Turning the Spectrometer On 3.1

If you are not sure how the electric power or water is connected, consult your local instrument or facilities manager.

1. **Turn on the power for the system.** How you do this depends on how the electric power was hooked up when the spectrometer was installed. Most likely you will activate the switch on the breaker box for the spectrometer. Breaker boxes are usually mounted on the wall. Consult the local instrument or facilities manager if you are not sure where the breaker box is.

2. **Turn on the tap water for cooling.** There are usually two valves, one for the supply and one for the return (or drain). Consult the local instrument or facilities manager if you are not sure where the valves are.

3. **Start the Xepr Program.** Log onto the UNIX workstation. If you are unfamiliar with UNIX, please refer to Appendix A or Appendix B for UNIX tips. We recommend using the xuser user account which is set up by the Bruker service engineer when the spectrometer is installed. The initial password for this account is user@xepr. Double-click the Xepr icon on the desktop to launch the program. (See Figure 3-2.)

Figure 3-2   The Xepr program desktop icon.

4. **Turn on the power for the console.** The power switch (green button) for the console is located in the upper left front corner of the console. (See Figure 3-3.) The acquisition server will boot and initialize all the modules of the Elexsys E 500 spectrometer. This process may take 30 seconds or longer: meanwhile you can continue with the next steps.
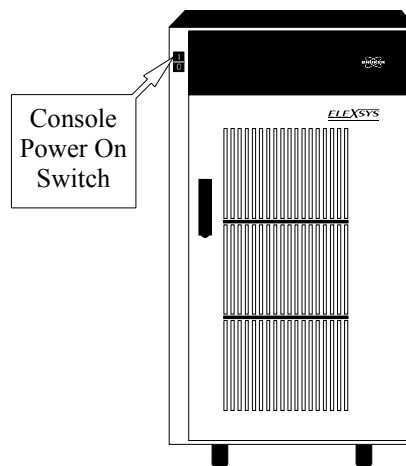

Console
Power On
Switch

Figure 3-3   The location of the console power switch.

5. **Turn on the heat exchanger and magnet power supply. (Instructions for Small Power Supplies.)** Follow this step if your power supply looks like the power supply in Figure 3-4. You must first turn the heat exchanger on by activating the power switch. The location of the power switch may vary depending on your heat exchanger. To turn the power supply on, push its POWER ON/OFF button. Go to Step 7.

Power Switch

Power On/Off Switch

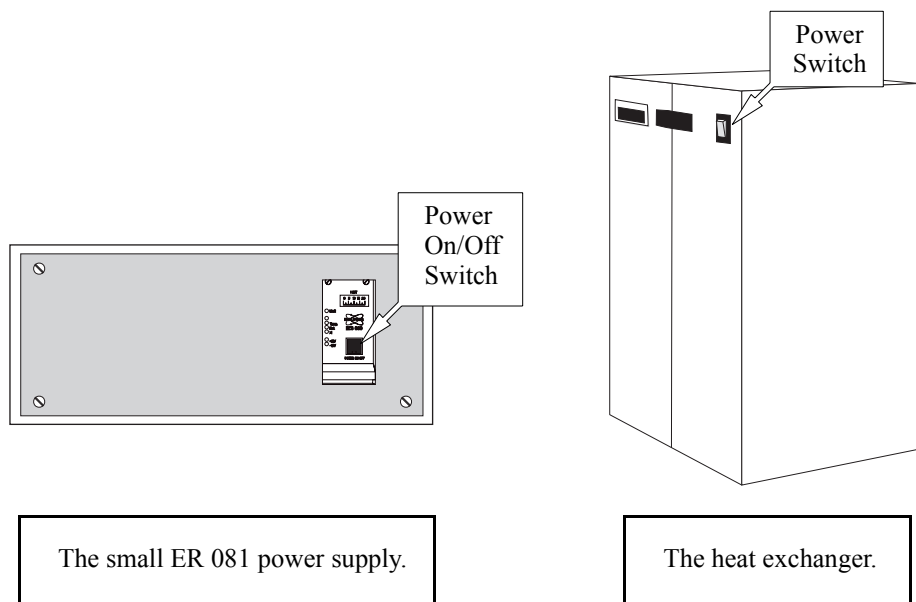The small ER 081 power supply.

The heat exchanger.

Figure 3-4   The small ER 081 power supply and the heat exchanger.

6. **Turn on the heat exchanger and magnet power supply. (Instructions for Large Power Supplies)**
Follow this step if your power supply looks like the power supply in Figure 3-5. On systems with large power supplies, you need to first press the ELECTR. ON button and then the POWER ON button. Pressing the POWER ON button also starts the heat exchanger. If not, make sure that the power switch on the heat exchanger is activated.

Electricity On

Power On

Power Switch

The large ER 082/083/085/086 power supplies.
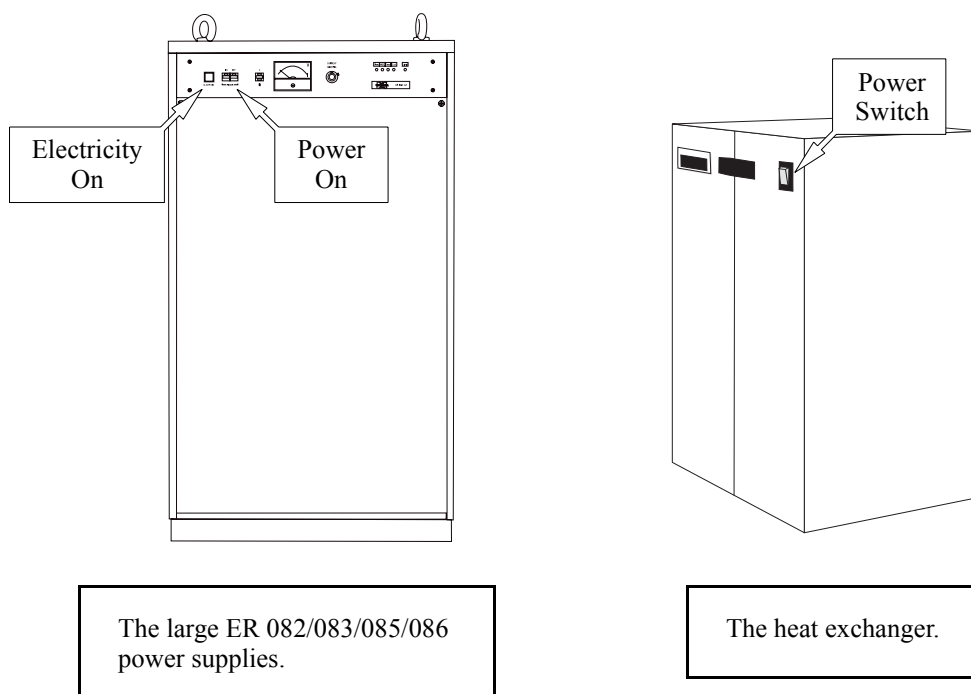
The heat exchanger.

Figure 3-5   The large power supply and the heat exchanger.

7.   **Connect to the spectrometer.** To control the spectrometer from the Xepr program you need to connect the workstation to the spectrometer. Click the Acquisition menu bar and then Connect to Spectrometer. A dialog box will appear with the Server Name: click OK. (See Figure 3-6.)
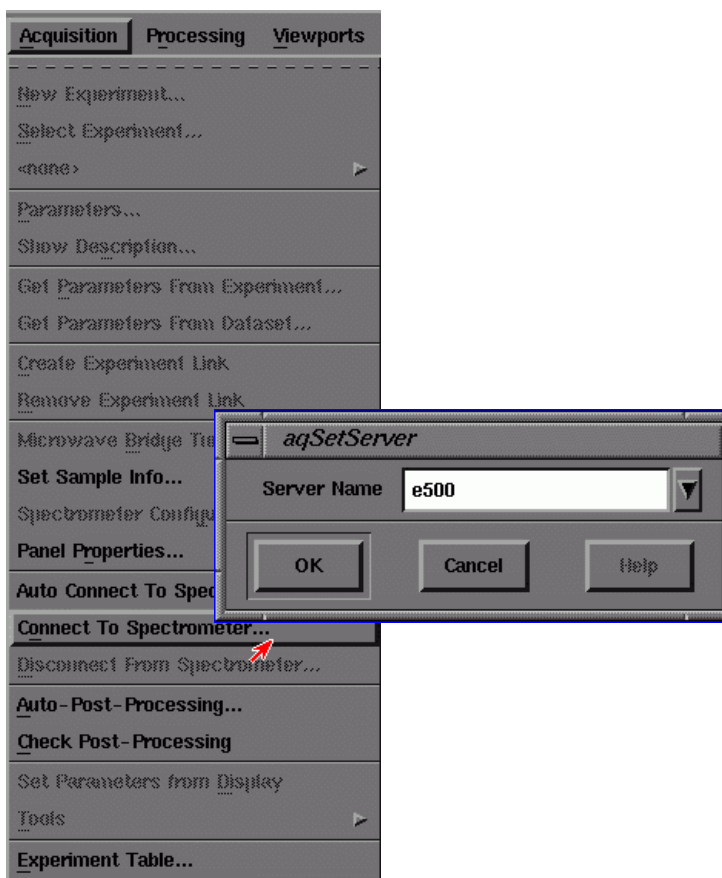

Figure 3-6   Connecting to the spectrometer.

When the connection is complete a monitoring panel will appear in the Xepr window. (See Figure 3-7.) The monitoring panel may appear on top of the viewport or at the bottom of the viewport depending on your default settings.

If you were not successful in connecting to the acquisition server, consult Chapter 17 of the Elexsys E 500 User's Manual: Advanced Operations.
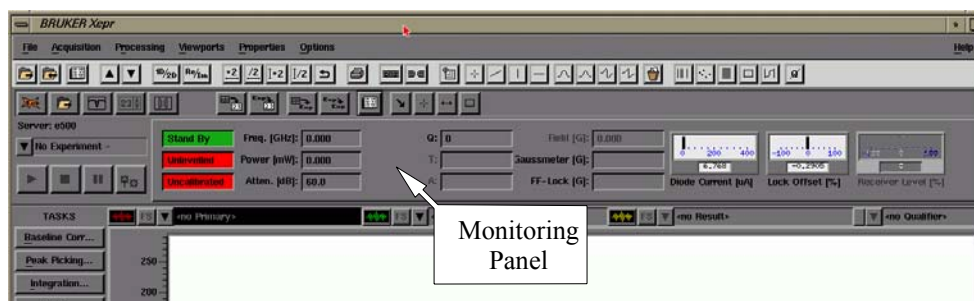

Figure 3-7   The Xepr window after connecting to the spectrometer.

8.   **Install an EPR cavity if there is not one presently installed.** The instructions in this chapter assume you are using a properly installed Bruker ER 4122SHQ or ER 4102ST cavity. If there is no cavity installed or the installed cavity is not one of them, seek the assistance of a knowledgeable EPR colleague or refer to Section 7.2 to learn how to install a cavity.

9.   Proceed to Section 3.2.

Tuning the Microwave Cavity and Bridge 3.2

1. **Open the Microwave Bridge Tuning dialog box.**
Click the Tuning button in the monitoring panel. The Microwave Bridge Tuning dialog box will then appear. (See Figure 3-8.)
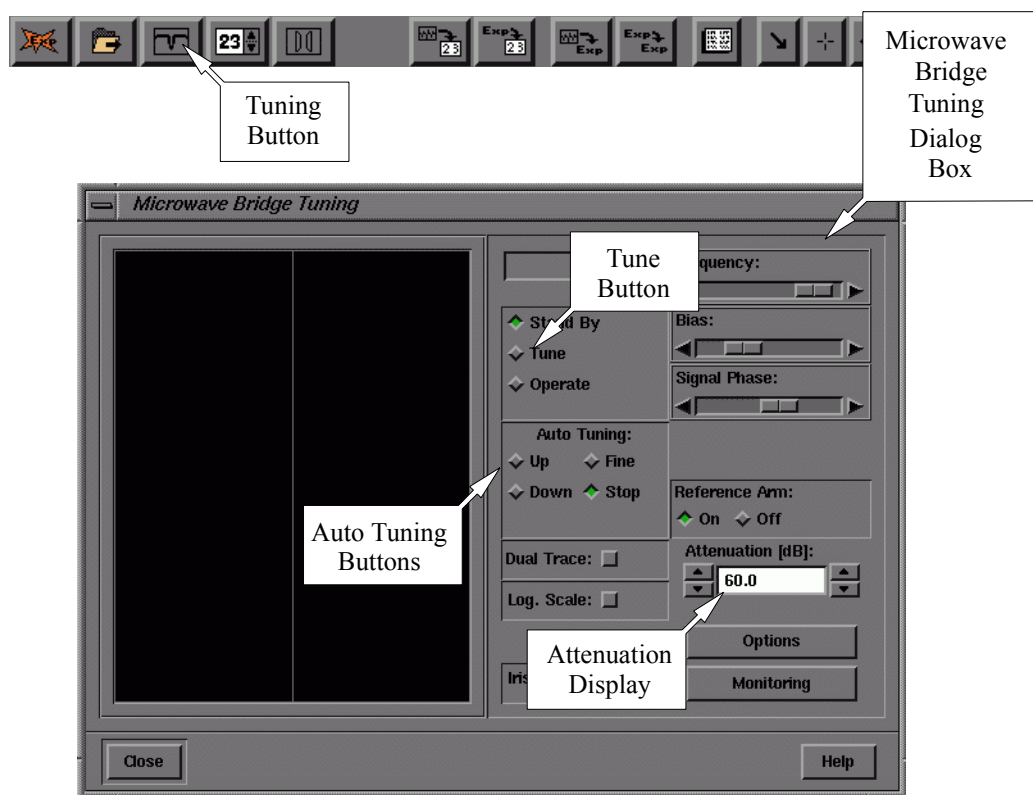

Figure 3-8   The Microwave Bridge Tuning dialog box.

There are three states or modes for the microwave bridge: StandBy, Tune, and Operate. When you connect to your spectrometer, the bridge is in Stand By, indicated by a green Stand By button. (See Figure 3-8.) If you have been acquiring spectra already, your bridge will probably be in Operate.

Take care if you are wearing an analog (mechanical) watch. The magnetic field in the air gap of the magnet is sufficiently strong to magnetize your watch! Therefore, to avoid damage to your watch, remove your watch before putting your hands in the magnet air gap.
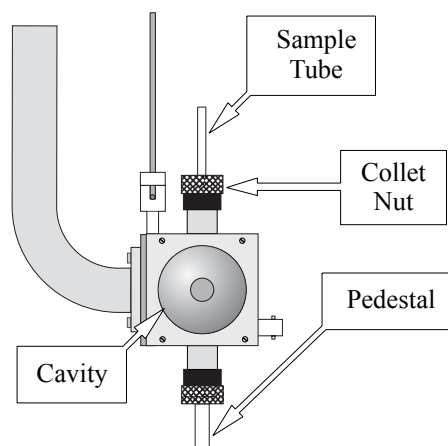
2. **Switch the microwave bridge to Tune.** Click the Tune button in the dialog box to change to Tune.

3. **Set the microwave attenuator to 30 dB.** The microwave attenuation is set by clicking the arrows on either side of the attenuation display in the dialog box. (See Figure 3-8.) The arrows on the left change the attenuator in 10 dB steps; those on the right in 1 dB steps.

4. **Remove the sample.** If there already is a sample in the cavity, remove it. Loosen the top collet nut (You do not need to remove the collet nut.) and carefully remove the sample from the cavity. Pulling the sample tube out as straight as possible prevents you from breaking the sample tube thereby destroying your valuable samples. (See Figure 3-9 and Figure 3-10 for details.) The ER 4122SHQ cavity is shown in the figures; however, the procedure of removing and inserting a sample is the same for ER 4102ST, ER 4122SHQ, and ER 4122SHQE cavities.

Figure 3-9   A Bruker ER 4122SHQ cavity.
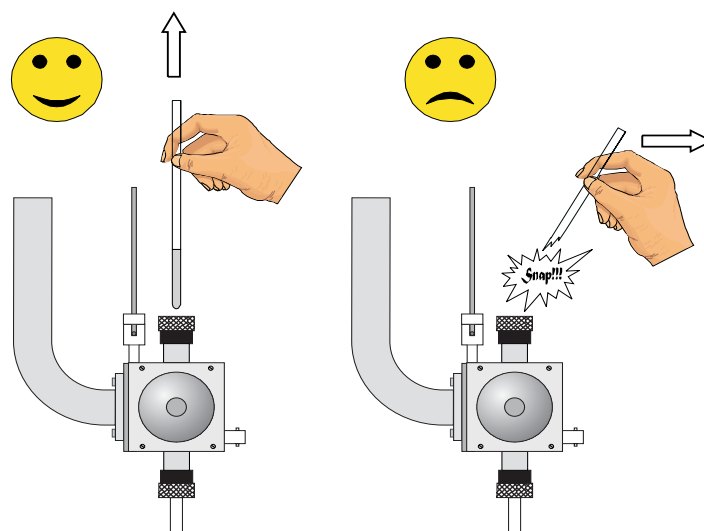
Figure 3-10   The right and wrong technique for removing a sample.

5.   **Clean the sample tube to be inserted into the cavity.** It is vital to avoid contaminating the microwave cavity with paramagnetic contaminants that produce spurious EPR signals or distorted base lines. Wiping the outside of the sample tube with tissue paper is usually adequate.

If this is your first time operating an Elexsys E 500 spectrometer, we recommend that you use the strong pitch sample supplied with your instrument. Our instructions in this chapter are based on using this sample.

Make sure that the pedestal is not in the cavity, as it can give an EPR signal and will also degrade the sensitivity.

6. **Insert the sample tube carefully into the cavity.** (See Figure 3-9 and Figure 3-10.) Make sure you have the appropriate collet size for your sample tube size. The tube should be slightly loose before you tighten the collet nut. The bottom of your sample should rest in the indentation on the pedestal. This ensures that your sample is centered horizontally. The height can be adjusted by loosening the bottom collet nut and moving the pedestal up and down. Tighten the top collet nut to firmly hold the sample tube in place and the bottom collet to firmly hold the pedestal.

7. **Tune the bridge and cavity.** Pressing either the Up button or Down button of Auto Tune starts the automatic tuning procedure. (See Figure 3-8.) The Up button starts by scanning the microwave frequency up in search of the cavity dip (or frequency where the cavity resonates). The Down button starts by scanning the microwave frequency down in search of the cavity dip. If you are not sure if the search should start up or down, do not worry. The frequency will be scanned until its limit is reached and then scan in the other direction until the cavity dip is found. The Auto-Tune routine adjusts the frequency, phase, and bias of the bridge and the coupling (matching) of the cavity. If there is an error message, try manual tuning described in Section 7.1. If you still have difficulty in tuning, it usually means that you have a lossy or conductive sample. Refer to Chapter 9 for trouble shooting.

8. **Close the Microwave Bridge Tuning dialog box.** Click the Close button. The Microwave Bridge Tuning dialog box will then disappear. (See Figure 3-8.)

9. **Proceed to the next section to learn how to acquire spectra.**

Acquiring Spectra   3.3

1.   **Follow the instructions of Section 3.1 through Section 3.2.** You should have the spectrometer turned on, a Bruker ER 4102ST standard cavity, ER 4122SHQ or ER 4122SHQE cavity installed, a strong pitch sample inserted in it, and the microwave bridge and cavity tuned.
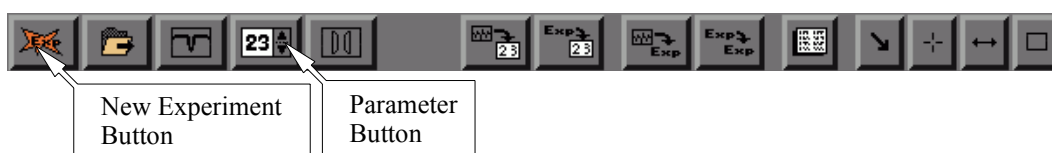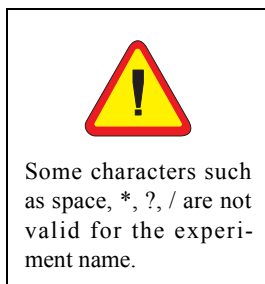

Figure 3-11   The New Experiment and Parameter buttons.

2.   **Create a New Experiment.** In order to acquire a spectrum, you need to define an experiment. To create a new experiment, click the New Experiment button in the monitoring panel. (See Figure 3-11.) In the New Experiment dialog box, enter a name for the new experiment. By default the name is Experiment. Click the C.W. tab. Choose Field for Abscissa 1 and None for Abscissa 2. If the Ordinate is not Signal Channel select Signal Channel in the Ordinate drop-down list. If the buttons for Temp Unit (the variable temperature unit), Goniometer, and Gradient Unit are activated (green) click on it to deactivate it. (See Figure 3-12.) Click Create and then the dialog box will close. The spectrometer will now be configured with these settings.

Some characters such as space, *, ?, / are not valid for the experiment name.
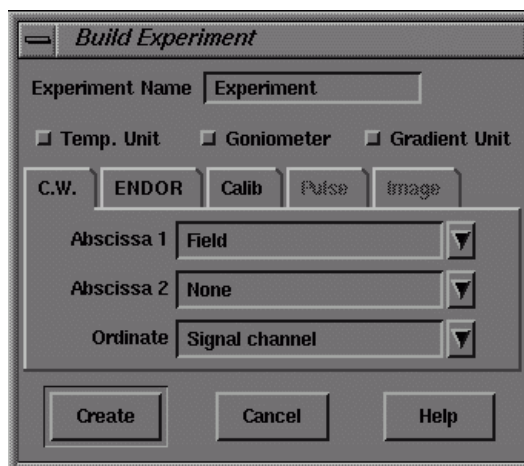
Figure 3-12 The New Experiment configuration.

It is very important to enter the sample information so that you maintain a record of the sample used to acquire the spectrum.Comments are very useful because you can keep track of such things as sample preparation details.

3. **Set Sample Information.** Click Acquisition in the menu bar, and then click Set Sample Info. A sample information dialog box opens in which you can type information regarding your sample. Enter your sample information and then click OK to close the window.
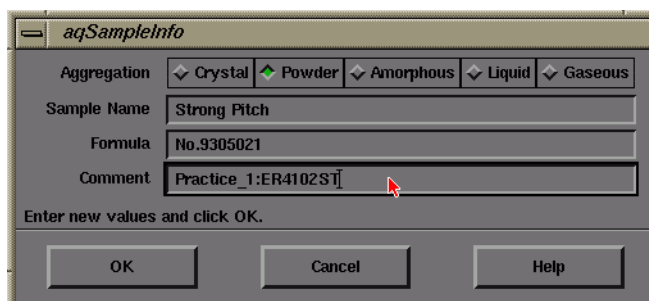
Figure 3-13 The sample information dialog box.

4. **Choose the Interactive Parameter Setting mode.**
Press the Activate button. (See Figure 3-14.) This button toggles between interactive and non-interactive parameter setting modes. As you edit parameters in the parameter panel in interactive mode, the new parameters are immediately activated on the spectrometer. In the non-interactive mode, you can edit instrument parameters in the parameter panel without activating them on the instrument. In this mode, the new parameters are only activated when you click the Run button.
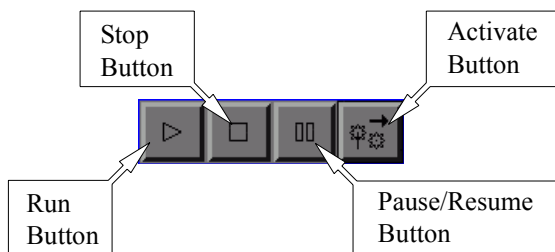
Figure 3-14   The experiment control tools.

5. **Open the Acquisition Parameters dialog box.**
Click the Parameter button in the tool bar to open the dialog box. (See Figure 3-11.)

If the Calibrated button is not green (activated) you need to load a proper calibration file. Consult the local facility manager or refer to Section 7.2 to find out how to load the calibration file.

6. **Check the Signal Channel parameters.** Click the Signal Channel tab. The signal channel parameters are further split into four tabs. Click the Detection tab. Edit the parameters in the dialog box so that they match those shown in Figure 3-15. The other tabs in the Signal Channel tab are for advanced options and their default settings are OK for this experiment.

Figure 3-15   The signal channel parameters.

7. **Check the Field parameters.** Click the Absc.1: Field tab. (See Figure 3-16.) Edit the parameters in the dialog box so that they match those shown in Figure 3-16.
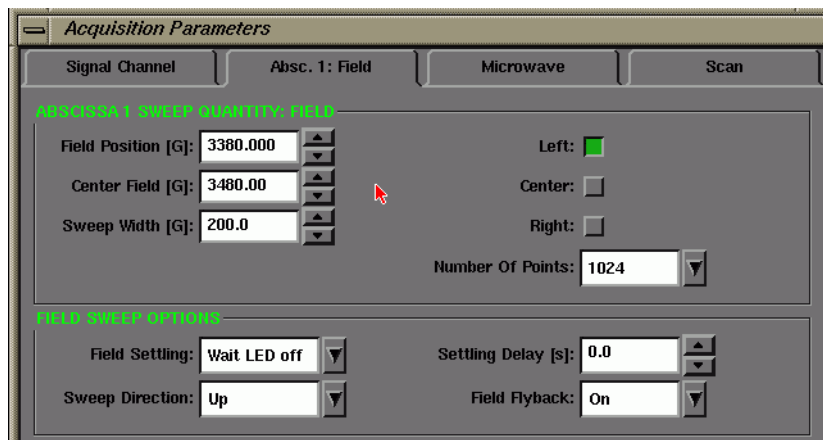

Figure 3-16   The magnetic field parameters.

8. **Check the Microwave parameters.** Click the Microwave tab. (See Figure 3-17.) Set the Attenuation to 30.0 dB.

Figure 3-17   The microwave parameters.

9. **Check the Scan parameters.** Click the Scan tab. (See Figure 3-18.) Edit the parameters in the dialog box so that they match those shown in Figure 3-18. After you have finished all the above parameter settings click the Close button.
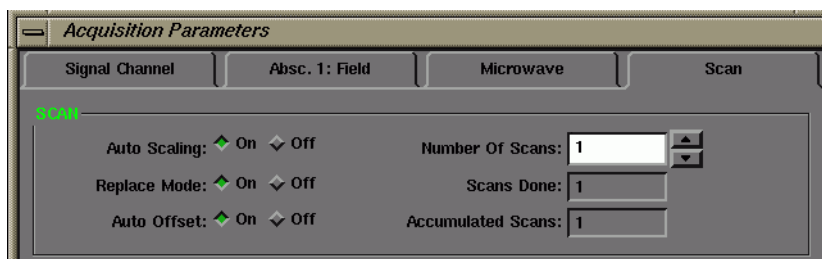

Figure 3-18   The scan parameters.

10. **Acquire a spectrum.** Click the RUN button to start an acquisition. (See Figure 3-19.) If you have a spectrum similar to the one in Figure 3-20, congratulations! You have successfully acquired an EPR spectrum.
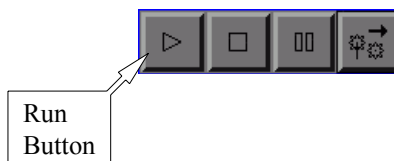

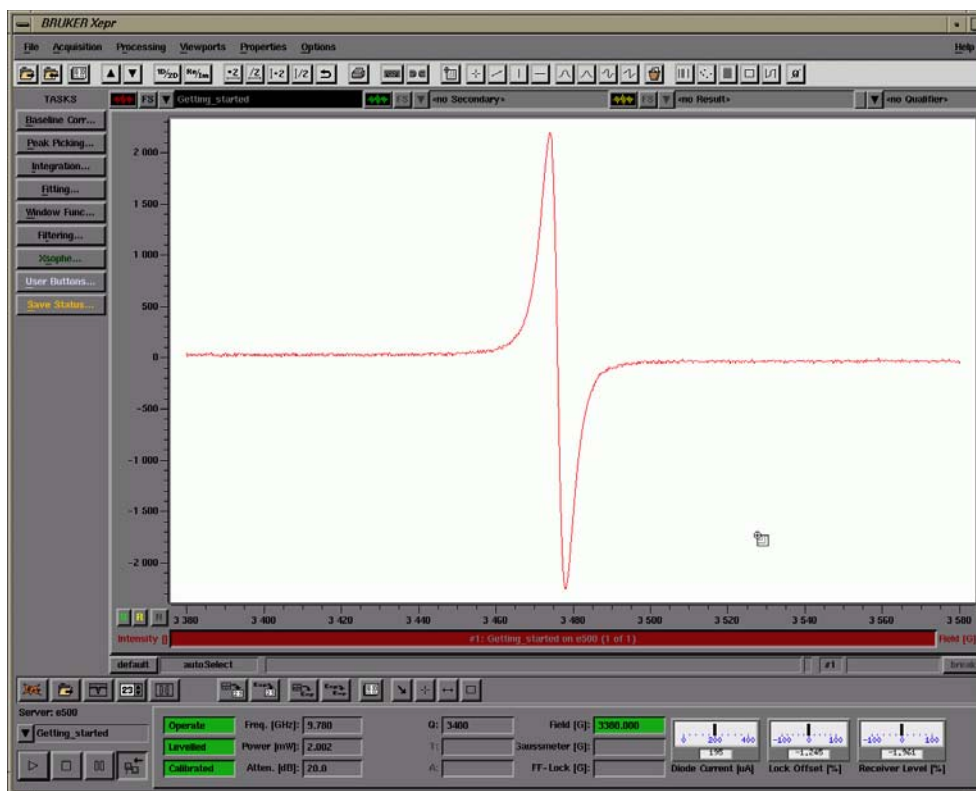Figure 3-19   The Run button.

Figure 3-20   An EPR signal of strong pitch.

11.   **Center the spectrum.** You may notice that the EPR line is not nicely centered. The next step will help you center your spectrum. To interactively set the center field, click Acquisition > Tools, and then the Center Tool in the menu bar. (See Figure 3-21.)
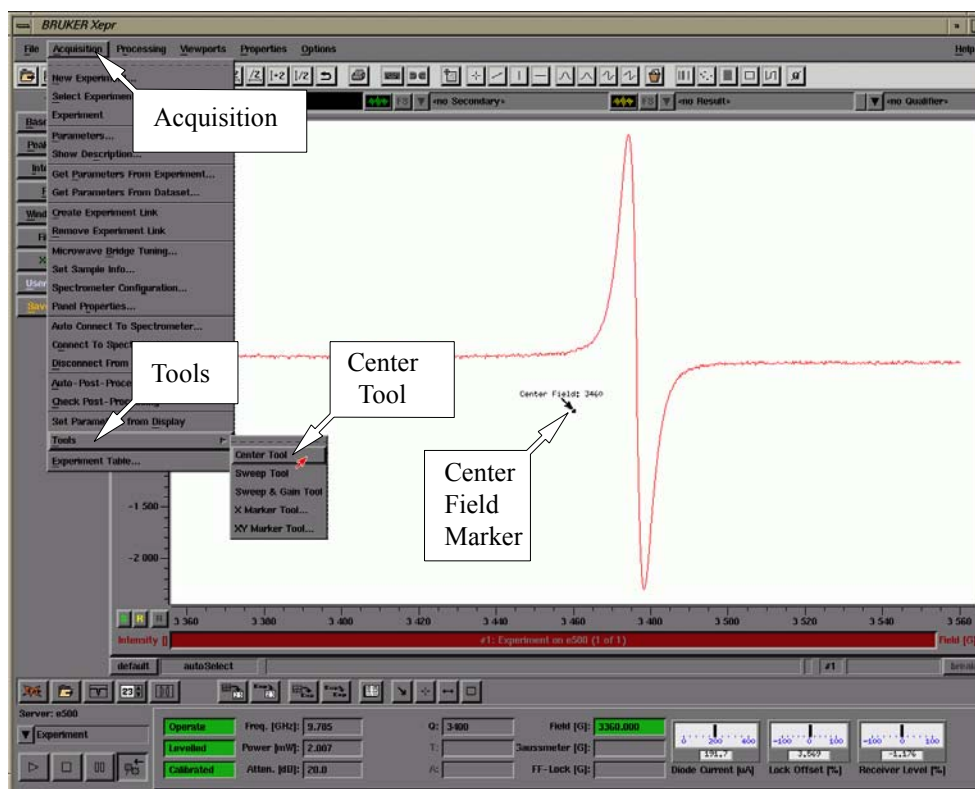
Figure 3-21   The Center Tool.

A center marker in the viewport appears. Drag the marker and place it where you would like the center field to be. (See Figure 3-22.)
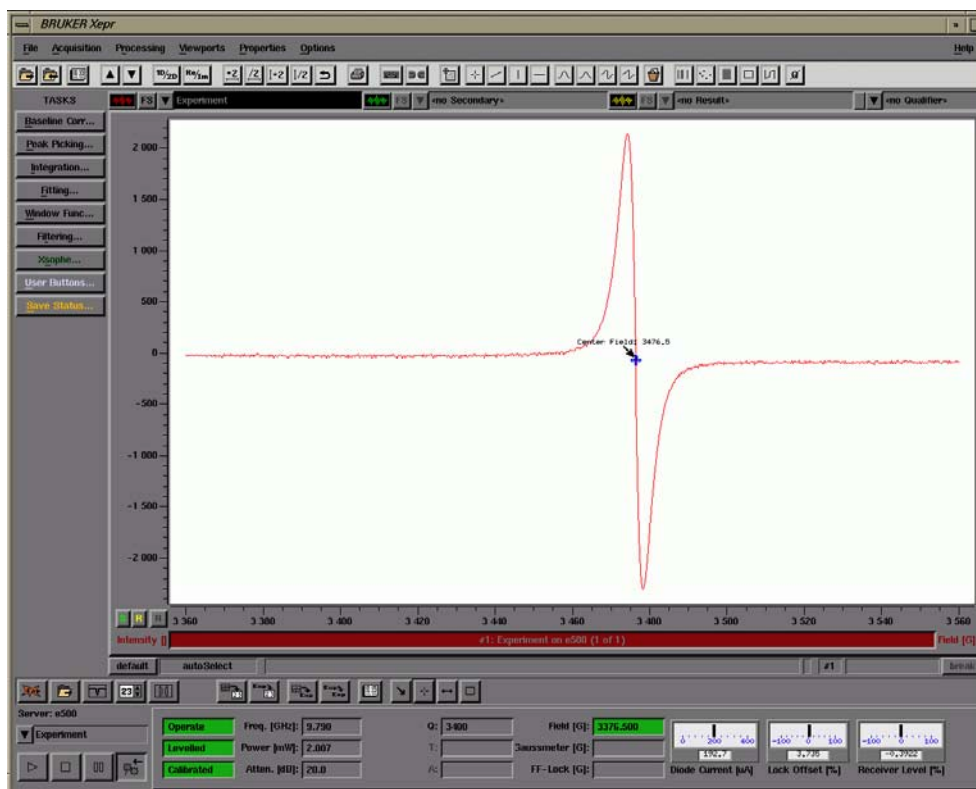

Figure 3-22   Placing the center marker.

This action replaces the center field value with the magnetic field position of the marker. To acquire the spectrum with the new center field, click the RUN button in the monitoring panel. The newly acquired spectrum will then be nicely centered. (See Figure 3-23.)
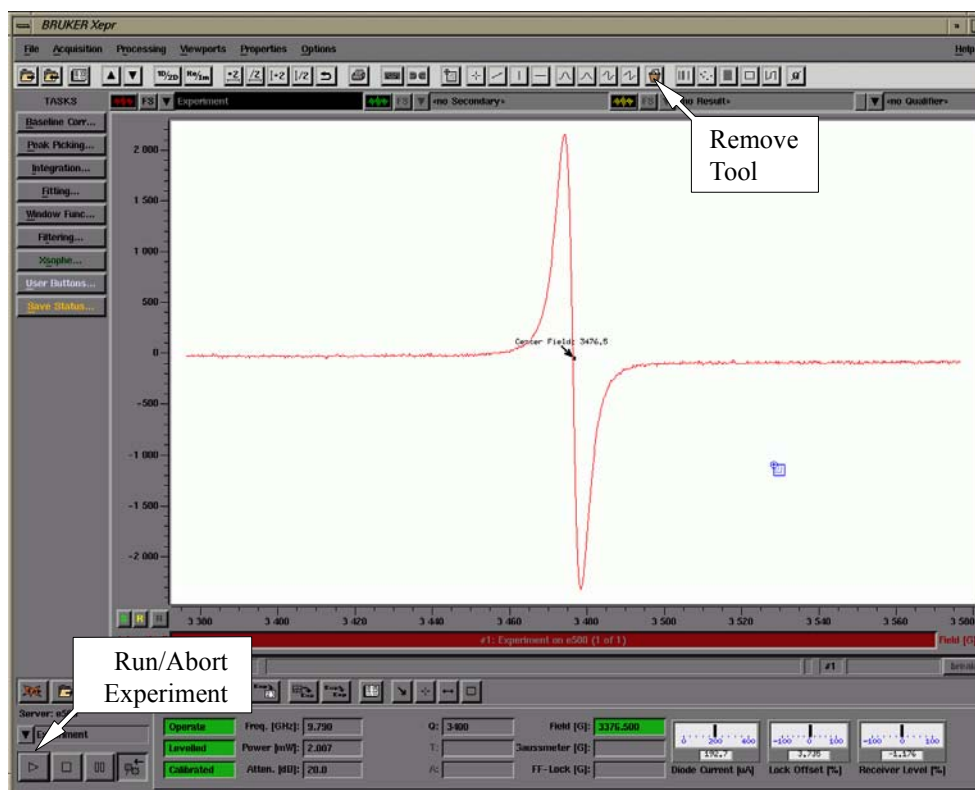

Figure 3-23 The centered spectrum.

If the tool cannot be removed in an active viewport it may be because the tool is inactive. Click the tool to activate it and then click the Remove Tool button to remove it.

12. **Remove the center marker.** You may remove the center marker by clicking the Remove Tool button in the tool bar. (See Figure 3-23.) The Remove Tool function removes the current active tool.

Storing and Saving the Spectrum 3.4

After you have acquired your spectrum, you may wish to save or store it. What is the difference between these two commands? The Store command stores the spectrum temporarily in memory which means it is lost when you exit Xepr. The Save command saves a permanent data file on your hard disk for future reference. Typically, the Store command is used to store intermediate results and the Save command is used to permanently save the results of your data processing.

1. **Store your spectrum.** Click the menu button in the viewport. Click Store in the drop-down menu. (See Figure 3-24.) The Store dialog box allows you to enter a descriptive title for the dataset. The presently active spectrum is stored in memory when you click Store.
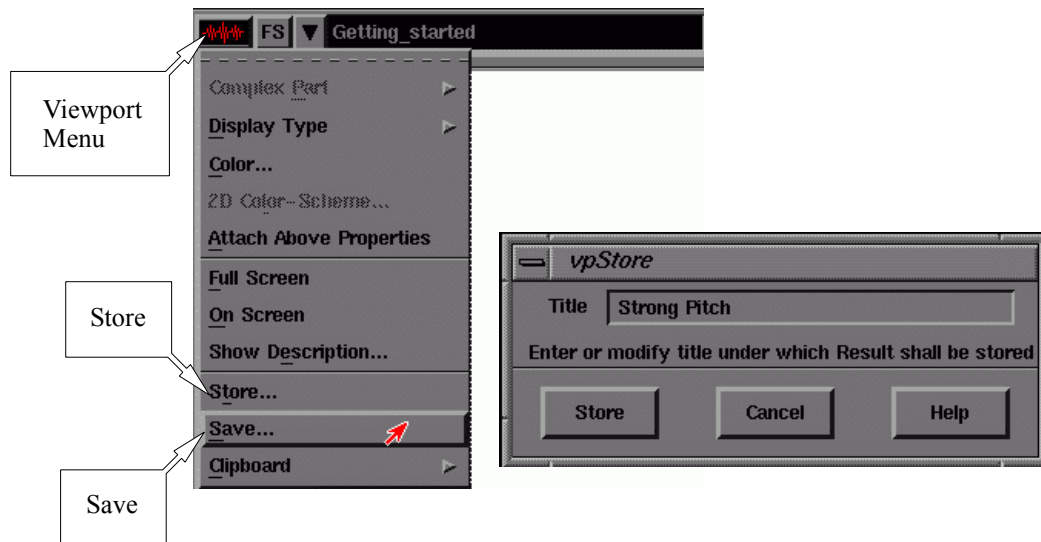

Figure 3-24   The Save, Store buttons and Store dialog box.

Do not save data in the ../xeprFiles/Data/ sharedData directory. This is a protected directory and your data will not be saved. You will receive an error message.

Some characters such as space, *, ?, / are not valid for file names.

2.   **Save your spectrum.** Click the menu button in the viewport. Click Save in the drop-down menu. (See Figure 3-25.) Enter a descriptive title for the dataset in the Title box. The Save dialog box lets you enter a file-name and destination directory. (See Figure 3-25.) To select the desired directory, click the appropriate paths under Group. Clicking on .. brings you to the parent directory. Enter the file name in the File box. Clicking OK saves the presently active spectrum on the hard disk. Note that the spectrum is also stored in memory when saved.
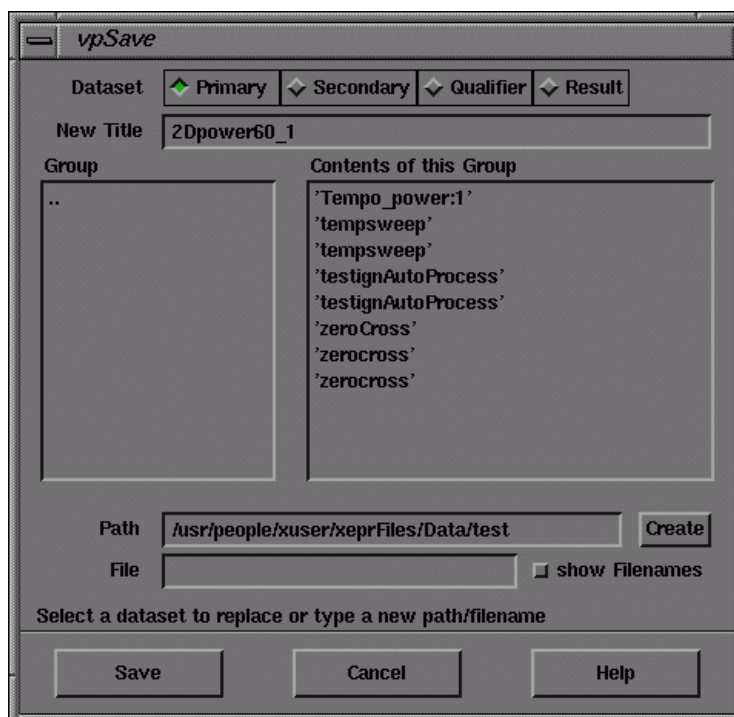
Figure 3-25   The Save dialog box.

If the chosen filename is already used for another file in the same directory, a warning box gives you the opportunity to decide whether to overwrite the existing file with the present spectrum. (See Figure 3-26.) Clicking No cancels the save process and allows you to select another name or directory.
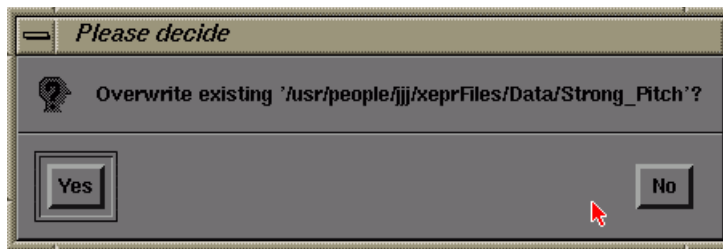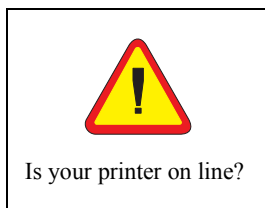

Figure 3-26   The warning dialog box for overwriting files.

Printing the Spectrum   3.5

1. **Prepare to print the spectrum.** Turn the printer on. Make sure that the paper is loaded. Refer to your printer documentation for details. Make sure the printer is properly set up. (See Sections 15.8 or 16.8 of the Elexsys E 500 User's Manual: Advanced Operations.)

Is your printer on line?

2. **Print the spectrum.** Click File in the menu bar and then Print. A dialog box will then appear in which you select desired options and print your spectrum. (See Figure 3-27.)

If you experience problems printing, consult the local facility manager or refer to See Sections 15.8 or 16.8 of the Elexsys E 500 User's Manual: Advanced Operations. to find out how to setup a printer.
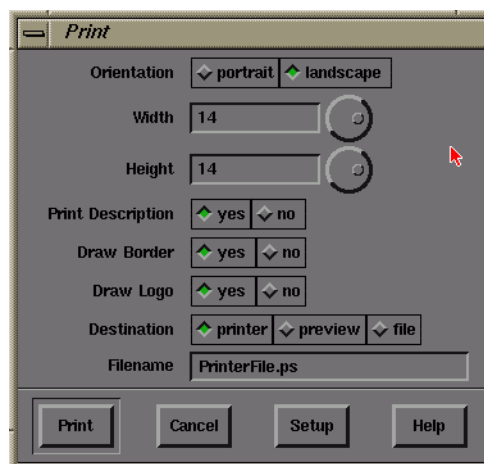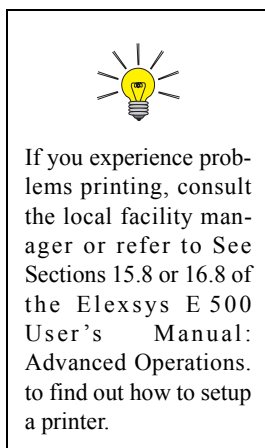

Figure 3-27   The Print dialog box.

Select the options shown in the above figure. Clicking Print starts the document printing and closes the dialog box. The output from the printer will look similar to Figure 3-28.
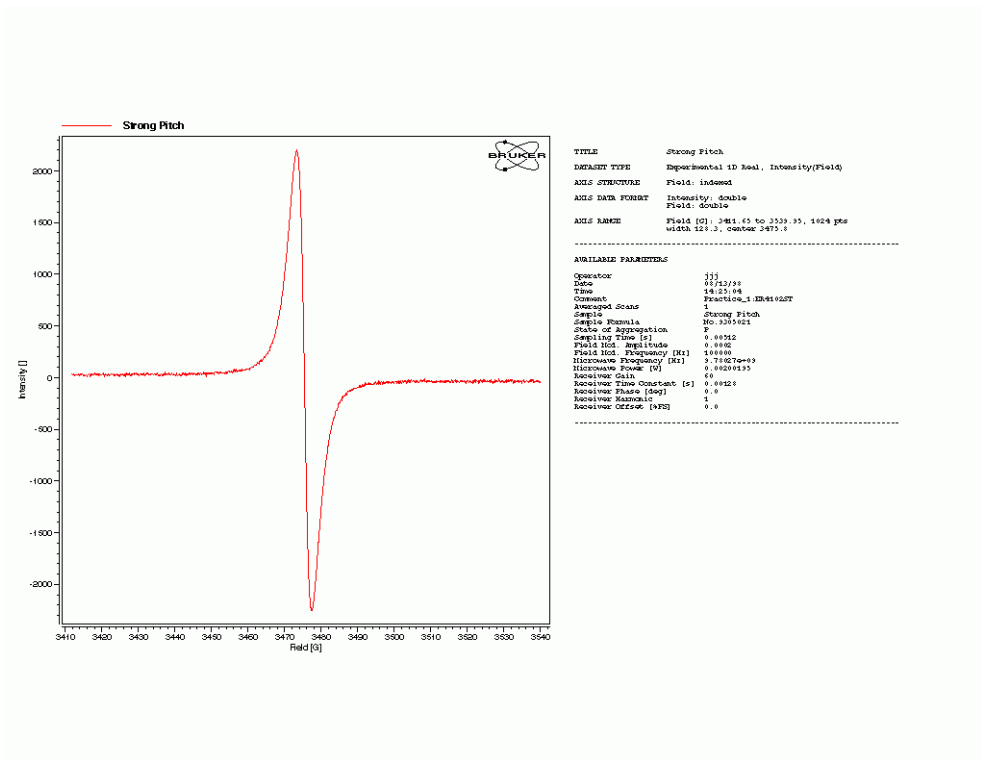
Figure 3-28   Typical output from the printer.

Turning the Spectrometer Off   3.6

1. **Open the Microwave Bridge Tuning dialog box.** If this window is not already open, click its button in the tool bar. The Microwave Bridge Tuning dialog box will then appear. (See Figure 3-29.)
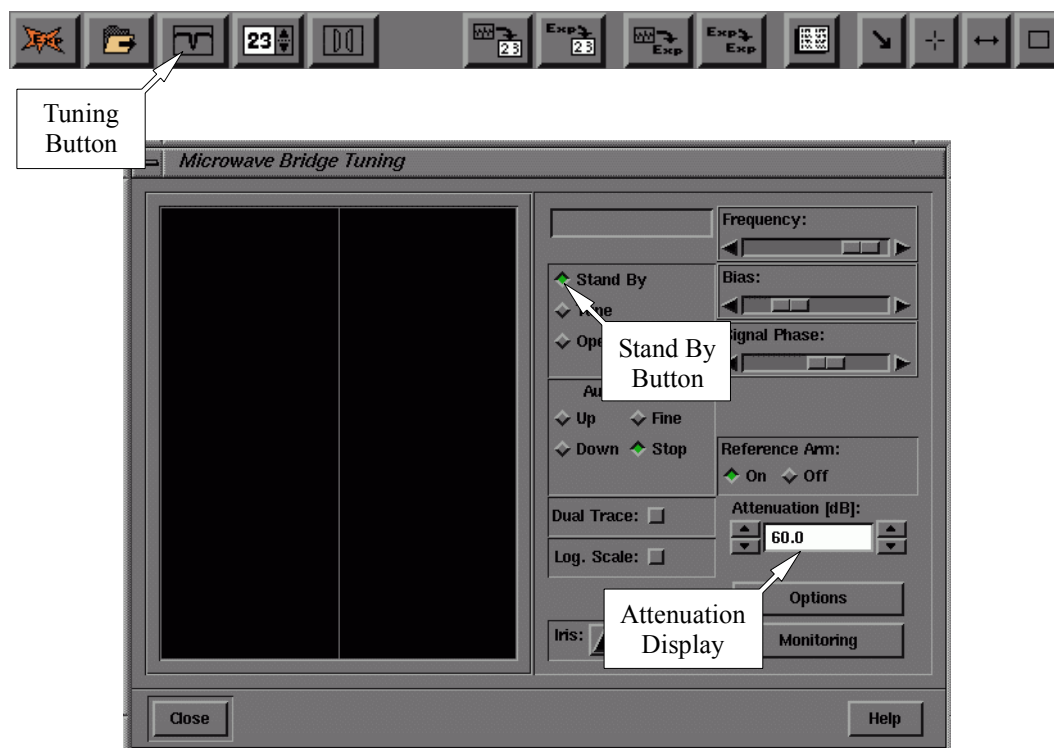

Figure 3-29   The Microwave Bridge Tuning dialog box.

It is important to exit the software in an orderly manner (*i.e.* don't just turn the computer off before disconnecting the instrument and exiting the software) because many instrument parameters are set to specific values for a safe shut-down of the spec-

Exit the software first before logging out.

2. **Switch the microwave bridge to Stand By.** Click the Stand By button in the dialog box to change to Stand By. (See Figure 3-29.) The microwave attenuator will be automatically set to 60 dB.

3. **Close the Microwave Bridge Control dialog box.** Click the Close button in the dialog box. The Microwave Bridge Tuning dialog box will then disappear.

4. **Remove the sample from the cavity.** See Section 3.2 for details on how to do this.

5. **Cover the upper collet or insert a solid collet plug.**

6. **Disconnect from the spectrometer.** Click the Acquisition menu bar and then click Disconnect From Spectrometer. The monitoring panel will disappear.

7. **Exit the Xepr program.** Click the File menu bar and then click Exit. You will be asked if you wish to save the changes.

8. **Log Out**. Click the right mouse button (for SGI O2 workstations) or click on the Gnome icon on the bottom of the screen (for the Linux workstations) and click Log Out in the drop-down menu. You need to confirm this by clicking Yes or select the proper radio button and click OK.

9.   **Turn off the heat exchanger and magnet power supply. (Instructions for Small Power Supplies.)** Follow this step if your power supply looks like the power supply in Figure 3-30. To turn the power supply off, push the POWER ON/OFF button. Turn the heat exchanger off by pressing the power switch. The location of the Power ON/OFF button may vary depending on your heat exchanger. Go to Step 11.

Power Switch

Power On/Off Switch

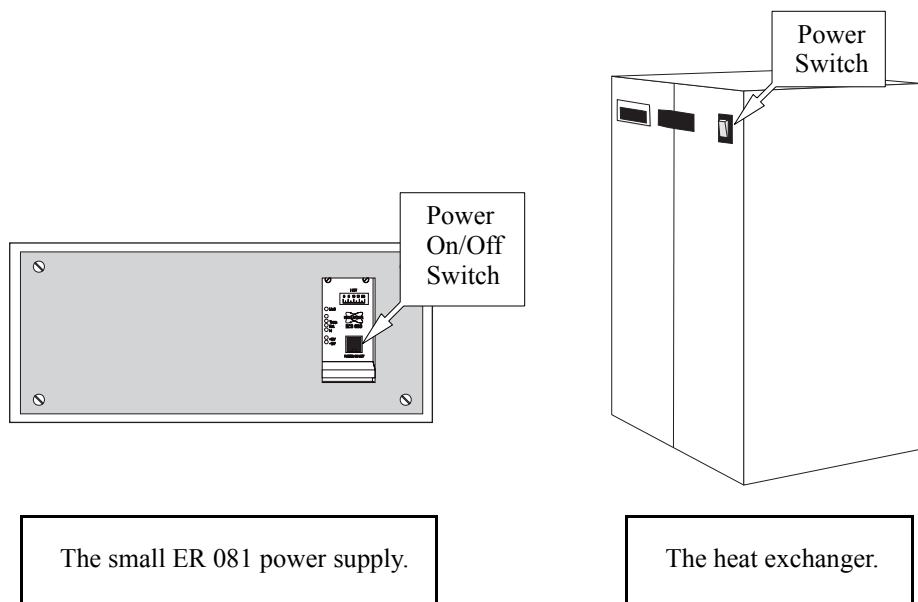The small ER 081 power supply.

The heat exchanger.

Figure 3-30   The small ER 081 power supply and the heat exchanger.

10. **Turn off the heat exchanger and magnet power supply. (Instructions for Large Power Supplies)** Follow this step if your power supply looks like the power supply in Figure 3-31. On systems with large power supplies, you need to first press the Power OFF button and then the ELECTR. ON button. Pressing the POWER OFF button also turns the heat exchanger off.

Electricity On
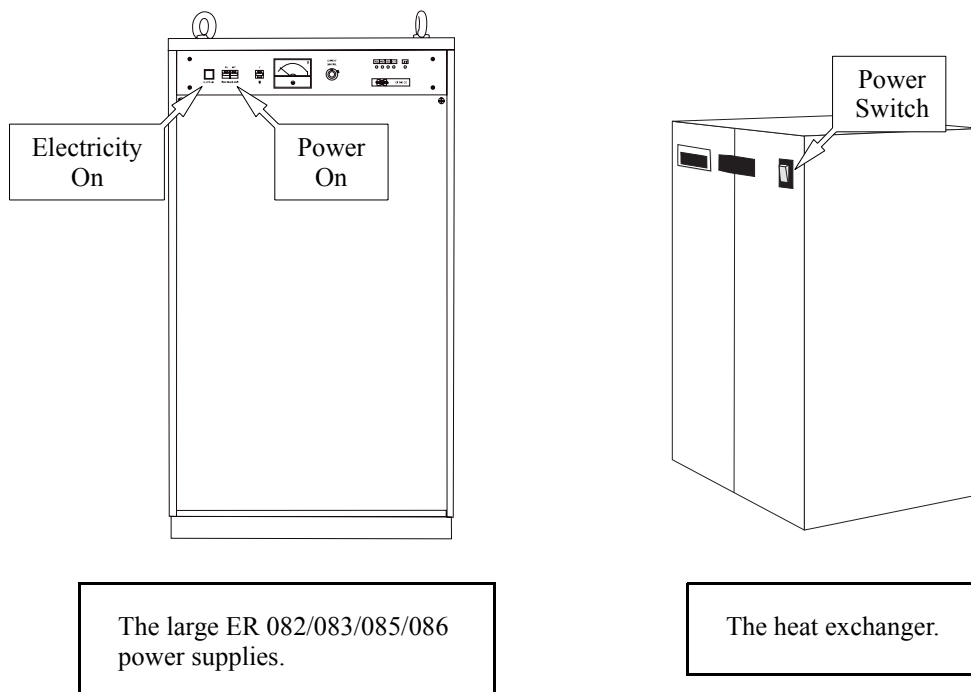
Power On

The large ER 082/083/085/086 power supplies.

Power Switch

The heat exchanger.

Figure 3-31   The large power supply and the heat exchanger.

11. **Turn off the tap water for cooling.** There are usually two valves, one for the supply and one for the return (or drain). Consult the local instrument or facilities manager if you are not sure where the valves are.

12. **Turn off the power for the console.** The power switch (red button) for the console is located in the upper left front corner of the console. (See Figure 3-32.)
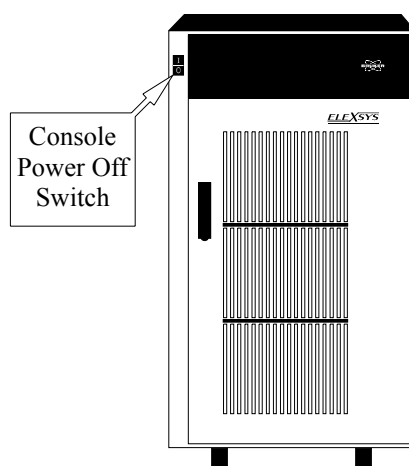
To reduce wear and tear on the hardware it is best to leave the workstation on unless it is not going to be used for a long time or there will be a power outage. Consult the workstation documentation or the SGI manual to learn how to turn off the workstation.

Console Power Off Switch

Figure 3-32   The location of the console power switch.

13. **Turn off the power for the system. (Optional)** How you do this depends on how the electric power was hooked up when the spectrometer was installed. Most likely you will deactivate the switch on the breaker box for the spectrometer. Breaker boxes are usually mounted on the wall. Consult the local instrument or facilities manager if you are not sure where the breaker box is.

If you have many power outages or electrical storms, it is a very good idea to shut off the power to the spectrometer.