IMSERC PULSE X-band Manual

CONTENTS

INTRODUCTION

The Bruker pulse X-band EPR spectrometer is equipped with a pulsed microwave bridge, X-band resonator, and low-temperature cryogenic system for advanced pulse EPR measurements. The system enables electron spin relaxation measurements, including T_1 and T_2, as well as echo-detected EPR, ESEEM, and other experiments. This instrument is suitable for studying spin dynamics, local electronic structure, and transition-metal complexes, spin-labeled biomolecules, and paramagnetic materials.

SAFETY

All users of IMSERC must review the general safety policies at http://imserc.northwestern.edu/about-policies.html. Plus, users should know the points below:

- Users with pacemaker are not allowed
- Emergency Exits
- Location of shower and eyewash
- No food and drink
- Dressing code

DATA MANAGEMENT

The personal data folder is created during training. The data were under the user's personal folder, which must be located under the supervisor's group folder.

EPR data on this instrument are copied on 'imsercdata.northwestern.edu' under 'data2/pcm/xband' within a few seconds. Please follow instructions at http://imserc.northwestern.edu/about-general-faq.html#data for details about data access.

SOFTWARE

The EPR experiment on this instrument is performed with Bruker Pulse X band.

MATERIALS & METHODS

The default measurement mode of pulse X band with the 4 mm Bruker resonator. If user want to run an experiment in a different resonator, they will contact the staff. The staff will also need to tune back to the default resonator after the experiment. (these will be charged via staff time)

The default working condition of INSTRUMENT-NAME is as follows:

1. Computer screen is by default deactivated. Users must start the reservation through NUCore in order to be able to turn on the computer screen.
2. Leave the acquisition software open when user complete with the measurement.

EXPERIMENT SETUP

STEP 1. SAMPLE PREPARATION

Sample Tubes:

a) Standard EPR tube size = 4 mm OD, however, sizes do vary based on different resonators. 1.6 mm OD capillary tube can be used a second tube to avoid sample explosion

b) EPR tubes are made out of quartz whereas NMR tubes are made out of Pyrex, which contains some iron that may or may not, depending on the sample, contribute to the EPR signal.

Tubes can be purchased by various vendors. A couple of options are,

Wilmad Glass: https://sp-wilmadlabglass.com/product/3-mm-thin-wall-quartz-epr-sample-tube-250mm-l/

Aqueous Samples (also true for highly polar and conducting samples): The water in aqueous samples absorbs the electric part of the microwaves and begins to heat (like your microwave at home), and therefore, the sample size needs to be limited, specifically the surface area of the sample size. Current, IMSERC uses capillary tubes to adsorb the liquid sample and wax to seal each open side. Then input the capillary tube with sample into the 1.6 mm quartz tube. The concentration of liquid sample is about 100 – 200 uM.

The liquid sample should be also pre-freeze in capillary tube to avoid the explode.

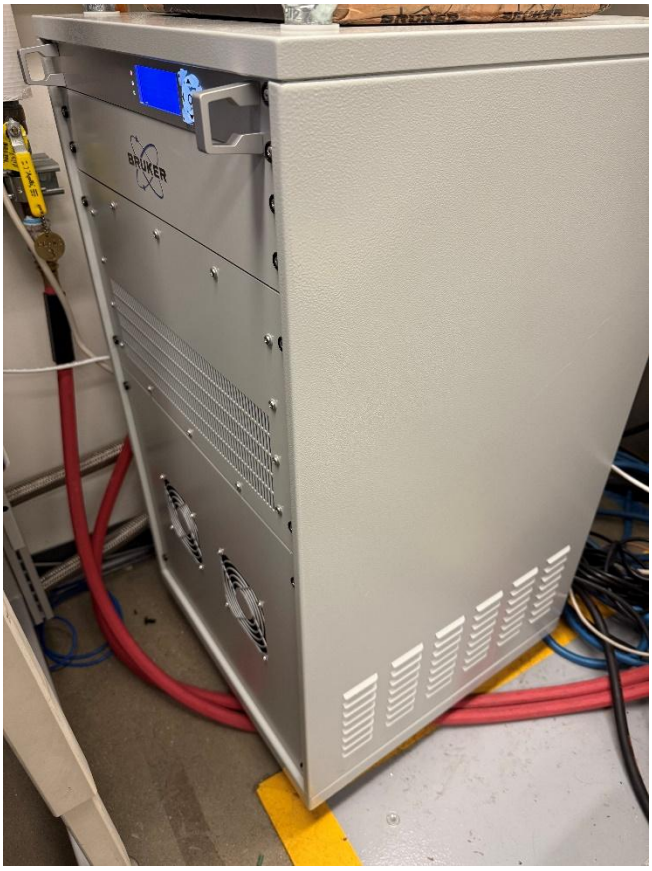
Powder samples: the capillary tube was sealed first, and power was packed into the tube.

STEP 2. TURN ON THE SPECTROMETER AND OPEN THE SOFTWARE

1) Turn on the following (if not already on):

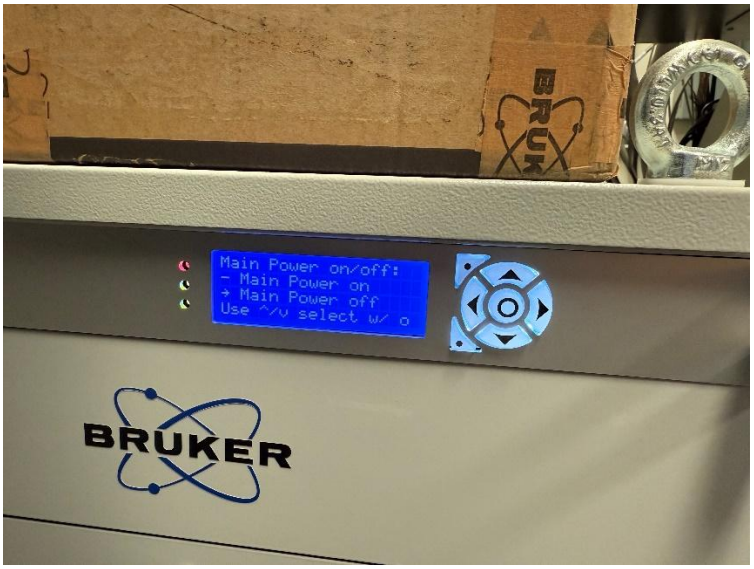
Console (if it is on, then skip)

Power Supply of the magnet (press center circle button and chose the main power on/off, switch between on/off with arrow and circle button)


BRUKER


ER083 Power Supply
23.86U 8.142A
Power-on state
Hall controlled
BRUKER


Choose operation:
^ Set Current
→ Main Power on/off
Use ^/v select w/ o
BRUKER



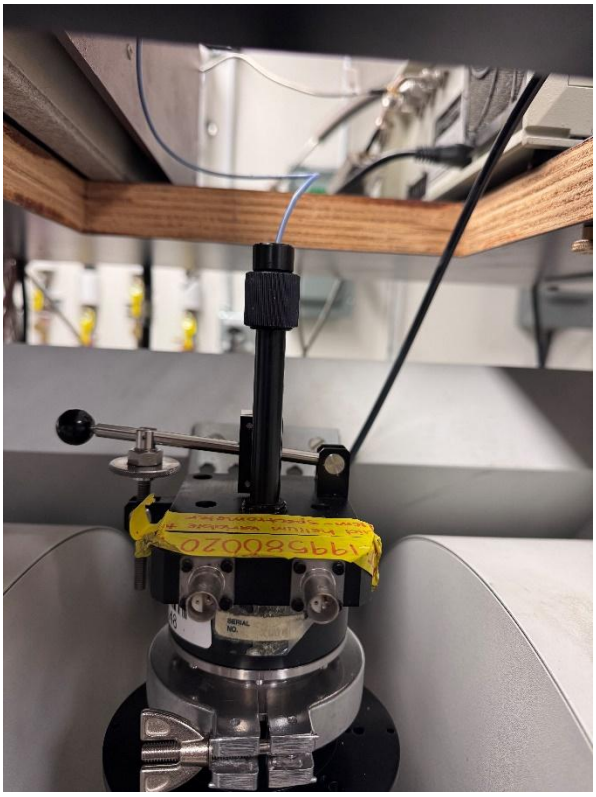
2) turn on the reservation on Nucore. Open the Acquisition software “XEPR” located on the desktop.

STEP 3. INSERT SAMPLE

1) Added around 6 mm to 15 mm sample to the 4 mm OD tube. Then assemble the 4 mm OD tube with the sample holder. (at least, we need minimum 6 mm sample). The distance from sample holder to the center of sample is about 54 mm for QD2 resonator (38 mm for MS3 resonator). Connect the sample holder to the sample rod.

2) Wipe the outside of the tube with acetone or isopropanol.

3) Losen the plug cap in the top of resonator and remove the whole plug. Quickly insert the sample rod with sample into the resonator. The whole stuff will go straight down to the end and then tighten the screw nut to seal the rod to the resonator.



STEP 4. SETTING TEMPERATURE

The temperature should be optimized for each sample type, but 10 K should be a good starting approximation. – control, set temperature, auto PID

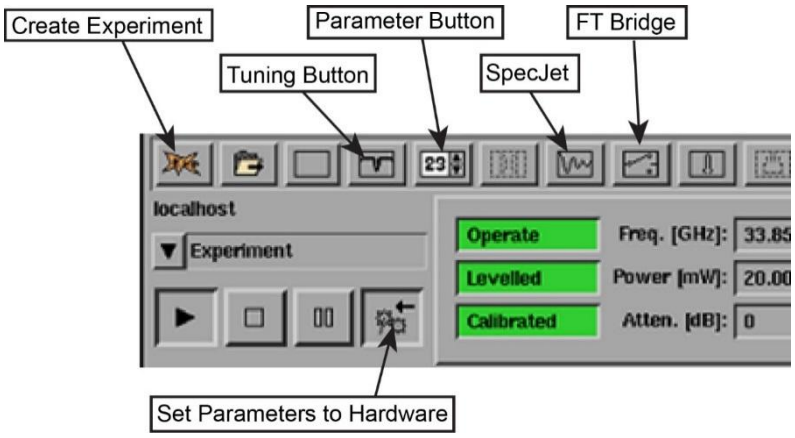
STEP 5. CONNECT THE SPECTROMETER AND CREATE NEW EXPERIMENT

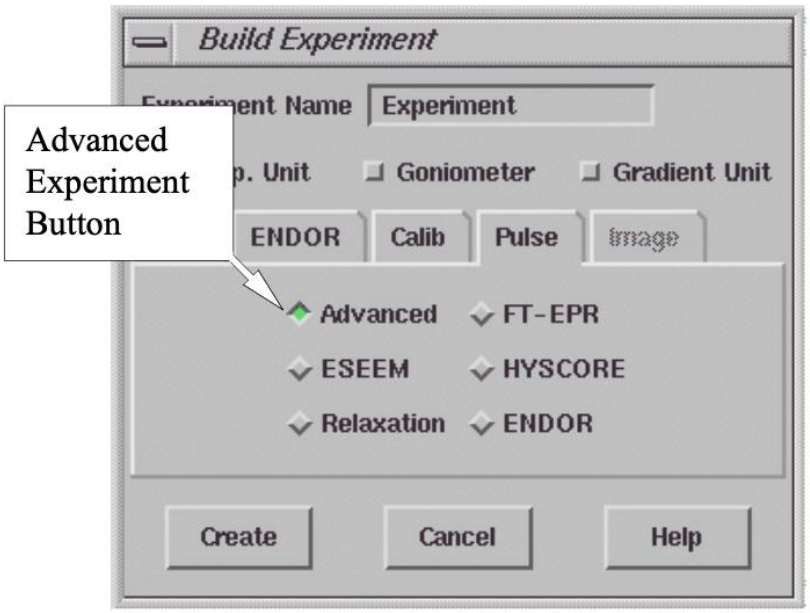
1) Open XEPR.
2) Go to connect to the spectrometer. The led light on the bridge should be on.

   Where: “Acquisition” → “Connect to spectrometer” → “local host” → “ok”
3) Create a new experiment and set parameters to hardware.

   Where: “Create Experiment” → “pulsed” → “Advanced” “create”

   Click “send parameters to hardware”





STEP 6. TUNING THE SPECTROMETER

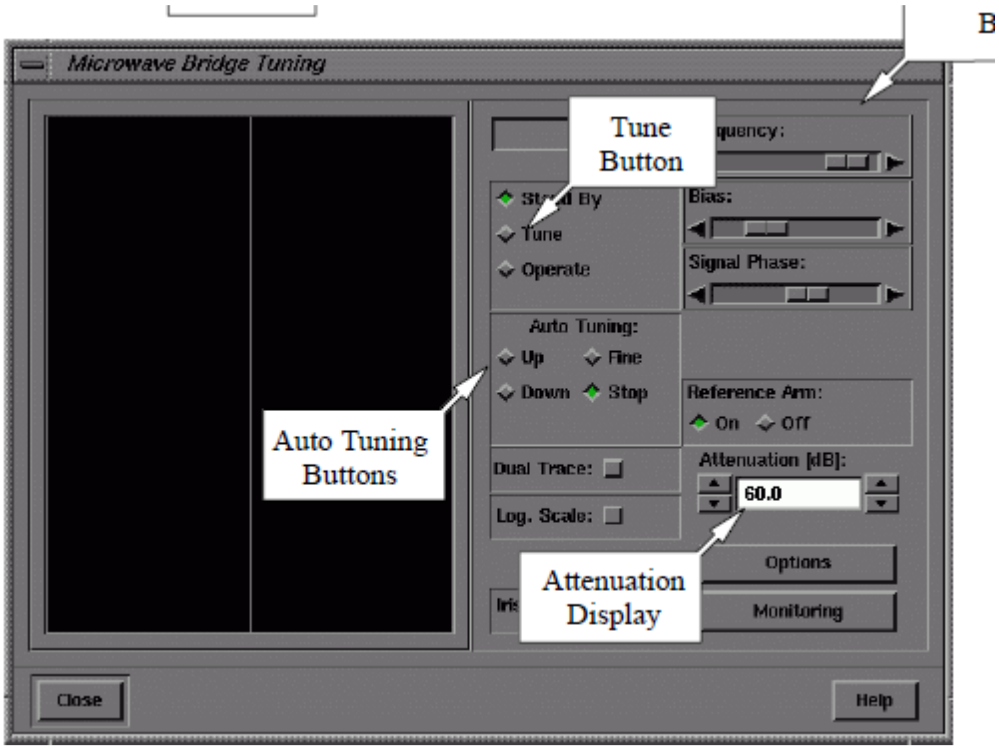
Go to the “Bridge Tuning”, the attenuation should be at 60 dB.

In the tune mode adjust the attenuation to 20dB and find the dip. Align the dip to the center frequency of the “tune” window. For the empty resonator, the tuning dip is around 9.74 GHz.

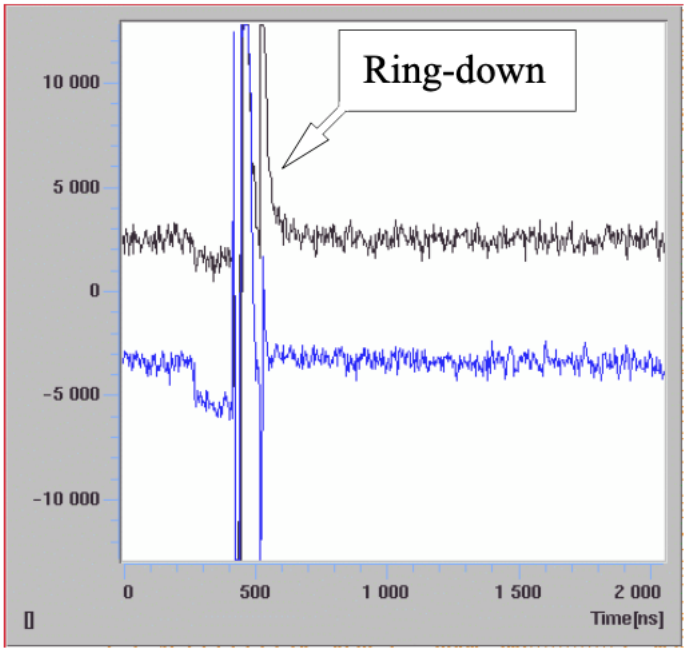
When sample is inserted, the dip will drop to lower frequency. The true dip should respond to the sample movement and the iris screw rotation (changing the resonator coupling). Adjust frequency bar to match the dip of tuning. Then turn the iris coupling to over coupled. (The iris should be turn up until the dip totally disappears, then you achieve the over-coupled condition.)

Make sure the resonator is over coupled, this will give us the better performance due to the large bandwidth.

Overcoupling the resonator also prevent receiver damage due to intense reflections.



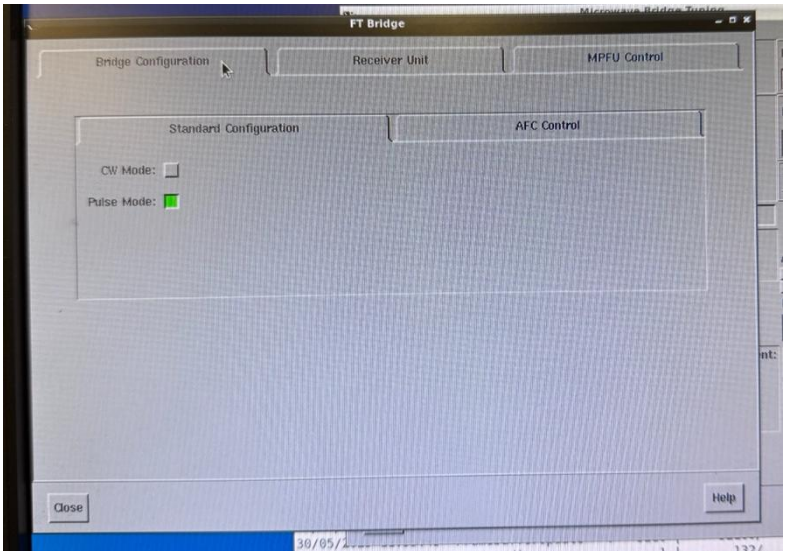
If the resonator is critical-coupled and you will find a significant ring-down effect when you check the echo signal later. Such as this. If you see this, please change the iris to over-coupled.



STEP 7. CHECKING DEFENSE PULSE

1) Switch to Pulse mode.

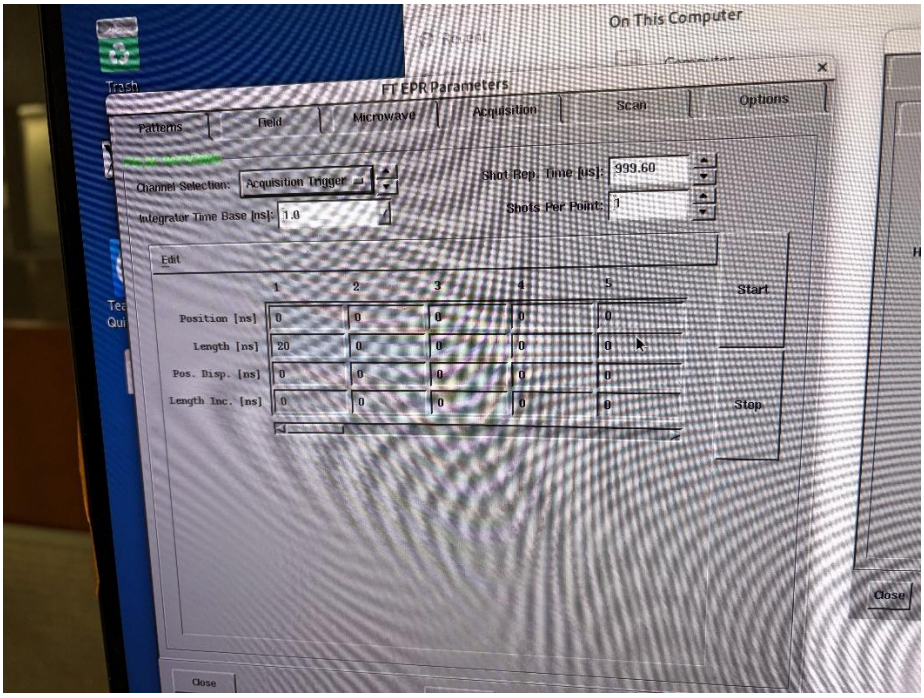
   Where: “FT Bridge” → “Bridge Configuration” → from “CW mode” to “pulse”



2) Check for the defense pulses.

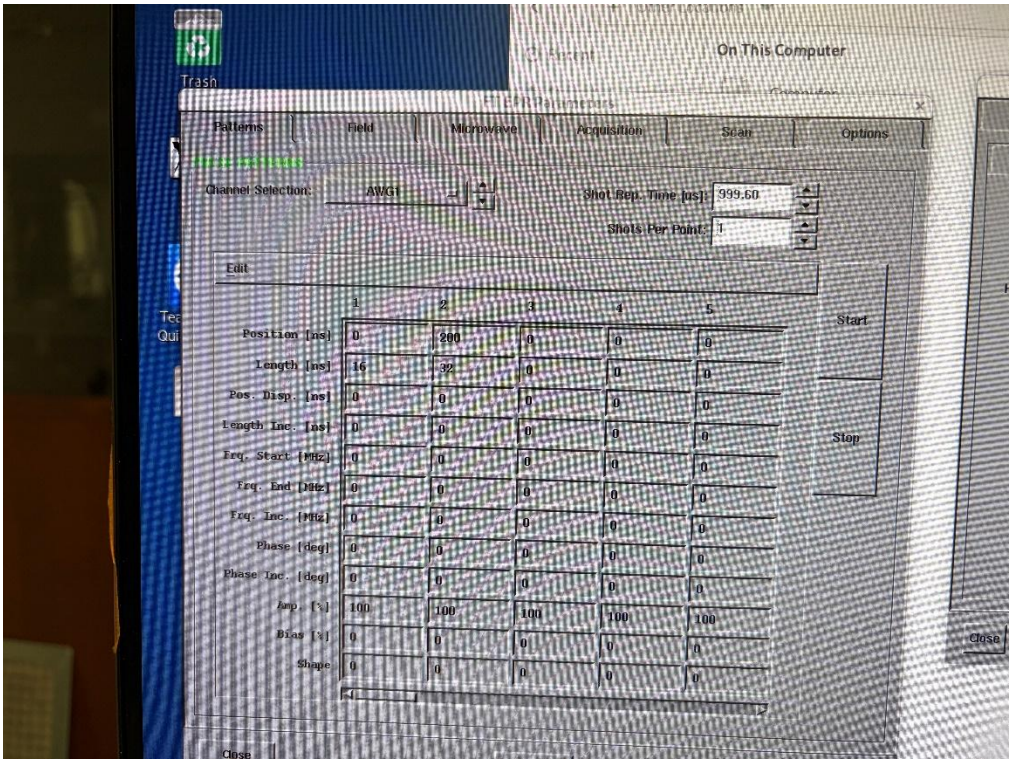
Open “Parameters button” → “Patterns”

Under channel “Acquisition Trigger”, program a 20 ns (length, first column) acquisition trigger, position as “0”. Change integration time to 1.

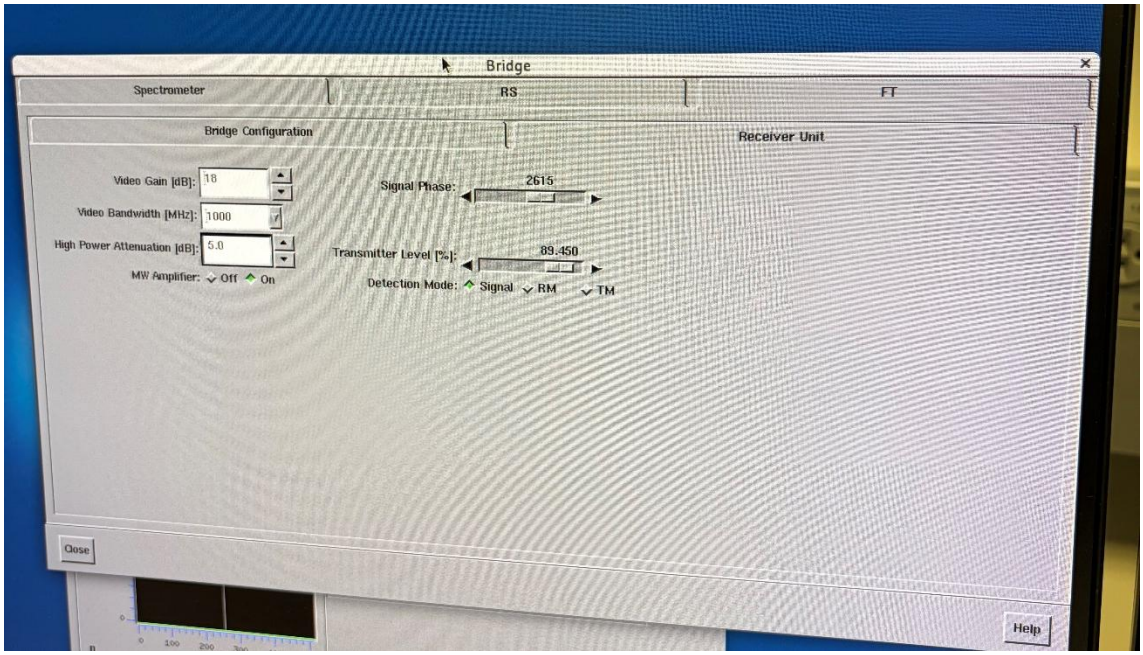


Under channel “AWG1”, program echo detection pulse.

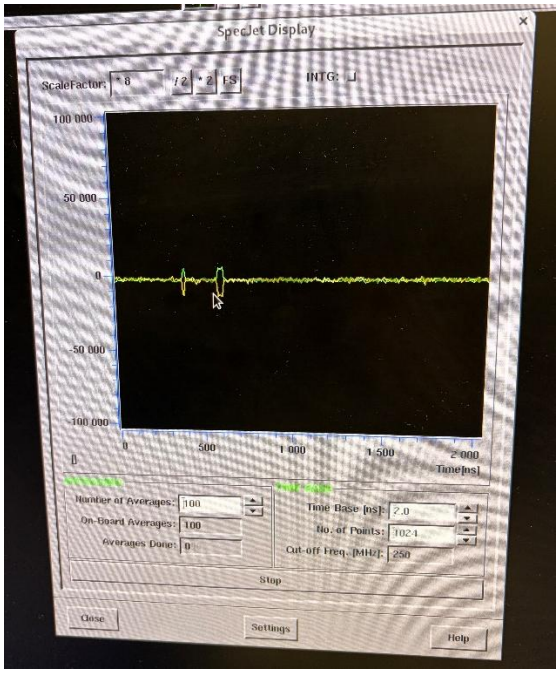
	Pulse 1	Pulse 2
Position	0 ns	200 ns
Length	12 ns	24 ns



Open “FT parameters” → “Microwave”, adjust video gain to ~ 30 db. Keep the attenuation at 60 dB.



Click “Start” under “Patterns”. Open “SpecJet” and click “Run”. Set Number of averages in SpecJet to 100. Change the time constant to 2 ms. Adjust the number of points, such as 2048 for that.



In the "SpecJet" window, you will find the two pulse that you just used. However, it is hard to tell the defense pulse based on the pulse. To ensure there is defense pulse, in the Bridge tab, receiver unit, switch between "Signal" and "TM". If the pulse's amplitude in "TM" is 10 time stronger than "signal", then the system has the correct "defense" pulses.

STEP 8. TURN ON TWT

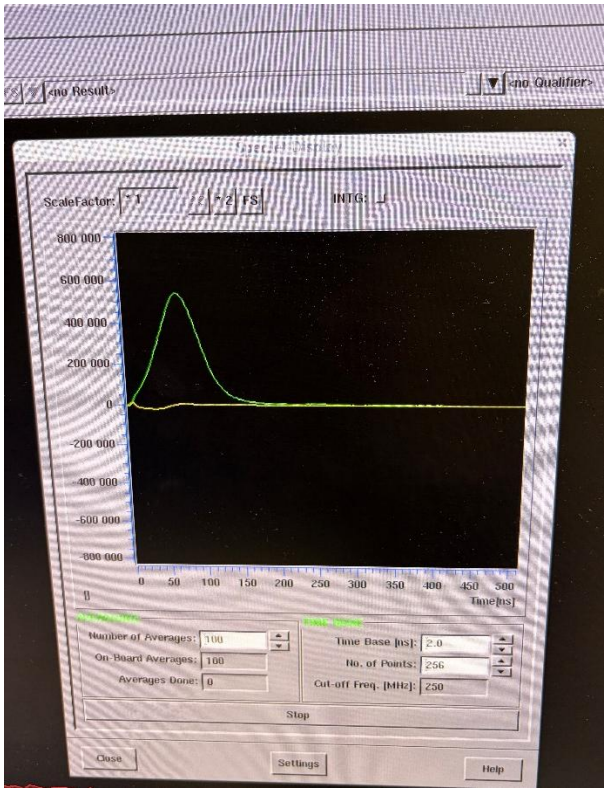
If you successfully checked the defense pulse, then you can turn on the TWT. To turn on the TWT, please press the on/off, wait until the TWT shows "standby". When TWT is "standby", press "operate" to turn on the HV. But *if the defense pulse is not visible then do not turn on TWT*.

STEP 9. OPTIMIZE SIGNAL

1) Decrease Attenuation "FT EPR Parameters" →"Microwave" → "attenuation" OR the "Receiver unit" in "FT bridge"
    a. You should see an echo start to form. You may have to adjust the video gain to decrease the signal as you go. The signal will initially become stronger when you lower the attenuator then usually goes through a maximum and starts to decrease., Find the attenuation value for the maximum signal.

    b. If you do not see an echo, you possible miss the correct value of the magnetic field for your ESR signal. In this case, you need to adjust the magnetic field value to find the echo signal.

    For a usual nitroxide sample with a g factor of ~ 2.006 at the frequency of v=9.5 GHz, the magnetic field vaue is ~ 3400 Gauss. The magnetic field position is related to the ESR frequency as B=hv/gβ, where h=6.626x10-27erg/s is the Planck and β=9.274x10-21erg/G is the Bohr magneton.



2) When the echo signal is found, change the position of the acquisition trigger to the beginning of the echo, ~ 780, to skip the defence pulses. Reduce the no of points to 256 and you will observe the signal only.

3) Phase the echo using “FT Bridge” → “Receiver Unit” → “Signal Phase”, make all the signal go to the “green curve”, and make the “yellow curve” small, almost flat
Adjust the field position to the maximum of the signal.

   Adjust the video gain so that the echo does not exceed the specjet windows.

   Adjust the attenuation to the maximal signal, usually between 1 and 7 dB depending on the sample size, sample properties, resonator coupling and temperature.

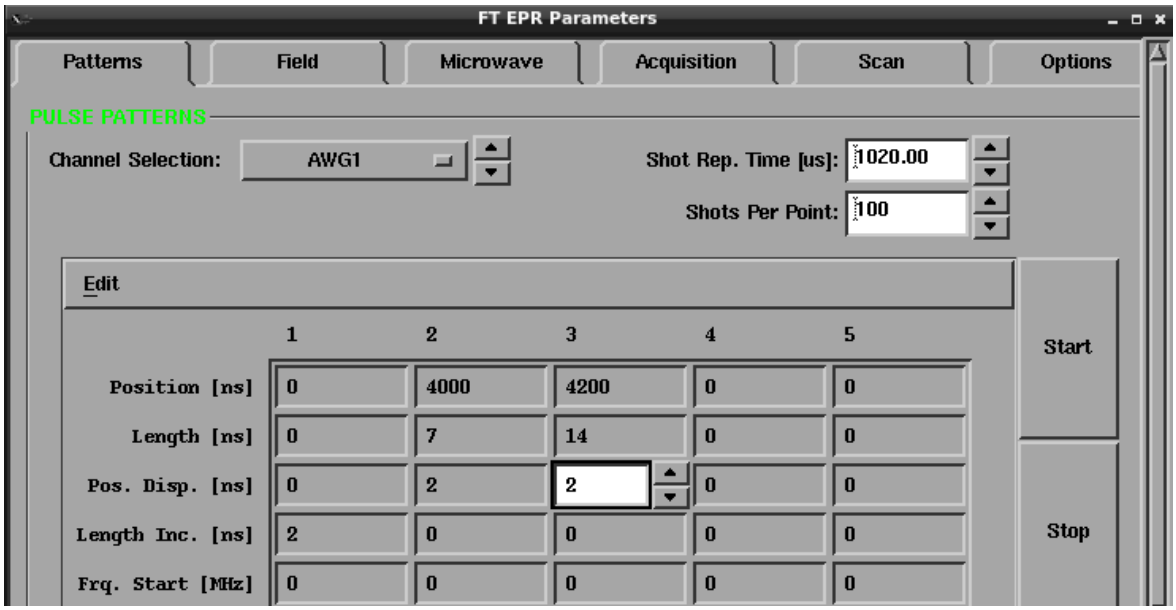
   Use arrow button with crtl and change the gain/attenuation by 1 directly. Use arrow with shift button to change the gain/attenuation by 10 directly.

   Here are some typical pulse length and power attenuation.

When attenuation is 5 dB, the pi/2 pulse is about 16 ns, pi pulse is about 32 ns.
When attenuation is 2 dB, the pi/2 pulse is about 12 ns, pi pulse is about 24 ns.

STEP 10. OPTIMIZE SIGNAL WITH A NUTATION EXPERIMENT (OPTIONAL)

a. Determine the width of the echo to integrate over and put this number into the "Length" of the acquisition trigger.
b. Under "Acquisition Trigger"
    i. Add 4000 to "Position".
    ii. Set "Pos. Disp." To 2 ns.
c. Under "AWG1"
    iii. Click "Edit" and "Insert Column"
    iv. Add 4000 ns to the position of pulse 2 and 3.
    v. Set pulse 1 "Length Increment" to 2 ns.
    vi. Set "Position Displacement" of pulse 2 and 3 to 2 ns.



d. Under "FT Bridge" → "Patterns" set "Shots per point" to 10
e. Under "FT Bridge" → "Acquisition" set "X-axis Size" to 256
f. Change X-Axis Quantity to "Time"
g. Change Acquisition mode to "Run from Tables"

h. Click “start”

STEP 12. COLLECTING 1D EPR SPECTRA

(1) Under “FT parameters” → “Acquisition” set Acquisition mode to “ran from Table”. **Do not run the filed sweep by the pulse program. It will frozen the software.**

(2) Measure the time position of the point at the beginning of the echo signal which is located at approximately ¼ of the hight. (“position” in the acquisition)

(3) Measure the echo signal width at approximately ¼ height. (“length” in the acquisition)

(4) Under “FT parameters” → “Acquisition” set x axis quantity “Magnetic field”

(5) Under “FT parameters” check the filed center and sweep width.

(6) Set the correct number in program for 1D magnetic field sweep

(7) Click the “run” in the “left bottom corner”. You should get a 1D ESR absorption spectrum

(8) Click “save” under “file” to save the spectra data.

STEP 6. SAVING THE DATA

As a File

File => Save as… => My Computer => D Disk => User’s name folder

As an ASCII file

File => Export… => Destination (notice the file type is ASCII format)

Open this file in Excel and it is delimited with tabs; format as necessary

ENDING WORK

1) Change the attenuation to 60 dB
2) change the TWT to standby by pressing the standby button, wait until it achieves standby condition
3) change the mode from “pulse” to “CW” (FT bridge)
4) In the tuning bridge, change to “tune” then change it to “standby”

5) Turn off the magnetic field off
6) Disconnect the spectrometer (acquisition tab in software)
7) Close the XEPR software
8) Change the setting of Temperature back to 10 K.
9) Move your sample (take out from the resonator). Put the black holder back to the resonator and tighten it.
10) Keep desk top clean
11) Logout from NUcore

TROUBLESHOOTING

For any problems in pulse X-band, please contact Dr. Jinlei Cui

REVISIONS

V 1.0 2025/02/16	Release of original version of IMSERC Pulse x-band V1.0
V 2.0 2025/02/16	Update of original version of IMSERC Pulse x-band V1.0, reflecting the upgraded of pulse X hardware.

APPENDIX A: HOW TO RUN WITH PULSE SEQUENCE EPR

For many experiments, such as T1, T2 and ESEEM, it is more convenient to run from the pulse sequence.

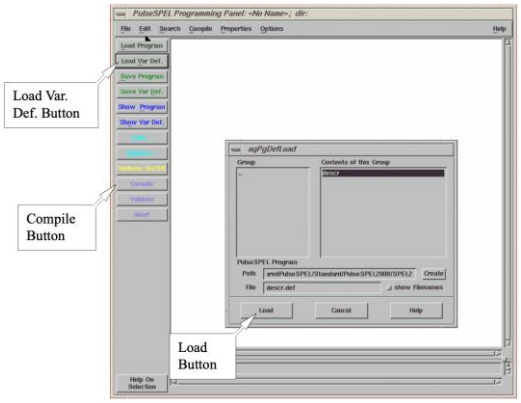
For 1D echo field sweep, and T2 relaxation.

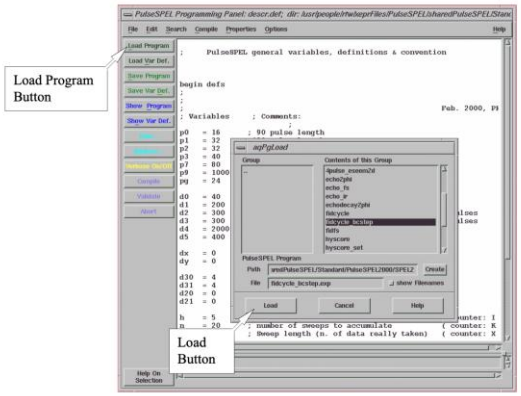
Load Program
i. /home/xuser/xeprFiles/PulseSPEL/IMSERC/IMSERC_Xband.exp
Load Variable Definitions
ii. /home/xuser/xeprFiles/PulseSPEL/IMSERC/IMSERC_Xband.def

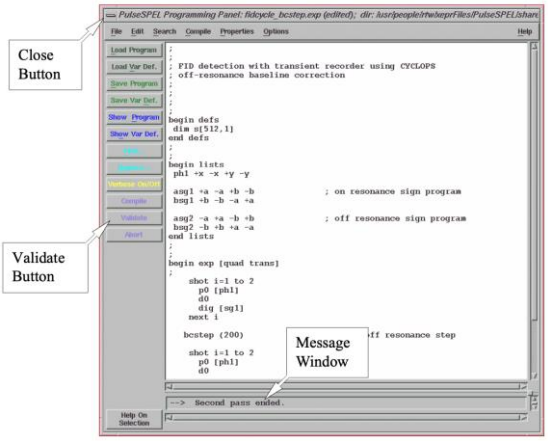
For T1.

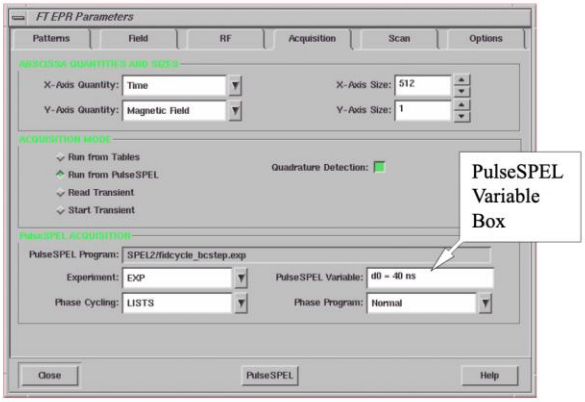
Load Program
i. /home/xuser/xeprFiles/PulseSPEL/IMSERC/T1_measurement.exp
Load Variable Definitions
ii. /home/xuser/xeprFiles/PulseSPEL/IMSERC/T1_measurement.def

The difference between T1_measurement.exp and IMSERC_Xband.exp is the former one uses the spg channel and the latter one uses the awg channel.


Load Var.
Def. Button
Compile
Button
Load
Button


Load Program
Button
Load
Button


Close
Button
Validate
Button
Message
Window


PulseSPEL
Variable
Box

APPENDIX B: T_1 AND T_2 MEASUREMENT

For T_1 and T_2 measurement, please first follow the previous steps to acquire the 1D EPR spectra. Then follow Appendix A to load the pulse sequence and parameters. Then please take the following steps to run the T_1 and T_2 measurement.

T_2 measurement:

a. Under "FT parameters" → "Acquisition" set Acquisition mode to "ran from PulseSpel"
b. In the "experiment" chose "2P ESE set up", phase cycle " 2 step". Make sure the set p0 and p1, attenuation and gain from previous steps.
c. In pulseSPEL variable, type d0 and set it to 0.
d. Type d1 and set it to 180 ns.
e. Type srt, set it to 1000*1.02 ns
f. Click the "run" in the left bottom corner and record the echo signal. Measure the time position of the point at the beginning of the echo signal which is located at approximately ¼ of the hight.
g. After that, change the d0 to adjust the beginning of echo to this point.
h. Measure the echo signal width at approximately ¼ height.
i. Type pg in pulseSPEL variable and set this number for integration of echo signal, for example 74 for the nitroxide echo.
j. Switch pulse sequence from "2p ESE set up" to " 2P ESE decay"
k. Type d30 and set it to 4 ns.
l. Change the dim2 to correct number for the echo decay. It should allow for seeing the whole echo decay to nearly zero.
m. Make sure P0 and P1 are correct.
n. Compile variable definitions and validate the program.
o. Change the phase cycle to "2 steps"
p. Click the "run" in the "left bottom corner". You should get an ESR echo decay curve.

T_1 measurement:

a. Load the program IMSERC/T1_measurement.exp
b. Do the same steps from step a to step i in T2 measurement.
c. Switch pulse sequence from "2p ESE set up" to " saturation recovery"
d. Type d30 and set it to 2000 ns. d19 to 8000 ns and d2 to 750 ns.
e. Change the dim4 to correct number for the saturation. It should allow for seeing the whole saturation recovery.

f. Make sure P0 and P1 are correct.
g. Set srt >2*d30*dim4
h. Compile variable definitions and validate the program.
i. Change the phase cycle to “2 steps”
j. Click the “run” in the “left bottom corner”. You should get an saturation recovery curve.
k. If you need run inversion recovery, then do the above steps, but make sure dim 5 is correct size, and set srt >2*d30*dim5. Also make sure P2 = pi pulse.