picoSpin 45 - Installation and Setup

⚠ WARNING

Avoid personal injury or equipment damage. Read and understand the accompanying documentation and all safety labels before attempting to install or use this equipment.

Install detailed information about the picoSpin from the documentation DVD that came with the system, or go to http://www.thermoscientific.com/picoSpin.
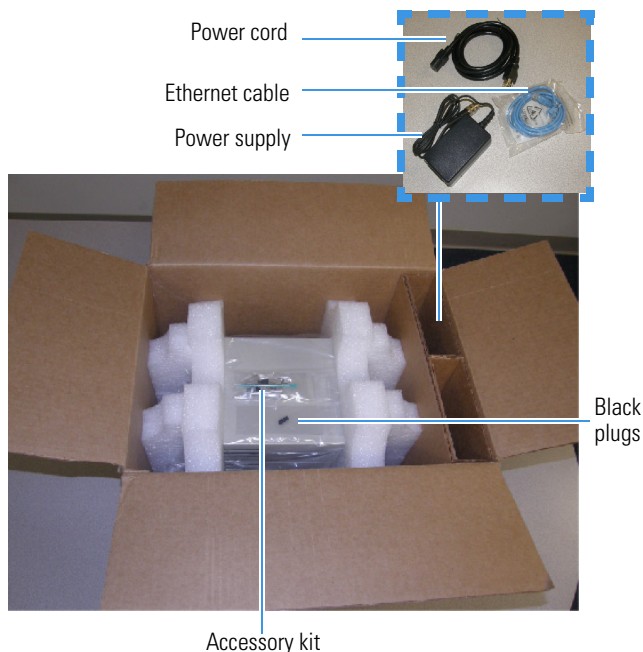
CAUTION Eye protection should be worn at all times while using this instrument. Please refer to OSHA standards on EYE and FACE protection when handling chemicals.

Introduction

The Thermo Scientific™ picoSpin™ NMR spectrometer is a complete pulsed Fourier transform liquid-phase proton NMR spectrometer. It includes a capillary cartridge, a permanent magnet, radio-frequency transmitters and receivers, digital data acquisition and signal processing, a programmable pulse sequencer, and a web-server user interface.
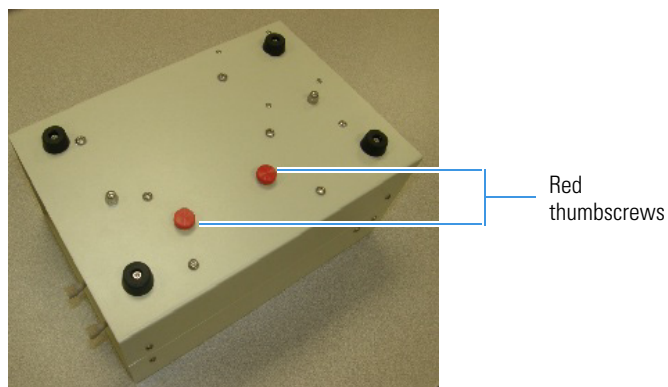
What's in the Box

Figure 1. Inside the box

Power cord
Ethernet cable
Power supply

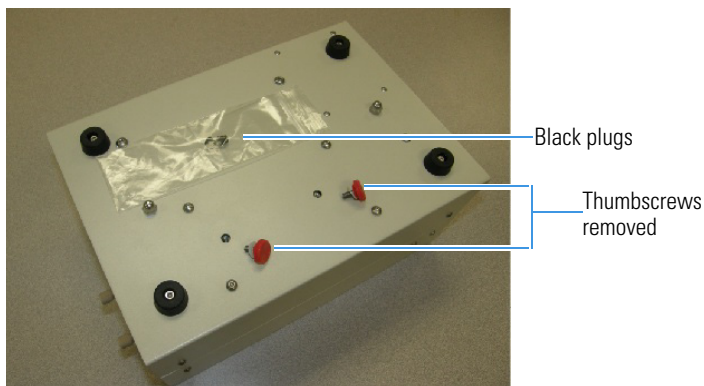Black plugs

Accessory kit

Unpacking the Spectrometer

1. Unpack the spectrometer.
2. Remove the shipping restraints.
   a. Turn the spectrometer over on its top.
   b. Turn the red thumbscrews counterclockwise until they can be removed.
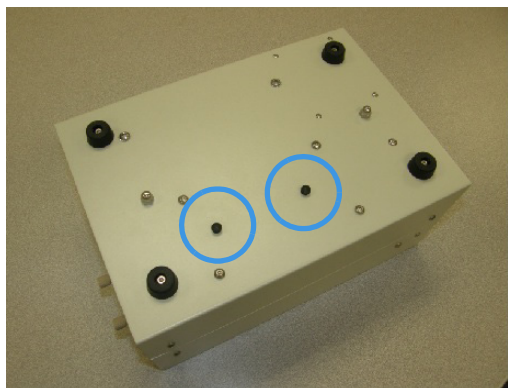
Figure 2. Bottom side of the spectrometer

Red thumbscrews

   c. Remove black plugs from the plastic bag.

Figure 3. Remove red thumbscrews

Black plugs

Thumbscrews removed

   d. Insert black plugs in the holes where the red thumbscrews were removed.

Thermo SCIENTIFIC

Figure 4. Black plugs inserted

e. Turn spectrometer over on its feet.

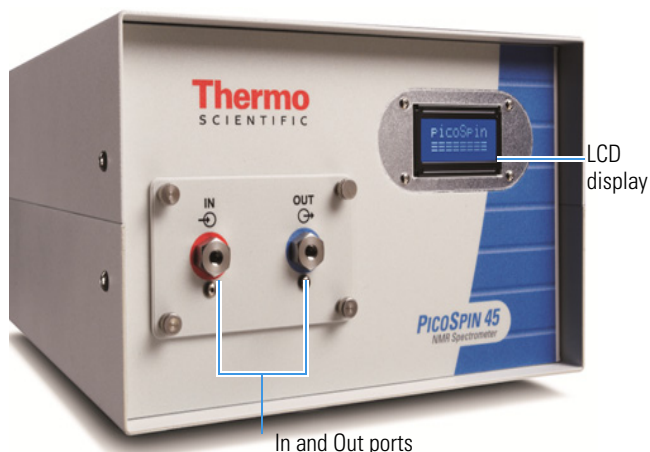## Spectrometer Components

Figure 5. Front of spectrometer

LCD display

In and Out ports

Figure 6. Back of the spectrometer

Vent

Auxiliary

Power switch

Serial number

Ethernet connector

DC Power connector

Rating label

Figure 7. Connecting the spectrometer cables

On/Off switch

Ethernet connect

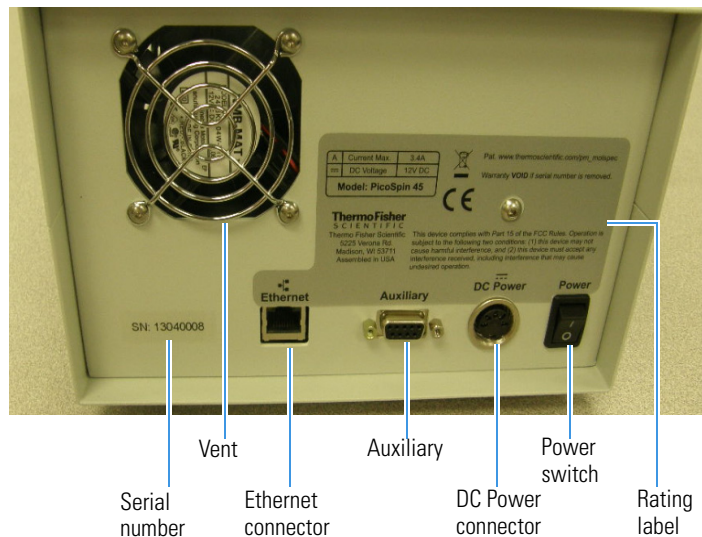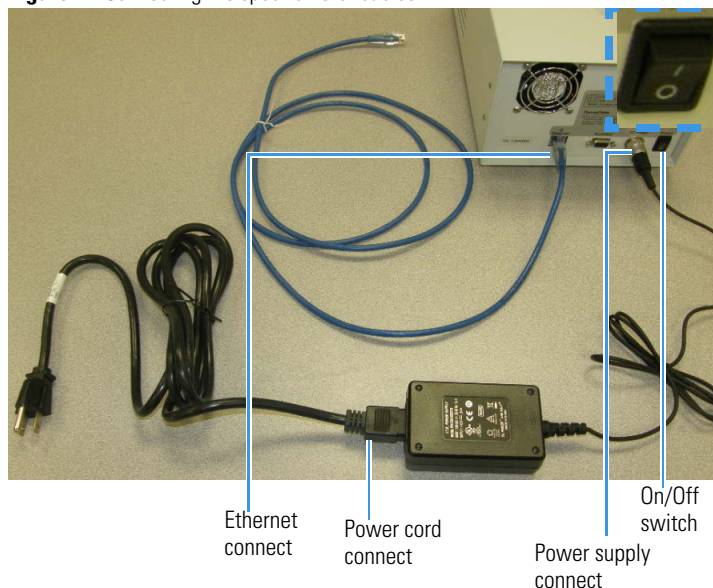Power cord connect

Power supply connect

1. Plug the power cord into the power supply.

2. Plug the ethernet cable into the computer or into your LAN, and connect the other end of the cable to the spectrometer.

3. Turn on the computer.

We recommend Mozilla's free Firefox™ web browser for best compatibility with our software, but most modern browsers will work equally well, including Google Chrome™ and Safari™, and Internet Explorer™ 10 and 11.

Ethernet Connection

Connecting through a LAN

1. Switch on the unit while watching the front panel LCD display.

   After about one minute an IP address appears on the front panel. This address has been assigned by your LAN using DHCP. (See "Setting up a Computer to Use a Static IP Address" if the address displayed is 192.168.42.31.)

   Type the IP address in the navigation toolbar of your web browser.

Figure 8. picoSpin 45 welcome screen

Thermo Scientific™ picoSpin™ 45 — *compact NMR spectrometer*

For example, if the address displayed is 192.168.2.12, type http://192.168.2.12 into the address field of the browser.

– Automatic assignment of the IP address by the LAN will only be successful if the unit is switched on after it has been connected to an active Ethernet port on your LAN, and then only if your LAN has DHCP capability.

– If the unit is not assigned an IP address by DHCP it will display a factory default IP address of 192.168.42.31.

In most cases, you will not be able to communicate through your LAN to this address because LANs are configured to communicate only with a subset of all IP addresses.

For example, your LAN might only allow communication between IP addresses of the form 192.168.2.x, where x is a number between 0 and 255. If you see the default IP address and you are sure that your LAN does support DHCP, try using the same cable to connect another device to the LAN Ethernet port you have chosen to make sure it is active.

– If the unit is assigned an IP address by DHCP and you cannot connect to it with your browser, then additional login credentials may be required. For guidance, consult your IT administrator on completing the LAN connection.

After the welcome screen has been displayed for a moment, it will be replaced by the Run page, which is used to enter experiment parameters, start experiments and monitor their progress.

The orange text links at the upper right of the page are used to navigate between pages.

If you are unable to connect to the spectrometer through your LAN, make a direct connection.

Direct Connection

The computer must have an available Ethernet port with a working network adapter. If you are unsure whether the Ethernet port is working, try using it to connect to a network.

1. Turn the spectrometer power switch on.

2. After about a minute, the front panel LCD will display the factory default IP address of 192.168.42.31.

3. Configure the Ethernet port on the computer so it can communicate with the default IP address.

   To do this, set the IP address of the computer to another address on the same subnet. See "Setting up a Computer to Use a Static IP Address."

   For example, set the subnet mask to 255.255.255.0 and the IP address to 192.168.42.10.

   The details of setting up Ethernet ports and network adapters on different computers vary widely. For help, consult your computer's documentation or your IT support staff. See "Setting up a Computer to Use a Static IP Address." for more detail.

4. Once the port has been set up, type **http://192.168.42.31** into the address field of your browser.

Setting up a Computer to Use a Static IP Address

In this section we outline how to set up your computer for a direct Ethernet connection to the picoSpin. In this mode of operation you must connect an Ethernet cable from the Ethernet port on the back of the spectrometer directly to the Ethernet port on your computer.

Windows 7 and Windows 8

1. Navigate to the networking configuration.
   a. Select **Control Panel** > **Network and Internet** > **Network and Sharing Center**.
   b. Select **Change adapter settings**.
   c. Select **Local Area Connection**.
   d. Select **Internet Protocol Version 4 (TCP/IPv4)**.
   e. Click **Properties**.
   f. Select **Alternate Configuration**.
   g. Select **User configured**.

2. Type in the following fields:
   IP address: **192.168.42.10** Subnet mask: **255.255.255.0**

3. Click **OK**.

4. Click **Close**.

   To check the configuration, launch the Command Prompt and type ipconfig. The output should indicate the IP address you entered above.

Linux Debian

In Debian edit the file /etc/networking/interfaces for the wired network interface card you intend to connect to the unit. For computers with one network card the interface is typically called eth0 and that section would look like:

iface eth0 inet static

address 192.168.42.1

netmask 255.255.255.0

network 192.168.42.0

broadcast 192.168.42.255

gateway 192.168.42.1

Magnet Temperature Control

After start up, it may take from 20 minutes to 1 hour for the magnet to stabilize at the setpoint temperature, depending on the starting temperature of the magnet.

❖ **To check the status of magnet temperature stabilization**

Click the **Temperature** link at the upper right to navigate to the temperature controller page.

A setpoint of 42.0 °C was used when the unit was tested at the factory. Both the magnet Larmor frequency and the optimal shim settings are a function of the magnet setpoint temperature, so it is important to use the factory setpoint temperature for initial setup.

If the magnet is not under temperature control, check the controller settings.

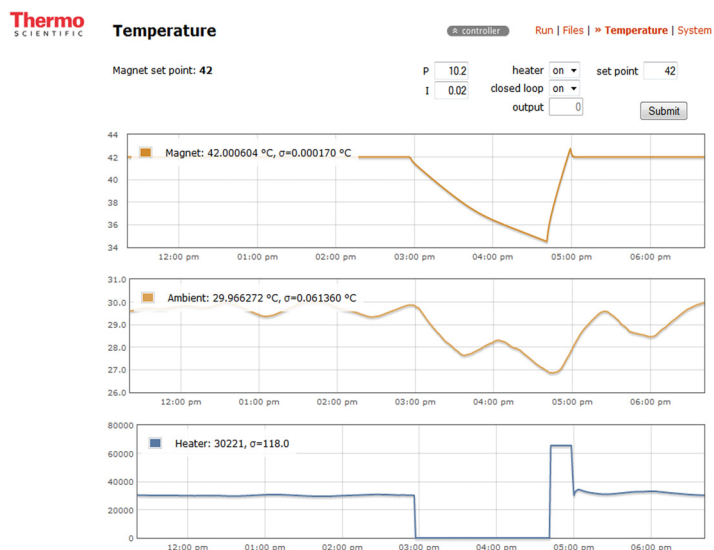❖ **To check the controller settings**

On the **Temperature** page:

1. Click ⌄ controller near the top of the page to display the temperature controller settings.

2. Verify the temperature controller parameters match those in Figure 9.

    a. P = 10.2, I = 0.02

    b. heater = on

    c. closed loop = on

    d. setpoint = 42

3. Click [Submit] .

Figure 9. Temperature page for a typical warm up

Note For best performance and temperature stability, it is recommended that the spectrometer be left turned on at all times.
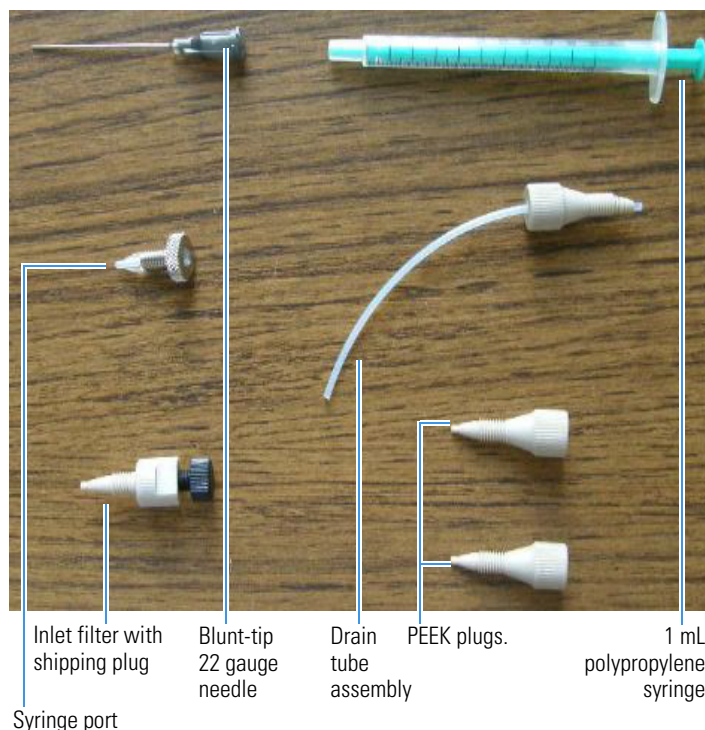
Injecting a Sample

WARNING Avoid personal injury.
- Needles and syringes should be considered regulated waste regardless of use
- Follow your local EH&S guidelines for disposal
- Never throw these items into the regular trash or dumpster

Figure 10. Accessory packet

Inlet filter with shipping plug   Blunt-tip 22 gauge needle   Drain tube assembly   PEEK plugs.   1 mL polypropylene syringe

Syringe port

CAUTION Avoid personal injury.
- Wear eye protection at all times when handling liquid chemicals
- Do not breathe hazardous vapors
- Avoid skin contact with hazardous liquids and vapors
- Eliminate ignition sources and prevent significant waste volume buildup

1. Remove the protective tape on the inlet and outlet fittings.
2. Insert the drain tube into the outlet fitting on the front panel.
3. Gently finger-tighten the PEEK nut.

   The drain tube should be directed into a small bottle.
4. Remove the shipping plug from the inlet filter.
5. Screw the filter into the inlet fitting, tightening it gently.
6. Screw the syringe port into the inlet filter but do not tighten it.
7. Put the blunt-tip needle onto the syringe and draw a few hundred microliters of fluid into the syringe, being careful not to draw up any air bubbles.

   Use water for initial shimming. Alternatively, you can use acetone in place of water but note that the signal amplitude will be reduced to 37% of the values in the test report.
8. Insert the needle into the syringe port and gently tighten it.
9. Gently inject fluid into the cartridge until you see it flowing out of the outlet and no air bubbles are visible in the drain tube.

Figure 11. Injecting fluid into cartridge

> **NOTICE** Do not exceed the 100 psi (700 kPa) maximum pressure of the cartridge. This pressure corresponds to a force of 3 lbf (1.4 kgf) applied to the 1 mL syringe plunger. If you use a syringe with a smaller plunger area, the maximum pressure will be generated with a smaller force

– Take care not to inject air into the cartridge
– If an air bubble develops at or near the NMR RF coil, the signal may be weak or it may be completely absent
– Any time you fail to find an expected signal keep in mind that it could be due to an air bubble

10. Remove the syringe port and the drain tube.
11. Plug the inlet and outlet with the two PEEK plugs from the accessory package.
12. Gently finger-tighten the plugs to seal the ports.

> **NOTICE** When the spectrometer is in continuous use it is sufficient to plug just one of the ports. However, it is poor practice to leave the instrument like this for long periods. Cartridges will last longest if they are filled with clean solvent and with both ports plugged.

Finding the Signal

1. Go to the **Temperature** page and check that the magnet temperature has stabilized to within a few millidegrees of the setpoint.

   The current temperature can be found in the upper left corner of the magnet temperature plot.

2. Go to the **Files** page.

   The Files page provides access to previously saved experiments, together with their settings and data. It also allows you to select shim settings files and experiment scripts.

3. Click the **autoShim QuickStart** saved run.
4. Click **use these settings at Run**.

   You will be transferred to the Run page, which will display settings similar to those shown in Figure 12.

5. Click `Start Run`.

A signal in the spectrum window appears as shown in Figure 14

a. Verify parameters match those in Figure 12.
   – Do not change the pulse length, it is the optimized 90 degree pulse length for your instrument
   – The test run box should remain checked

> **Note** The auto tx and auto tx offset parameters allow the spectrometer to track and change the transmitter frequency (tx frequency) during shimming. • Auto tx uses the auto tx offset parameter value to place the weighted average of signals at the offset frequency.
>
> • When checked, the tx frequency discovered using auto tx becomes a global value that is shared with other experiment scripts, such as the onePulse script.
> • Turning off auto tx allows for manual control of the transmitter frequency.
> • Checking the test run box in the autoShim script overrides auto tx selection even if checked. While test run is checked auto tx will not make changes to the tx frequency.
> • Test run is designed for evaluating parameter values without changing them.
> • Unchecking test run reestablishes auto tx control to discover and change the tx frequency.

For shimming with water, the offset value should be set between 200 and 500 Hz. The default QuickStart auto tx offset value is set to 200 Hz.

b. Verify the signal appears in the spectrum window.

   It should appear at the auto tx offset value.

c. If any parameters are changed click `Start Run` again.

   The true signal position is indicated in the text window at the bottom of the page; it is found next to the words spectral center (Figure 13). The auto tx feature will change the tx frequency after the second RF pulse and will write a new tx frequency value to all experiment scripts when the script completes, or when the script is aborted.

Figure 12. autoShim with QuickStart parameters

d. If no signal appears in the spectrum window, reinject water and verify the bandwidth and min. and max freq. to plot values (Figure 12).

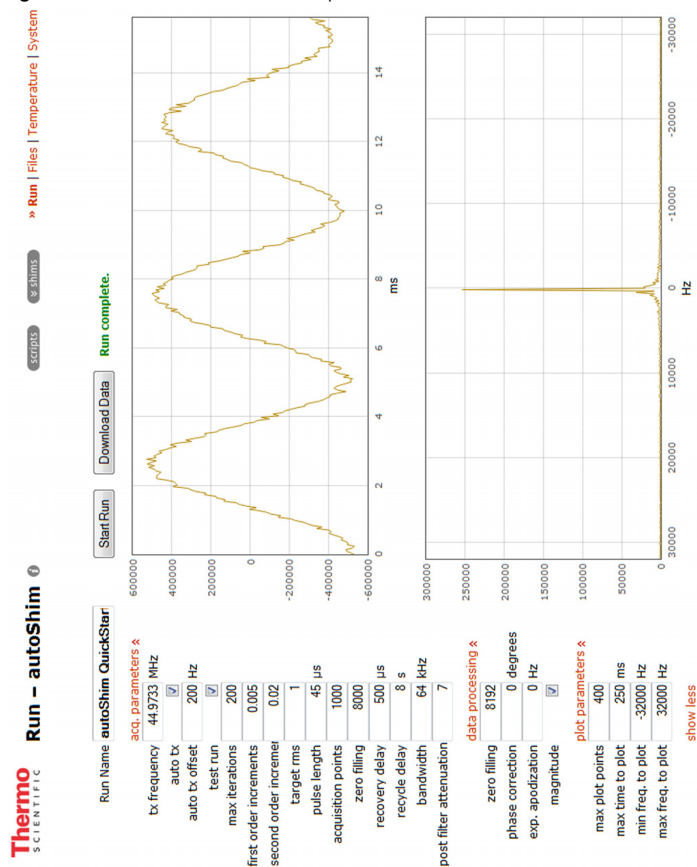e. Click **Start Run** again.

f. Change **max iterations** to **1**.

g. Uncheck the test run box and click **Start Run**.

Your true transmitter frequency will now appear in the tx frequency field. Proceed to the Automatic Shimming section.

Figure 13. Spectral center

```
setting shims: [-0.01033, -0.03945, 0.03
spectral center = +1767 Hz
spectrum peak height: 149030
script autoShim completed successfully
```

Figure 14. autoShim with QuickStart parameters

Automatic Shimming

Note Allow approximately 20 to 30 minutes for each 100 iterations run in the autoShim script.

1. If you are not already in the autoShim script, go to the **Run** page and select the **autoShim** script from the **scripts** drop-down menu at the top right of the page.

2. Verify parameters match those in Figure 15.
   - Do not change the value of pulse length, it is the optimized 90 degree pulse length for your instrument
   - The test run box should remain checked
   - If any parameters are changed, click **Start Run** again

Figure 15. Phase I autoShim script parameters
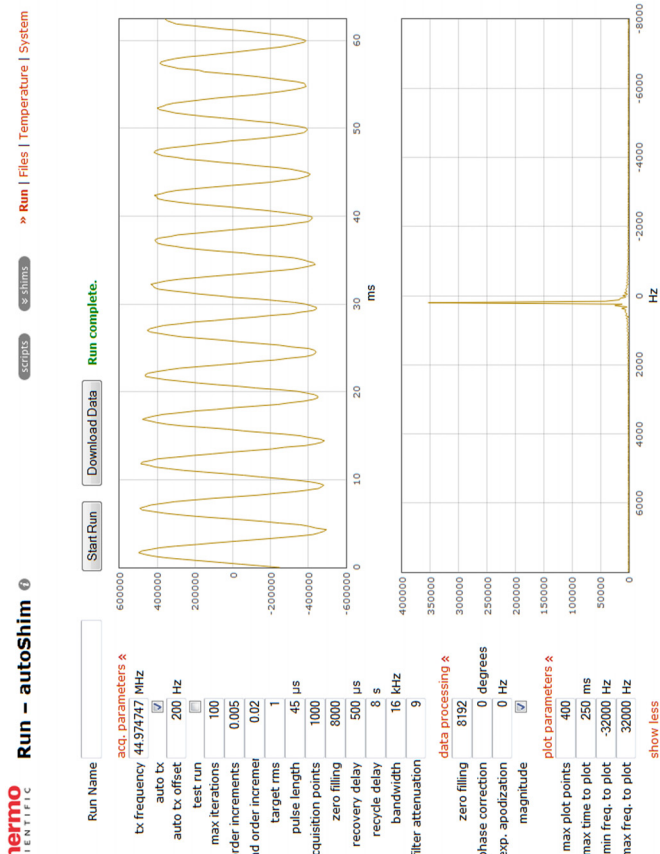

3. Verify the signal appears in the spectrum window and looks similar to Figure 14.

   The signal appears at the auto tx offset value.

   If any parameters are changed click Start Run .

Figure 16. autoShim after phase I shimming

4. Click Start Run to start phase I shimming.

5. Upon completion of phase I shimming, a screen similar to Figure 16 appears.

6. Recheck the **test run** box.

7. Adjust the parameters to match those in Figure 17.

Figure 17. Phase II autoShim script parameters

8. Click Start Run .

9. Uncheck the **test run** box and click Start Run to begin phase II shimming.

   Upon successful completion of phase II shimming, a screen similar to Figure 18 appears.

❖ **To determine if the instrument is properly shimmed**

1. Evaluate the FID and spectrum signal - look for a slow decay of the FID (intensity of the FID should decay to half the initial intensity).

2. Spectrum peak height is reported in the text at the bottom of the Run page upon completion of the autoShim script.

   Compare the spectral peak height displayed in the message window of the web browser to the value provided in the factory test report. The spectrum peak height should be within ~15% of the factory test report value; the factory spectrum peak height was obtained fully relaxed water.

   > **Tip** If the unit has been moved, power cycled, or subjected to temperature extremes since it was tested at the factory, it may be necessary to run the autoShim script several times before the performance found at the factory is reestablished.

❖ **To save your shims**

The autoShim script automatically saves the new shim values to the working memory, but it does NOT save the shims to a named shim file when it completes unless a name has been entered in the **Shims Name** field.
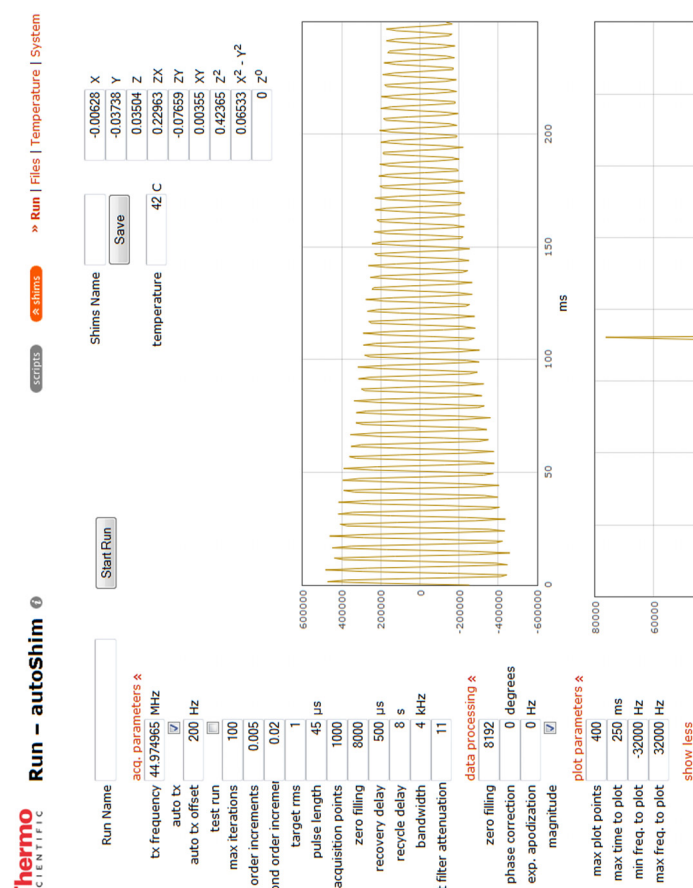
Tip It is recommended that you save a named shim file at least once a week.

1. When autoShim completes, save the shims by typing a name into the **Shims Name** field and click **Save**.

   The new shim file will then appear on the Files page.

   NOTICE Anytime Start Run is pressed, shim values are written to the shim electronics. If a file name is present in the Shims Name field, clicking Start Run will also cause the named shims file to be written and saved to memory. Saved shim files can be overwritten if the Shims Name field is not cleared; no file overwrite warning message will appear. It is possible to overwrite good shim values by clicking Start Run in execution and test run mode if the Shims Name field is not cleared.

Figure 18. autoShim screen after Phase II shimming

Your First Spectrum

After shimming, if the auto tx box was checked, the tx frequency discovered in the autoShim script will be written to the tx frequency field in the onePulse script.

1. Follow the instructions on Injecting a Sample, fill the cartridge capillary with the sample fluid.

   Ethyl acetate is a good choice.

2. Click **scripts** in the upper right corner of the screen.

3. Select the **onePulse** script from the scripts dialog window that appears.

   If this is the first time using the onePulse script, the factory default onePusle QuickStart values will populate the parameter fields.

   If in doubt, go to the **Files** page, click the **onePulse QuickStart** saved run, and click **use these settings at Run**.

   You will see settings similar to those shown in Figure 19.

Figure 19. onePulse with QuickStart parameters

4. Verify the parameters match those in Figure 19.
   – Do not change the value of the pulse length, it is the optimized 90 degree pulse length for your instrument
   – If any parameters are changed, click Start Run again
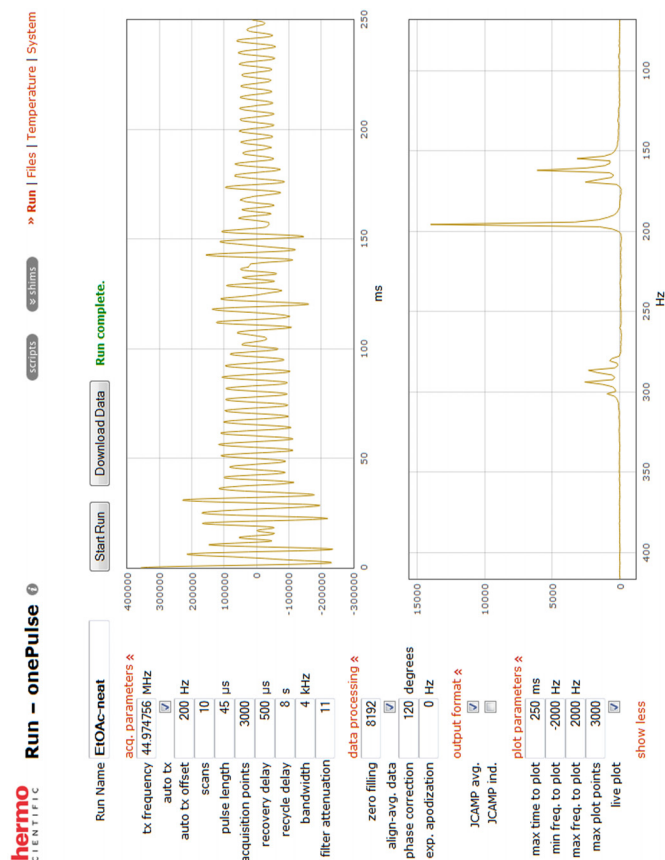
5. Click Start Run and examine the plots.

   Tip Plotting fewer points reduces the load on the server so that live plots can be viewed during the run without extending the desired recycle time. The plot settings have no effect on the data that is saved.

   Make adjustments to the auto tx offset value and phase correction until the spectrum looks similar to Figure 20.

   Keep the **min.** and **max freq. to plot** fixed at ±2 kHz and use the interactive mouse zoom feature to zoom spectrum region of interest.

   Note The auto tx feature uses a weighted average of signals present in the full bandwidth, regardless of user supplied min. and max freq. to plot values.

Figure 20. onePulse script with ethyl acetate

With a neat sample you will be able to see the main spectral features from a single FID. In this case, we see a CH_3 singlet, a CH_3 triplet, and a CH_2 quartet.

6. Once you have the desired settings, set scans to 10.

7. Name the Run, such as "EtOAc-neat."

8. Click Start Run.

9. After the run finishes a Download Data button (Download Data) will appear next to Start Run button.

10. Click Download Data and, in Firefox, a file dialog window will appear.

11. Select **Save File** and click **OK**.

    This will save the file to your computer's download directory.

> **Tip** In Firefox, you can also select the option Open with, select Mnova from the drop-down menu and check the **Do this automatically...** and the files will automatically open in Mnova.

12. Open the file EtOH-neat-avg.jdx in Mnova; it contains the 10 scan averaged FID data in JCAMP-DX format.

Mnova

Every picoSpin comes with a one-year free license to the Mnova NMR data analysis package from Mestrelab Research. Mnova is a powerful package that makes all common NMR analysis tasks fast and easy. To learn the basics of Mnova, download the manual from the Mestrelab site and read the first 5 sections of Chapter VIII, *Processing Basics.*
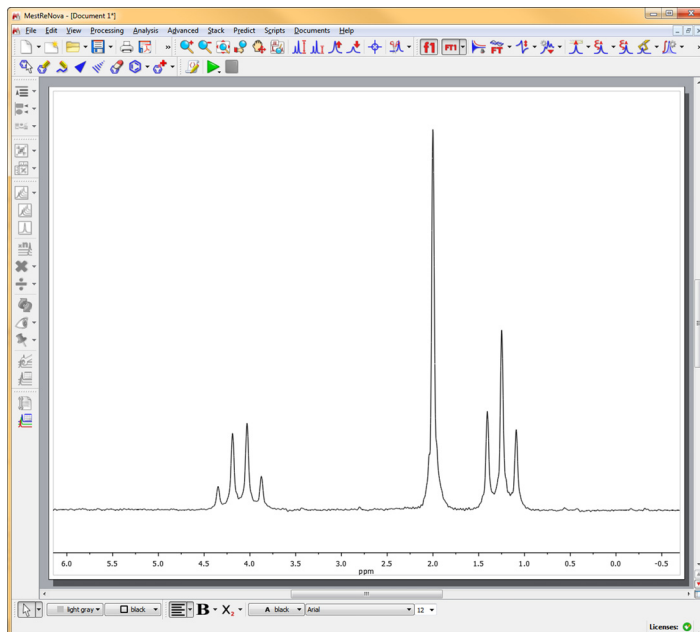
Figure 21. Ethyl acetate in Mnova

Figure 21 shows the result of processing the EtOAc-neat-avg.jdx file in Mnova as follows:

- open the JCAMP file
- set zero filling to 32,000
- LP filling: forward LP
- backward LP: -2 to 0
- apodization: 0.5 First Point
- adjust zero-order phasing
- zoom axes
- reference the methyl singlet to 2.0 ppm

These steps were done manually but Mnova can perform these tasks automatically with processing scripts and can perform many other useful processing steps, including multiplet analysis, peak integration and peak fitting.

Notes:

Notes:

Contact Information

For U.S. Technical Support, please contact:

Thermo Fisher Scientific
5225 Verona Road
Madison WI 53711-4495 U.S.A.
Telephone: 1 800 532 4752
E-mail: us.techsupport.analyze@thermofisher.com

For International Support, please contact:

Thermo Fisher Scientific
Telephone: +1 608 273 5017
E-mail: support.madison@thermofisher.com

Trademark Information

Microsoft, Windows and Internet Explorer are either trademarks or registered trademarks of Microsoft Corporation in the United States and/or other countries. Mnova is a trademark of Mestrelab Research, Inc. and its subsidiaries. Safari is either a trademark or registered trademark of Apple Inc. in the United States and/or other countries. Firefox is a trademark of the Mozilla Foundation in the United States and/or other countries. Google Chrome is a trademark of Google Inc. All other trademarks are the property of Thermo Fisher Scientific Inc. and its subsidiaries.