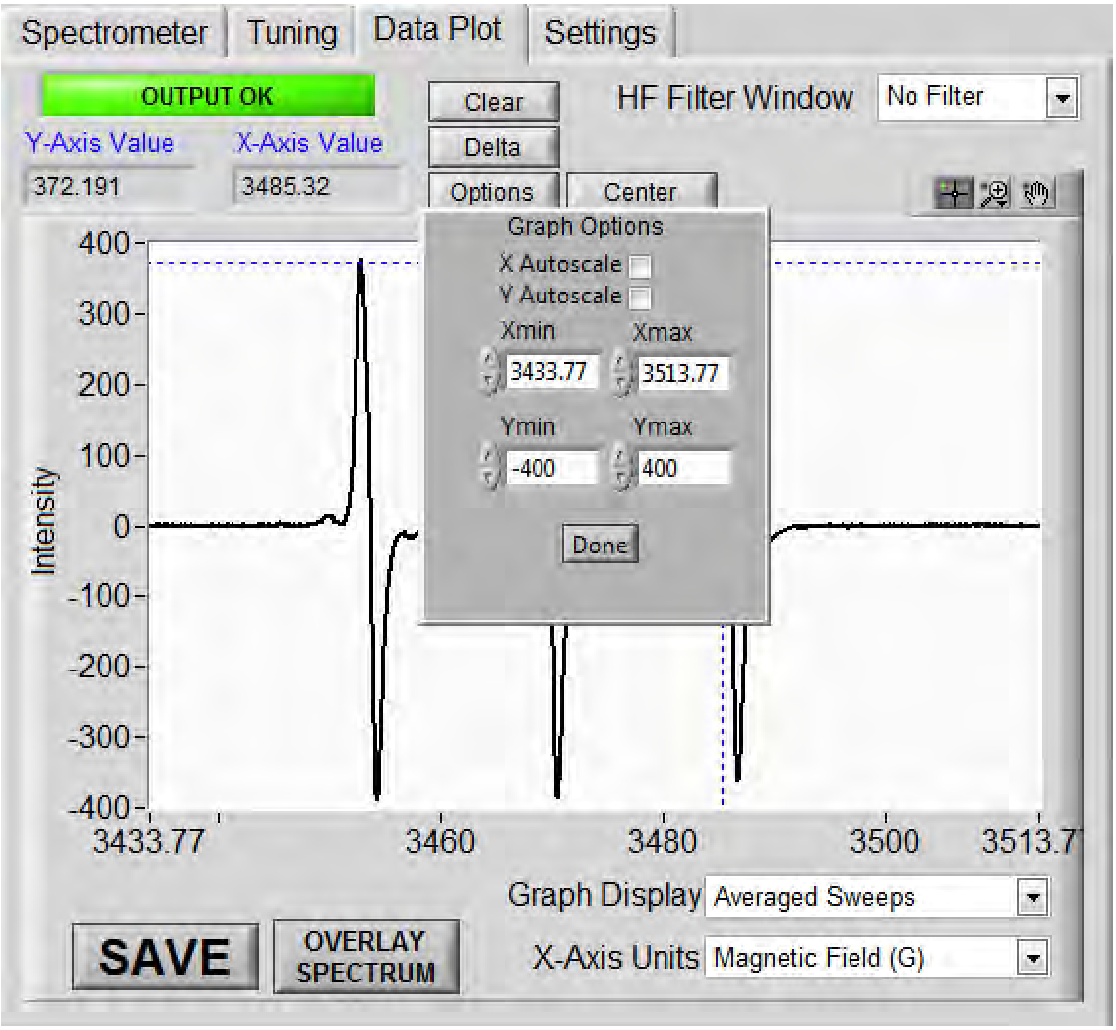


Figure 8.43: Autoscale must be de-selected for the expand tool to function

- Click on the center icon.
- Select top left icon from pop-up:

Figure 8.44: Pop-up displayed when the center icon is selected. Select upper left icon to expand spectrum.

- Drag to select area to expand:

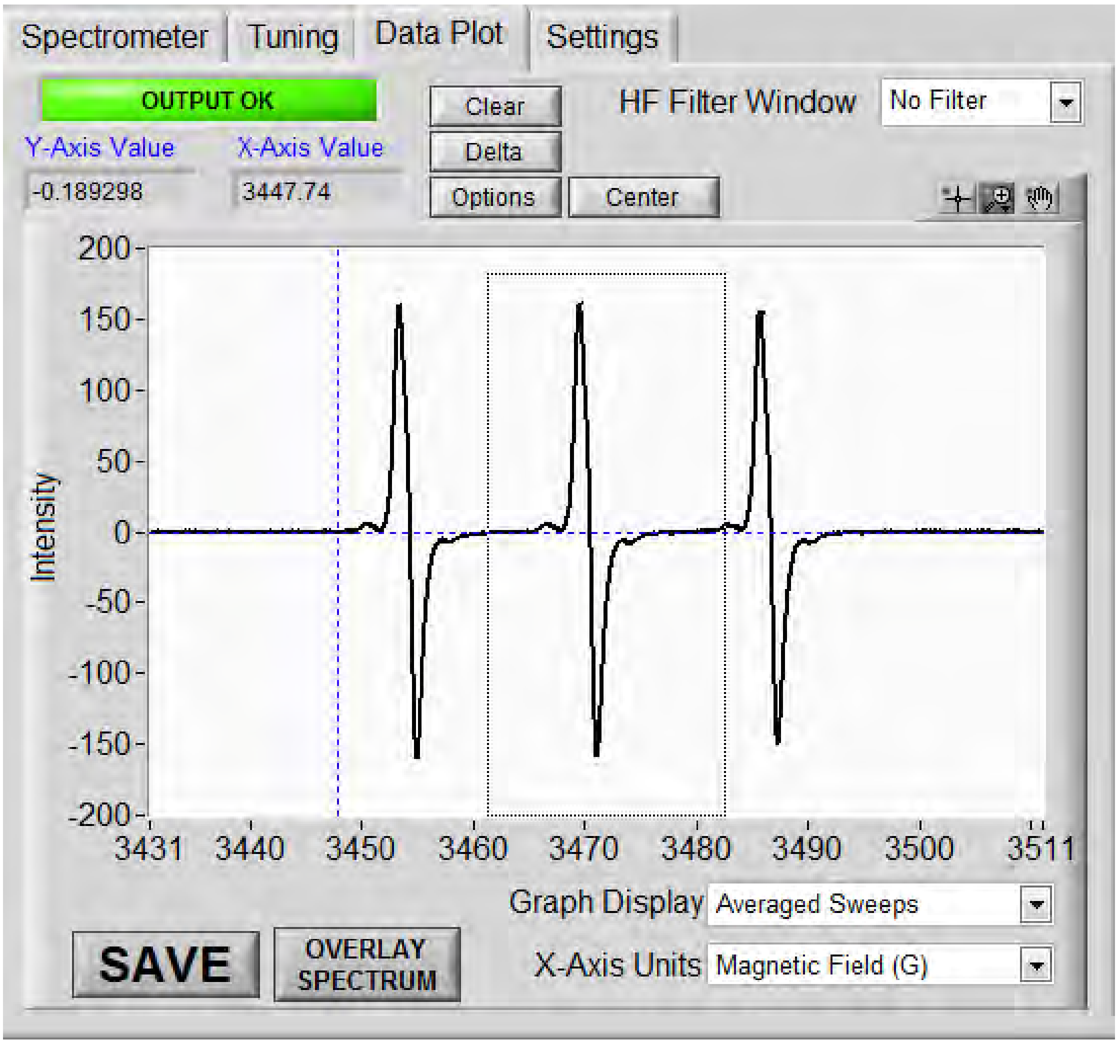


Figure 8.45: Drag mouse to select the area to expand

- Unclick the mouse button and the expanded spectrum will be displayed:

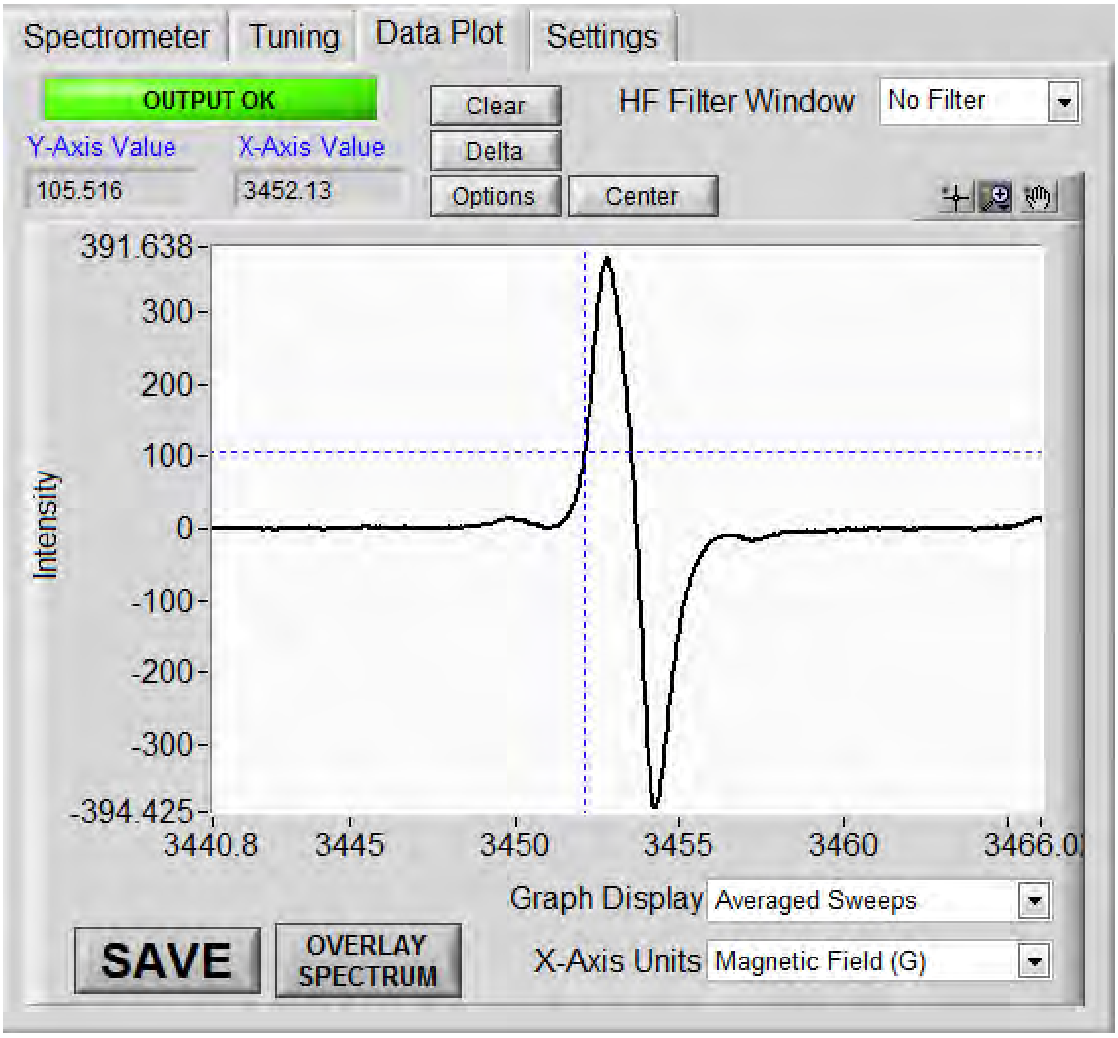


Figure 8.46: Unclick mouse to view expanded area

The bottom left icon in the pop-up (*Figure 8.44 [▸ 71]*) returns the spectrum to its standard size.

The left most icon (*Figure 8.42 [▸ 70]*) must be highlighted for the cursors to be active.

See “Guide to microESR Analysis and Processing Software” for more information on the tool.

Graph Display

Allows the data to be displayed as the averaged accumulated sweeps or the last single sweep. The Extended Range Benchtop also has a Partial Waveform option.

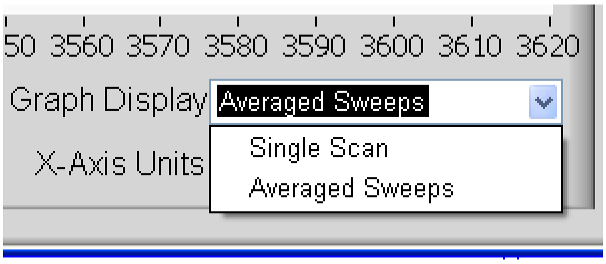


Figure 8.47: Graph Display Options

X-Axis Units

The X-axis can be displayed in magnetic field units of Gauss or mTesla, or g-Factor units (see *Appendix [▶ 111]* for examples).

SAVE

Option to save filtered data to a new file. Clicking on the **SAVE** button on the lower left will create another data file with the filtering turned on. The file will be named SampleName_FILT_date_time.ESR. A pop-up window will confirm that the file has been saved.

Figure 8.48: Saved File Message

OVERLAY SPECTRUM

Load a previously saved spectrum, and overlay on current plot. If the **Clear** option has been selected, then the spectrum will be displayed on the blank page.

Clicking on the **Overlay** button will bring up a file system window.

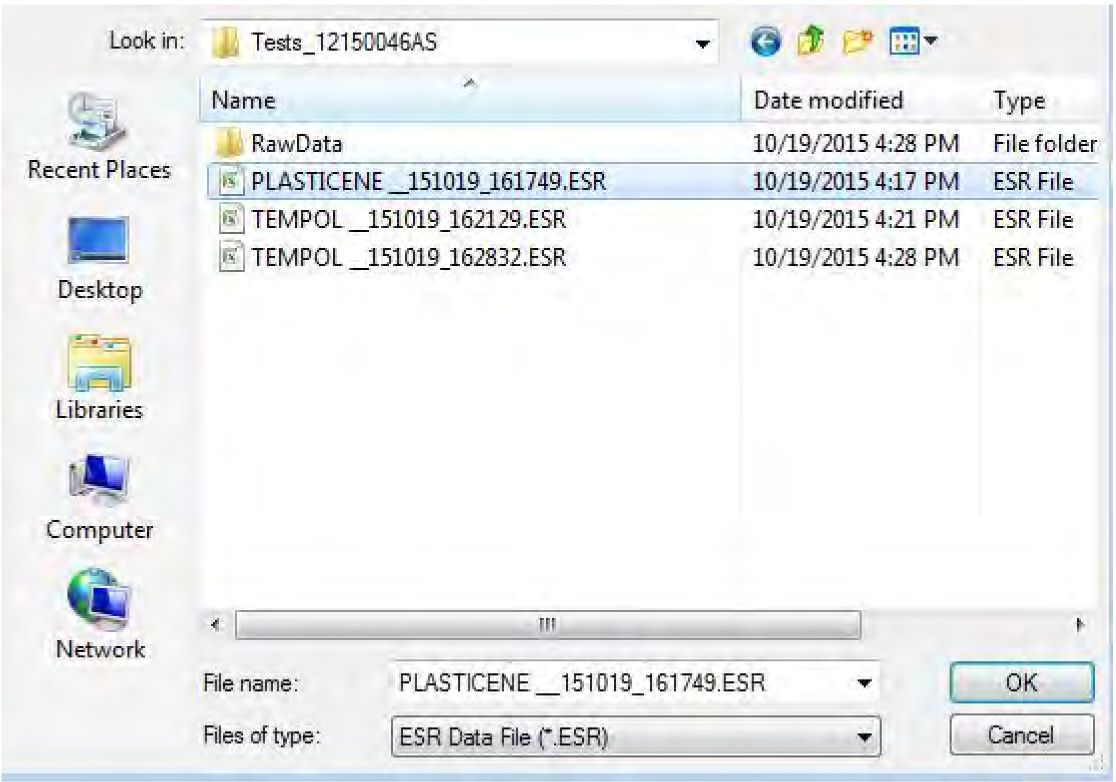


Figure 8.49: Overlay Data Set

- Select the file to be displayed and click **OK**.

The selected spectrum will be displayed on top of the existing spectrum. The original spectrum is in black, and the overlaid spectrum is in red:

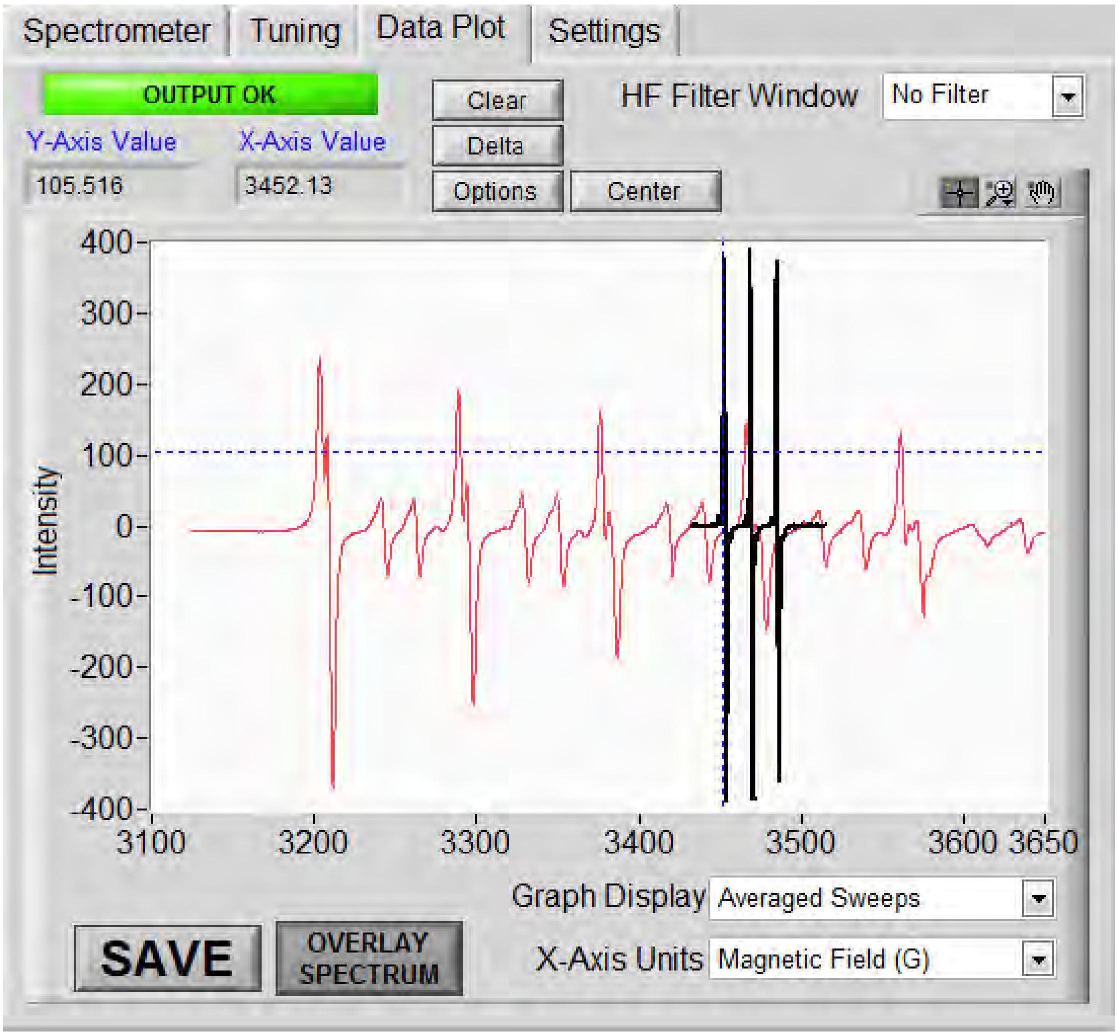


Figure 8.50: Overlayed Spectra

Clicking the **OVERLAY SPECTRUM** button (now dark) a second time, will remove the second spectrum. The button is a toggle.

The field of the display is always scaled to the largest field, and will return to the original scale when the second spectrum is removed:

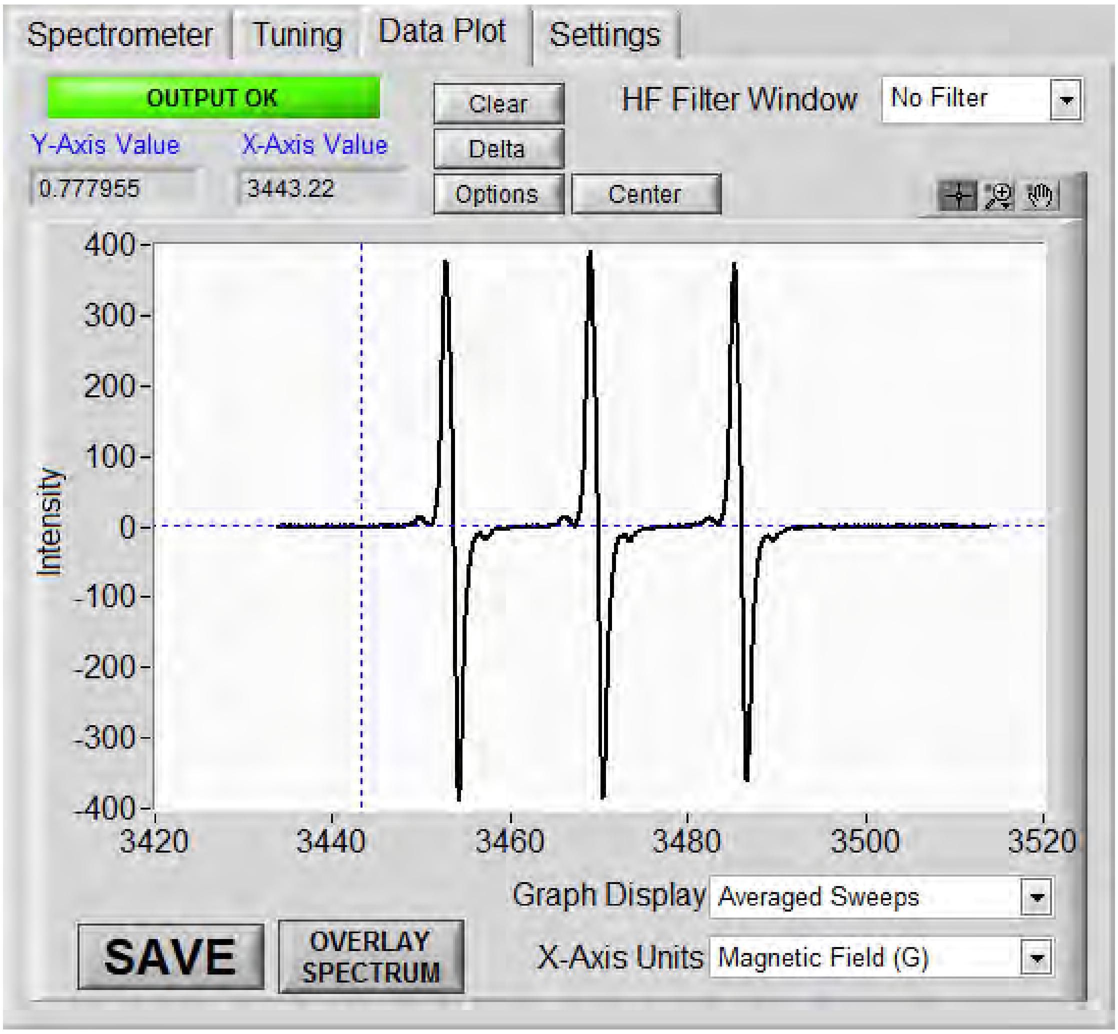


Figure 8.51: Auto-Scaled Spectrum

Clicking the **Clear** button will clear the screen. If **OVERLAY SPECTRUM** is now selected, only the second spectrum will be displayed.

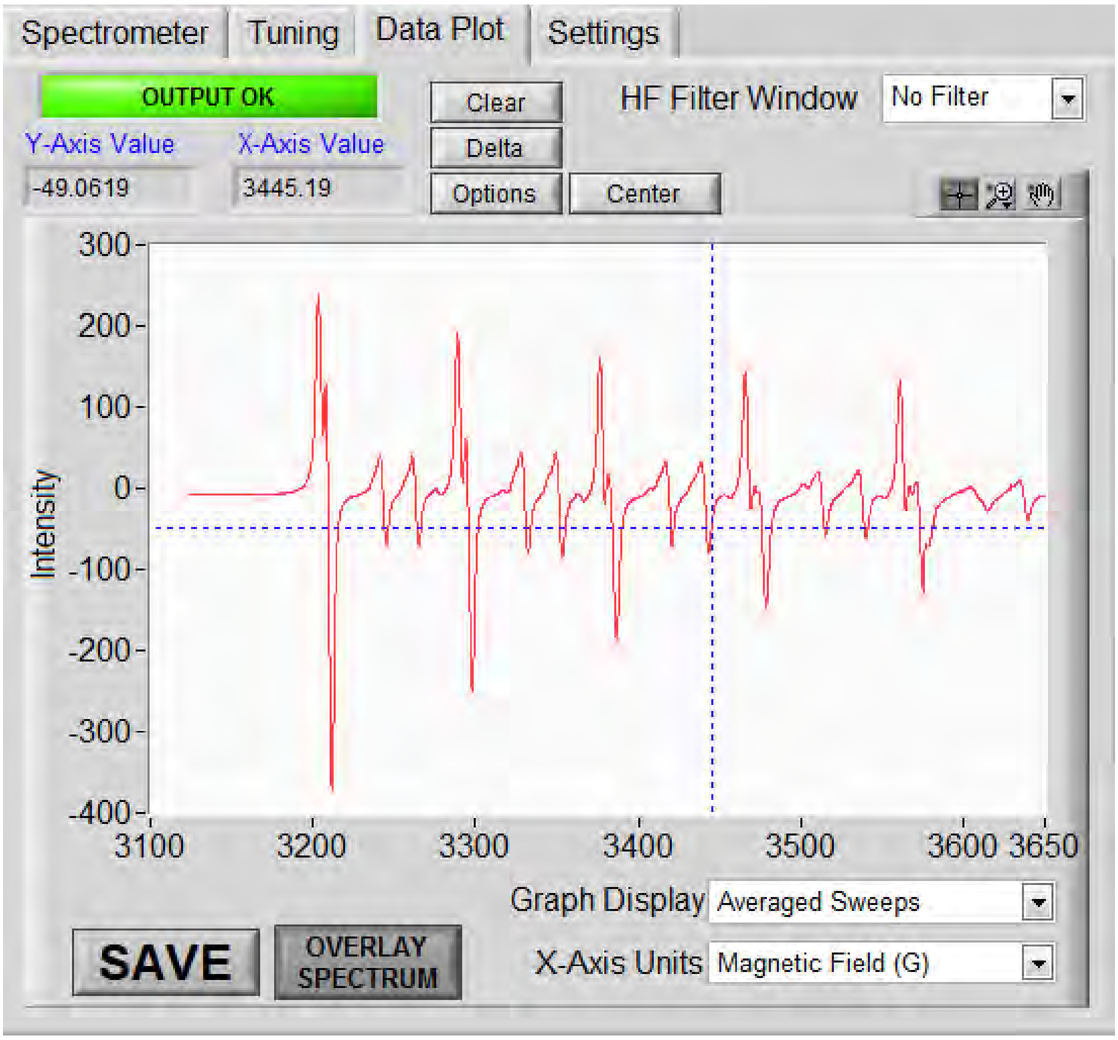


Figure 8.52: Spectrum Overlay

The **OVERLAY SPECTRUM** feature works while the spectrometer is acquiring.

A second spectrum can be displayed or removed while the acquisition is in progress.

8.4 Settings Tab

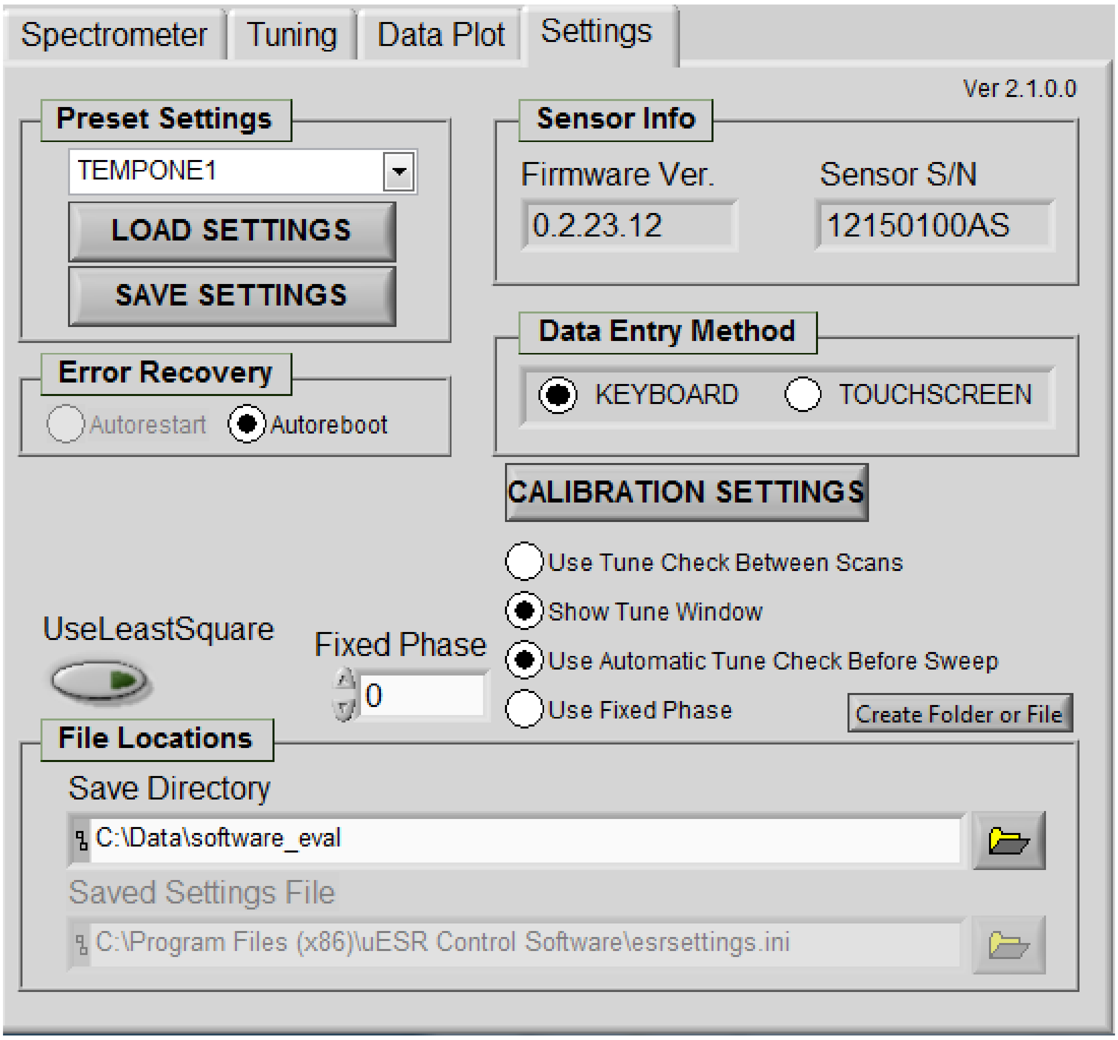


Figure 8.53: Settings Tab

8.4.1 Preset Settings

- Select from the pull down menu a previously saved parameter set.

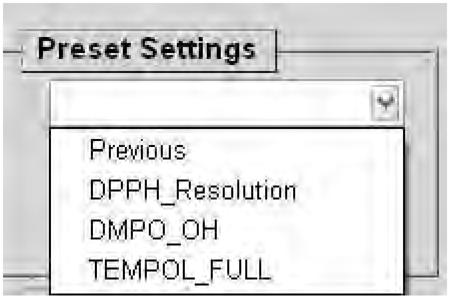


Figure 8.54: Preset Settings

LOAD SETTINGS

Loads the settings from the selected parameter set.

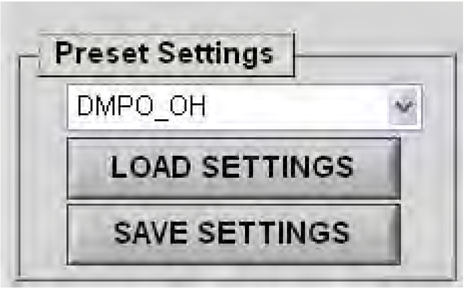


Figure 8.55: Preset Settings

SAVE SETTINGS

Saves parameters from the current parameter set. Clicking on **Save Settings** will make a pop-up window appear. Enter a name for the parameter set, and click **Done**.

Figure 8.56: Save Preset Settings

All saved parameter sets, or settings, are saved in the esrsettings.ini file. An example file is in the appendix.

8.4.2 Error Recovery

Autorestart

Restarts the acquisition in the event of an error.

Autoreboot

Reboots Windows in the event of an error.

8.4.3 Use Least Squares

Uses a least squares fit program when the green arrow is highlighted. By default, this feature is not turned on.

8.4.4 File Locations

Save Directory

Specifies path to where data will be saved.

Saved Settings Files

Path to where the saved settings (parameters) are stored.

8.4.5 Sensor Info

Firmware Version

Current version of the uESR control software.

Sensor S/N

Serial number of the spectrometer.

8.4.6 Data Entry Method

Keyboard

Allows values to be typed in using a keyboard.

Touchscreen

When parameter field is selected, a pop-up will appear allowing values to be changed using the touchscreen. Refer to the next two figures.

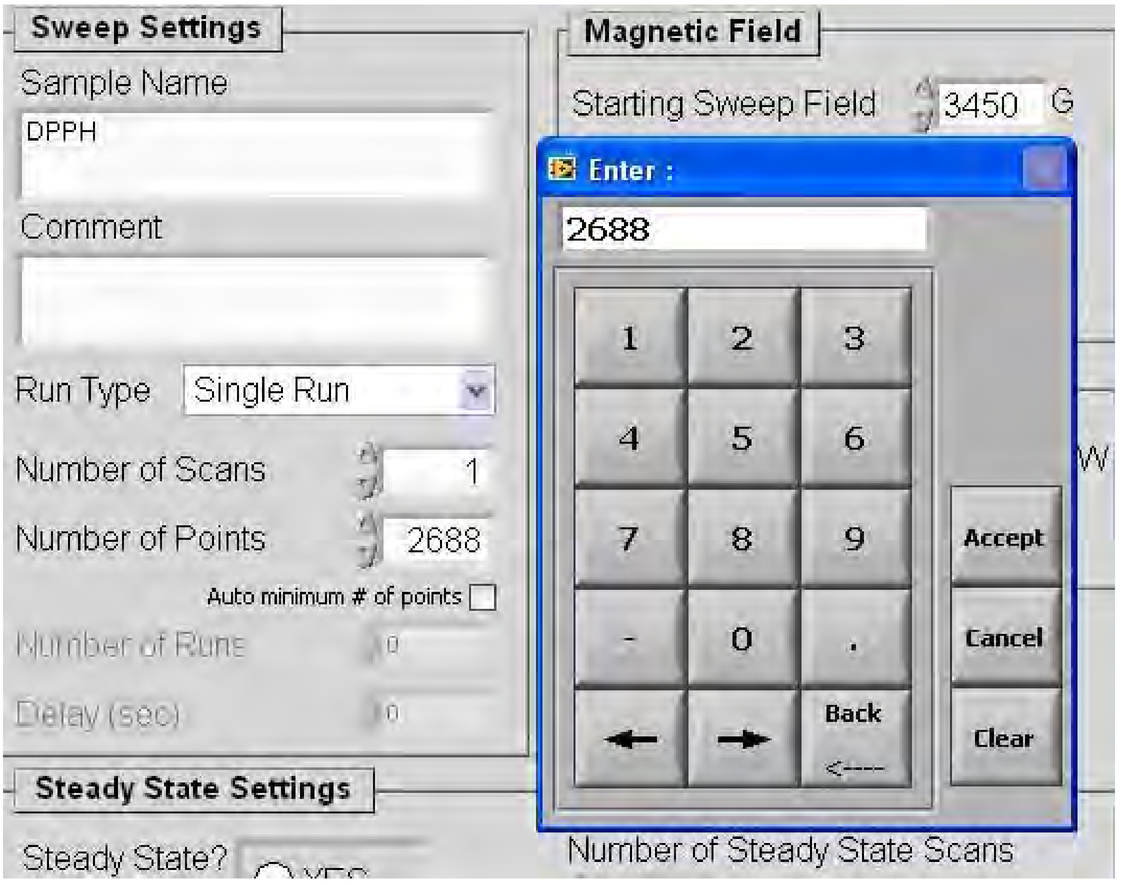


Figure 8.57: Pop-Up Keypad

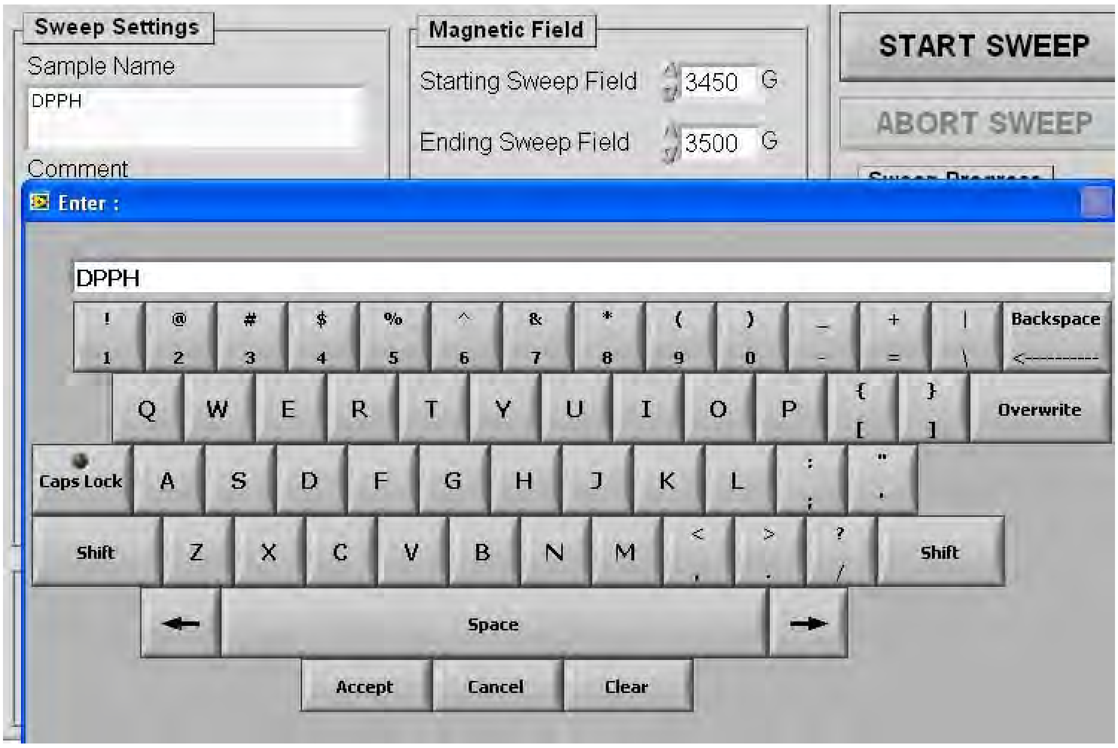


Figure 8.58: Pop-Up Keyboard

The full key pad is displayed for text, and the numerical key pad for entries requiring only numbers.

CALIBRATION SETTINGS

Brings up a window that displays the calibrations that are in the firmware:

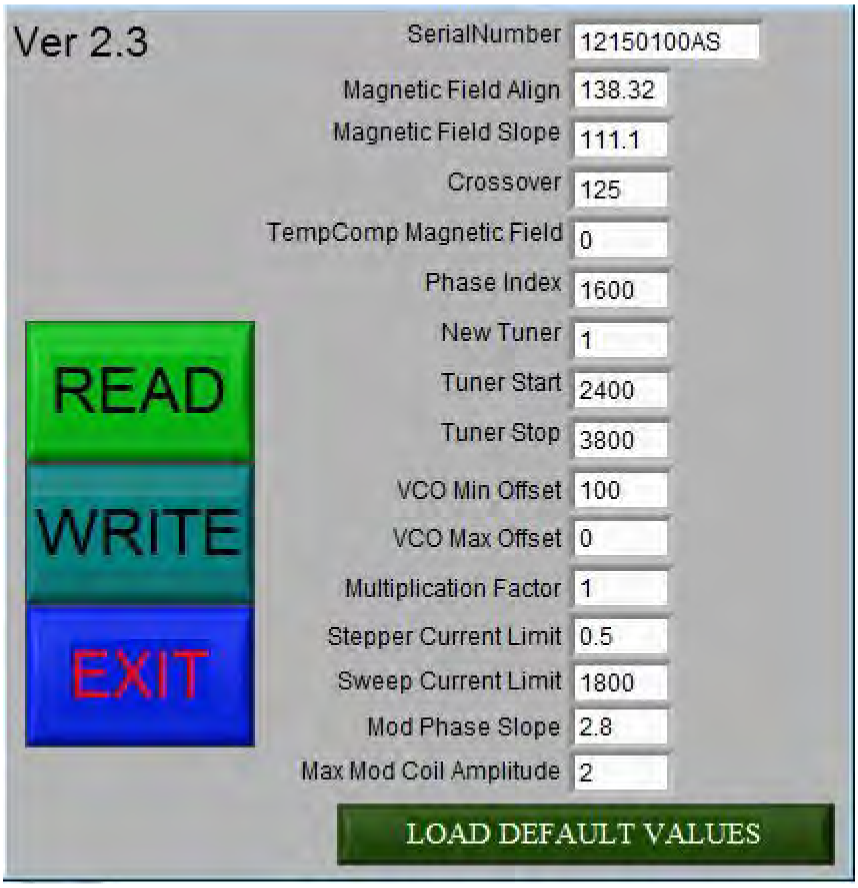


Figure 8.59: Calibration Interface for Firmware

These calibrations are set in the factory, and can be changed from this window. We do not recommend changing these settings without guidance from a field service engineer. See the *Appendix [▸ 111]* for details about calibrations and how to change them.

Use Tune Check Between Scans

When this button is checked, the software will check the minimum reflected tune power before each scan. This feature should be used if the sample may be slowly changing temperature over the run. The sample needs to be close to thermal equilibrium before tuning is started; however, small temperature changes can be compensated for. The **Use Automatic Tune Check** button must also be selected for this feature to work.

Show Tune Window

Displays the tune window when checked. See section *Auto Tune Routine [▸ 57]*.

Use Automatic Tune Check

When either Continuous Runs or Multiple Runs with Delay have the tune before each run selected, the software will check the minimum reflected power and the noise floor before automatically tuning. If the values are below the specified values, the acquisition starts without retuning. If either value is higher, then Fast Re-Tune is run. The values for minimum reflected power and noise floor are then rechecked. If either value is higher than the specified value, the standard tune routine will be run; otherwise, the acquisition will proceed without further tuning. The values are in the Tune pop-up.

Use Fixed Phase

When the Use Fixed Phase button is selected, the tune algorithm will set the phase to the value in the box to the left labeled “Fixed Phase”. The algorithm will then eliminate the fine tuning step. NOTE: If the selected phase is not correct, the spectrometer will not tune. This feature is NOT recommended for most uses.

Create Folder or File

Allows the user to create a folder or file in windows from the software.

- Click on the **Create Folder** or **File** button (see *Settings Tab [▸ 78]*).
- Type the exact path where the folder or file is to be created.
- Enter the file or folder name.
- File names need an extension or the software will create a folder:

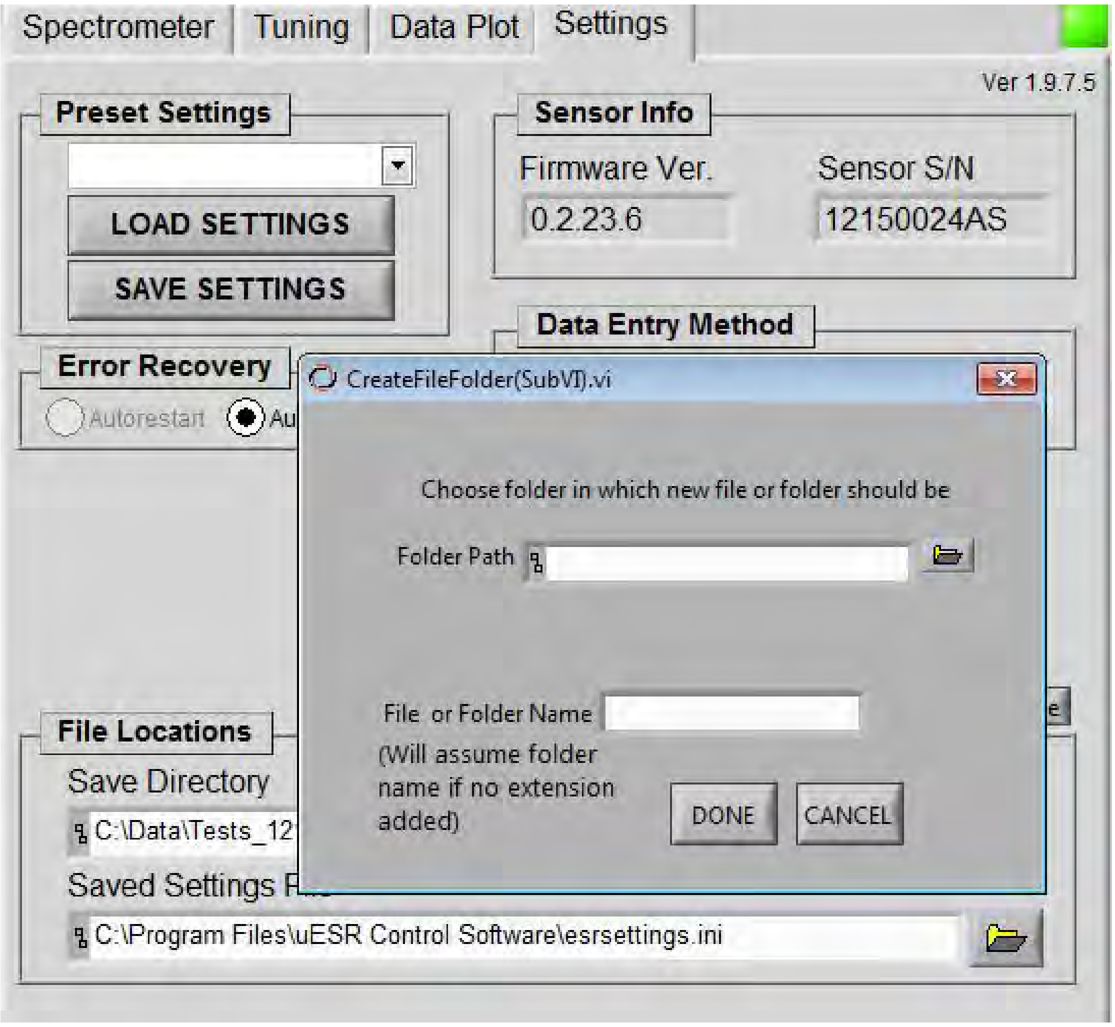


Figure 8.60: Pop-Up to Create Folder or File in Windows

8.5 Sweep Control Buttons

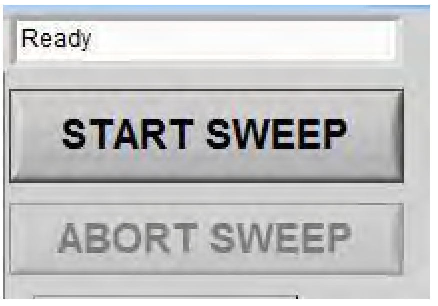


Figure 8.61: Sweep Control Buttons

8.6 System Status

Displays the current status of the system.

Ready

System is idle and ready to use (see *Sweep Control Buttons [▸ 84]*).

Preparing to Run:

Figure 8.62: Sweep Status – Preparing to Run

Warming Up…

Running Steady State scans. The system is on scan 1 of 4 requested scans.

Figure 8.63: Sweep Status – Warming Up

Sweeping Spectrum Run # n

Acquiring data and writing to disk. If multiple runs have been selected, the software keeps track of the current run number (n):

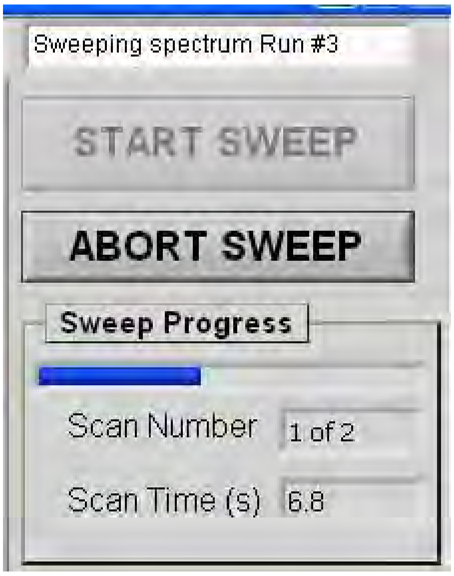


Figure 8.64: Sweep Status – Sweeping Spectrum Run #-n

START SWEEP

Starts acquisition. Will automatically start acquisition if **Start Sweep After Successful Lock** was selected in the AutoTune window.

ABORT SWEEP

Stops the acquisition. The button will be grayed out (as shown in *Spectrometer Tab [▸ 39]*) unless the spectrometer is running.

8.7 Status Window and Output Data

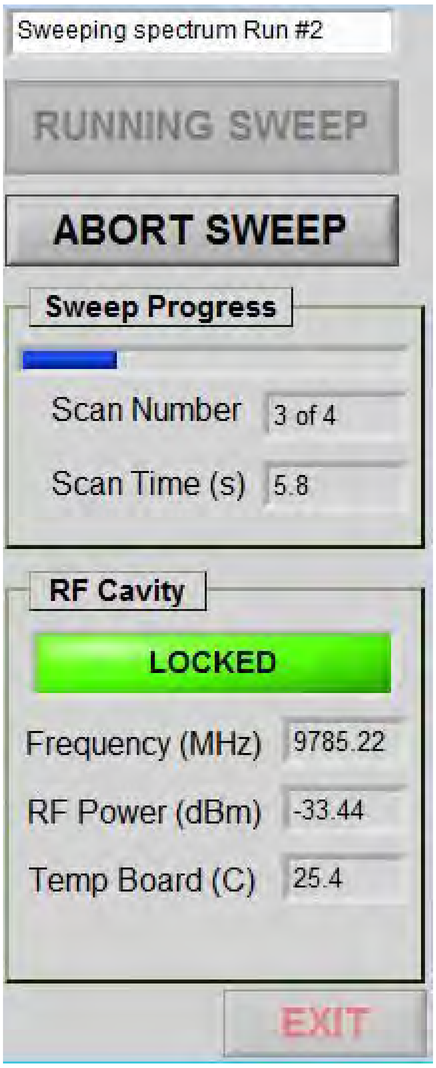


Figure 8.65: Status Window

8.7.1 Sweep Progress

If the multiple run option has been selected (section *Sweep Settings [▹ 40]*), the current run number is listed.

The blue line travels across in real time as the field is swept.

Scan Number

Lists the current scan the spectrometer is running out of the total number of scans requested. If the status says Warming Up (see *Figure 8.63 [▹ 85]*), then the numbers displayed will be the current steady-state scan the system is running out of the total requested steady-state scans.

Scan Time

The total time for one complete scan to run in seconds. The time will be a function of the sweep width and the number of points selected. The time per scan is about 50 msec per point.

RF Cavity

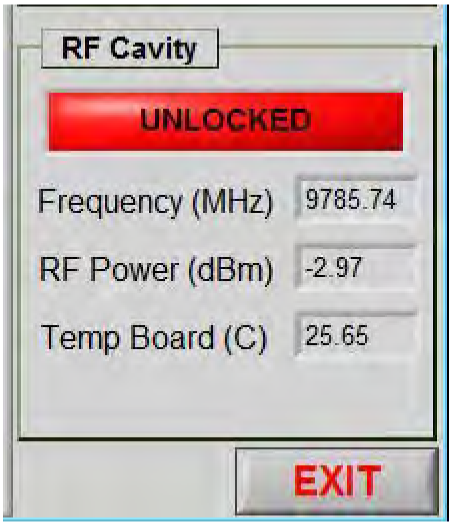


Figure 8.66: Unlocked RF Cavity

LOCKED

AFC is locked to resonator. See *Figure 8.65 [▸ 86]*.

UNLOCKED

The green **LOCKED** button will turn red and say UNLOCKED if critical coupling has been lost.

No useable data will be acquired if the spectrometer is not locked. The spectrometer will not start acquisition if not successfully locked from the Auto Tune routine; however, clicking on the **Start Sweep** button will start acquisition if the spectrometer is locked or not.

Frequency (MHz)

The locked resonator frequency.

RF Power (dBm)

Power of the RF carrier signal in the resonator. The lower this number, the better the critical coupling.

Temp Board (C)

Reading from the TEC temperature controller inside the magnet.

9 Cavity Dimensions

The figure below shows the dimensions of the spectrometer sample chamber. The small circle in the middle is the resonator cavity. For optimal results, the cavity should be fully loaded with sample. You can use this diagram to position an O-ring on the sample tube such that the sample is centered in the resonator cavity. The position of the O-ring will depend on the length of the sample tube, and the amount of sample in the tube. Not all samples will require an O-ring.

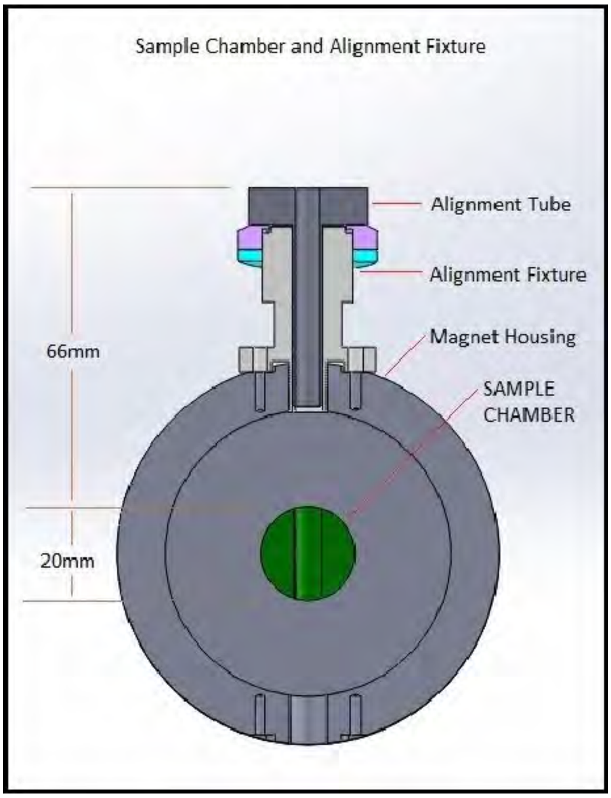


Figure 9.1: Cavity Dimensions

With no O-ring, the blue area of the tube will be in the resonator when a standard tube guide is used. If the sample depth is greater than 30 mm, the cavity will contain the maximum amount of sample. If the sample is shorter than 30 mm, then an O-ring should be used to center the sample in the resonator. Use the diagram to determine where to place the O-ring.

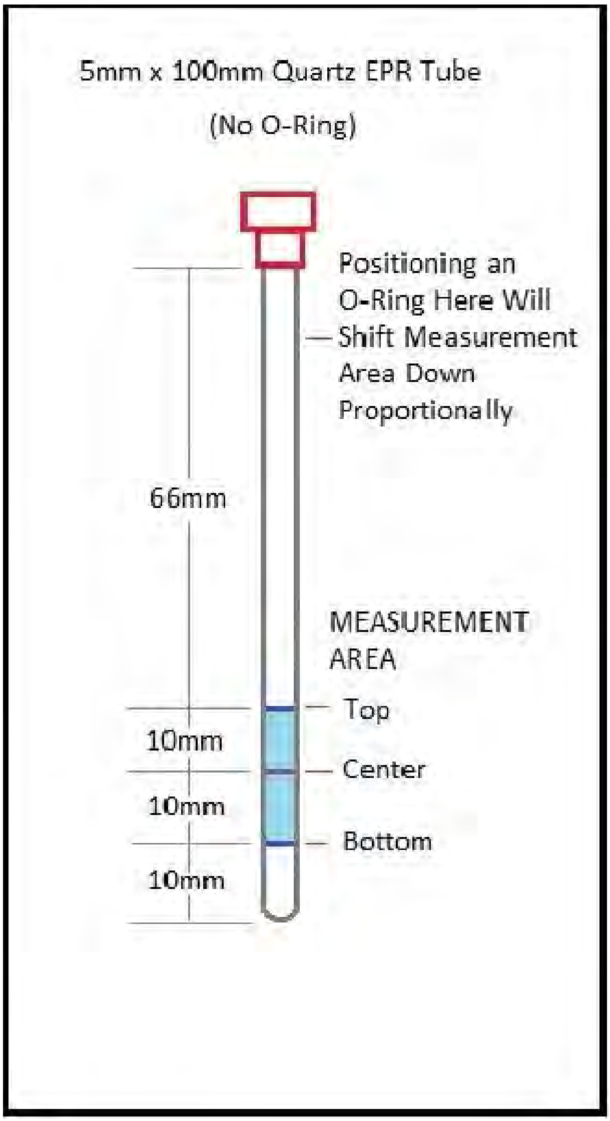


Figure 9.2: Standard 5 mm ESR Tube Dimensions.

10 Optional Accessories

10.1 ESR at 77K Using the Finger Dewar

This application allows the user to easily run samples at 77 K using the liquid nitrogen finger dewar.

Sample preparation and handling

- Samples need to be loaded into 1.7 mm capillaries.
- Samples can be liquid, solid, emulsions, etc. Gas phase samples would need special tubes.
- We recommend freezing the sample in the capillary in a LN2 bath before placing in the dewar.
- Partially fill the dewar with LN2, insert the sample, and then refill the dewar.
- The dewar can be topped off while the experiment is running.
- An O-ring is used to adjust the height of the dewar.
- The figure below shows the liquid nitrogen dewar and capillary. The O-ring is used to adjust the height of the dewar.

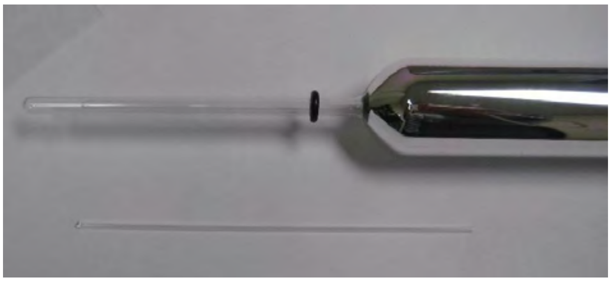


Figure 10.1: The LN2 Dewar and Capillary

- The next figure shows the spectrum of crude oil at 298 K (A) and 77 K (B). Note the increase in the vanadium signal at 77 K.

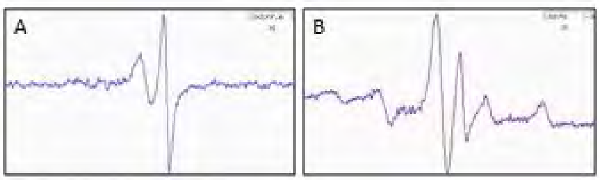


Figure 10.2: Crude Oil at 298 K (A) and Crude Oil at 77 K (B)

10.2 ESR with Ultra Violet Irradiation

The fiber optic connection supplied with this kit allows the user to irradiate the resonator cavity with an ultra violet (UV) light source. UV light has enough energy to initiate many radical reactions, so this feature enables the user to initiate reactions in situ. Light sources other than UV can also be used, but may require a different fiber optic cable. All fittings in this kit are standard SMA.

Sample preparation and handling

There is no special sample prep required.

Installing the fiber optics

- Shut down the spectrometer, and turn off the power.
- Unplug the spectrometer from its power source.
- Remove the four screws from the base of the spectrometer, and remove the base.
- Gently, place the spectrometer on its side.
- If you purchased the UV kit after the spectrometer, skip to the next section.
- There will be a port with an SMA connector on the bottom of the magnet. Screw the SMA on the fiber optic cable to the SMA connector on the bottom of the magnet.
- Thread the fiber optic cable out of the AUX port on the back of the microESR.
- Be careful not to exceed the bend radius on the fiber optic cable as it could damage the cable.
- Replace the base and the four screws.
- Attach the other end of the fiber optic cable to your light source.
- Operate the microESR normally.

The following figure shows the fiber optic cable with the SMA connector, that is screwed into the adapter at the bottom of the magnet. The adapter will already be mounted to the bottom of the magnet if the spectrometer was purchased with the UV Kit (section in the red circle).

Figure 10.3: The Fiber Optic Cable, with SMA Connector and Adapter

Installing the fiber optics if the UV kit was purchased after the spectrometer

- The fixture that mounts to the magnet will need to be installed.
- The figure above shows the fixture not yet mounted to the magnet. The fixture should be mounted to the magnet before the fiber optic cable is attached.
- The process is straight forward, and the directions are included with the kit.
- Once the fixture is installed, proceed as described above.

Removing the fiber optic cable

- Shut down the spectrometer, and turn off the power.
- Unplug the spectrometer from its power source.
- Remove the four screws from the base of the spectrometer, and remove the base.
- Gently, place the spectrometer on its side.
- Unscrew the SMA on the fiber optic cable from the fixture on the bottom of the magnet.
- The fixture can remain in place, but if there is a need to remove it (as to install something else), then just unscrew the nut on top of the fixture, and remove the attachment with the SMA connector.
- Make sure to store the fiber optic cable in a manner such that the bend radius is not exceeded.
- Replace the base and the four screws.

Other frequency light sources

- Other frequency light sources can be used.
- If a lower frequency light source is used, the optimal fiber optic will be different. Any fiber optic cable with a standard SMA connection will work.

References

- UV Kit Manual.

10.3 microESR VT Control Using the ThermoCube

The ThermoCube is a thermoelectric heater/ chiller unit that can be interfaced to the microESR. The ThermoCube allows temperature regulation in the range of 10 to 55 °C. The unit comes with a custom adapter kit which allows the unit to interface with the microESR and an inert gas source. The unit needs about 80 psi of dry compressed air or nitrogen gas and a flow rate 1-2 L/min.

10.3.1 Sample Preparation and Handling

- Samples need to be placed in 2 mm quartz EPR tubes which fit the custom tube holder.
- There is no other special preparation or handling required.

10.3.2 Start-up

- Turn on the power, and wait for windows to boot.

- Double click on the Bruker microESR icon on the desktop. This will start the acquisition software. The Spectrometer Tab in the Acquisition Software allows the user to set up experimental parameters:

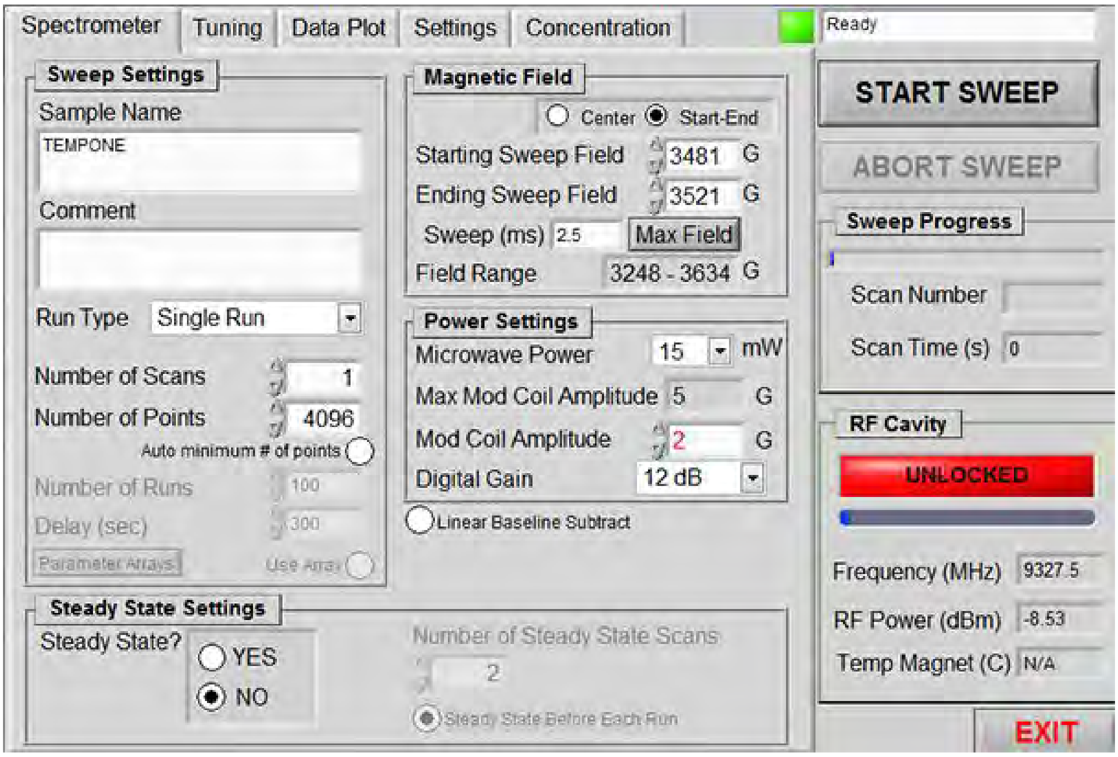


Figure 10.4: Spectrometer Tab in the Acquisition Software

10.3.3 Setting Up the ThermoCube

Figure 10.5: The Thermo Cube Thermoelectric Heater Chiller

- The ThermoCube is approximately the same size and mass (~ 10 kg) as the microESR spectrometer.

- It is important to position the ThermoCube as close to the microESR as possible. This helps reduce loss in heat or cooling from the ThermoCube to the sample.
- Fittings are all CPC, so are easy to attach and detach.
- The air temperature in controlled by the ThermoCube, but the sample temperature can be monitored via a thermocouple.
- The airflow adapter is easily installed on top of the spectrometer replacing the standard sample holder and fitting. A cover (not shown in the next figure) screws onto the top of the assembly.

Figure 10.6: Airflow Adapter Shown Installed on the microESR

- A PTFE tube surrounds the sample.
- An O-ring holds the tube securely in place.
- The hose from the output of the ThermoCube attaches to the fitting on the airflow adapter via a CPC fitting:

Figure 10.7: Attaching the Air Hose from the TermoCube via CPC Adapter to the Air Flow Adapter (shown with cover)

- The next figure shows the hose used to connect the ThermoCube to the microESR. On one end is the coupling body and on the other end the coupling insert.

Figure 10.8: Hose Connecting the ThermoCube to the microESR

- The figure below shows the air connections on the ThermoCube. PROCESS OUT is the air going to the microESR and PROCESS IN is the air coming from the compressed air source.

Figure 10.9: ThermoCube Connections

10.3.4 Starting Up the ThermoCube

- Plug line cord into 110 VAC or 230 VAC, 50/60 Hz
- Turn on the switch located on the left side. The front display should read the current process gas temperature.
- Start the Process Gas flow.

10.3.5 ThermoCube Operation

The ThermoCube is operated via the control panel located on the front panel.

The control panel has a 16-character LCD display and four input keys: UP, DOWN, ENTER, and START. These keys work as follows:

Key	Action
UP	Pressing the UP key raises the parameter value displayed.
DOWN	Pressing the DOWN key lowers the parameter value displayed.
ENTER	Pressing the ENTER key momentarily enters the parameter changed.
ENTER	Pressing and holding the ENTER key for 3 seconds changes the LCD display menu.
START	Pressing the START key turns on temperature control.
START	Pressing the START key while the chiller is operating turns off temperature control.

The ThermoCube comes with preset operating parameters that will work well for most applications.

If temperature control at one temperature is desired, follow the steps below:

- Turn on ThermoCube and wait for display to read TEMP.
- Press the **UP** or **DOWN** keys to change SETTEMP 1 to the desired set point.
- Press the **START** key.

The ThermoCube will now control to the set point temperature.

To change the set point temperature, press the **UP** or **DOWN** keys again to change SETTEMP 1 to the new set point, followed by the **START** key.

To program multiple temperatures, see the ThermoCube 300/400 Air/N2 Manual that ships with the system.

10.3.6 References

- The VT Installation Manual, shipped with the VT Kit.
- The ThermoCube 300/400 Air/N2 Manual, SOLID STATE COOLING SYSTEMS, 167 Myers Corners Road, Wappingers Falls, NY12590. Delivered with system.

10.4 Directions for Testing the ThermoCube

10.4.1 Factory Set-up

The factory should set up an attachment to an 80 psi dry air or N_2 source that can be connected to the Process In port on the ThermoCube.

The factory should also have a hose that plugs into the Process Out port of the ThermoCube for testing.

The ThermoCube does not need to be connected to the microESR to test.

10.4.2 ThermoCube Set-up

- Remove the ThermoCube from the box.
- Connect the hose from the PROCESS IN connection to the house air or nitrogen.

Figure 10.10: Connections on the ThermoCube

- Connect the “dummy” hose to the PROCESS OUT port of the ThermoCube.
- Plug the power cord into the 110 VAC or 230 VAC, 50/60 Hz connection.
- Turn on the switch located on the left side of the unit. The front display should display the current process gas temperature.
- Start the process gas flow.

10.4.3 Testing the ThermodCube

- Set the SETTEMP1 to 37 °C.
- Wait until the temperature stabilizes.
- Measure the temperature at the PROCESS OUT hose with a thermocouple, and note the difference between the measured temperature and the SETTEMP.
- Set the SETTEMP1 to 37° + the measured difference in temperature.
- Once the temperature has stabilized, measure the temperature at the PROCESS OUT hose. It should now measure 37 °C.
- Make sure the thermocouple is securely in the air stream.
- Press **START** and make sure the temperature regulation turns off (note: the unit will still be powered on).

10.4.4 ThermoCube End Test Protocol

System: ______________________ Date: ______________ Test Engineer: __________________

Software Version: ______________ Firmware Version: _______________

ThermoCube			
	Comment	**Target**	**Actual**
Power On		Pass/Fail	
Responds to Keys		Pass/Fail	
Temperature Stable at 37 °C	On ThermoCube TEMPSET	Pass/Fail	
Temperature at PROCESS OUT Hose	This will likely be a bit lower than 37 °C because of thermal loss through the hose. Will depend on hose length	Report temperature at the PROCESS OUT hose.	
37 °C at PROCESS OUT	Temperature reading stable on ThermoCube.	Yes/No	
	Actual Temperature Reading at PROCESS OUT.	Record value.	
Temperature Control Turns OFF	Pressing **START** when the temperature is regulated turns off the heater/chiller.	Yes/No	

microESR Side			
	Comment	**Target**	**Actual**
Internal VT Parts	Install the second tube alignment fixture, diffuser, and thermocouple attachment.		
Assemble Flow Adapter	Assemble the flow adapter with O-rings and CVC couplings.	See figure below.	
Check Assembly with PTFE Tubing.	Insert the PTFE tubing and install the airflow adapter fixture.	Check that the PTFE tubing fits, and is easily removed.	
Ship with Standard Fixtures	Uninstall the VT fixture from the top of the spectrometer, and ship with the standard black beveled ring and nut.		

Accessories		
Accessory	**Comment**	**Actual**
ThermoCube with CPC Connectors.		

Accessories		
Accessory	**Comment**	**Actual**
Assembled Flow Adapter.	Ship with cap screwed on.	
White Tapered Washer.		
PTFE Tubes (2).		
Vacuum Tube (1).	Expensive and fragile.	
O-rings -1.5 X 2.0 VT.		
O-rings - 2.4 X 5.6 VT.		
2 mm x 250 quartz ESR tubes (10).		
CPC Brass Coupling Inserts (2).		
1/4” ID x 3/8 OD Vinyl Hose.		

Figure 10.11: Assembled Flow Adapter without Cap

Documents		
Document	**Comment**	**Actual**
Installation Manual.		
ThermoCube SOP.		
ThermoCube 300/400 Air/N2 Manual.	Comes with ThemoCube.	

11 Maintenance

This unit has no user-serviceable parts, and can only be repaired by a qualified service engineer. Never attempt to open the spectrometer. In the event of a malfunction, please contact Bruker.

11.1 Cleaning

Before cleaning:

- Stop the device from doing any action.
- Switch the power off.
- Disconnect the power supply.

11.1.1 Cleaning the Outside of the Device Chassis and Units

Do not use any detergent or other cleaning solvents. Use only water or neutral cleaning fluids. Usage of volatile cleaners like thinner or benzene may damage the surface of the unit.

1. Clean the outside of the device chassis and units with a soft, lint-free cloth dampened in water.
2. Wait until the unit is completely dry before you reconnect the power cable!

11.1.2 Other Cleaning Operations

For all other cleaning operations contact Bruker service for advice and support. It may be necessary to send in the device for a cleaning service.

No special precautions have been taken in the device to avoid contamination from leaking samples. Bruker accepts no responsibility for any damage which may occur when samples are used containing radioactive or other hazardous materials.

In case of an accident with toxic, radioactive, explosive, or biologically active substances, the device and associated equipment must be cleaned in such a way that no danger emanates from the device and associated equipment, especially for all uninformed personnel. If a device has to be cleaned of all remains of a substance for safety reasons, contact Bruker service for advice and support.

Note that in serious cases it may be necessary for the owner to exchange the device with a new one, contact Bruker service for details.

11.2 Preventative Maintenance

All parts in the device have been designed to work reliably without preventative maintenance.

12 Troubleshooting

12.1 Basic

It is best to have a known sealed, stable sample to use for troubleshooting. The Cr(III) Oxide sample as well as the Mn(II) impurity in plasticene that comes with the spectrometer can be used.

A spectrum of both samples should be acquired at the time the instrument is received, and saved. The specific samples used should also be put somewhere safe.

The spectrometer also comes with a 50 µM aqueous TEMPONE sample which should be run when the spectrometer is received. This sample does not have an indefinite lifetime.

The parameters used to acquire the spectra of the standard sample have been saved on the spectrometer.

The spectrometer will come with the following directory which will contain the ESR data from your standard samples that were run in the factory:

C:\Data called Calibration_Standards_serial_no

12.2 Tuning

If the tuning window does not appear, go to the settings page, and select the Show Tune Window button. The tune window should be displayed as tuning should proceed as described in section *Automation Controls [▸ 56]*.

Clicking on **Autotune** causes the spectrometer to reboot.

Go to **Devices and Printers** under the Windows **Start** menu, and verify that the Phidget driver is present. The Phidget driver will be displayed under **Unspecified** and is labeled 1067_0.

12.3 Locking

The most common reason for the AFC to fail to lock to the resonator is that the sample is too lossy.

If the system fails to tune, try to run the Auto Tune routine with an empty quartz tube. If the system fails to tune with an empty tube, call your service representative.

If the system tunes with an empty cavity, use a smaller diameter tube for your sample.

If the system is having trouble locking, try turning down the microwave power.

Make sure the Tune window is displayed, and check that a dip appears in the lower and upper left windows (*Auto Tune Routine [▸ 57]*).

Also, check that the noise level reported is below what is specified in the Auto Tune Pop-Up (*Figure 8.22 [▸ 56]* and *Figure 8.30 [▸ 60]*).

If there is no dip in the lower left window, widen the sweep range in the Auto Tune Pop-Up.

12.4 Signal to Noise

- Make sure that the system is locked, and the RF power is below 25 dBm (*Figure 8.65 [▸ 86]*).
- Remove the sample from the cavity and check the alignment. Make certain that the sample is centered in the resonator.
- Check both the microwave and mod coil amplitude power settings (*Figure 8.1 [▸ 39]*).
- Run a standard sample.
- Load the parameter set used to acquire the original data.
- Run the Auto Lock routine, and acquire the data.
- Use the **Overlay Spectrum** button under the **Data Plot** tab to display the original spectrum along with the newly acquired spectrum.

When **the two spectra of the standard sample are the same**, or close, then the poor signal to noise is not a spectrometer issue, but a sample issue.

NOTE: Since the above suggested samples are solids, the spectra may not be identical.

If the signal to noise is significantly lower in the new spectrum, contact your local service representative.

- Look at the RF power when the sample is locked. The lower this number, the better the critical coupling, and the better the critical coupling, the better the signal to noise.
- Try lowering the microwave power (*Figure 8.1 [▸ 39]*). It is possible the sample is being saturated; also, the critical coupling should improve with lower microwave power.
- Try adjusting the RF phase from the **Tune** tab to check noise floor (see *Noise Floor [▸ 62]*).
- Try manually changing the tuner position in steps of 5 such that the RF power reading is minimized (*Figure 8.21 [▸ 55]*).
- Try using a smaller diameter tube. Although there will be less sample, better critical coupling may give a better signal.
- Try increasing the microwave power, especially if the RF power is already low.
- Try another sample tube.
- Check the frequency at which the sample is locked; if it is below 9.6 GHz, the spectrometer may not be locked at the correct frequency. See *Tuning [▸ 103]* troubleshooting.

12.5 Output Distorted

The signal is too large.

- Turn down the digital gain.
- Turn down the mod coil amplitude.
- Turn down the microwave power.
- Decrease the sample volume and or concentration.

13 Replacement of Parts

This unit has no user-serviceable parts, and can only be repaired by a qualified service engineer. Never attempt to open the spectrometer. In the event of a malfunction, please contact Bruker.

13.1 Returning the Unit for Repair

If the Bruker Hotline diagnoses an instrument failure that requires a part to be returned for repair, please follow the procedure listed here:

1. Contact your local Bruker office to start the repair process (see *Contact [▸ 131]*). Repair is always handled by your local Bruker office. Their reply will contain all necessary information for the subsequent repair process steps.
2. They will provide you with details on the shipping address, and also in most cases a “Return Merchandise Authorization” number (RMA number) that allows references to the repair case. Always refer to this RMA number in case of questions.
3. Send the defective part to the local Bruker office and include the following documents:
   - RMA sheet (if RMA number was assigned).
   - Signed Equipment Clearance Form. The Equipment Clearance Form will be sent to you as part of step 1 (see above) with information about the returned part (part number, serial number, your contact details) already filled in.
4. Attach the relevant papers to the *outside* of the packaging, for instance in a transparent polybag.

The unit should be returned using the original container and packing assembly. If this packaging is no longer available, contact your local Bruker office for further instructions.

14 Dismantling and Disposal

Following the end of its operational life, the device must be dismantled and disposed of in accordance with the environmental regulations.

Installation, initial commissioning, retrofitting, repairs, adjustments or dismantling of the device must only be carried out by Bruker Service or personnel authorized by Bruker. Damage due to servicing that is not authorized by Bruker is not covered by your warranty.

14.1 Dismantling

Before starting dismantling:

1. Shut down the device and secure to prevent restarting.
2. Disconnect the power supply from the device; discharge stored residual energy.
3. Remove consumables, auxiliary materials and other processing materials and dispose of them in accordance with the environmental regulations.
4. Clean assemblies and parts properly and dismantle in compliance with applicable local occupational safety and environmental protection regulations.

14.2 Disposal Europe

Environmental information for laboratory and industrial customers within the EU (European Union)

This laboratory product is developed and marketed for Business-to-Business (B2B), so does not fall under article 6 clause 3 of the German Act ElectroG. To meet the demands of the European Directive 2012/19/EU WEEE 2 (Waste of Electrical and Electronic Equipment) and the national Equipment Safety Act, electrical and electronic equipment that is marked with this symbol directly on or with the equipment and/or its packaging must not be disposed of together with unsorted municipal waste or at local municipal waste collecting points. The symbol indicates that the equipment should be disposed of separately from regular industrial/ domestic waste.

Correct disposal and recycling will help prevent potential negative consequences for the environment and risk to personal health. It is your responsibility to dispose of this equipment using only legally prescribed methods of disposal and at collection points defined by government or local authorities in your area.

The WEEE register number can be found on the product label of the equipment. If you need further information on the disposal of equipment or collection and recovery programs available, contact your local Bruker BioSpin sales representative. Local authorities or professional waste management companies may also provide information on specific waste disposal services available in your area.

Disposal - End of Life (EoL) information: the common procedure as defined in the sales contract with Bruker BioSpin

After the lifespan of an electrical and electronic product, Bruker BioSpin takes responsibility for final disassembly and correct disposal in accordance with the European directive 2012/19/ EU WEEE 2.

Bruker BioSpin offers to take back the equipment (only for deliveries after 23.03.2006) after termination of use at the customer site upon request by the customer. This request must be affirmed when the equipment is ordered from Bruker BioSpin. Additional costs for dismantling and transport service will apply!

Only 100% pre-decontaminated equipment can and will be accepted by Bruker BioSpin. A release document for decontamination can be inquired from your nearest Bruker BioSpin contact site, also to be used when repairs, going back to Bruker sites, are requested.

In compliance with WEEE II directive: **2012/19/EU**

14.3 Disposal USA and Other Countries

Disposal of these materials may be regulated due to environmental considerations. For disposal or recycling information, please contact our local office or your local authorities, or in the U.S.A., contact the Electronics Industry Alliance web site at *www.eiae.org*.

15 Technical Data

15.1 Power Supply

The internal power supply plugs into an external standard 115/230 volt power source.

15.2 Instrument Dimensions and Operating Conditions

- Instrument Dimensions: 30.5 x 30.5 x 30.5 cm^3
- Instrument Weight: 10 kg

15.3 Operating Conditions

- Supply Voltage Range: 100-240 VAC
- Supply Frequency Range: 50/60 Hz
- Operating Temperature Range: 15 C - 30 °C
- Operating Humidity Range: 40 - 60% RH

15.4 Rating Plate

The rating plate is located on the rear of the unit and includes the following information:

- Type
- Manufacturer
- Voltage
- Fuses
- WEEE Number
- Year of Production
- PN: Part Number
- SN: Serial Number
- ECL: Engineering Change Level

Figure 15.1: An Example of a microESR Rating Plate

16 Appendix

16.1 Filtering

Filters, or any window function, are applied after the data has been acquired. They do not change raw data.

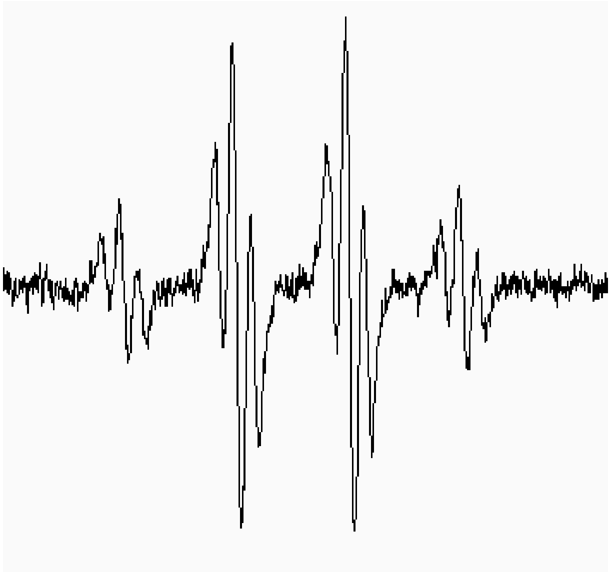


Figure 16.1: BHT in Mineral Oil with no Filtering

Figure 16.2: BHT in Mineral Oil with Savitksy-Golay Filtering

16.2 Display Modes

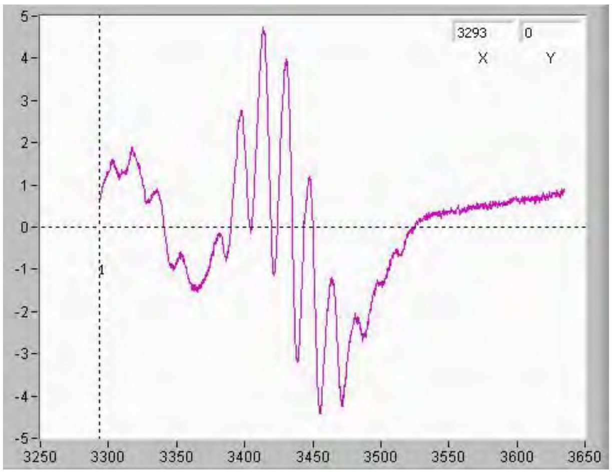


Figure 16.3: Cu(II)TPP in Benzene, Magnetic Field, G

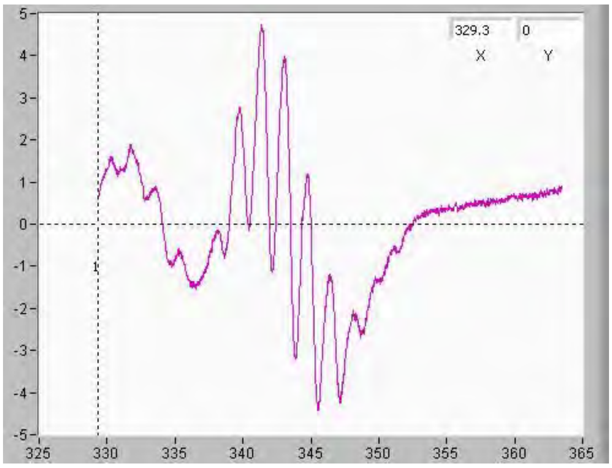


Figure 16.4: Cu(II)TPP in Benzene, Magnetic Field, mT

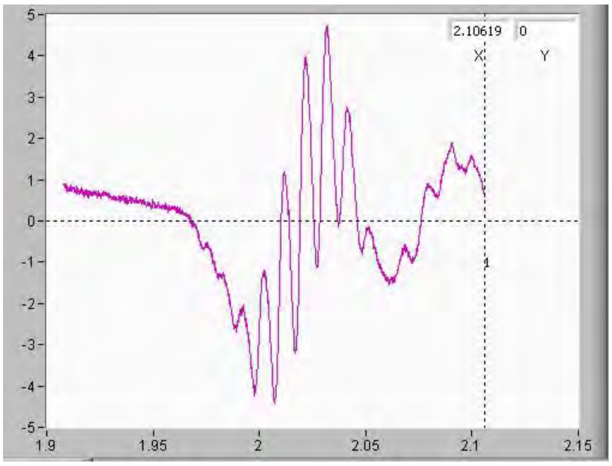


Figure 16.5: Cu(II)TPP in Benzene, gFactor

16.3 Tuning and Signal to Noise

A well tuned sample will have much better signal to noise than a poorly tuned sample. The next two figures are both spectra of the same aqueous 50 μM TEMPOL sample. The spectrometer was well-tuned in the first figure, but poorly tuned in the next.

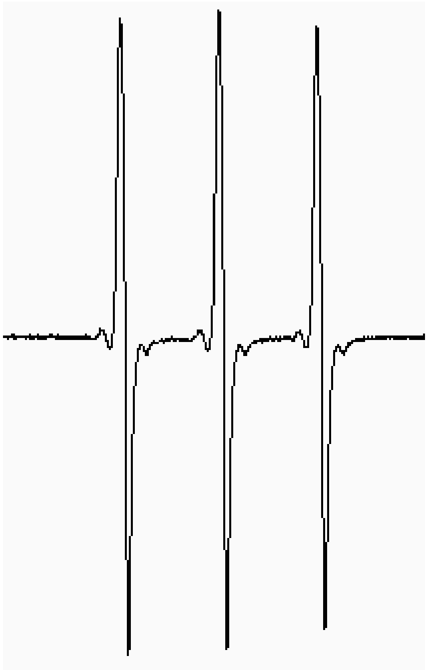


Figure 16.6: Well-Tuned TEMPOL Signal

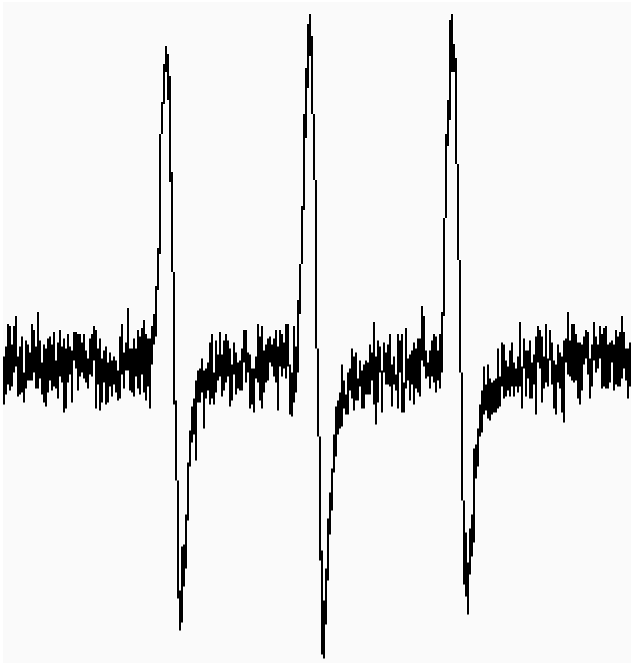


Figure 16.7: Poorly Tuned TEMPOL Signal

16.4 Standard Samples

EPR/ESR does not have clearly defined standards as does, say, NMR, but below are some suggestions. The most important thing is to have a sample that is stable over time, or can be reproducibly made.

Mn(II) impurity in plasticene

This sample will last indefinitely. As long as the same sample is used, data can be compared over time. The next figure is a spectrum of the Mn(II) in plasticene. Depending on the center of your spectrometer, you may only see five of the six lines. Since ^{55}Mn has nuclear spin I=5/2, there are 6 lines total. The smaller resonances in between the large resonances are forbidden transitions*. Manganese has 5 unpaired electrons, significant spin-orbit coupling, and zero-field splitting which make its spectra more difficult to interpret.

* J. A. Weil, et al, *Appl. Magn. Reson.*, 24, 113-125 (2003)

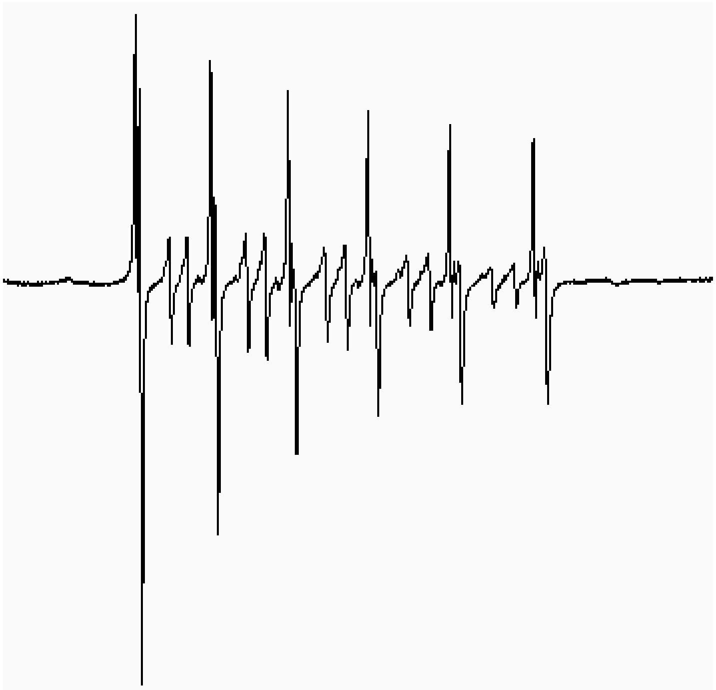


Figure 16.8: Mn(II) in Plasticene

DPPH (solid) in arabinose

This sample comes with the spectrometer, and is also not quantitative, but the signal should remain the same over time. Since the sample is a solid, the signal amplitude and resolution may change with the rotation of the sample tube due to anisotropy. As long as the same sample is used, spectra can be compared over time. The figure below shows a spectrum of DPPH in arabinose with the cursors set to display the resolution of the signal in mT.

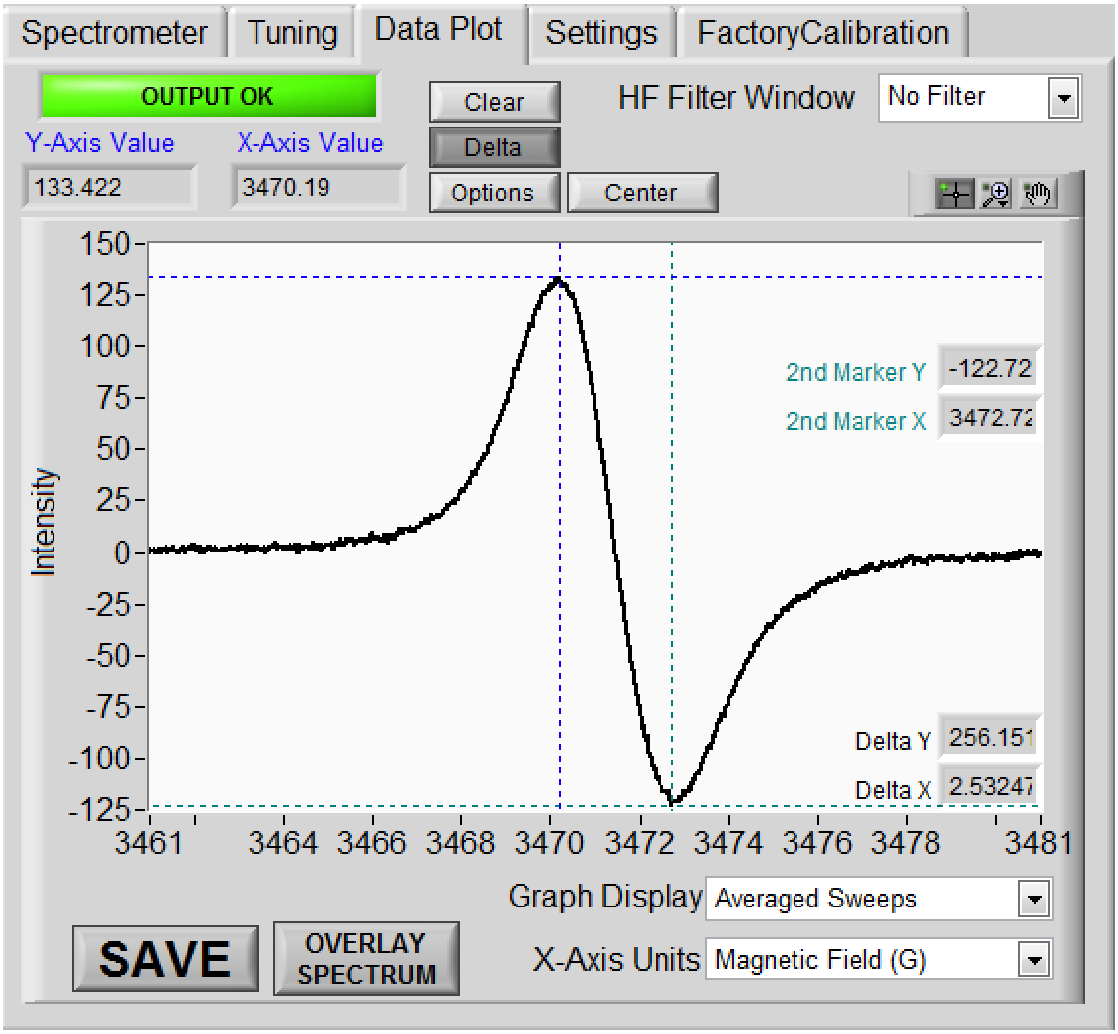


Figure 16.9: DPPH Spectrum (Field)

The next figure shows the same spectrum displayed in g-Factor.

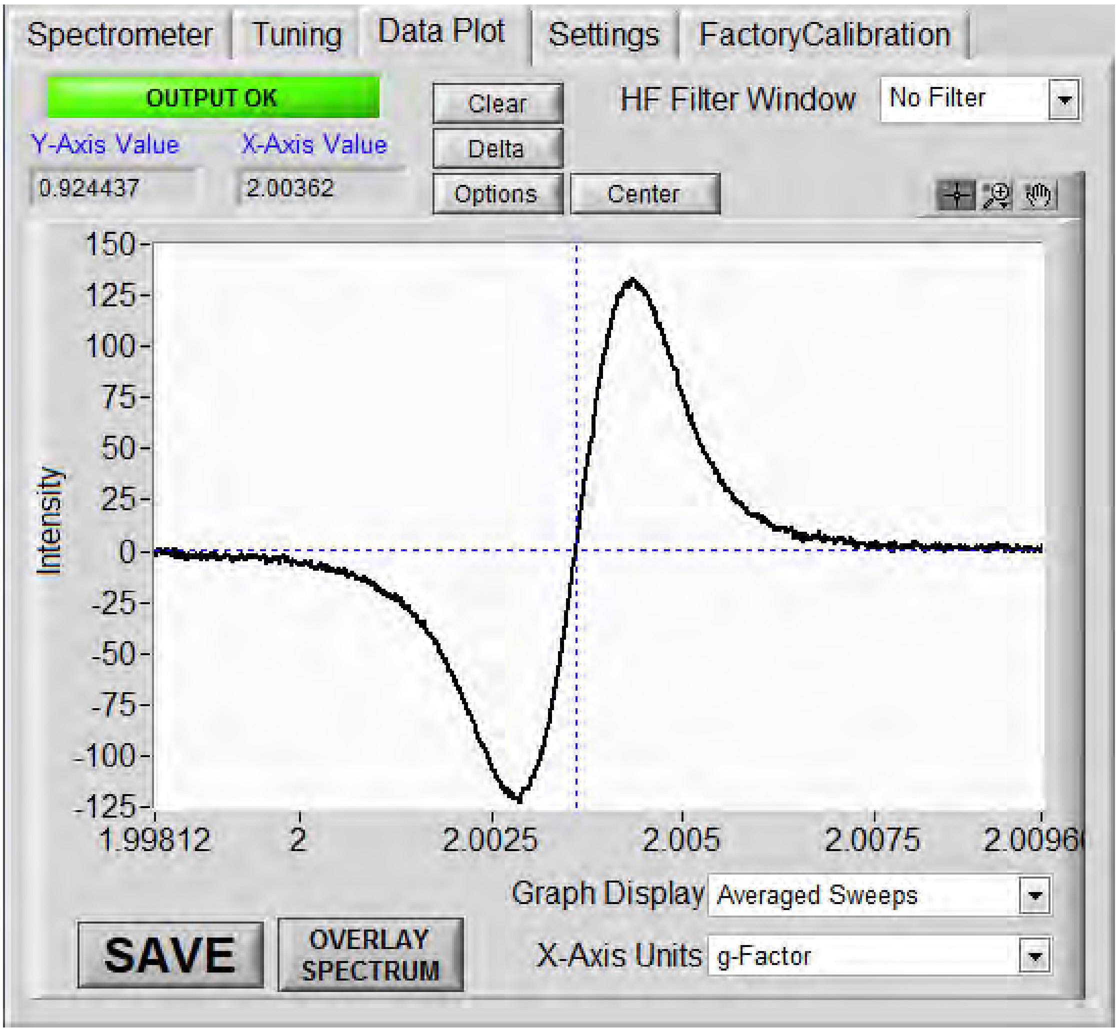


Figure 16.10: DPPH spectrum (g-factor)

Mn(II) impurity in commercially available CaO

This sample cannot be made quantitatively, but any given sample will last indefinitely. As long as the same sample is used, data can be compared over time. The next figure is a spectrum of the Mn(II) impurity in CaO. Depending on the frequency range of your spectrometer, you may only see five of the six lines. The small peaks are forbidden transitions, and the broad hump is the signal from another radical.

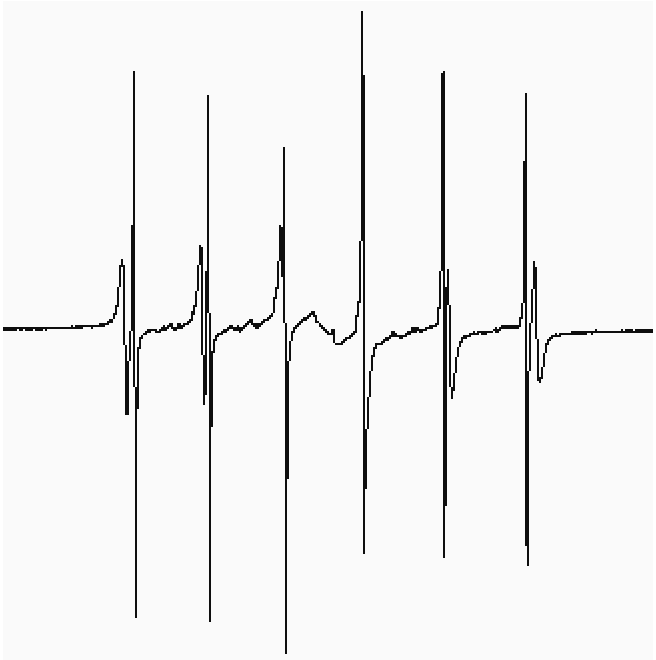


Figure 16.11: Mn(II) impurity in CaO

DPPH in benzene

This sample will not keep unless it is in a sealed tube. This sample is best used to illustrate the hyperfine splitting from the two almost equivalent ^{14}N atoms (I = 1) in the DPPH. This sample can only be used to compare signal intensities over time if the sample is in a sealed tube.

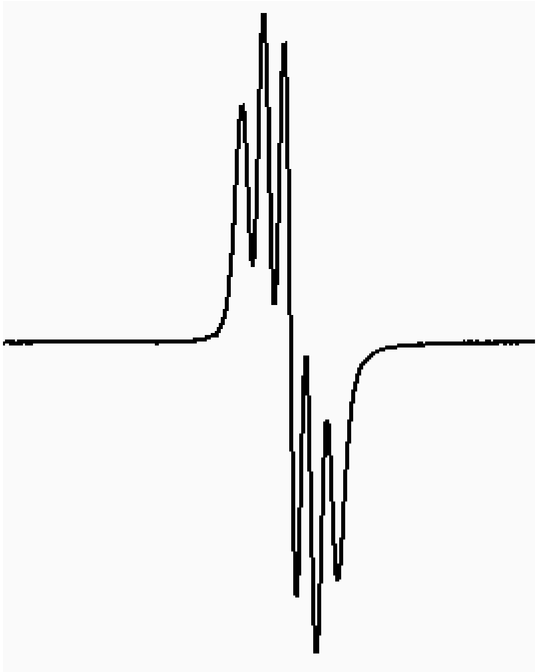


Figure 16.12: DPPH in Benzene

TEMPOL, **TEMPO**, **TEMPONE**, and other similar compounds

Can be used as standards; however, for best results, these samples should be made just before use. Also, care needs to taken in the choice of solvent: TEMPOL gives much narrower signals in water than other solvents because of lower solubility of molecular oxygen in water. Molecular oxygen is paramagnetic and provides an effective relaxation pathway for excited electrons. *Figure 16.15 [▸ 119]* shows overlapping spectra of TEMPOL in toluene and in water. The samples are the same concentration. The signal in water is much narrower.

TEMPONE has a much narrower natural line width than the other nitroxides mentioned because it is much more rigid due to the carbonyl group. TEMPOL also gradually oxidizes to TEMPONE in solution. A normal ESR spectrum of TEMPOL has three equally spaced lines with equal amplitude. A “TEMPOL” spectrum in which the center resonance is greater in amplitude than the two outer resonances, is actually an overlap of the spectra from both TEMOL and TEMPONE. As more TEMPOL is oxidized, the signal becomes more distorted as shown in (C) below. The finer structure in spectrum (C) can only be seen at low modulation amplitudes.

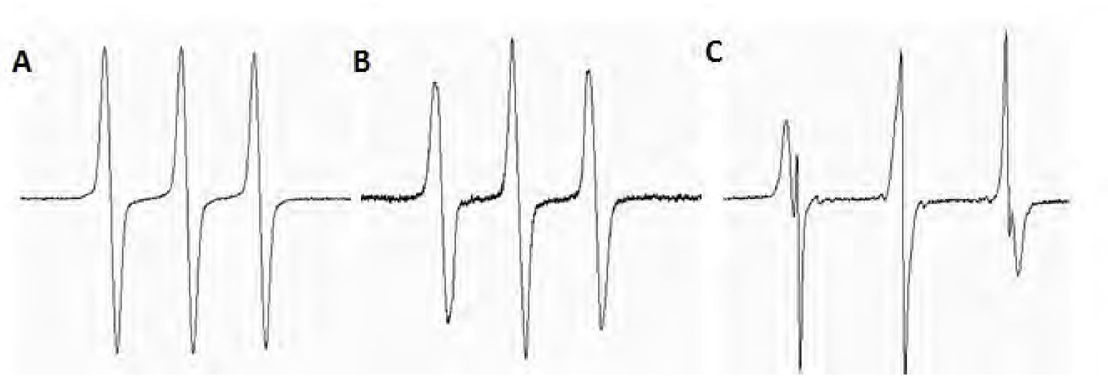


Figure 16.13: TEMPOL oxidizing to TEMPONE. (A) A normal TEMPOL spectrum. (B) TEMPOL oxidizing to TEMPONE. (C) The spectra of both TEMPOL and TEMPONE are observed.

It is also easy to observe the ^{13}C satellites in aqueous TEMPONE because of its narrow lines as shown:

Figure 16.14: 200 µM TEMPONE in Water Showing the 13C Satellites

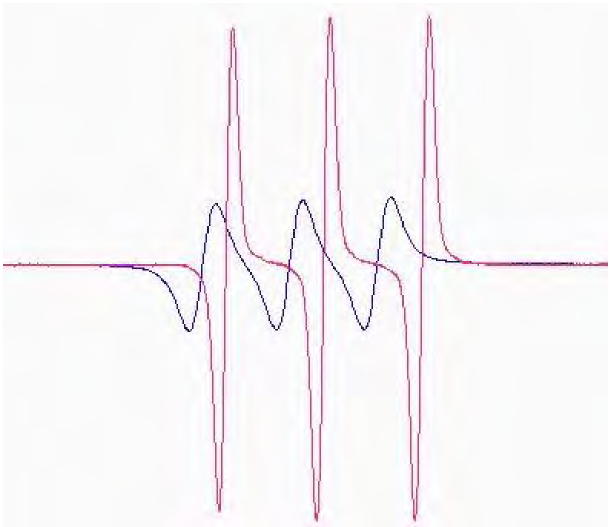


Figure 16.15: TEMPOL in Water (red) and in Toluene (blue). Both Samples are the Same Concentration.

16.5 Mod Coil Settings

The perylene radical cation is an ideal sample to use to look at the effect of the modulator amplitude on ESR spectra. The modulation amplitude should be less than the narrowest line*. There is, of course, a trade-off between resolution and sensitivity. The lower the modulation amplitude, the better the resolution, but signal to noise may also be lower.

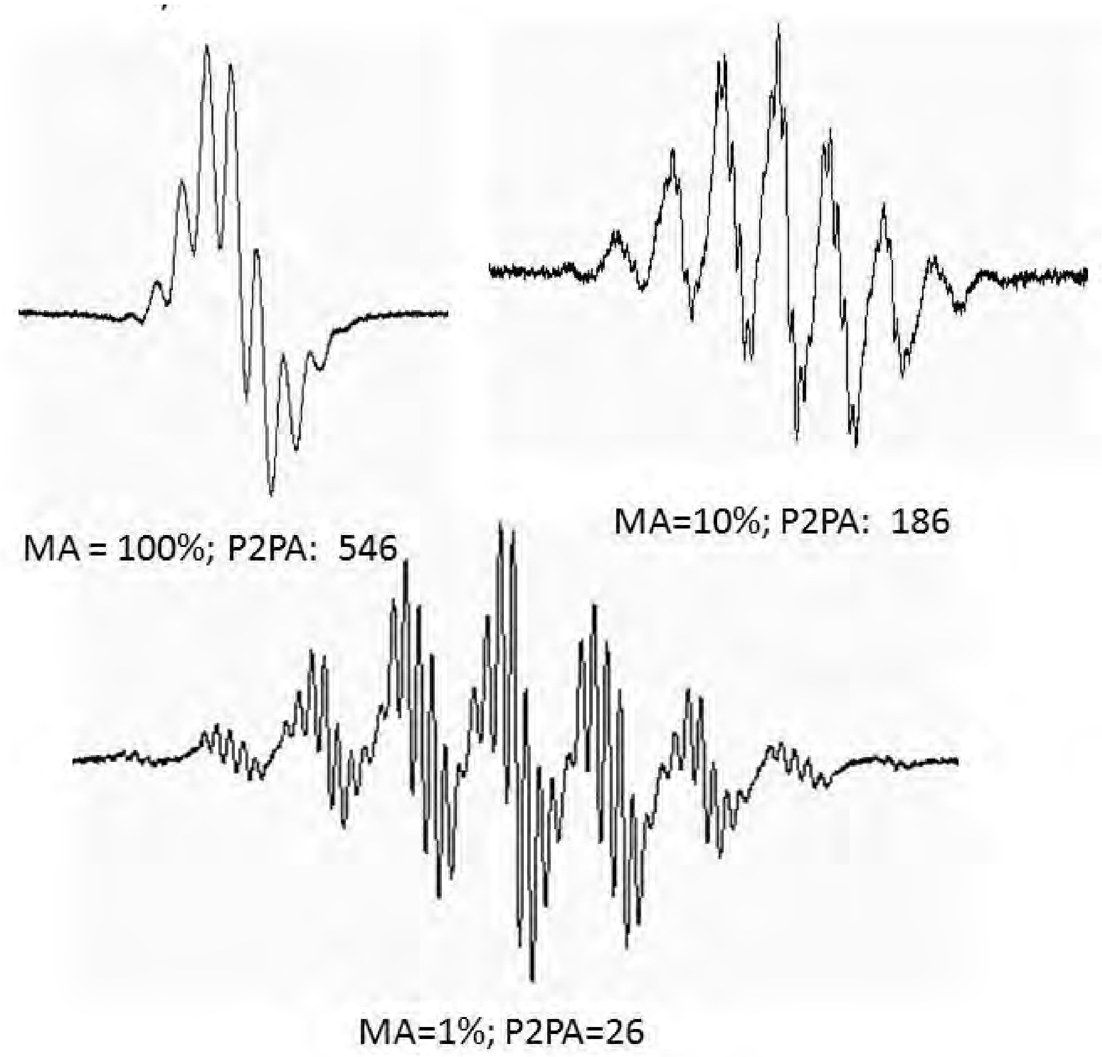


Figure 16.16: Perylene Radical Cation with Different Modulation Amplitudes.

The modulation amplitude on the Micro-ESR was set to 100%, 10%, and 1% in the above figures. P2PA is the peak to peak amplitude

*For optimum resolution and true lineshape, the modulation amplitude should be 0.2 times the natural linewidth. (John A. Weil and James R. Bolton, "*Electron Paramagnetic Resonance: Elementary Theory and Practical Applications*", Wiley, 2007, pp 554-556.

16.6 The Configuration Settings File

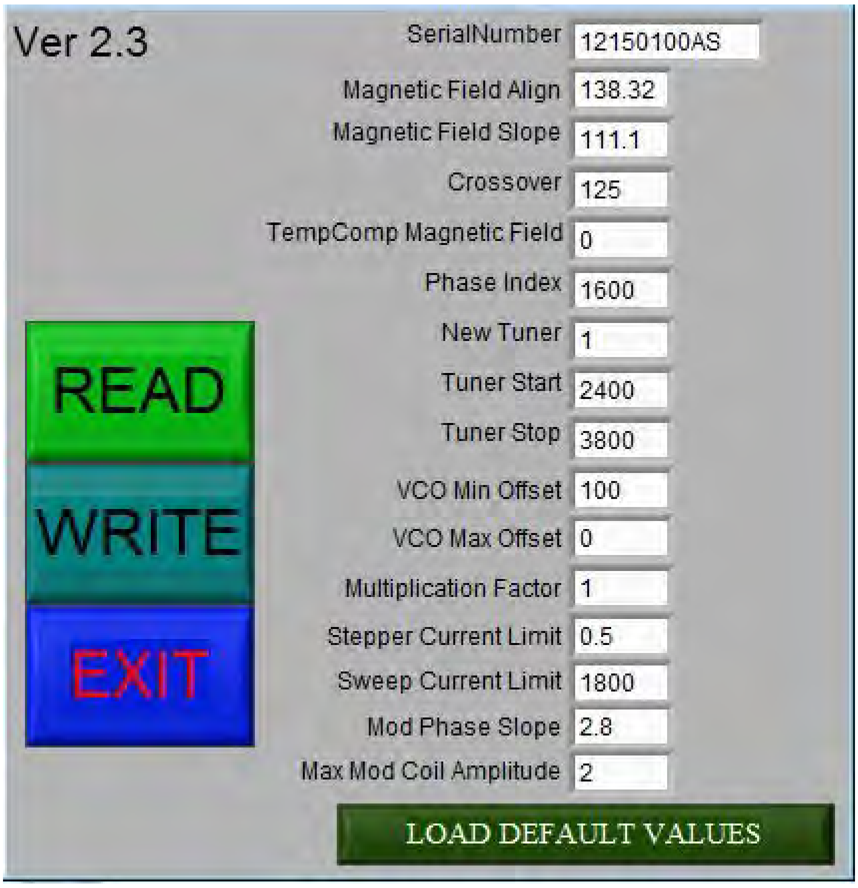


Figure 16.17: Calibration Interface for Firmware

READ

Reads the data from flash and displays it in human readable format in the first column. The second column, labelled Raw Data is directly from the flash.

WRITE

Writes the data in column 1 to the flash.

EXIT

Exits program.

Serial Number

The Serial Number must be 10 characters.

Magnetic Field Align

Is calibrated in the factory to give a g-factor of 2.0056 to TEMPONE in water. This parameter is sensitive to temperature, and can be adjusted before running experiments where accurate g-values are needed.

Magnetic Field Slope

Is calibrated in the factory to give a hyperfine splitting constant of 16.13 Gauss to TEMPONE in water.

Crossover

Time correction for when sweep current switches direction (microESR only).

Temp Comp Magnetic Field

Compensates for magnetic field shifts due to temperature changes. Used only in automation.

Phase Index

The value used for the mod coil setting for a phase of 90 degrees (*Figure 8.21 [▸ 55]*). The selected value should give you maximum signal amplitude.

Tuner Start

Index where tuner starts tuning.

Tuner Stop

Index where tuner stops tuning.

New Tuner

Can have a value between 0 and 2, and is set in the factory.

VCO Min Offset

Sets the minimum sweep frequency for tuning.

VCO Max Offset

Sets the maximum sweep frequency for tuning.

Multiplication Factor

Is a scaling factor that scaled the output signal. It will increase the signal size, but also the noise. It does not change signal to noise.

Sweep Current Limit

Sets the maximum current to drive the modulation coil.

Mod Phase Slope

The Mod Phase Slope is a linear function of Modulation Amplitude, and the slope is the proportionality constant.

Max Mod Coil Amplitude

Calibrated in the factory based on the linewidth of a 200 uM TEMPONE sample in water.

16.7 Changing the Configuration File

If you select to change the configuration file, click on the **Calibration Settings** button under the Settings Tab.

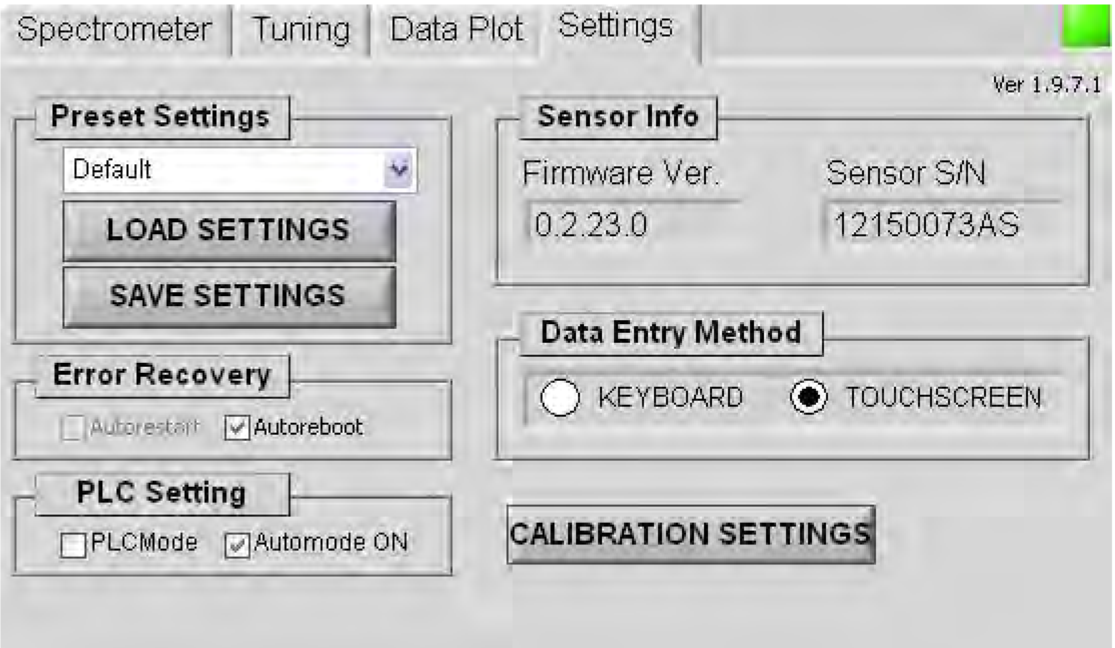


Figure 16.18: Calibration Settings Button under the Settings Tab.

- Click on the box next to the item you wish to change. In this example, **Tuner Start** was selected:

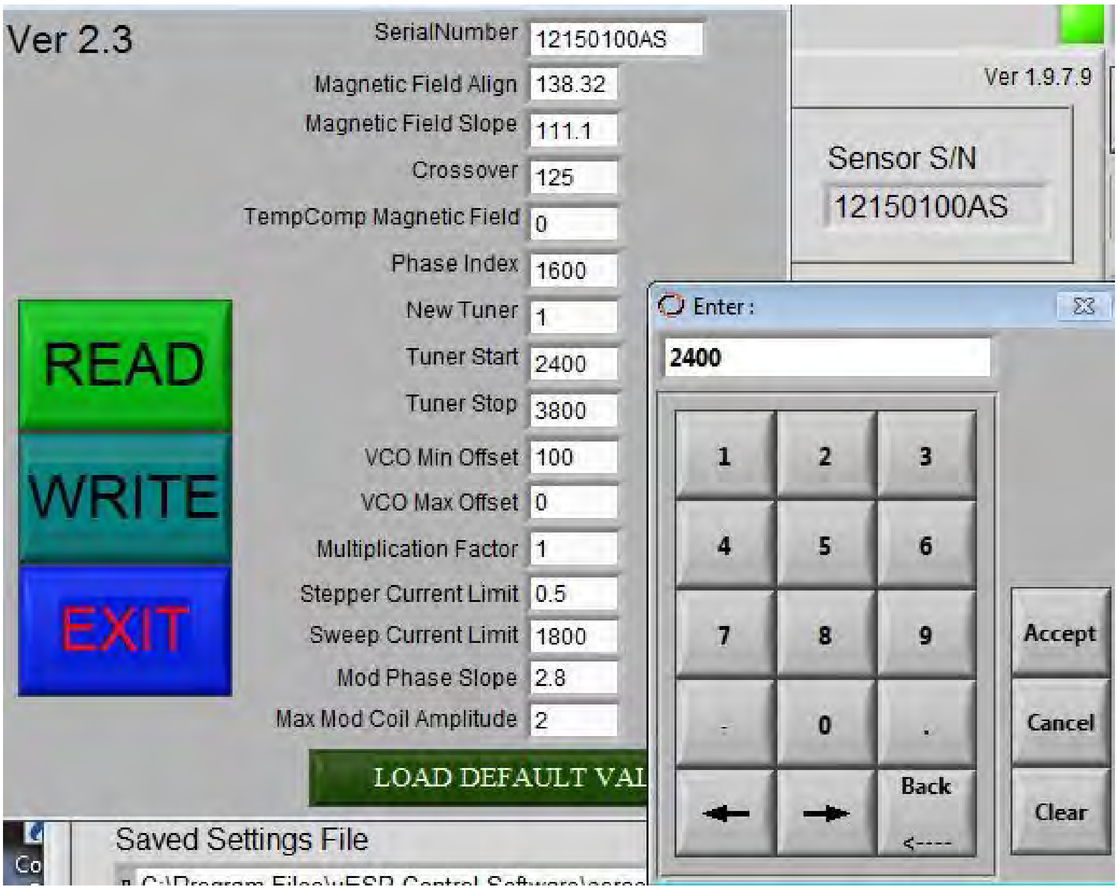


Figure 16.19: Pop-up Appears after Clicking in Box next to Tuner Start

- A keypad pop-up will appear. Use the keypad to change the value.

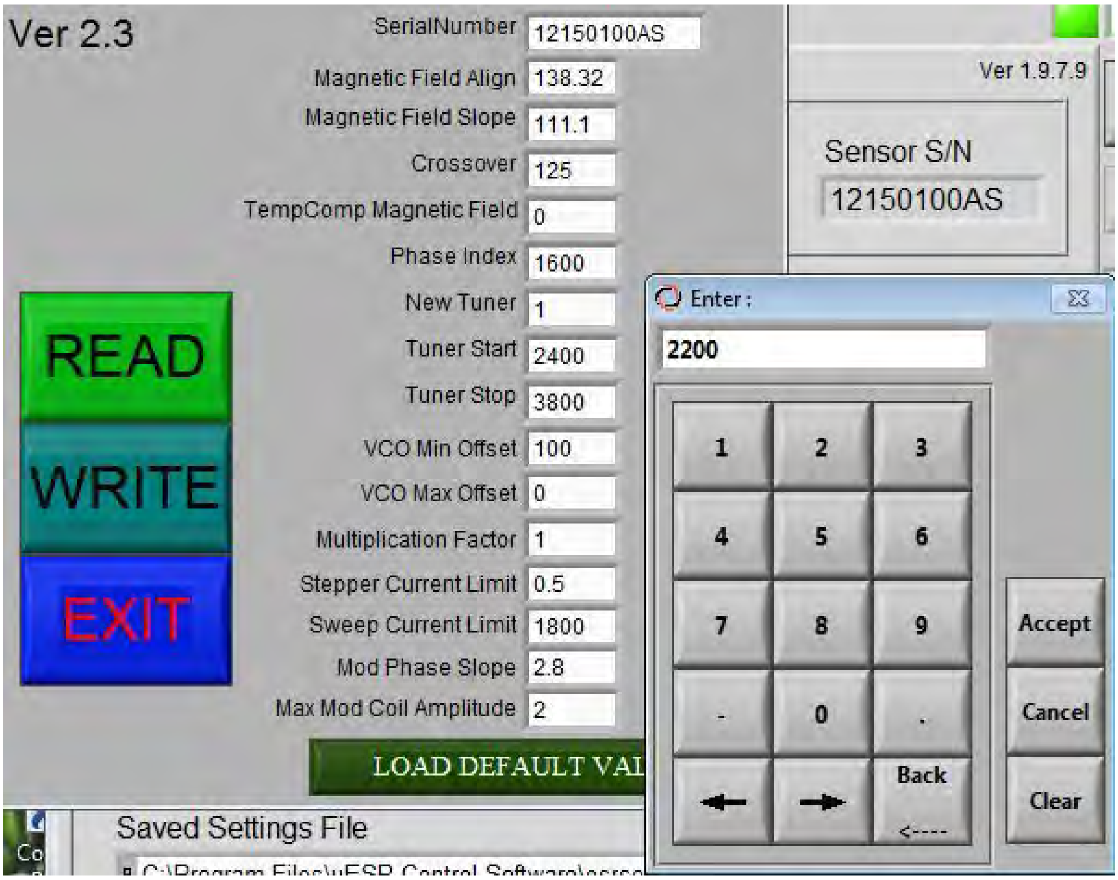


Figure 16.20: Keypad with Changed Tuner Start Value Entered.

- Click **Accept** when change is complete. The pop-up will disappear:

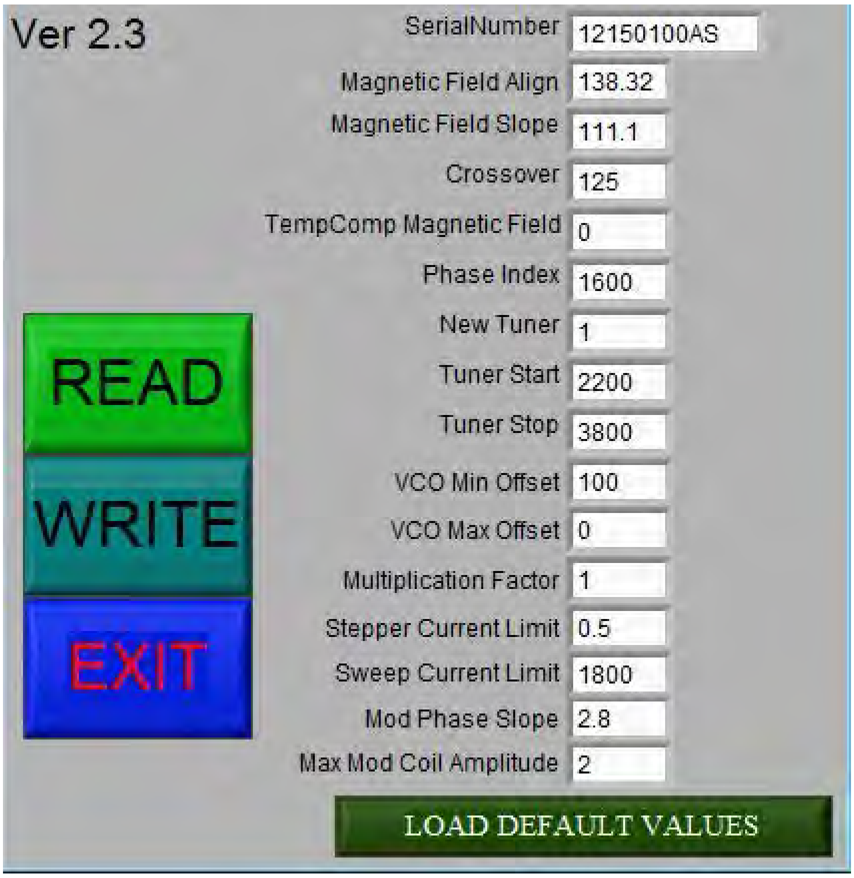


Figure 16.21: Calibration Interface after Changing the Tuner Start.

Note that the value has been changed to 2200, the number entered. This number has not, however, been written to the flash yet.

- Click on the **WRITE** button to write the data to FLASH

Multiple values may be changed simultaneously. LOAD DEFAULT VALUES loads factory calibratn values stored in FLASH.

- Click on **LOAD DEFAULT VALUES**.

The Calibration interface will appear with the values loaded in the factory. Keep any values that you want (for example, you have recalibrated the g-factor, and want to keep your value of magfieldalign rather than reload the factory value).

- Click **WRITE** to write the values to FLASH.

16.8 Calibrating the g-factor

The **magfieldalign** parameter controls the g-factor.

We recommend using 200 µM aqueous TEMPONE; however, any sample that you know the g-factor for precisely will work. Keep in mind that solid samples are very sensitive to orientation. The g-factor for TEMPONE in water at 22 º C is 2.00562, and A_N = 16.13 Gauss

(Ref. J.J. Windle, J. *Magn. Reson.*, 45, 432-439 (1981).

- Using the cursors in the Data screen with the axis display set to g-factor, place one cursor at the current g-factor.
- Place the second cursor at the correct g-factor value.
- Switch back to Magnetic Field (G) and look at the difference.
- Add the difference to the current value of Maximum Magnetic Field.

  [(field at g=correct) – (field at first cursor setting)] + *[current Magfieldalign value]*
- Open the CONFIGURATION SETTINGS file.
- Enter the new value next to magfieldalign, and click accept in the pop-up.
- Click **WRITE** and then **EXIT** in the calibration setting window.
- Rerun spectrum, and measure g-factor.

This may require more than one iteration.

16.9 ESR Settings File

The ESR *setting.ini* file saves parameter values such as spectral width, maximum reflected tune power, sweep width, and many others. The file also controls the visibility of some features.

The *esrsettings* file is in *C:\Program Files\ uESR Control Software* (there are spaces in the directory names).

There is a great deal of information in the *esrsettings* file. We do not advise editing things in this file unless you understand what they are. If you do decide to edit the file, with the exception of the four parameters listed below, make a back-up copy of the original file.

The following four lines allow some parameters to be hidden:

DigitalGainVisible = TRUE	Digital Gain (*Figure 6.3 [▸ 27]*; *Figure 8.1 [▸ 39]*).
TempSetPointControl = TRUE	Control of TEC set point (*Figure 8.21 [▸ 55]*).
SweepDelayVisible = TRUE	Control of Sweep Delay (*Figure 6.3 [▸ 27]*; *Figure 8.1 [▸ 39]*).
d1Visible = TRUE	Allows the use of a preacquisition delay (*Figure 6.3 [▸ 27]*; *Figure 8.1 [▸ 39]*).

These parameters will appear in the section of the file labeled global.

When the argument is TRUE, the corresponding parameter will be visible. When the argument is FALSE, the parameter will be hidden.

If a change is made, save the file. The acquisition software must be exited and restarted for the changes to be visible.

Please contact Bruker with any questions about the *esrsettings* file.

```
[Global]
LimitSwitchStepBack = 1500
AutocalStepBack = 175
#of Tuning attempts = 9
Mod Freq = 43300
Data Entry Method = 0
TempSetPointControl = TRUE
SweepDelayVisible = TRUE
DigitalGainVisible = TRUE
2ndChannel = FALSE
Autorestart = FALSE
Autoreboot = TRUE
d1Visible = TRUE
TempBoardEnable = FALSE
useFastFineTune = FALSE
LimiterEnable = FALSE
FactoryCalibration = FALSE
Use Fixed Phase = FALSE
```

Figure 16.22: The Global Section of the esrsettings.ini File

16.10 Data File Contents

After each sweep, the data is saved to a tab-separated value file on the host computer in the directory specified in the **Settings** tab. This file has three sections: a main header, a sweep-specific header, and a data table. The main header has the following information:

- SAMPLE NAME
- COMMENT
- SENSOR SERIAL NO
- DATE/TIME
- MICROWAVE POWER (mW)
- MOD COIL SETTING (%)
- STARTING SWEEP FIELD (G)
- ENDING SWEEP FIELD (G)
- NUMBER OF POINTS
- SWEEP TIME (s)
- NUMBER OF SWEEPS

The sweeps each have a header as well:

- SWEEP NUMBER
- TIME
- FREQUENCY (MHz)
- REFLECTED POWER (dBm)
- TEMPERATURE (C)

Finally, the spectrum data is listed with the corresponding magnetic field. The first column is the averaged values of all sweeps.

	A	B	C	D	E
1	SAMPLE NAME	50uM_TEMPOL			
2	COMMENT				
3	SENSOR SERIAL NO	12150100AS			
4	DATE/TIME	4/3/2015 14:36			
5	MICROWAVE POWER (mW)	15			
6	STARTING SWEEP FIELD (G)	3276			
7	ENDING SWEEP FIELD (G)	3599			
8	NUMBER OF POINTS	4096			
9	SWEEP TIME (s)	23.5			
10	NUMBER OF SWEEPS	16			
11	MODCOIL SETTING	100			
12	BASELINE SLOPE		1	1	1
13	BASELINE OFFSET		0	0	0
14					
15	SWEEP NUMBER	AVERAGED	SWEEP 1	SWEEP 2	SWEEP 3
16	TIME	2:29:34 PM	########	########	########
17	FREQUENCY (MHz)	9806.728	9806.728	9806.728	9806.728
18	REFLECTED POWER (dBm)	-40.690919	-40.8645	-40.6068	-40.3777
19	TEMPERATURE (C)	26.184527	25.88976	26.5186	26.98703
20					
21	FIELD (G)	INTENSITY			
22	3276	-1.258987	0	-3.01608	-2.74291
23	3276.078877	-1.199448	0	-2.25123	-2.66336
24	3276.157753	-1.313763	0	-1.04115	-2.63649
25	3276.23663	-1.123006	0	-0.56899	-1.94443
26	3276.315507	-0.994164	0	-0.96942	-0.64975
27	3276.394383	-0.960416	0	-1.69473	-0.96113

Figure 16.23: Example of a Partial ESR Data File

The figure above is an example of a partial ESR data file. Since this data set had 64 sweeps, the entire data set would show a column for each sweep: You see columns for SWEEP1 and SWEEP2; the entire document has 64 such columns out to SWEEP 64.

There are 1600 entries (total number of points) for the FIELD, INTENSITY, and each of the SWEEP columns.

The column labelled INTENSITY is the average of all the SWEEP columns.

16.11 File Name Format

The name of the saved file is a combination of the sample name, the date (in **D**ay/**M**onth/**Y**ear format), and the time (in **H**our/**M**inutes/**S**econds):

[SAMPLENAME]__DDMMYY_HHMMSS.ESR

17 Contact

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WEEE DE43181702

EPR Hotlines

Contact our EPR service centers.

Bruker BioSpin EPR provide dedicated hotlines and service centers, so that our specialists can respond as quickly as possible to all your service requests, applications questions, software or technical needs.

Please select the EPR service center or hotline you wish to contact from our list available at:

https://www.bruker.com/service/information-communication/helpdesk.html

List of Figures

Figure 4.1:	The Benchtop microESR	19
Figure 5.1:	Back Panel Ports	22
Figure 5.2:	Front USB Ports	22
Figure 5.3:	Windows Desktop and uESR Software Icon [microESR]	23
Figure 6.1:	Various sized ESR tubes and Sample Holders	25
Figure 6.2:	Loading a Sample	26
Figure 6.3:	Spectrometer Tab in the uESR Control Software	27
Figure 6.4:	Run Type Option	27
Figure 6.5:	Auto Tune Window	28
Figure 6.6:	Initializing the Tuner, Stepping the Tuner, and Moving Tuner to Critical Coupling.	29
Figure 6.7:	Locking the AFC to the Resonator, Fine Tuning, and Sweeping for Optimal Phase.	30
Figure 6.8:	Spectrometer Locked, Sweeping the Field has Started.	31
Figure 6.9:	Saved File Location	32
Figure 7.1:	Standard 5 mm Quartz ESR Sample Tube	33
Figure 7.2:	Adjustment O-Ring on the Tube	33
Figure 7.3:	1.7 mm Capillaries	34
Figure 7.4:	O-Ring Used to Keep the Smaller Tube Aligned Inside the 5 mm Tube	34
Figure 7.5:	Rectangular Tube	35
Figure 7.6:	Tube Alignment Holder	35
Figure 7.7:	Pins on the Bottom of the Rectangular Tube Holder	35
Figure 7.8:	Groove of the Alignment Socket	36
Figure 7.9:	4 mm Quartz ESR Sample Tube	36
Figure 8.1:	Spectrometer Tab	39
Figure 8.2:	Sweep Settings – Single Run Selected	40
Figure 8.3:	Single Run Sweep in Progress	41
Figure 8.4:	Continuous Runs Selected	42
Figure 8.5:	Multiple Runs Selected	43
Figure 8.6:	Multiple Run Sweep in Progress	44
Figure 8.7:	Parameter Array Pop-up	45
Figure 8.8:	Parameter Array Pop-Up with a Non-Zero Array Size for Microwave Power	46
Figure 8.9:	Microwave Power Array Set Up	46
Figure 8.10:	The Sweep Settings Displaying the Minimum Number of Scans for an Array with Four Values.	47
Figure 8.11:	Set up to Array the Mod Coil Amplitude	48
Figure 8.12:	Use Keypad to Enter Mod Coil Amplitude Values	48
Figure 8.13:	Parameter Array Pop-Up Set Up to Array the Mod Coil Amplitude	49
Figure 8.14:	Pull Down Menu for each Element	50
Figure 8.15:	Example of an Array of both Microwave Power and Mod Coil Amplitude.	50
Figure 8.16:	An Example of a Diagonal Array.	51

Figure 8.17: Steady State Warm-Up ... 52
Figure 8.18: Steady State Settings ... 52
Figure 8.19: Magnetic Field Settings ... 53
Figure 8.20: Power Settings, Baseline Correction and Preacquisition Delay. ... 54
Figure 8.21: Tuning Tab ... 55
Figure 8.22: Auto Tune Pop-Up ... 56
Figure 8.23: Ready Light and Message ... 56
Figure 8.24: Minimize Button ... 57
Figure 8.25: Auto Tune Display ... 58
Figure 8.26: Stepping Tuner ... 58
Figure 8.27: Moving to Critical Coupling ... 59
Figure 8.28: Locking to Resonator ... 59
Figure 8.29: Sweeping for Optimal Phase ... 59
Figure 8.30: Successful Auto Tune ... 60
Figure 8.31: Tuner Settings ... 60
Figure 8.32: Other Tuning Options ... 61
Figure 8.33: Signal with Correct Mod Phase ... 63
Figure 8.34: Signal with Incorrect Mod Phase ... 64
Figure 8.35: Data Plot Tab ... 65
Figure 8.36: Output Distorted ... 66
Figure 8.37: Delta Option ... 67
Figure 8.38: Graph Options ... 67
Figure 8.39: Cursor Selects the Desired Center, and Center Pop-Up Allows User to Set Range ... 68
Figure 8.40: New Spectral Width Displayed after Center ... 69
Figure 8.41: Magnetic Field Settings ... 69
Figure 8.42: Tools to Allow the User to Expand and Move the Spectrum ... 70
Figure 8.43: Autoscale must be de-selected for the expand tool to function ... 70
Figure 8.44: Pop-up displayed when the center icon is selected. Select upper left icon to expand spectrum. ... 71
Figure 8.45: Drag mouse to select the area to expand ... 72
Figure 8.46: Unclick mouse to view expanded area ... 73
Figure 8.47: Graph Display Options ... 73
Figure 8.48: Saved File Message ... 74
Figure 8.49: Overlay Data Set ... 74
Figure 8.50: Overlayed Spectra ... 75
Figure 8.51: Auto-Scaled Spectrum ... 76
Figure 8.52: Spectrum Overlay ... 77
Figure 8.53: Settings Tab ... 78
Figure 8.54: Preset Settings ... 78
Figure 8.55: Preset Settings ... 79
Figure 8.56: Save Preset Settings ... 79
Figure 8.57: Pop-Up Keypad ... 80
Figure 8.58: Pop-Up Keyboard ... 81

Figure 8.59: Calibration Interface for Firmware 82
Figure 8.60: Pop-Up to Create Folder or File in Windows 83
Figure 8.61: Sweep Control Buttons 84
Figure 8.62: Sweep Status – Preparing to Run 84
Figure 8.63: Sweep Status – Warming Up 85
Figure 8.64: Sweep Status – Sweeping Spectrum Run #-n 85
Figure 8.65: Status Window 86
Figure 8.66: Unlocked RF Cavity 87
Figure 9.1: Cavity Dimensions 89
Figure 9.2: Standard 5 mm ESR Tube Dimensions. 90
Figure 10.1: The LN2 Dewar and Capillary 91
Figure 10.2: Crude Oil at 298 K (A) and Crude Oil at 77 K (B) 91
Figure 10.3: The Fiber Optic Cable, with SMA Connector and Adapter 92
Figure 10.4: Spectrometer Tab in the Acquisition Software 94
Figure 10.5: The Thermo Cube Thermoelectric Heater Chiller 94
Figure 10.6: Airflow Adapter Shown Installed on the microESR 95
Figure 10.7: Attaching the Air Hose from the TermoCube via CPC Adapter to the Air Flow Adapter (shown with cover) 95
Figure 10.8: Hose Connecting the ThermoCube to the microESR 96
Figure 10.9: ThermoCube Connections 96
Figure 10.10: Connections on the ThermoCube 98
Figure 10.11: Assembled Flow Adapter without Cap 100
Figure 15.1: An Example of a microESR Rating Plate 109
Figure 16.1: BHT in Mineral Oil with no Filtering 111
Figure 16.2: BHT in Mineral Oil with Savitksy-Golay Filtering 111
Figure 16.3: Cu(II)TPP in Benzene, Magnetic Field, G 112
Figure 16.4: Cu(II)TPP in Benzene, Magnetic Field, mT 112
Figure 16.5: Cu(II)TPP in Benzene, gFactor 112
Figure 16.6: Well-Tuned TEMPOL Signal 113
Figure 16.7: Poorly Tuned TEMPOL Signal 113
Figure 16.8: Mn(II) in Plasticene 114
Figure 16.9: DPPH Spectrum (Field) 115
Figure 16.10: DPPH spectrum (g-factor) 116
Figure 16.11: Mn(II) impurity in CaO 117
Figure 16.12: DPPH in Benzene 118
Figure 16.13: TEMPOL oxidizing to TEMPONE. (A) A normal TEMPOL spectrum. (B) TEMPOL oxidizing to TEMPONE. (C) The spectra of both TEMPOL and TEMPONE are observed. 118
Figure 16.14: 200 µM TEMPONE in Water Showing the 13C Satellites 119
Figure 16.15: TEMPOL in Water (red) and in Toluene (blue). Both Samples are the Same Concentration. 119
Figure 16.16: Perylene Radical Cation with Different Modulation Amplitudes. 120
Figure 16.17: Calibration Interface for Firmware 121
Figure 16.18: Calibration Settings Button under the Settings Tab. 123

Figure 16.19: Pop-up Appears after Clicking in Box next to Tuner Start 124
Figure 16.20: Keypad with Changed Tuner Start Value Entered. 125
Figure 16.21: Calibration Interface after Changing the Tuner Start. 126
Figure 16.22: The Global Section of the esrsettings.ini File 128
Figure 16.23: Example of a Partial ESR Data File... 129

List of Tables

Table 3.1: Symbols on the Packaging 15

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