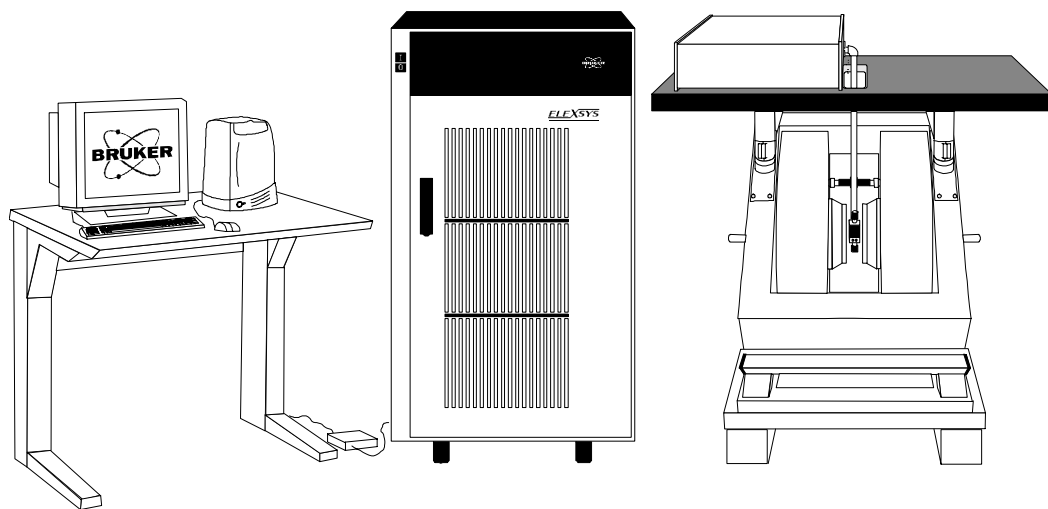


ELEXSYS E 500

EPR Spectrometer User's Manual Advanced Operations



ELEXSYS E 500 USER'S MANUAL Advanced Operations

Authors: Dr. JinJie Jiang, Dr. Ralph T. Weber

Illustrations: Aaron A. Heiss, Dr. Ralph T. Weber, Dr. JinJie Jiang
EPR Division

Bruker BioSpin Corporation
Billerica, MA USA

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This manual is part of the original documentation for the Bruker ELEXSYS spectrometer.

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e-mail: epr_applications@bruker-biospin.com

FAX: 978-670-8851

Tel. 978-663-7406

**mailing
address** EPR Division
Bruker BioSpin Corporation
19 Fortune Drive
Manning Park
Billerica, MA 01821 USA

Thank you for your help.

Electrical Safety

0.1

Do not remove any of the protective covers or panels of the instrument. They are fitted to protect you and should be opened by qualified service personnel only.

Power off the instrument and disconnect the line cord before starting any cleaning work in the spectrometer. Never operate the instrument with the grounding cord disconnected or by passed. Facility wiring must include a properly grounded power receptacle.

Chemical Safety

0.2

Individuals working with hazardous chemicals, toxic substances, or enclosed liquid samples must take every precaution possible to avoid exposure to these agents. As a general rule, **THINK OF THE CHEMICAL LABORATORY AS A HAZARDOUS ENVIRONMENT IN WHICH YOU MUST CONTINUALLY MAINTAIN A HIGH STANDARD OF VIGILANCE.** Do not assume a cavalier attitude -- the substances with which you work present very real, and very serious threats to your health and safety.

Adhere to all currently recommended guidelines for standard laboratory safety as promulgated by governmental codes and contemporary laboratory practice. Inform yourself about the specific risks that are present when you handle actual or potential carcinogens (cancer-causing agents), explosive materials, strong acids, or any liquids that are sealed in glass containers.

Specifically:

- Be extremely careful when you handle sealed glass samples that are rapidly heated or cooled. The rapid cooling of some samples may result in the formation of a solid bolus in the sample tube that may make the tube prone to explosive rupture.
- Educate yourself about the temperature at which chemicals evaporate. When a sample gets close to the temperature at which it evaporates, it may quickly become volatile.
- In general, the safety threat posed by flying glass and violently escaping gases and liquids should not be underestimated.
- Wear safety glasses, face masks, and other protective clothing whenever there is any risk of spillage, breakage, or explosion. Protective shields should also be employed when there is any risk of explosion.
- Be sure that both storage and working areas are properly ventilated. They should be equipped with powerful blowers and fume heads.
- Store chemicals safely. Avoid integrating containers of chemicals that may result in dangerous combinations.
- Practice good housekeeping in work and storage areas. Clean up spills and refuse promptly. Do not leave volatile, combustible, or acidic liquids exposed on counters, benches, or other work areas.
- Make certain all chemical containers are properly labeled and classified, and that especially hazardous materials are appropriately designated with clearly understood decals or warnings.
- Never taste or inhale unmarked chemicals.

- All laboratories should be equipped with fire doors, fire extinguishers, fire smothering materials, and sprinkler systems or showers, as well as a detailed fire safety plan.

Microwave Safety

0.3

As long as the microwaves are contained in metal structures, microwaves can be very safe. Here are some precautions which, if followed, will eliminate the possibility of injury due to the microwaves.

- Do not have an open waveguide when the microwave power is on.
- Switch the bridge to standby when you remove or change EPR cavities.
- Never look down an open waveguide when there is microwave power. The eyes are very susceptible to damage from microwaves.

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We have described the basic operations of the Bruker Elexsys E 500 EPR (Electron Paramagnetic Resonance) spectrometer in the Elexsys E 500 User's Manual: Basic Operations. We strongly suggest users read that manual first before reading this manual. The Bruker Elexsys E 500 EPR spectrometer features many advanced functions that facilitate a wide variety of sophisticated experiments. This manual introduces you to the many advanced functions of the spectrometer. Owing to the abundant features of the Elexsys E 500 spectrometer, it is almost impossible to describe every experimental situation or operation in one volume. We encourage you to explore the information in this manual to discover the methods and techniques that best suit your experimental needs.

How to Find Things

1.1

- Chapter 2** This chapter describes the procedure for calibrating cavities and signal channels so that you may obtain reproducible, quantitative, and high sensitivity spectra.
- Chapter 3** In order to maintain your spectrometer in optimal working condition, it is a good idea to periodically test your system's performance. Chapter 3 describes how to measure the sensitivity of the spectrometer.
- Chapter 4** This chapter describes how to configure the spectrometer. It is important to properly configure the spectrometer especially, when you use accessories and perform advanced experiments.
- Chapter 5** Calibrating the magnetic field precisely is vital for accurately measuring g-values. We describe the procedure for calibrating the magnetic field as well as determining g-values.

- Chapter 6** In addition to the signal channels, the Elexsys E 500 EPR spectrometer includes a fast digitizer (ADF) for rapidly acquiring transient EPR signals. This chapter describes how to perform both rapid field and time scan experiments.
- Chapter 7** The Elexsys E 500 spectrometer has many predefined experiments. As an example, we introduce you to non-linear scans in this chapter. You can learn how to acquire a non-linear 1D field sweep spectrum and a 2D non-linear scan spectra. We hope this introduction stimulates your interest to explore other built-in as well as user- defined experiments.
- Chapter 8** The built-in macros of Xepr simplify a complicated sequence of operations to a simple mouse click. Chapter 8 provides you information on how to create your own macros.
- Chapter 9** Automatic post-processing provides automatic data processing and storage after an acquisition. This chapter provides detailed instructions for this feature.
- Chapter 10** By making macros and other functions, you can customize your Xepr software. To guide other users to use your customized functions you can implement your own help files for the Xepr help system. This chapter shows you how to do so in a simple way.
- Chapter 11** Many different microwave bridges are available. This chapter explains how to change microwave bridges.
- Chapter 12** Bruker provides many optional accessories to help customers succeed with their experiments. The Elexsys E 500 accommodates a wide variety of accessories. In this chapter you can find information on how to set up the most commonly used accessories, such as an external frequency counter, goniometer, variable temperature unit, field frequency lock, and teslameter.

Chapter 13 This chapter describes the function and operation of the many indicators, inputs, and outputs of the modules in the console.

Chapter 14 Many sophisticated experiments such as double modulation, quadrature detection, or externally triggered experiments can be realized. This chapter describes how to set up these advanced experiments.

Chapters 15 & 16 These chapters describe many system administration issues related to the Xepr program. Chapter 15 covers the SGI IRIX operating system and Chapter 16 deals with Linux. Installation and setup of Xepr is described. The computer workstation is equipped with full networking capabilities. Not only can you transfer data through the network but you can also control the spectrometer remotely. This chapter demonstrates how to perform remote operations. It also offers advice on setting up and troubleshooting printers.

Chapter 17 This chapter describes diagnostics for troubleshooting the acquisition server.

Typographical Conventions

1.2

Fonts

1.2.1

Special fonts are used in the text to differentiate between normal manual text and the text displayed in the program.

Times This is the font used for the normal text in the manual.

Helvetica This is the font used for text that is displayed by the program or must be entered into the program by you.

Special notes

1.2.2

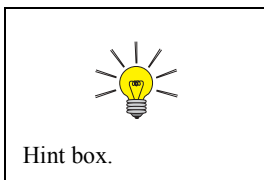
Some special notation is employed in this manual to simplify the descriptions.

< ... > The content between the brackets needs to be substituted with proper entries by the user.

> The right bracket indicates sequential selection of the menu entries. For example, **Processing > Filtering > Smoothing** means clicking the **Processing** button in the menu bar, followed by clicking **Filtering** in the sub-menu, and then clicking **Smoothing**.



You will see a warning box sometimes in the lefthand margin. These are meant to point out critical information. In particular, it warns you about any procedures or operations that may be dangerous to the spectrometer or you. Always read and follow this advice.



In addition, there are also hint boxes in the lefthand margin. These are meant to be helpful hints and point out important information.

For many experiments, it is vital that your spectrometer is carefully calibrated. For example, it is essential to know the precise values of the magnetic field modulation amplitude in order to obtain quantitative EPR spectra. The calibration procedures in this chapter enable you to measure the experimental conditions produced by the spectrometer with considerable accuracy.

This chapter is not meant to be a general overview of spectrometer calibration and quantitative EPR. Therefore, we highly recommend the following references which discuss the topic in much greater detail:

- Poole, C.P. *Electron Spin Resonance, a Comprehensive Treatise on Experimental Techniques*: First Ed., Interscience, New York, 1967.
- Poole, C.P. *Electron Spin Resonance, a Comprehensive Treatise on Experimental Techniques*: Second Ed., Wiley, New York, 1983.
- Alger, R.S. *Electron Paramagnetic Resonance*: Interscience, New York, 1968.

Standard Samples

2.1

Standard samples are useful for system performance tests, spectrometer calibration, and quantitative concentration measurements. Ideally the standard sample should contain stable, long lived paramagnetic species, be easily prepared under consistent and controlled methods, and should be fully characterized with respect to all spectroscopic parameters such as relaxation times and hyperfine and fine structure splittings. In addition, the resonance line should be narrow and preferably homogeneous. Unfortunately, the universal standard sample has not been found. Many standards have been suggested and each has its own particular merit. The standard samples supplied with every Bruker spectrometer are discussed below.

DPPH (α , α' - diphenyl- β -picryl hydrazyl)

2.1.1

DPPH serves as a reference both in the solid state and in the liquid state when dissolved in benzene or toluene/mineral oil. The line width measured from the solid is subject to exchange narrowing and thus, varies from under 1 gauss to over 4 gauss, depending on the solvent that was used for recrystallization. It has a g factor of 2.0036 ± 0.0003 . When dissolved in solution, a quintet with unresolved hyperfine couplings is observed because the spin exchange narrowing is reduced as the sample is diluted. A small single crystal of DPPH is an ideal sample for calibrating the phase and the field modulation amplitude of the signal channel of an EPR spectrometer. DPPH has been studied extensively by:

- Möbius, K. and R. Biehl. *Multiple Electron Resonance Spectroscopy*: Plenum Press, 1979.
- Dalal, N.S., D.E. Kennedy, and C.A. McDowell. *J. Chem. Phys.*: **59**, 3403 (1979).



DPPH exhibits g-anisotropy, *i.e.* the g-value depends on the orientation of the sample with respect to the magnetic field.

- Hyde, H.S., R.C. Sweed, Jr., and G.H. Rist. *J. Chem. Phys.*: **51**, 1404 (1969).
- Dalal, N.S., D.E. Kennedy, and C.A. McDowell. *J. Chem. Phys.*: **61**, 1989 (1974).
- Dalal, N.S., D.E. Kennedy, and C.A. McDowell. *Chem. Phys. Lett.*: **30**, 186 (1975).

Weak and Strong Pitch Samples

2.1.2

Pitch in KCl has emerged as a standard because of its long-lived paramagnetic radicals and low dielectric loss. Because of the long life of the radicals, it is unsurpassed as a test of spectrometer sensitivity. The pitch is added to a powder of KCl and the mixture is carefully mechanically mixed to obtain a homogeneous sample. After mixing, the sample is heated, pumped, and sealed under vacuum. Pitch is generally prepared in two concentrations: strong pitch which is 0.11% pitch in KCl, and weak pitch which is 0.0003% pitch in KCl.

To correct for variations in spin concentration, each weak pitch sample is compared to a “standard” and assigned a correction factor. The peak to peak line width is typically 1.7 G with a g-factor of 2.0028. The size (very weak) of the signal renders pitch ill suited for modulation amplitude calibration. The weak pitch samples from Bruker Biospin have an estimated concentration of 10^{13} spins per centimeter. The samples are calibrated and the correction factor is printed on the side of the tube. This sample is prepared for the purpose of measuring instrument performance owing to its high stability, however, it is not meant as a quantitative spin-counting standard.

Theory of Signal Channel Calibration 2.2

You need to carefully calibrate your spectrometer's signal channel reference phase and modulation amplitude in order to obtain maximum sensitivity, minimum distortion, and quantitatively reproducible measurements. The SCT/H and SCT/L signal channels in conjunction with the Xepr software make this calibration easy to perform. The results of the calibration are saved on disk for future use. We recommend recalibration at least once a year to ensure quantitative and reproducible results. Each cavity or resonator has its own individual calibration file, therefore, this procedure must be followed for each cavity.

Introduction 2.2.1

Calibration of the signal channel involves two separate yet interdependent procedures. The first procedure is to calibrate the peak to peak modulation amplitude. For the sake of brevity, modulation amplitude will be used in place of peak to peak modulation amplitude. The second procedure is to calibrate the phase difference between the reference signal and the modulated EPR signal. Because the calibration and adjustment of the modulation amplitude can affect the phase difference, the first procedure is performed first.

Amplitude Calibration

2.2.2

You calibrate the modulation amplitude by overmodulating a narrow EPR signal. A crystal of DPPH, with a line width of approximately 1 G, is a very good sample to use. When the modulation amplitude is large compared to the line width, the magnetic field modulation brings the sample into resonance before and after the magnet has reached the field for resonance. This results in a broadening and distortion of the EPR signal. (See Figure 2-1.) In the limit of an infinitesimally narrow EPR signal, the peak to peak width of the first derivative EPR signal will be approximately equal to the peak to peak modulation amplitude.

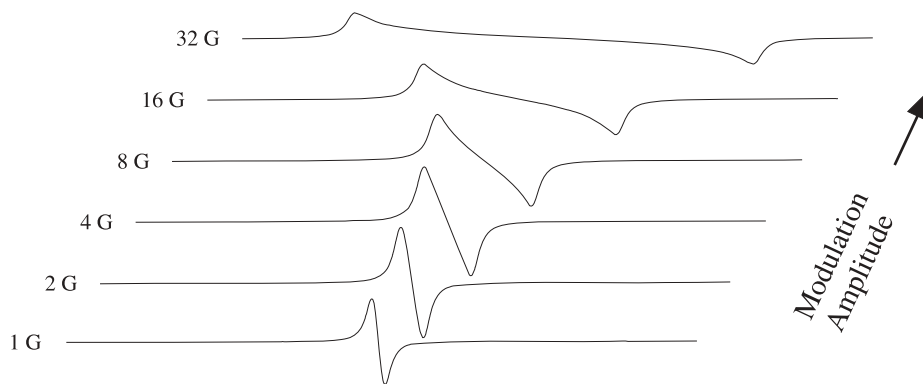


Figure 2-1 The signal shape of the DPPH EPR signal as a function of the field modulation amplitude.

The first step of calibrating the modulation amplitude involves choosing the correct tuning capacitors. The modulation amplifier needs a bit of help to obtain large modulation amplitudes at modulation frequencies greater than 50 kHz. This is a consequence of the decreasing skin depth with increasing frequency.

The modulation coils on the cavity are tuned, or made resonant, by adding a tuning capacitor in series with the modulation coil.

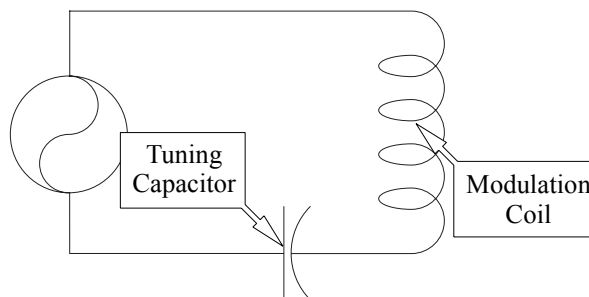


Figure 2-2 The LC resonant circuit for high frequencies.

The calibration routine switches various tuning capacitors in and out of the circuit until the modulation amplitude is maximized. The optimal capacitor for that particular frequency as well as the modulation amplitude for full gain of the modulation amplifier are recorded and saved with the calibration file. Once this data is available, the signal channel will then vary the input signal to the modulation amplifier to produce the modulation amplitude that you have selected.

Those familiar with older EPR spectrometers will remember the tuning boxes for tuning the modulation coils. The above mentioned software and hardware makes these tuning boxes unnecessary as well as adding great flexibility in the choice of modulation frequencies.

Phase Calibration

2.2.3

Once the modulation amplitude has been calibrated, the reference phase is easily calibrated by studying the phase angle dependence of the signal intensity. The intensity of the output signal is proportional to the cosine of the phase difference between the reference signal and the modulated EPR signal. (See Figure 2-3.) It is most convenient to determine where the 90° phase difference occurs because first, the absence of a signal ($\cos(90^\circ) = 0$) is easy to detect and second, the cosine function (and hence the intensity) changes rapidly with respect to the phase angle at 90° . In the calibration routine, spectra are acquired at several different values of the reference phase and the 90° phase difference is extrapolated from the signal intensities. The phase angle resulting in maximum signal intensity for that particular frequency is recorded and saved with the calibration file.

The phase difference between the modulated EPR signal and the reference signal depends on several experimental conditions. The length of the cable leading to the modulation coils, the inductance of the coils in the particular cavity, the gain setting of the modulation amplifier, the tuning capacitors, and the signal channel used can all change the phase difference. However, the reference phase calibration is performed automatically during the routine described in this section.

The two editions of the book by C.P. Poole that are mentioned at the beginning of this chapter are very good references for the details on the theory of phase sensitive detection and the calibration of signal channels. We encourage you to explore this topic further to learn more about calibration.

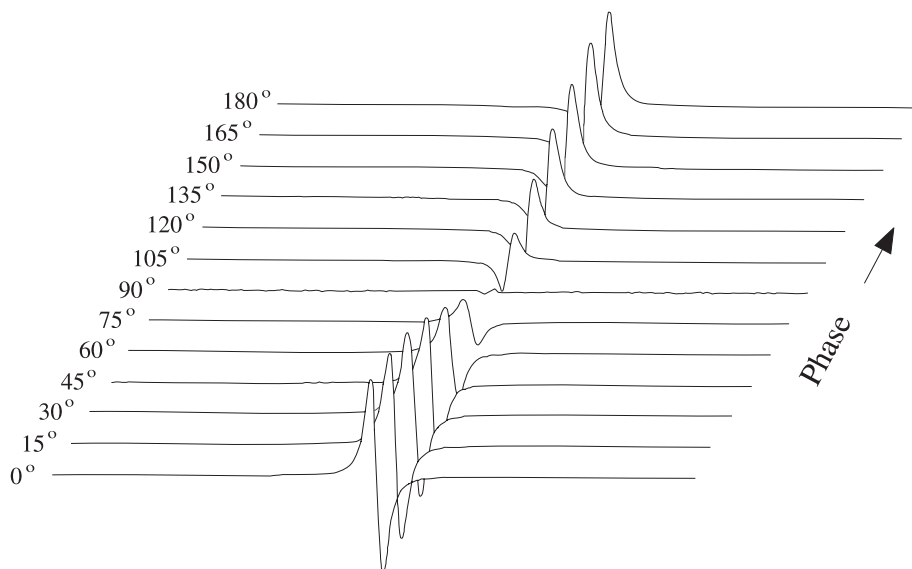


Figure 2-3 The signal intensity as a function of the reference phase angle.

Calibrating the Signal Channel

2.3



Do not attempt to calibrate a cavity with an ER 4112HV or ER 4113HV helium cryostat installed in the cavity. The cryostat sample holder prevents the DPPH sample from being centered properly.

1. **Get ready for the calibration routine.** Follow the instructions of Section 3.1 and 3.2 of the Elexsys E 500 User's Manual: Basic Operations. You should have the spectrometer turned on, the cavity properly installed with a Bruker standard DPPH sample in it, and the microwave bridge and cavity tuned. Remove cryostats from the cavity because it is easier to position the DPPH sample properly in the cavity. (Except for the FlexLine resonators: it is necessary to use the ER 4118CF cryostat when calibrating FlexLine resonators.) Another advantage is that the resonant frequency of the cavity will be approximately 9.8 GHz without the helium cryostat and the field for the DPPH signal will be known (approximately 3500 Gauss). The DPPH sample is a point sample so it is important to have it roughly centered in the cavity. The sample has a mark on it indicating where the DPPH is. The distance from the collet to the center of the cavity is approximately 70 mm.

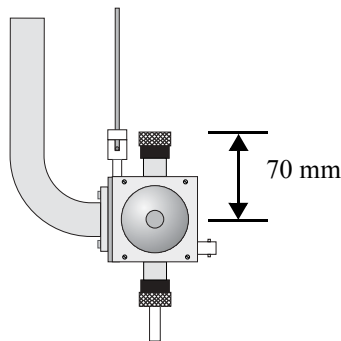


Figure 2-4 The approximate distance from the collet to the center of the cavity.

2. **Create a calibration experiment.** Click the New Experiment button in the monitoring panel and the dialog box will appear. (See Figure 2-5.) Enter a name for the experiment (calibration for example) in the Experiment Name column. Click the Calib tab. Make sure the Standard push button is activated (green). The R.F. button is for ENDOR calibration. Click the Create button to close the dialog box.

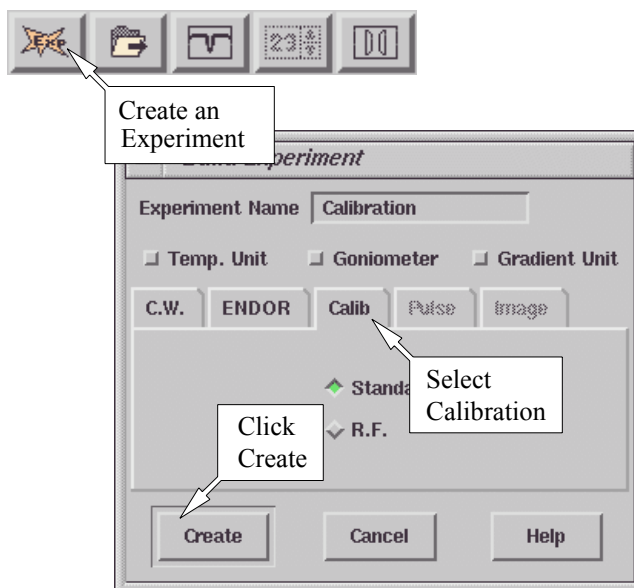


Figure 2-5 Creating a calibration experiment.

3. **Open the parameter dialog box.** Click the Parameter button to open the Calibration Parameters dialog box. If the Calibration folder is not in front, click its tab to bring it to the front. (See Figure 2-6.)

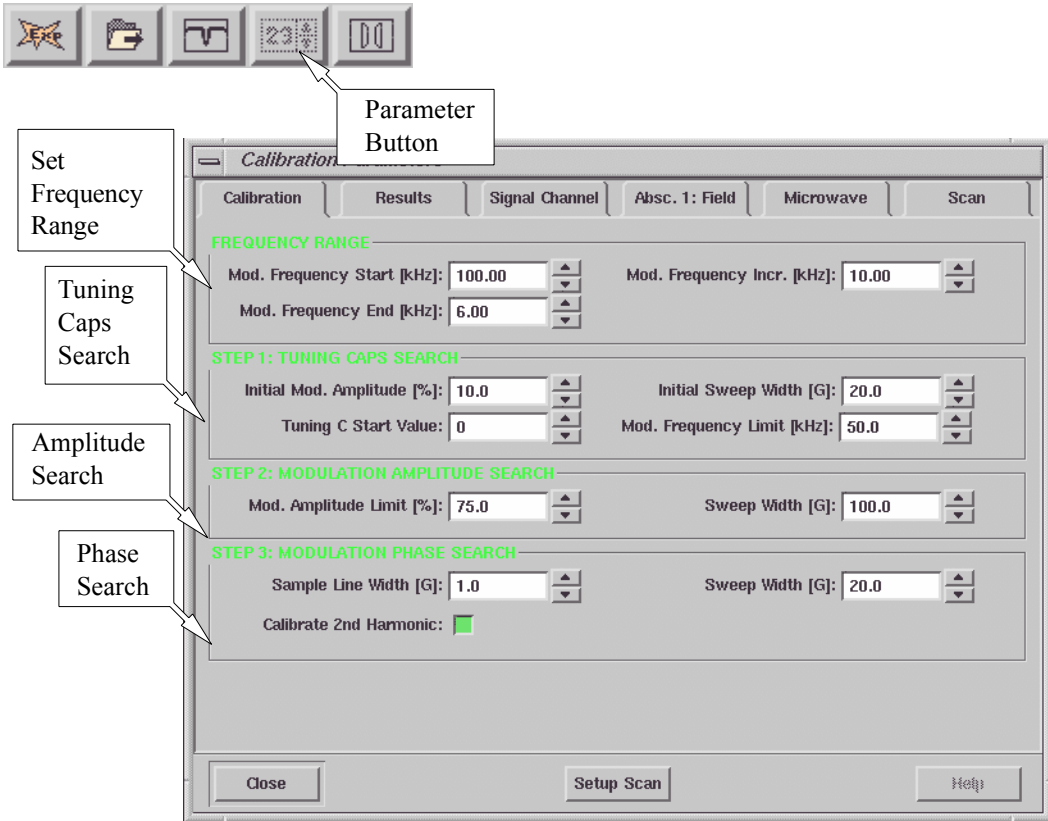


Figure 2-6 The Calibration Parameters dialog box.



The EN 801 ENDOR resonator should not be used above 25 kHz. Consult the Elexsys E 560 documentation for instructions on calibrating this cavity.



The ER 4122SHQE and ER 4119HS resonators should only be calibrated one modulation frequency at a time! At lower modulation frequencies, the modulation amplitude can get sufficiently large to damage the coils. The Mod. Amplitude Limit needs to be adjusted separately for each modulation frequency.

4. **Set the calibration frequency range.** A calibration is required for each modulation frequency that you intend to use. The SCT/H signal channel, has a range of 100 kHz to 6 kHz in 0.1 kHz steps. The SCT/L signal channel covers the frequency range of 6 kHz to 100 Hz in 1 Hz steps. Most people will normally run all their spectra using 100 kHz modulation, but under some special circumstances, other frequencies may be desirable. A good approach to take is to calibrate the signal channel every 10 kHz from 100 kHz to 10 kHz. A sufficiently large range of frequencies is then covered for most EPR experiments. Enter the Mod. Frequency Start [kHz] (usually 100 kHz), Mod. Frequency End [kHz] as well as the Mod. Frequency Increment [kHz] values. The start value must be greater than the end value. (See Figure 2-6.) Note that you should set the start and end values equal to each other for ER 4122SHQE or ER 4119HS cavities so that only one frequencies at a time is calibrated. (See the warning in the lefthand margin.)
5. **Set initial values for the tuning caps search.** The parameters in this section set the starting values for the tuning capacitor search. (See Figure 2-6.) The **Initial Modulation Amplitude** defines the modulation amplitude used when optimizing the tuning capacitors. The default value, 10%, is appropriate for most cavities and resonators. The default **Initial Sweep Width** of 20 G works well for a DPPH sample. If you know an approximate optimal value for the tuning capacitors at 100 kHz, you can enter that value in **Tuning C Start Value**. This will save you time because otherwise the searching routine will start from 0 nF. **Mod Frequency Limit** is always set at 50 kHz because skin depth poses less of a problem at lower frequencies.



It is not necessary to calibrate every 100 Hz from 100 kHz to 10 kHz. In particular, FlexLine resonators in a cryostat with no gas flow can become sufficiently warm to damage the resonator if you calibrate more than 20 frequencies in one experiment.



Better safe than sorry. It is a good idea to calibrate the phase of the second harmonic when running the calibration routine.

6. **Adjust the Mod. Amplitude Limit.** The Mod. Amplitude Limit parameter limits the size of the signal sent to the modulation amplifier. This prevents damage to the coils owing to excessive current. Enter a value of 75% for an ER 4122 SHQ cavity. For Flex-line resonators, a value of 10% is more appropriate. A value of 30% works well for ER 4122SHQE and ER 4119HS cavities. It is always safe to set this value a bit low as you will adjust this value later. A 100 G Sweep Width is sufficient for most resonators. (See Figure 2-6.)
7. **Enter values for the modulation phase search.** The parameters in this step depend on your calibration sample. The default values of 1 G Sample Line Width and 20 G Sweep Width work well with the Bruker DPPH standard. The signal channel can produce either a first harmonic (first derivative) or a second harmonic (second derivative) spectrum. If you have no need for second harmonic spectra and wish to save a bit of time in the calibration routine, you may deselect this option by clicking the **Calibrate 2nd Harmonic** button. The green color indicates that the option is selected. (See Figure 2-6.)
8. **Enter the filename for the calibration file.** Click the **Results** tab. (See Figure 2-7.) The calibration file name usually consists of two to four letters that identify the type of cavity (ST for ER 4102ST or SHQE for ER 4122SHQE) followed by the serial number of the cavity. This number is found on the back or front of the cavity. For FlexLine series resonators the serial number is found at the top part of the module block. Enter the calibration file name into the **Calibration Data Set** box. If you are re-calibrating, you can click the file name in the drop down list of the **Calibration Data Set**. The new values will be appended to the calibration file or replace previous values in the file. If you want to start fresh, click the **Delete Data Set** button to delete the old calibration data.

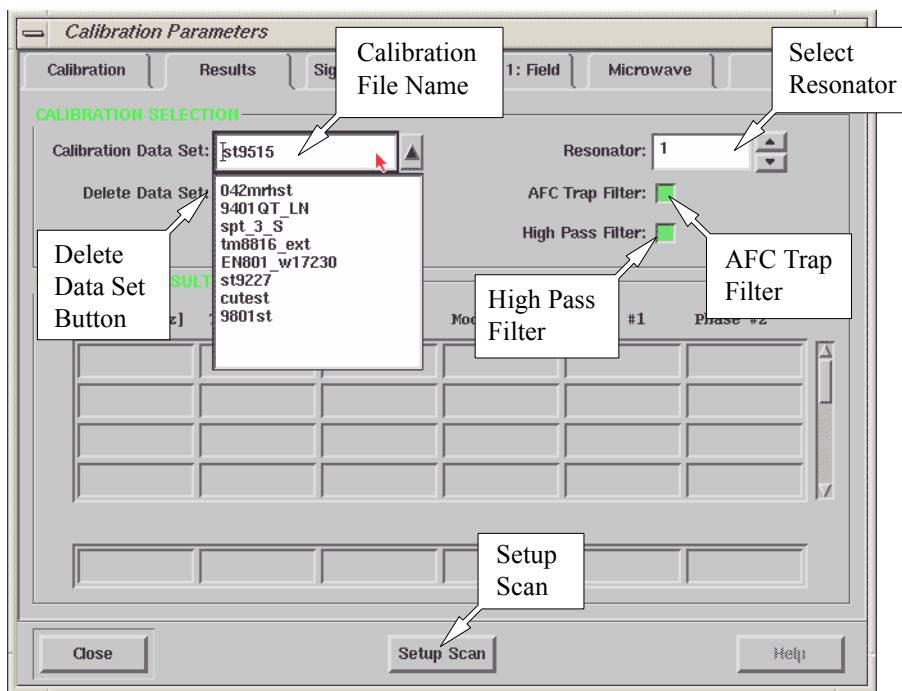


Figure 2-7 The Results folder of the Calibration Parameters dialog box.

9. **Select the resonator.** In almost all cases, the 1st Resonator should be selected. (See Figure 2-7.) The ER 4105DR dual cavity has two sets of modulation coils. By selecting 1st Resonator or 2nd Resonator, you are selecting the set of modulation coils that are to be calibrated.



The ER 4105DR dual cavity is different from the ER 4116DM dual mode cavity. The ER 4116DM has only one set of modulation coils.

10. **Turn on the AFC Trap and High Pass Filters.** The AFC trap filter blocks any signal components at the AFC modulation frequency that may contribute to noise in the EPR signal. The high pass filter suppresses low frequency signal components that also contribute to added noise in the EPR signal. These two filters influence the calibration values of the signal channel. By default they are both selected. Ensure that both options are selected. (See Figure 2-7.) Only under very rare circumstances would you acquire spectra without these filters.
11. **Start the Setup Scan.** When the Setup Scan function is activated, the magnetic field is swept rapidly (up to 50 Gauss at about a 30 Hz rate) to provide a “real time” display of the EPR spectrum on the screen. Click the Setup Scan button in the Results folder. (See Figure 2-7.)

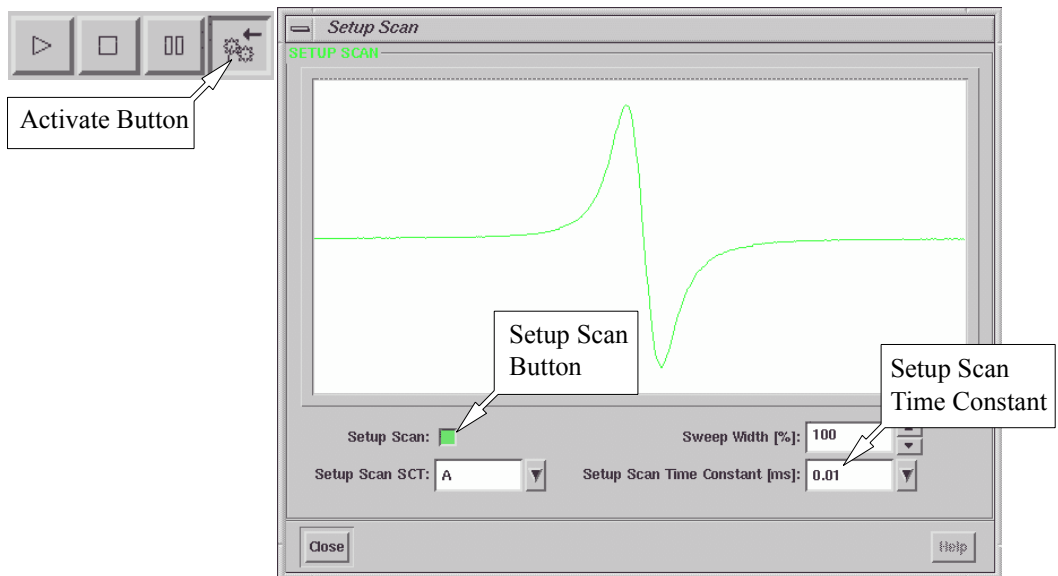


Figure 2-8 The Setup Scan window.

A **Setup Scan** window will appear. Click the **Activate** button in the monitoring panel if it is not already activated. Then click the **Setup Scan** button in the **Setup Scan** window to activate it. (See Figure 2-8.) Leave this window open so you can adjust the parameters interactively.

12. **Set the Microwave Attenuator to approximately 25 dB.** Click the **Microwave** tab. Set the microwave power to 25 dB. (See Figure 2-9.)

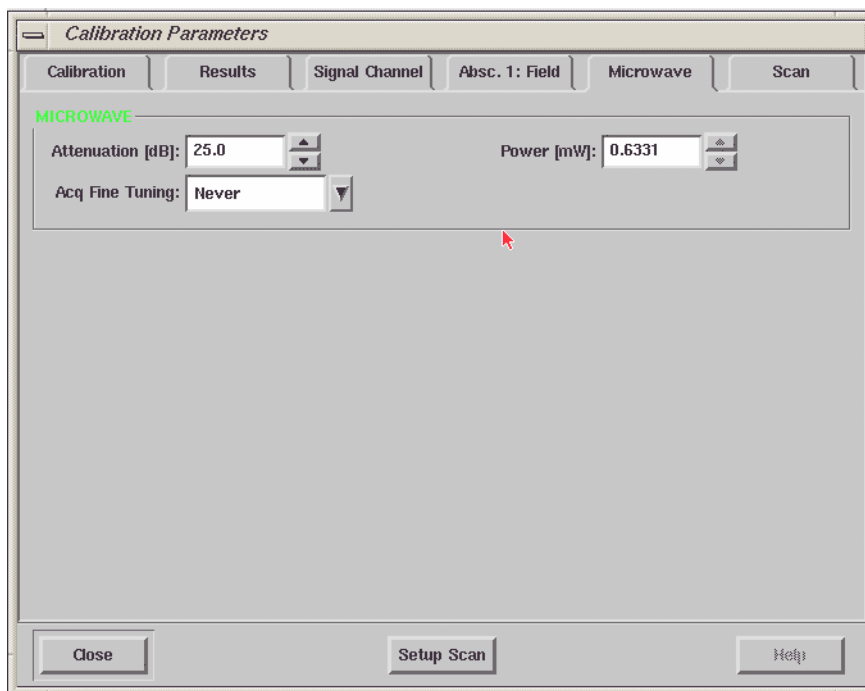


Figure 2-9 The Microwave folder of the Calibration Parameter dialog box.



You should be able to see the DPPH signal by now. If you do not see the signal, change the modulation phase by 90 degree. If you still can not find the signal, refer to Chapter 8 of the Elexsys E 500 User's Manual: Basic Operations

13. **Center your DPPH spectrum in the display.** Click the Absc.1: Field tab. (See Figure 2-10.) Arrange the Calibration Parameters window and the Setup Scan dialog box so that you can see both of them at the same time. Make sure that the Center box is selected (green). Adjust the Center Field value by entering a field value or clicking on the up and down arrows until the signal appears centered in the Setup Scan window. For a microwave frequency of about 9.8 GHz, DPPH resonates at 3500 Gauss. Reducing the Sweep Width in the Setup Scan window can help you to center the signal.

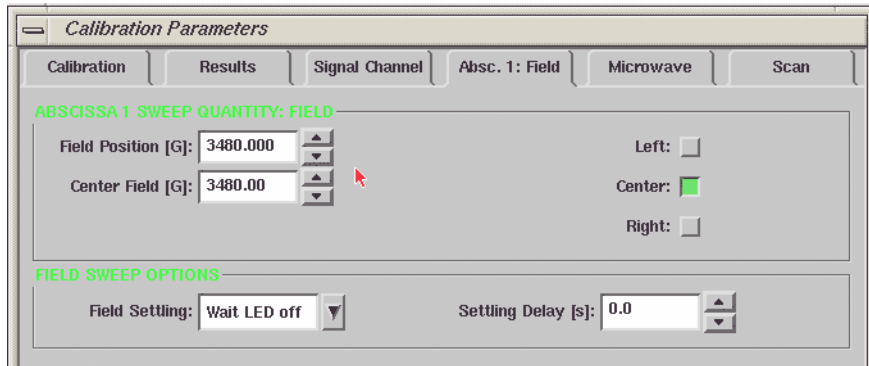


Figure 2-10 The Absc.1: Field folder of the Calibration Parameters dialog box.



The EN 801 ENDOR resonator should not be used above 25 kHz. Consult the Elexsys E 560 documentation for instructions on calibrating this cavity.

14. **Adjust other parameters.** Adjust the Receiver Gain so that the signal fills approximately half of the vertical display range in the **Setup Scan** window. Make sure that the signal channel is set to 100 kHz modulation (or the maximum modulation frequency you entered in the **Calibration** folder) and first harmonic detection. A 1 gauss **Modulation Amplitude** usually is sufficient.
15. **Optimize the DPPH sample position.** Move the sample tube up and down until the maximum signal intensity is attained. (See Figure 2-11.) Avoid moving the sample from side to side. Perhaps the best technique is to loosen the collet nuts for the pedestal and sample tube and move the sample too low. Then use the pedestal to slowly push the sample up. Sometimes the process of moving the sample tube in the cavity can cause the AFC to lose lock. Re-tune the frequency if this happens. If the signal is clipped, decrease the **Receiver Gain**. Rotate the sample tube until the maximum signal intensity is attained. When you have maximized the signal intensity, secure the sample tube by tightening the collet nuts. Check the matching if needed. You may need to re-center the signal by adjusting the **Center Field** in the **Absc.1: Field** tab.

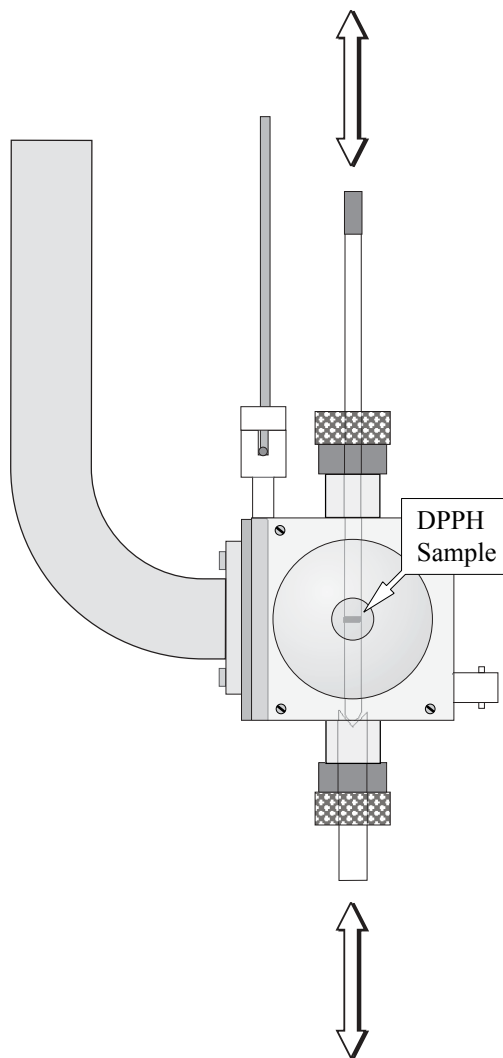


Figure 2-11 Fine adjustment of the DPPH sample height in the cavity.

16. **Close the Setup Scan window.** Click the Close button in the Setup Scan window.
17. **Reset some parameters.** After centering the DPPH sample, most of the parameters should be fairly close to what is needed for the calibration routine. Set the Receiver Gain to approximately 36 dB.
18. **Start the calibration routine.** Click the Result tab. Move the Calibration Parameters window to the corner so that you can see the acquisition and the calibration result at the same time. Click the Run button in the monitoring panel to start the calibration routine.

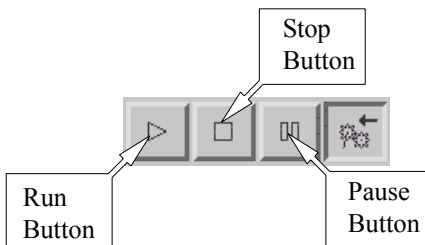


Figure 2-12 The Acquisition control buttons.

19. **Adjust the Receiver Gain.** Monitor the Receiver Level while the scan is running. (See Figure 2-13.)

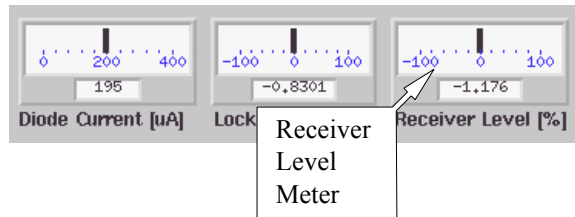


Figure 2-13 Monitoring the Receiver Gain.

If the needle deflects more than 1/4 of the display, stop the calibration routine by clicking the Run/Abort button. Lower the **Receiver Gain** and restart the calibration by clicking the Run button again. A warning message will appear asking if you want to overwrite the existing value. Click the **Yes** button to continue. Repeat until the needle does not deflect more than 1/4 of the display. You may have to repeat this last step a few times.

20. **Observe the calibration results.** The Results folder consists of a table of parameter values and settings for each modulation frequency. The first column is the modulation frequency. For modulation frequencies greater than or equal to 50 kHz, the optimal tuning capacitor value is listed in the second column. The third column contains the value of Mod Amp [% max]. Mod Amp [G] in the fourth column is the measured maximum modulation amplitude when the corresponding Mod Amp [% max] is used. Phase #1 and Phase #2, in columns five and six respectively, are the phases at which the first and second harmonic signals are nulled. (See Figure 2-14.)

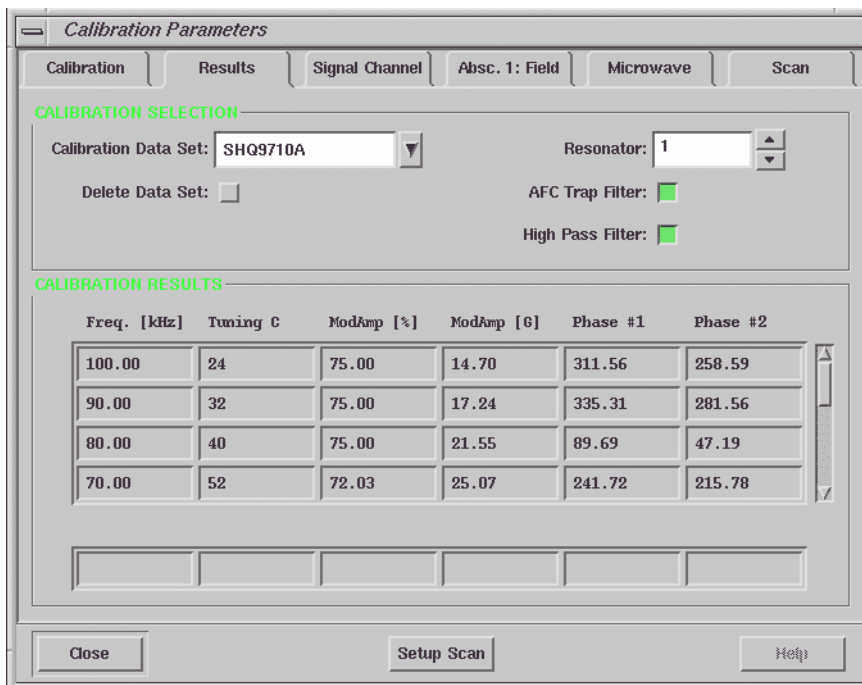


Figure 2-14 The calibration results.

21. **Check the Mod. Amp [G] at 100 kHz.** The calibration routine performs its task sequentially, starting with the highest modulation frequency and continuing for each selected modulation frequency. As each parameter is determined, it is displayed in the table. (See Figure 2-14.) Wait until the 100 kHz calibration is completed. Click the **Pause** button of the **Acquisition Control** buttons. Note the value in the fourth column of the table, **Mod Amp [G]**. Compare the **Mod Amp [G]** at 100 kHz with the values listed for your cavity in Table 2-1. If the value obtained by the calibration routine is slightly less than or approximately equal to the value listed in Table 2-1, allow the calibration routine to continue its task by clicking the **Pause** button again and proceed to Step 23. If the value exceeds the values listed in Table 2-1, stop the calibration routine by clicking the **Run/Abort** button in the **Acquisition Control** buttons. Values that are too high can allow excessive current to flow through the modulation coils of the cavity at the maximum modulation amplitude, resulting in damaged modulation coils.

Cavity	Maximum Mod. / Gauss at 100 kHz
ER 4102ST	32
ER 4105DR	32
ER 4104OR	32
ER 4116DM	10
ER 4103TM	16
ER 4108TMH	32
ER 4106ZRC	10
ER 4106ZRAC	10
ER 4107WZC	10
ER 4107WZAC	10
ER 4115ODC	10
ER 4115ODAC	10
ER 4119HS	25
ER 4122SHQ	15
ER 4122SHQE	25
ER 4114HT	7
ER 4117MX (or D-MVT)	10
ER 4117D-R	32
ER 4109EF	10

Table 2-1 Maximum modulation amplitude for EPR cavities.

22. **Reset the Mod. Amplitude Limit.** The Mod. Amplitude Limit parameter which was set in Step 6. limits the size of the signal sent to the modulation amplifier to prevent any danger of burning out the modulation coils. To calculate a safer value for Mod. Amplitude Limit use the following formula:

$$\text{Mod Amplitude Limit} = \text{Mod Amp}[\% \text{max}] \frac{\text{Max. Mod.}}{\text{Mod. Amp [G]}}$$

where Mod Amp[%max] and Mod. Amp [G] are the values listed in the calibration table and Max. Mod. is the maximum modulation amplitude listed for your cavity in Table 2-1. Enter this new value for Mod. Amplitude Limit in the Calibration tab. Click the Results tab to bring it to the front. Click the Run button to restart the calibration routine.

23. **Finish the calibration.** When the routine is finished the Run button will no longer be highlighted and the calibration data for the lowest modulation frequency is completed. (*I.e.* the calibration data in the Results tab are no longer -1.)
24. **Special Instructions for ER 4122SHQE and ER 4119HS cavities.** It was mentioned on page 12 that these cavities should be calibrated one frequency at a time. An additional requirement is that the Mod. Amplitude Limit be set properly at each modulation frequency. Follow Step 21. and Step 22. for each of the individual modulation frequencies after you have started the calibration routine for that frequency.

25. **Adding additional frequencies.** If you wish to add more frequencies to the calibration file, for example lower frequencies below 10 kHz, the remaining instructions guide you through the necessary steps. Click the **Calibration** tab. Change the **Mod. Frequency Incr.** (for example, to 2 kHz). Change the **Mod. Frequency Start** (for example to 5 kHz). Change the **Mod Frequency End** (for example to 1 kHz). (See Figure 2-15.) For modulation frequencies less than 10 kHz you need to reset the **Mod. Amplitude Limit** to 10%.

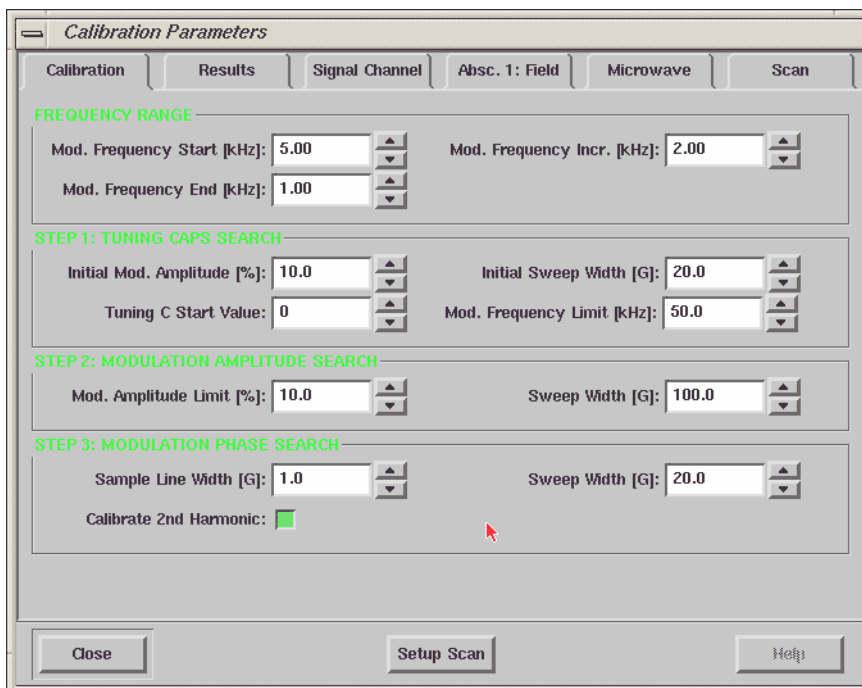


Figure 2-15 Changing the Frequency Range.

26. **Start the calibration routine.** Click the Run button to start the calibration routine. The low modulation frequency calibration data will be appended to the table. If there is one or more calibration frequencies already existing in the previous calibration file, a warning message will appear on the screen asking you if you want to overwrite the existing value. Click the **Yes** or **No** button (depending on your wishes) to continue the calibration routine. Notice that the previous calibration data for other frequencies are still kept in the file.
27. **Close the Calibration Parameters dialog box.** Click the Close button in the Calibration Parameters dialog box. The signal channel is now calibrated for your cavity and the data saved in the calibration file. The next time when you connect the Xepr software to the spectrometer, this calibration file will be the default calibration file.

This chapter describes procedures for testing the performance of your Bruker EPR spectrometer. The test measures the spectrometer's sensitivity. The procedure is especially designed to test as many of the components of the spectrometer as possible with one simple test. It therefore gives you a good indication of the overall health of your spectrometer. It is also an excellent criterion for comparing the sensitivity of different spectrometers. Should your spectrometer or cavity not meet specifications, first consult Chapter 9 of the Elexsys E 500 User's Manual: Basic Operations on troubleshooting. If none of the hints solve the problem, contact your Bruker EPR service representative.

Signal to Noise Ratio Test

3.1

The signal to noise ratio test is an important part in maintaining your spectrometer. It is also helpful in diagnosing possible problems you may encounter especially when you deal with very weak signals or quantify your EPR signals. The signal to noise ratio test uses the ER 4122SHQ or ER 4122SHQE cavity and the weak pitch sample that was shipped with your spectrometer. The test measures the EPR signal intensity (peak-to-peak height) of the weak pitch sample at low microwave power (23 dB) and then measures the noise level under the same conditions except higher microwave power (0 dB) and higher receiver gain to characterize the noise better. The formula for calculation of the signal to noise ratio is:

$$\frac{S}{N} = \frac{A_S}{A_N} \times \frac{G_N}{G_S} \times \sqrt{\frac{P_N}{P_S}} \times \frac{2.5}{\sqrt{T} \times C}, \quad [3-1]$$

where A_S and A_N are the peak to peak height of the weak pitch and amplitude of the noise respectively. G_S and G_N are the receiver gains used in signal and noise measurements respectively. We use their ratio to correct for the gain difference. P_S and P_N are the powers used in two measurements and we use the square root of the ratio of powers to correct for the power difference. The factor of 2.5 translates the peak-to-peak noise level to a RMS (Root Mean Square) noise level. T is the time constant (in seconds) and we use the square root of the time constant to normalize the S/N to a one second time constant. C is the weak pitch correction factor that is printed on the label of the weak pitch sample. The standard instrument settings for signal and noise measurements are listed in Table 3-1.

Parameter	Signal Measurement	Noise Measurement
Modulation Amplitude	6.0 G	6.0 G
Modulation Frequency	100 kHz	100 kHz
Receiver Gain	60 dB	80 dB
Phase	0	0
Time Constant	1310.72 ms	1310.72 ms
Conversion Time	163.84 ms	163.84 ms
Center Field	3510 G	1500 G
X Axis Setting	Field Sweep	Time Scan
Sweep Width	50 G	--
Resolution of X Axis	1024 points	1024 points
Microwave Attenuation	23 dB	0 dB

Table 3-1 Parameters for Signal/Noise Measurements with SuperX.

Preparing for the S/N Test

3.1.1



You need to purge nitrogen gas through the cavity to avoid possible oxygen signals owing to the high sensitivity of the cavity



The calibration factor is found on the weak pitch sample's label. It is listed as:

$C = C_0 \times \text{calibration factor}$

It is usually approximately equal to one and corrects for variations in the sample concentration.

1. **Install an ER 4122SHQ or ER 4122SHQE cavity.** (See Section 7.2 of the Elexsys E 500 User's Manual: Basic Operations for instructions.) The specification for the signal to noise ratio is based on an ER 4122SHQ or ER 4122SHQE cavity using the weak pitch sample. We strongly suggest using this cavity for the standard test and keep a record and verify the specification periodically. If you use other types of cavities to do the signal to noise ratio test the results and the settings will be different due to different Q values and the microwave field distributions of the cavities.
2. **Insert the weak pitch sample.** Copy down the calibration factor posted on the label of the weak pitch sample before you insert it. The weak pitch sample should be inserted in the cavity until the bottom of the label and tape on the sample tube is flush with the collet. You also should use the pedestal to hold the weak pitch sample rigidly.
3. **Turn on the instrument and tune.** Turn on the instrument if it is not on yet. Tune the microwave bridge and the cavity. It is best to wait several hours to let the spectrometer fully warm up because the spectrometer is most sensitive and stable after it has achieved thermal equilibrium.
4. **Optimize the AFC.** Refer to Section 7.3.2. of the Elexsys E 500 User's Manual: Basic Operations for instructions or hints.
5. **Check the Spectrometer Configuration.** Click Spectrometer Configuration in the Acquisition menu and click the Signal Channel tab. Make sure you have the correct calibration file for the cavity you are using. High Pass Filter and AFC Trap Filter should be acti-

vated. If you make any changes, click the Apply button and then the Close button.(See Figure 3-1.)

Spectrometer Configuration

Field Misc. Signal Channel ENDOR FT-Epr W-Band

CALIBRATION OPTIONS

Resonator: 1 AFC Trap Filter: High Pass Filter:

Standard Calibration Data RF Calibration Data

STANDARD CALIBRATION DATA

Calibration Data Set: SHQ9710A Delete Data Set:

Freq. [kHz]	Tuning C	ModAmp [%]	ModAmp [G]	Phase #1	Phase #2
100.00	24	75.00	14.70	311.56	258.59
90.00	32	75.00	17.24	335.31	281.56
80.00	40	75.00	21.55	89.69	47.19
70.00	52	72.03	25.07	241.72	215.78
60.00	76	66.44	27.61	311.41	301.41
50.00	110	63.81	32.51	341.88	0.62

Close Apply Reset Load Save Help

Figure 3-1 The Spectrometer Configuration dialog box.

Acquire the Signal Spectrum

3.1.2

1. **Create an experiment to measure the signal.** Click the New Experiment button to bring up the dialog box. (See Figure 3-2.) Click the C.W. tab and select Field for Abscissa 1. It is a good idea to assign the Experiment Name as Signal. Click Create to continue.

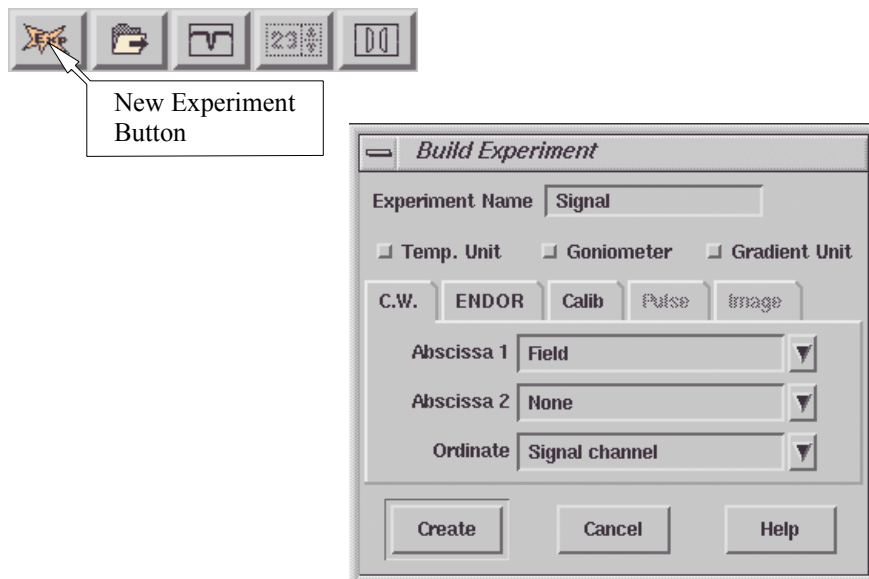


Figure 3-2 Creating an experiment to measure the weak pitch signal.

2. **Input the sample information.** Click Acquisition in the menu bar and then Sample Info. (See Figure 3-3.) Enter the weak pitch information. Click OK to continue.

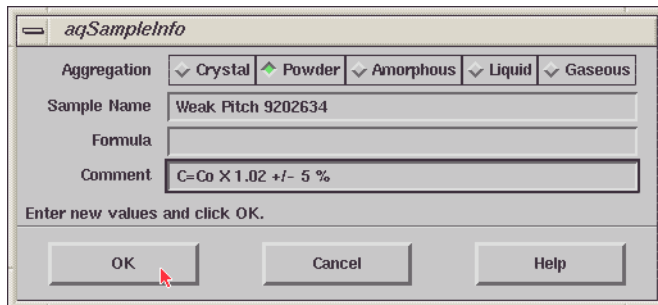


Figure 3-3 The sample information dialog box.

3. **Open the Acquisition Parameters dialog box.** Click the Activate button and then the Acquisition Parameters button to bring up the dialog box.

4. **Set the Microwave Power.** Click the Microwave tab and set the Attenuation to 23 dB. We also advise you to select the Before Each Slice Scan option in the Acq. Fine Tuning box to ensure the acquisition is made under proper coupling conditions. It will automatically fine tune the coupling each time a scan starts. (See Figure 3-4.)

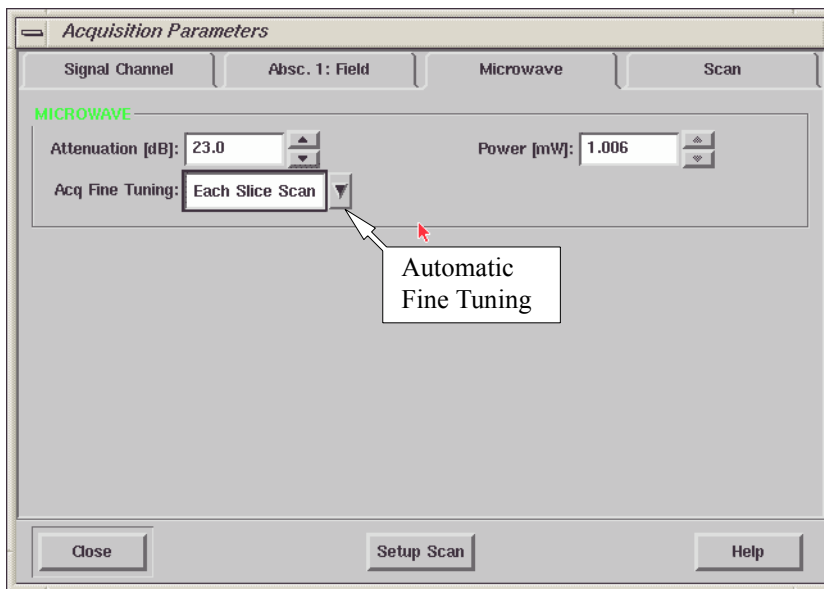


Figure 3-4 Setting the microwave attenuation.

5. **Set the magnetic field.** Click the Absc. 1: Field tab. Set the Center Field to 3510 G and the Sweep Width to 50 G. Set the Number of Points to 1024. Since a very long time constant will be used, set the field to the Left edge to avoid overshoots or undershoots in the first few data points when you acquire the spectrum. (See Figure 3-5.)

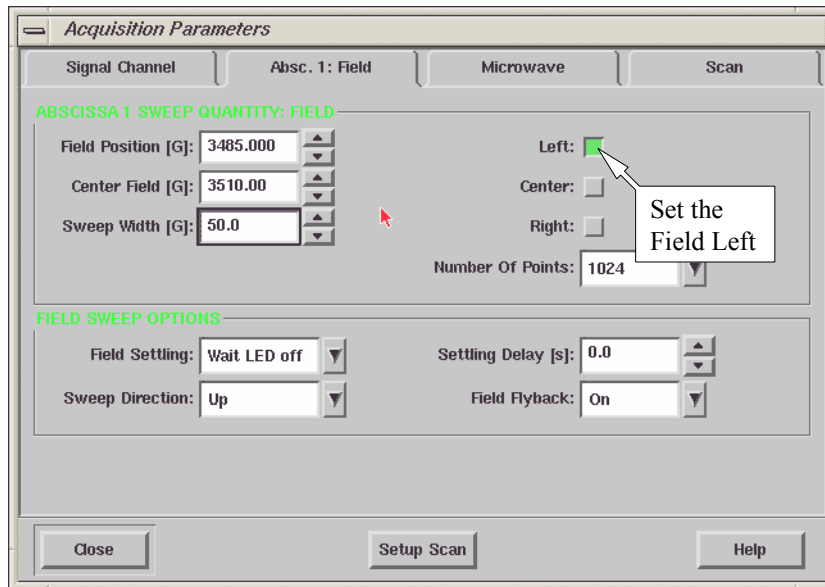


Figure 3-5 Setting the magnetic field.

6. **Set the Scan parameters.** Click the Scan tab and make sure that the Number Of Scans is set to one. (See Figure 3-6.)

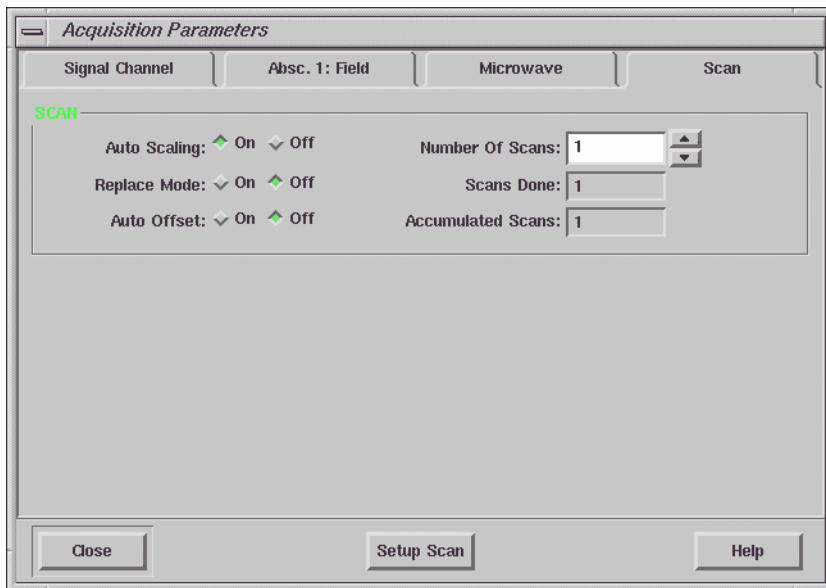


Figure 3-6 Setting the Scan parameters.

7. **Set the Signal Channel parameters.** Click the Signal Channel tab to bring the folder to the front. Set the parameters to those shown in Figure 3-7 or consult Table 3-1. If the Receiver Level meter is off center, change the Offset to bring the needle back to the center.

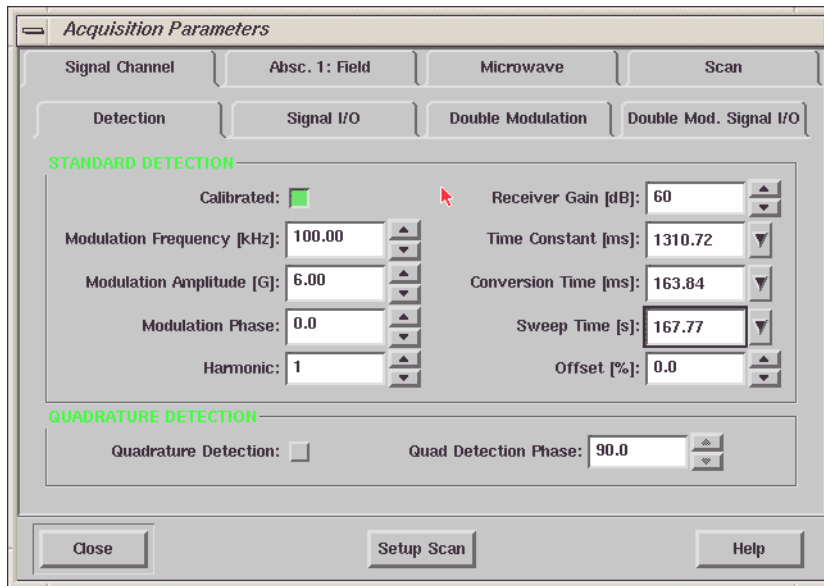


Figure 3-7 Setting the Signal Channel parameters.

8. **Acquire a signal spectrum.** Click the Run button to acquire a spectrum. It should look similar to Figure 3-8.

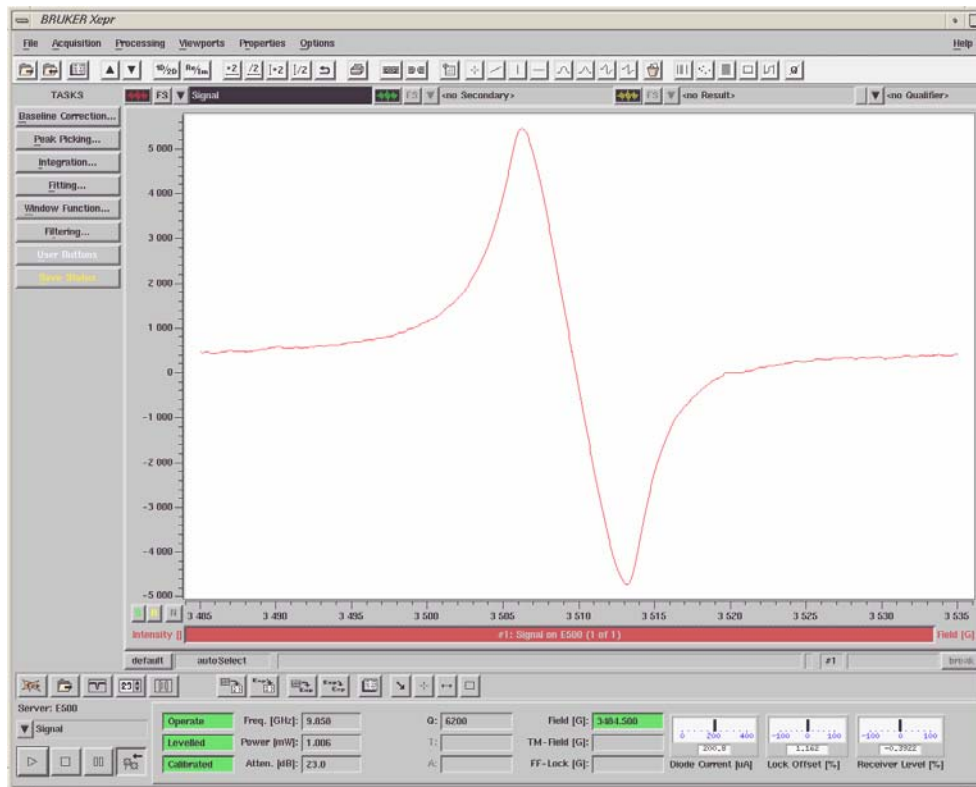


Figure 3-8 The weak pitch signal.

9. **Store the signal spectrum.** Click on the Primary Properties menu, select Store in the drop down menu to store the spectrum in memory. Name the spectrum and store it.

Acquire the Noise Spectrum.

3.1.3

1. **Create an experiment to measure the noise.** Click the New Experiment button to bring up the dialog box. (See Figure 3-9.) Click the C.W. tab and select Time for Abscissa 1. It is a good idea to assign the Experiment Name as Noise. Click Create to continue.

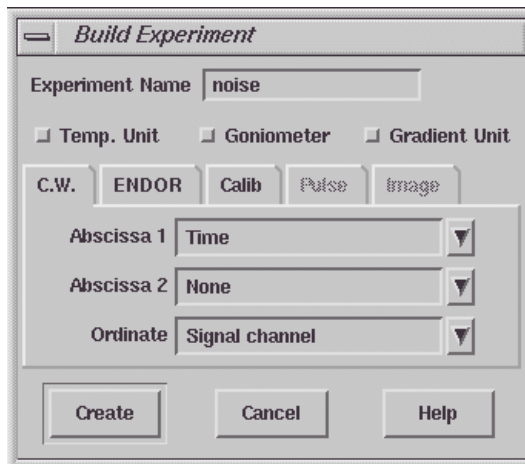


Figure 3-9 Creating a noise measurement experiment.

2. **Open the Acquisition Parameters dialog box.** Click the Activate button if it is not depressed and then the Acquisition Parameters button to bring up the dialog box.

3. **Set the Microwave Power.** Click the Microwave tab. Set the Attenuation to 0 dB. We also advise you to select the Before Each Slice Scan option in the Acq. Fine Tuning box to ensure the acquisition is made under proper coupling conditions. It will automatically fine tune the coupling each time a scan starts. (See Figure 3-10.)

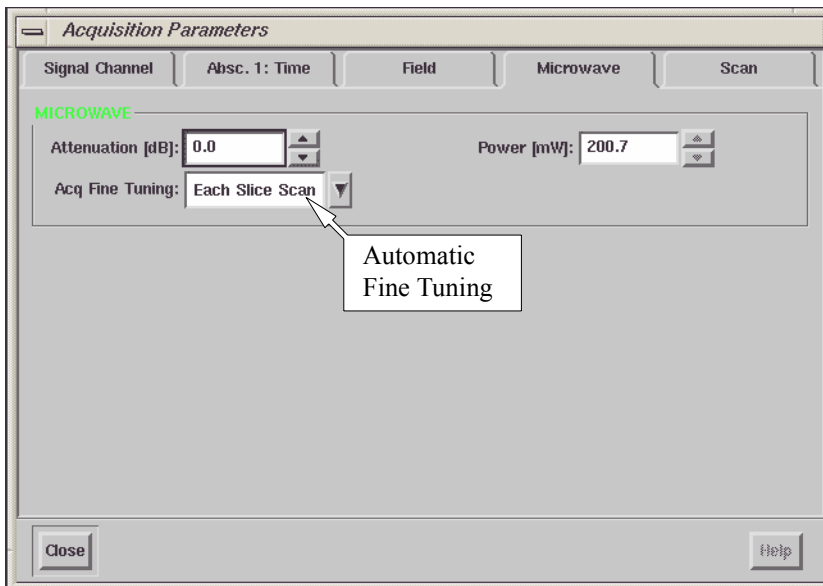


Figure 3-10 Setting the microwave power for the noise measurement.

4. **Set the Magnetic Field.** Click the Field tab. Set the Static Field to 1500 G. (See Figure 3-11.) Since a very long time constant will be used, set a settling delay time of 2-5 seconds to avoid overshoots or undershoots in the first few data points when you acquire the spectrum.

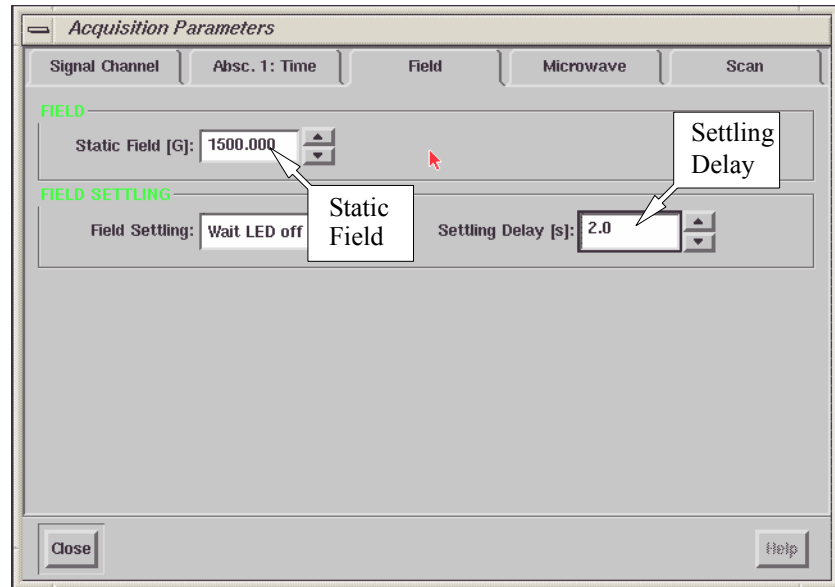


Figure 3-11 Setting the magnetic field.

5. **Set the time sweep parameters.** Click the Absc 1: Time tab. Set the Number of Points to 1024. Set the Sweep Time to 167.77 s. (See Figure 3-12.)

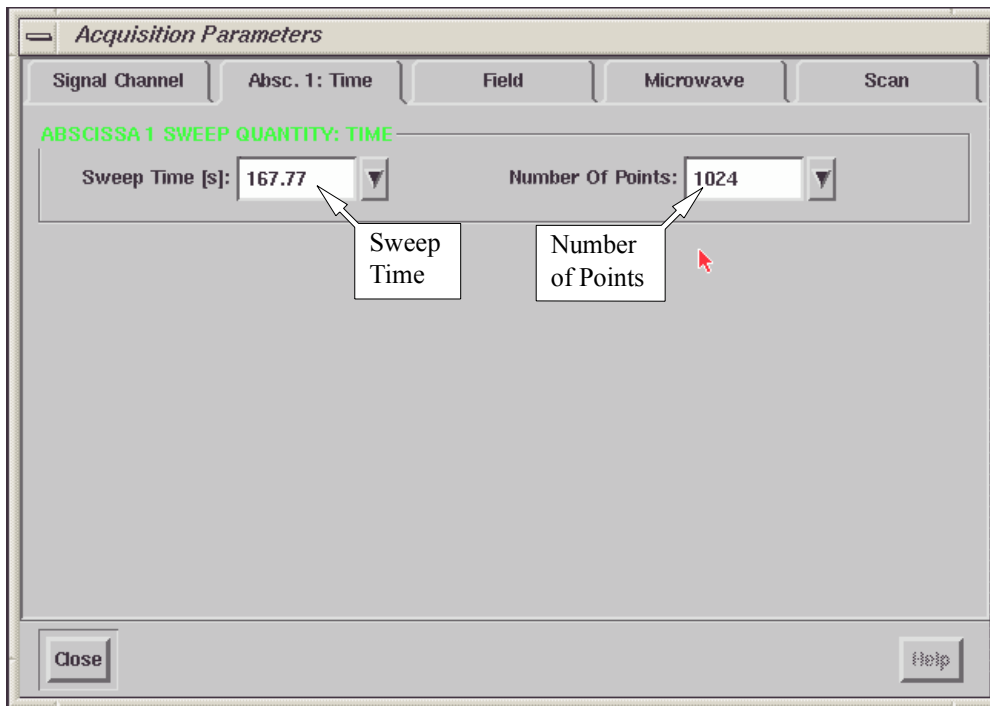


Figure 3-12 Setting parameters for the Absc.1: Time folder.

6. **Set the Scan parameters.** Click the Scan tab and make sure that the Number Of Scans is set to one. (See Figure 3-13.)

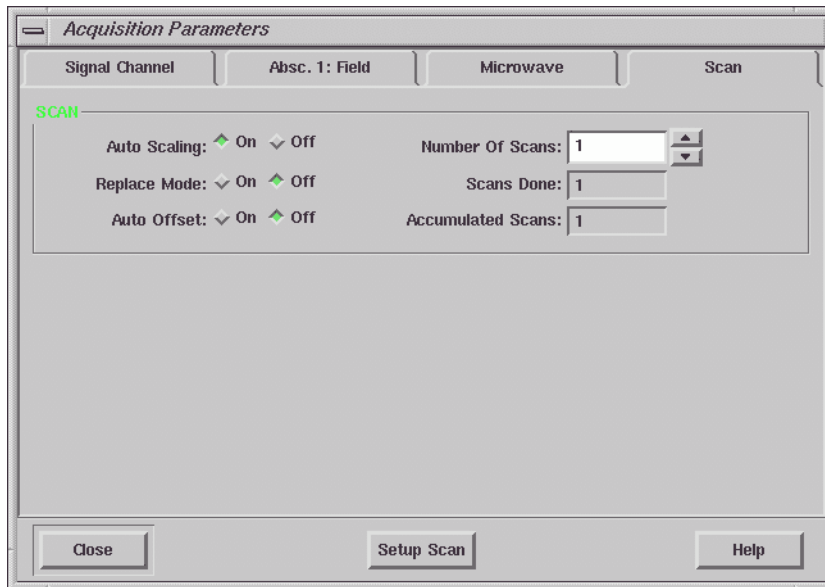


Figure 3-13 Setting the Scan parameters.

7. **Set the Signal Channel parameters.** Click the Signal Channel tab to bring the folder to the front. Set the parameters to those shown in Figure 3-14 or consult Table 3-1. If the Receiver Level meter is off center, change the Offset to bring the needle back to the center.

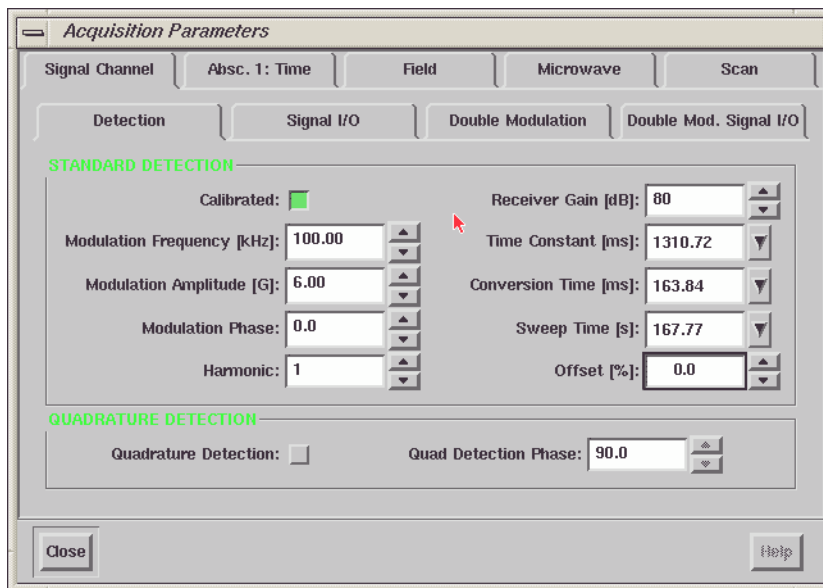


Figure 3-14 Setting the Signal Channel parameters.

8. **Get ready to acquire a noise spectrum.** Click the RUN button in the acquisition tool bar to acquire the noise spectrum. Frequently the baseline will drift since 200 mW microwave power is going to heat up the cavity and the sample. Wait a few minutes to achieve thermal equilibrium. Check the tuning and coupling of the system. Retune the system if necessary. You may also have a rather large offset due to the excessive power and high gain. Adjust the offset so that the indicator of the Receiver Level meter is in the middle.



If there is a linear drift, you do not need to re-acquire the noise spectrum. We shall subtract it out in the next step

9. **Acquire a noise spectrum.** Click the RUN button in the acquisition tool bar and acquire the noise spectrum again. (See Figure 3-15.) If there is no baseline drift, store the spectrum and continue to the next section. If there is drift, wait a bit longer and run the noise trace again.

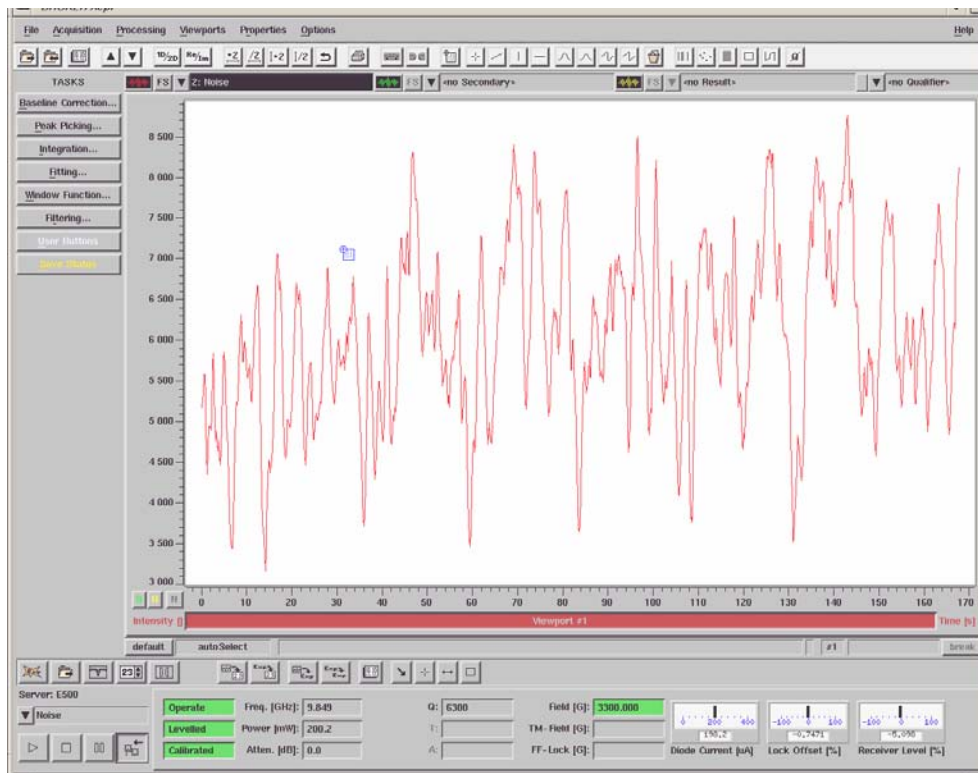


Figure 3-15 Acquiring a noise spectrum.

10. **Correct the linear drift of the baseline if necessary.** If the baseline drifts you can click the Baseline Corr. button in the Tasks bar and then click Polynomial. Click the 1st order button to perform a linear fit of the baseline. (See Figure 3-16.)

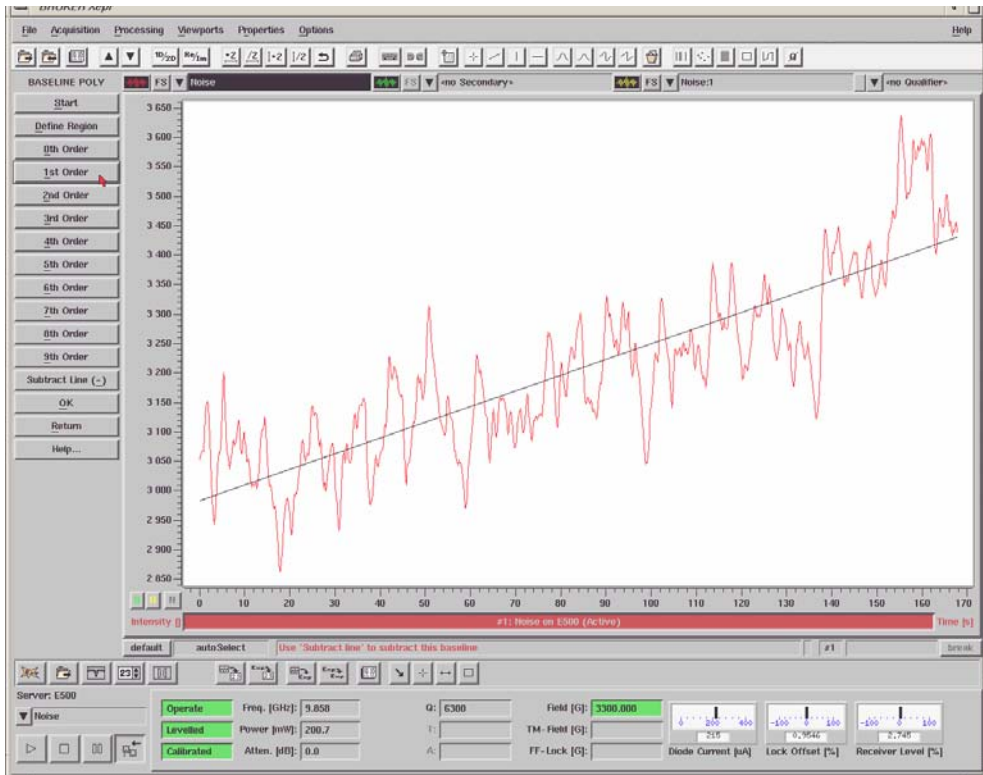


Figure 3-16 A linear baseline correction of the noise spectrum.

Click **Subtract Line (-)** to subtract the linear baseline from the noise spectrum to compensate for linear drifts. Click **OK** and store the resulting noise spectrum. (See Figure 3-17.) Click **Return** on the **Tasks** menu.

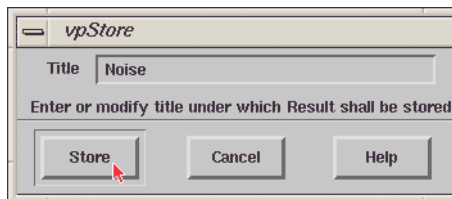


Figure 3-17 Storing the noise spectrum.

Measuring the Signal to Noise Ratio

3.1.4

1. **Display the noise spectrum in the Primary dataset.** If the noise spectrum is not in the Primary dataset, click the Primary Dataset List and select the noise spectrum.
2. **Display the signal spectrum in the Secondary dataset.** Click the Secondary Dataset List and select the signal spectrum.

3. **Load the Signal to Noise ProDeL program.** Click the Processing > ProDeL > Load Programs. (See Figure 3-18.)

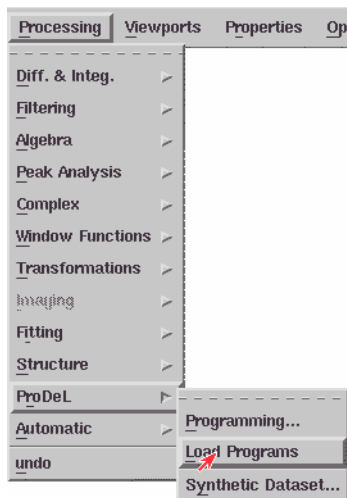


Figure 3-18 Loading the ProDeL program.

A dialog box will appear. Select the path <your home directory>/xepFiles/ProDeL/sharedProDeL/Standard and then the signal2noise file. In the Add Menu Entry selection, click the yes button. Click Load to continue. (See Figure 3-19.)

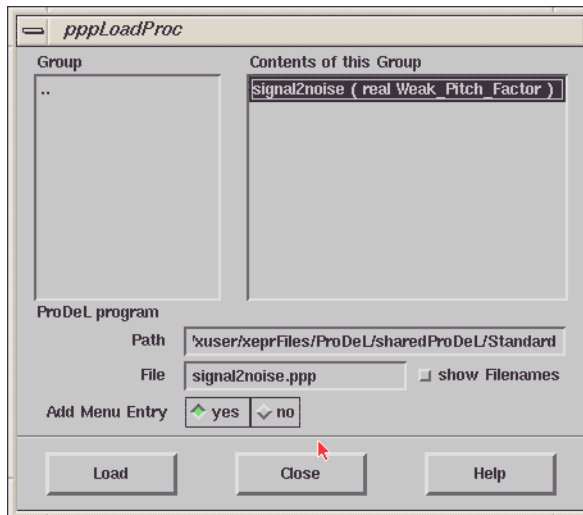


Figure 3-19 Selecting the signal to noise calculation program.

4. **Run the signal2noise ProDeL program.** Click Processing in the menu bar and then ProDeL. You will find signal2noise in the submenu of ProDeL entries. Click signal2noise to start the program. A dialog box pops up asking you to enter the weak pitch factor. Enter the number you copied down from the weak pitch label. Click Apply to continue. (See Figure 3-20.)

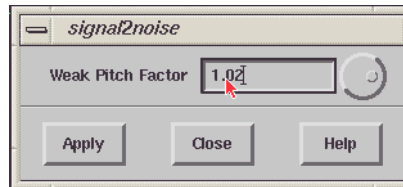


Figure 3-20 Entering the weak pitch factor.

5. **Check the S/N ratio.** The signal intensity, noise level, and the signal to noise ratio will be automatically measured and calculated. The results will be displayed in a message box. (See Figure 3-21.)

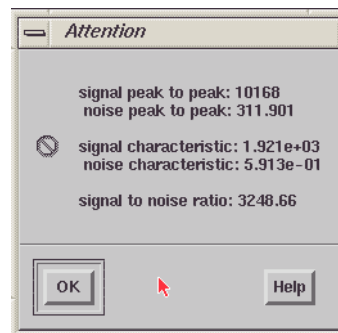


Figure 3-21 Displaying the signal to noise ratio.



This specification is only valid for an E500 spectrometer with a SuperX bridge and ER 4122SHQE or ER 4122SHQ cavity. For a normal bridge and an ER 4102ST cavity, please consult Section 3.1.5.

Copy down the signal to noise ratio for your records. The signal to noise ratio should be greater than 3000 to meet the specifications of the Bruker EPR instrument. If the result is lower than the specification value, consult Chapter 8 and 9 of the Eleksys E 500 User's Manual: Basic Operations. Sometimes a large cavity background signal can significantly decrease the test result. Quite often, waiting longer before acquiring the noise trace helps quite a bit. If those hints do not help, contact your Bruker service representative.

6. **Save the results.** Click **Close** to exit the signal to noise ProDeL program. For your records, you can save these two spectra in a designated directory on the hard disk.
7. **Make a hard copy.** Click on the **Secondary Properties Menu** and click **Align with Primary...** (See Figure 3-22.)

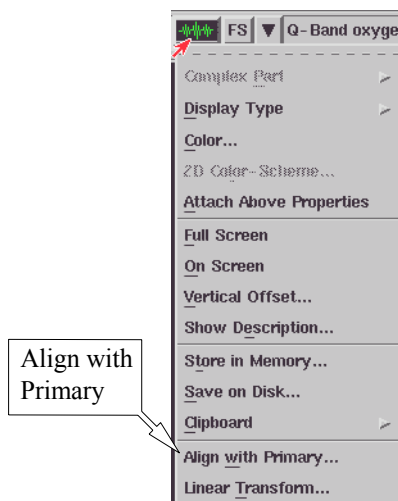


Figure 3-22 Aligning with the primary dataset.

Select Abscissa and then click Align.

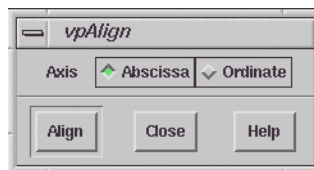


Figure 3-23 Aligning with the primary dataset.

Click on the FS button for the Secondary dataset. (See Figure 3-24.) Click the Print button to make a hard copy.

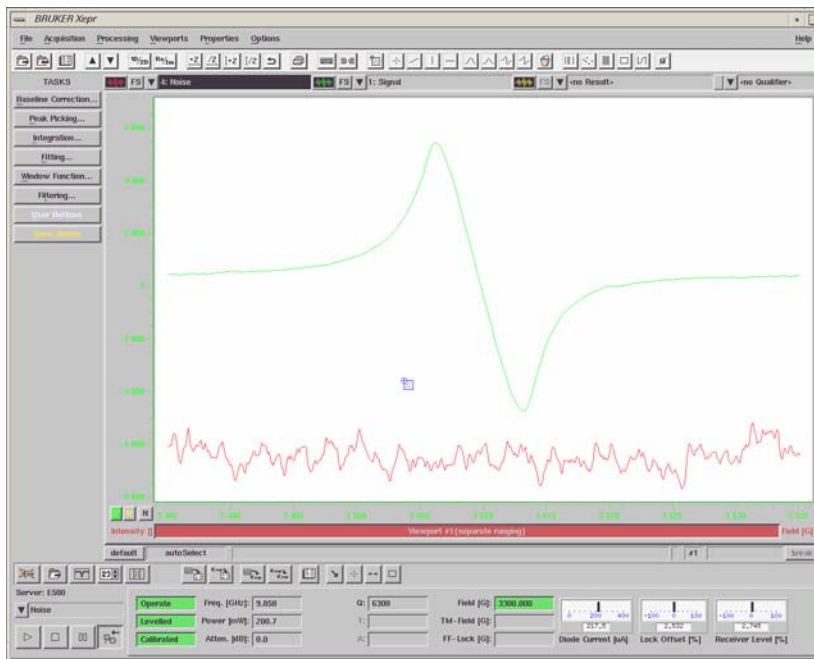


Figure 3-24 Scaling individual spectra to make a hard copy.

Signal to Noise Ratio Test for an ER 4102ST Cavity 3.1.5

The parameters and the specifications for the ER 4102ST cavity are different from those for the ER 4122SHQ cavity. The measurement procedure however is identical. Perform the operations of the previous section except with the following parameters.

Parameter	Signal Measurement	Noise Measurement
Modulation Amplitude	6.0 G	6.0 G
Modulation Frequency	100 kHz	100 kHz
Receiver Gain	60-70 dB	80 dB
Phase	0	0
Time Constant	1310.72 ms	1310.72 ms
Conversion Time	163.84 ms	163.84 ms
Center Field	3480 G	1500 G
X Axis Setting	Field Sweep	Time Scan
Sweep Width	50 G	--
Resolution of X Axis	1024 points	1024 points
Microwave Attenuation	12 dB	0 dB

Table 3-2 Parameters for Signal/Noise Measurements for an ER 4102ST cavity.

The signal to noise ration should be at least 360.

In order to use specific hardware and accessories such as the cavity, frequency counter, field frequency lock, teslameter, *etc.*, you need to configure your Bruker Elexsys E 500 spectrometer. To configure the spectrometer, connect the workstation to the spectrometer. Click Acquisition > Spectrometer Configuration to open the dialog box.

The Field Folder

4.1

This folder contains the parameters related to the magnetic field.

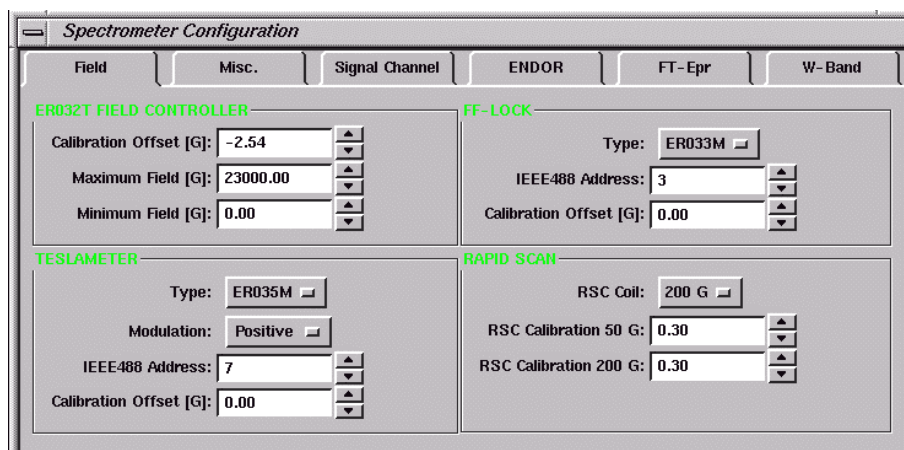


Figure 4-1 The Field folder.

ER032T Field Controller

4.1.1

Calibration Offset: This parameter corrects for the difference in the field value measured by the Hall probe and the actual field value that the sample sees. Refer to Chapter 5 to learn how to calibrate the magnetic field.

Maximum Field: This parameter is the maximum field your magnet can reach. It depends on the size of your magnet and the magnet power supply you have. Refer to the specifications of your magnet and power supply for the maximum field value.

Minimum Field: This parameter is the minimum magnetic field your magnet can reach. If you do not have the zero crossing option for your magnet power supply, the minimum magnetic field is 50 gauss. If you have the zero crossing option you can set this parameter to 0 gauss.

FF-Lock

4.1.2

Refer to Section 12.4 for more detailed instructions regarding setting up the optional E 033M FF-Lock accessory.

Type: If you purchased the optional FF-lock from Bruker, you need to select **ER033M** in the drop-down list. If you do not have an FF-lock, select **None** and go to the next section.

IEEE 488 Address: The acquisition server communicates with the FF-lock through the IEEE bus. The default address for the FF-lock is 3. Make sure to set the IEEE address on the FF-lock unit to 3 as well.

Calibration Offset: Since the FF-lock probe is mounted on the side of the magnet polepiece the field it detects is different from the magnetic field your sample experiences.

Teslameter (Gaussmeter)

4.1.3

Refer to Section 12.5 for more detailed instructions regarding setting up the optional E 035M teslameter accessory.

Type: If you purchased an optional teslameter from Bruker, select ER035M. Otherwise select **None** and go to the next section.

Modulation: The teslameter probe can operate with two modulation polarities. Select **Positive** here. If the magnet is wired incorrectly or the probe is flipped, you may need to select **Negative**.

IEEE 488 Address: The acquisition server communicates with the teslameter through the IEEE bus. The default address for the FF-lock is 7. Make sure to set the IEEE address on the FF-lock unit to 7 as well.

Calibration Offset: The field measured by the teslameter depends on where you place the teslameter probe. It does not necessarily reflect the actual field value that your sample experiences. You can calibrate the field offset by inserting an optional intra-cavity probe in the cavity (if so equipped) or using a standard sample (See Section 12.5.5.). Enter your calibration result here.

Rapid Scan

4.1.4

Refer to Section 6.4 for more detailed instructions regarding setting up rapid scan experiments.

RSC Coil: When performing rapid scan experiments, you need to select one set of rapid scan coils. If you use the scan coils mounted on the magnet, select **200 G**. If you use the scan coils assembled in the cavity, select **50 G**.

**RSC Calibration
50 G:** The sweep width of the rapid scan coils needs to be calibrated. Enter the calibration factor for the 50 G rapid scan coils here. Refer to Section 6.4 to learn how to calibrate the sweep width of the rapid scan coils.

**RSC Calibration
200 G:** Enter the calibration factor of the 200 G rapid scan coils here. Refer to Section 6.4 to learn how to calibrate the sweep width of the rapid scan coils.

The Misc. Folder

4.2

The Misc. (miscellaneous) folder of the Spectrometer Configuration dialog box contains configuration parameters for a variety of hardware components and accessories.

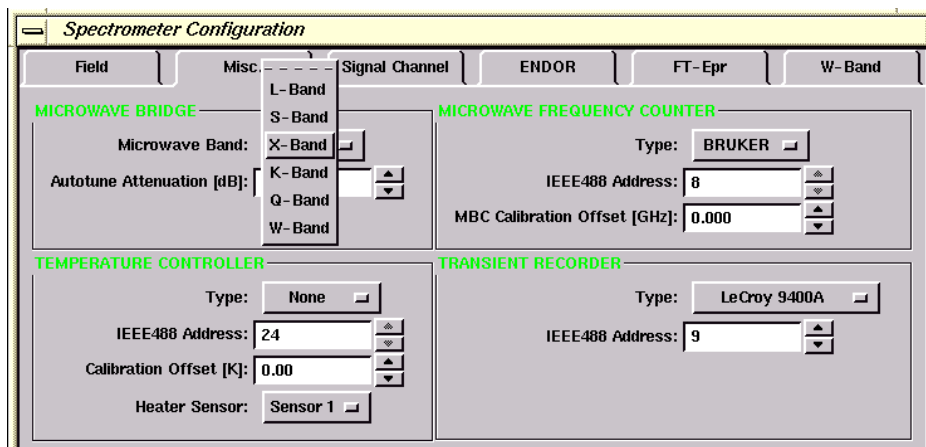


Figure 4-2 The Microwave Band section of the Misc. folder.

Microwave Band

4.2.1

Microwave Band: Depending on the microwave bridge you are using you can select from L-, S-, X-, K-, Q- or W-band.

Autotune Attenuation: This value sets the microwave power for the autotuning process. For a standard cavity, set it to 25 dB. For a resonator with a very high Q value you can set it to 30 dB or higher. Setting the Attenuation too low or too high may cause the autotuning process to fail.

Microwave Frequency Counter

4.2.2

See Section 12.1 for details on setting up frequency counters.

Type: If you do not have a frequency counter, select **MBC**. The **MBC** (Microwave Bridge Controller) board provides an approximate (three digits) microwave frequency. You can use an accurate frequency counter to calibrate the microwave frequency and enter the calibration result into the **MBC Calibration Offset**. If you need a more accurate microwave frequency reading, use either the Bruker frequency counter or another frequency counter provided by other vendors. For the Bruker frequency counter, select **Bruker** in the drop-down list. For other frequency counters you can select one that Xepr supports.

IEEE 488 Address: If you are using the frequency counters from vendors other than Bruker, you need to set the IEEE 488 address to 8. For the Bruker and **MBC** options, you do not need to set the IEEE address.

MBC Calibration Offset: This offset is for **MBC** board calibration only. Use a frequency counter to calibrate the difference between the frequency counter and the **MBC** board and enter the result here. See Section 12.1.1 for instructions on how to measure the offset.

Spectrometer Configuration

Field	Misc.	Signal Channel	ENDOR	FT	W-Band
MICROWAVE BRIDGE		MICROWAVE FREQUENCY COUNTER			
Microwave Band:	X-Band	Type:	BRUKER		
Autotune Attenuation [dB]:	25.0	IEEE488 Address:	HP5340A		
		MBC Calibration Offset [GHz]:	HP5342A		
			HP5350A		
			HP5351A		
			HP5352A		
			HP53150A		
TEMPERATURE CONTROLLER		TRANSIENT RECORDER			
Type:	None	Type:			
IEEE488 Address:	24	IEEE488 Address:	EIP545A		
Calibration Offset [K]:	0.00		EIP548A		
Heater Sensor:	Sensor 1		EIP625A		
			EIP628A		

Figure 4-3 The Microwave Frequency Counter section of the Misc. folder.

Temperature Controller

4.2.3

Refer to Section 12.2 and Section 12.3 for more details on setting up and using temperature controllers.

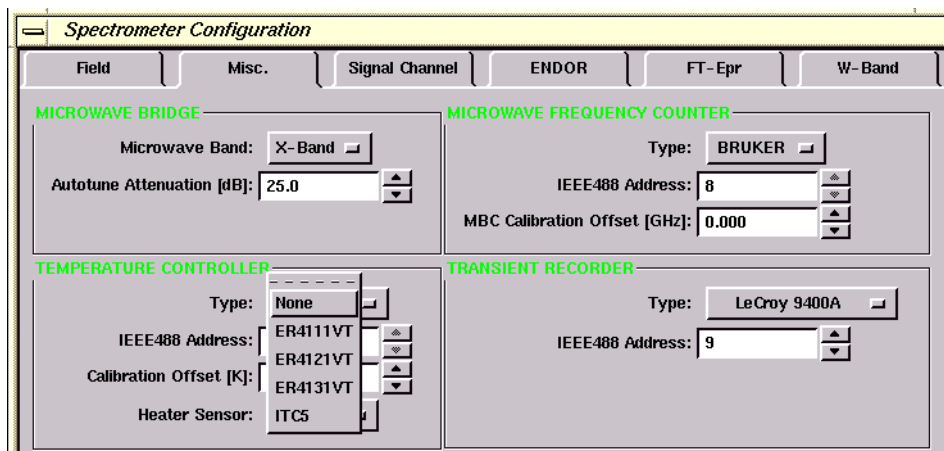


Figure 4-4 The Temperature Controller section of the Misc. folder.

Type: The Bruker Elexsys spectrometer supports two types of temperature controllers. ER 4111VT, ER 4121VT, and ER 4131VT variable temperature systems use liquid nitrogen whereas the ITC 5 system uses liquid helium. Select the temperature controller you wish to use. Refer to Section 12.2 for further details.

IEEE 488 Address: The acquisition server communicates with the ITC5 temperature controller through the IEEE bus. The default address for the ITC5 is 24. Make sure to set the IEEE address on the ITC5 temperature controller to 24 as well. The other controllers do not use the IEEE bus and the value does not affect their operation.

Calibration Offset: Because the sample and thermocouple are not in the same position, the temperature measured by the thermocouple may differ from the actual sample temperature. You can use a temperature

sensor to calibrate the actual sample temperature and enter the difference here. Refer to Section 12.2 Section 12.3 for details.

Heater Sensor: For the ITC temperature controller, there are several sensor inputs that can be used to control the heater. Refer to the temperature controller's manual to find out which sensor to use and select the correct sensor. The value does not affect the operation of the other temperature controllers.

Transient Recorder

4.2.4

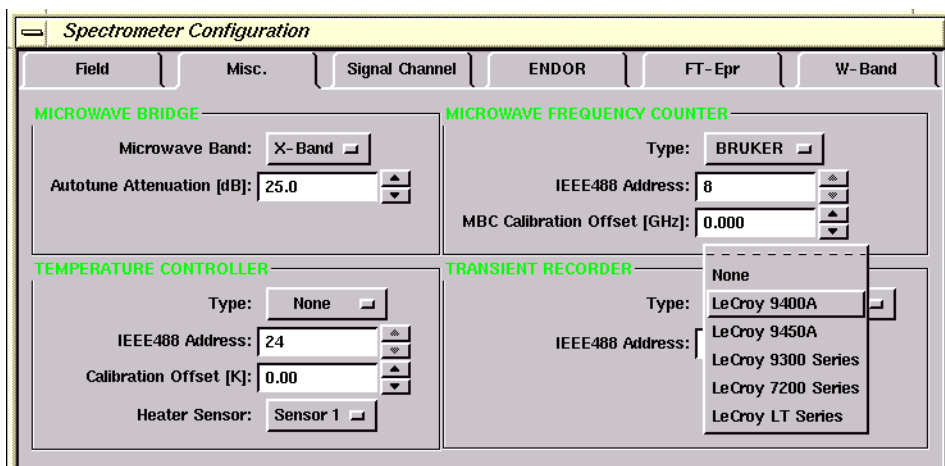


Figure 4-5 The Transient Recorder section of the Misc. folder.

Type: Xepx supports several models of optional transient recorders. If you are using a transient recorder that is listed in the drop-down list, select it.

IEEE 488 Address: The acquisition server communicates with the transient recorder through the IEEE bus. The default address is 9. Make sure to set the IEEE address on the transient recorder to 9 as well.

The Signal Channel Folder

4.3

Refer to Chapter 2 for details regarding signal channel calibration.

The screenshot shows the 'Spectrometer Configuration' dialog box with the 'Signal Channel' tab selected. The 'CALIBRATION OPTIONS' section includes a 'Resonator' dropdown set to '1', and checkboxes for 'AFC Trap Filter' and 'High Pass Filter', both of which are checked. Below this, there are two tabs: 'Standard Calibration Data' (selected) and 'RF Calibration Data'. The 'STANDARD CALIBRATION DATA' section features a 'Calibration Data Set' dropdown set to 'hs2002' and a 'Delete Data Set' checkbox. A table displays calibration data for various frequencies, with columns for Freq. [kHz], Tuning C, ModAmp [%], ModAmp [G], Phase #1, and Phase #2.

Freq. [kHz]	Tuning C	ModAmp [%]	ModAmp [G]	Phase #1	Phase #2
100.00	37	44.00	31.92	327.81	285.16
90.00	47	44.00	37.01	5.47	345.31
80.00	69	44.00	33.68	66.09	352.97
70.00	81	44.00	47.18	245.47	222.66
60.00	111	44.00	53.25	327.19	330.62
50.00	165	44.00	59.12	351.25	17.03

At the bottom of the dialog box, there are buttons for 'Close', 'Apply', 'Reset', 'Load', 'Save', and 'Help'.

Figure 4-6 The Standard Calibration Data section of the Signal Channel folder.

Calibration Options 4.3.1

Resonator The ER 4105DR resonator has two sets of modulation coils. Selecting 1 or 2 displays the calibration values for the Resonator1 or 2 channel of the modulation amplifier module.

Standard Calibration Data 4.3.2

Calibration Data Set: Every resonator needs signal channel calibration data for reproducible and quantitative results. The calibration data is stored in a file on the disk. Select the correct file for the resonator you are using. Each file contains information such as the modulation amplitude and phase calibration as well as modulation amplitude limits and tuning capacitor values.

RF Calibration Data. 4.3.3

This subfolder contains signal channel calibration data for the RF used in ENDOR experiments. Refer to the ENDOR documentation for further details.

Other Folders 4.4

There are other folders in the Spectrometer Configuration dialog box. They are for ENDOR, FT-EPR, and W-Band. Refer to their respective user's manuals for further information.

Because the sample and Hall probe are not physically in the same location in the magnet, the Hall probe measures a magnetic field different from that experienced by the sample. This field difference is often called the field offset. Carefully measuring the magnetic field offset is critical for precisely measuring g-values and other EPR parameters. There are several methods to calibrate the magnetic field. The most direct way of calibrating the magnetic field is to measure the magnetic field at the sample using a teslameter (or gaussmeter) with an intra-cavity probe which you can place in the sample position and then compare that value with the field value that the Hall probe detects. Alternatively, if you do not have a teslameter available you can use a frequency counter and a sample with known g-value. This method is described in this chapter. You can also use this method to measure the g-value of your sample.

Field Calibration Standards

5.1

A standard sample with an accurately measured g-value, which is used to mark the field value, is called a field calibration standard. It is also called a g-marker. The criteria for selecting such a standard sample are:

1. The g-factor has been accurately measured.
2. The EPR signal is isotropic and symmetric with good reproducibility.
3. The EPR signal has a relatively narrow line width. The narrower the line width, the more accurately the magnetic field or g-value can be determined.
4. The EPR signal is strong enough to avoid noise induced errors.

5. The sample is relatively stable.

Examples of such standard samples are potassium peroxyamine disulfonate ($K_2(SO_3)NO$) (known as Fremy's salt or PADS) in a 10% Na_2CO_3 degassed aqueous solution and a dilute perylene solution in degassed concentrated H_2SO_4 . In this chapter we use perylene radical cation as the standard sample. The g-factor has been reported to be 2.002569 ± 0.000006 . (B. G. Segal, M. Kaplan, and G. K. Fraenkel, *J. Chem. Phys.*, **43**, 4191 (1965); R. D. Allendoerfer, *J. Chem. Phys.*, **55**, 3615 (1971))

Measuring the Field Offset

5.2

Calibrating the Field Offset

5.2.1

1. **Insert the sample and tune the system.**
2. **Set the Field Offset to zero.** Click Acquisition > Spectrometer Configuration > Field and enter 0 in the Field Offset box. Click Apply and Close.
3. **Create a field sweep experiment.**
4. **Set appropriate parameters for your sample.** Figure 5-1 shows a parameter set that works well for a perylene sample. The microwave power is set at 0.05 mW for the perylene sample to avoid line broadening due to microwave saturation. The number of data points is set at 4096.

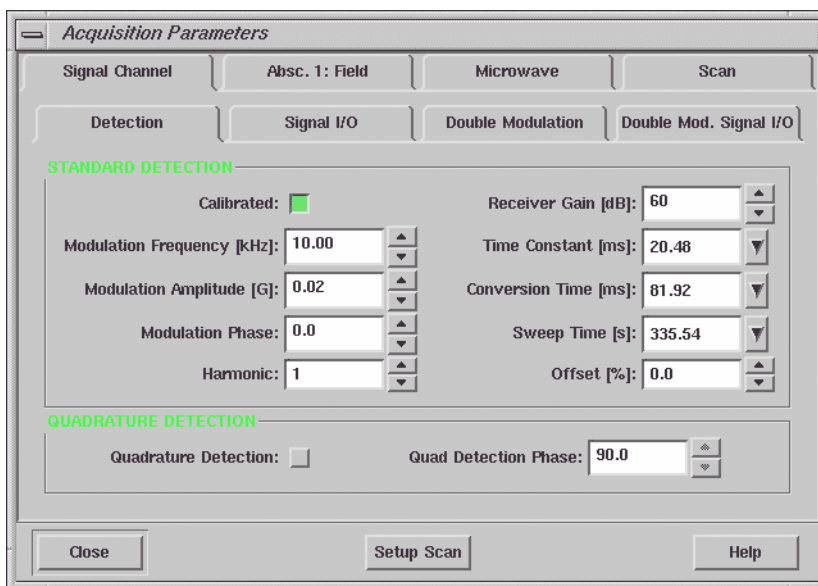


Figure 5-1 Appropriate parameters for a perylene sample.

5. **Acquire a spectrum.** Run the experiment
6. **Center the EPR signal.** Select a center field that centers the EPR signal in the sweep or use the sweep tool.
7. **Adjust the sweep width.** The following rule of thumb usually works well:

$$\frac{\text{sweep width}}{\# \text{ of points}} < 10 \cdot \text{peak-peak linewidth} \quad [5-1]$$

30 gauss works well for the perylene sample.

8. **Measure the magnetic field for the center line.** Zoom in on the center EPR line and move the cursor to the zero cross over point of the center EPR line. Read the field value, B_0 , from the Viewport Selection bar.

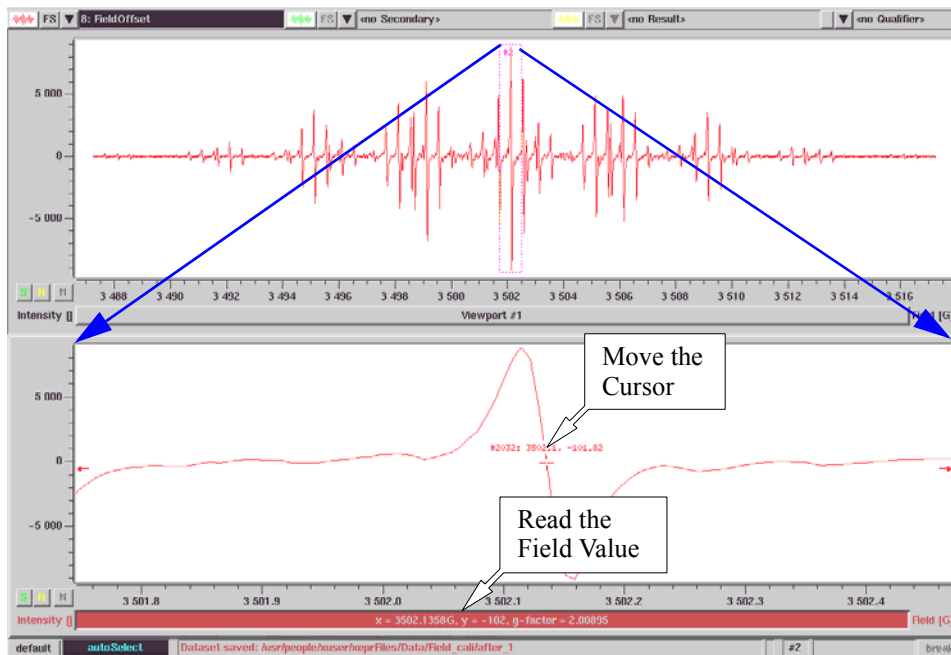


Figure 5-2 Zooming in to measure the center EPR line field value.

9. **Determine the field value for the known g factor.** Move the cursor until the g-factor reading from the Viewport Selection bar matches the known sample g-factor. Read the magnetic field value, B_0' , of that point.

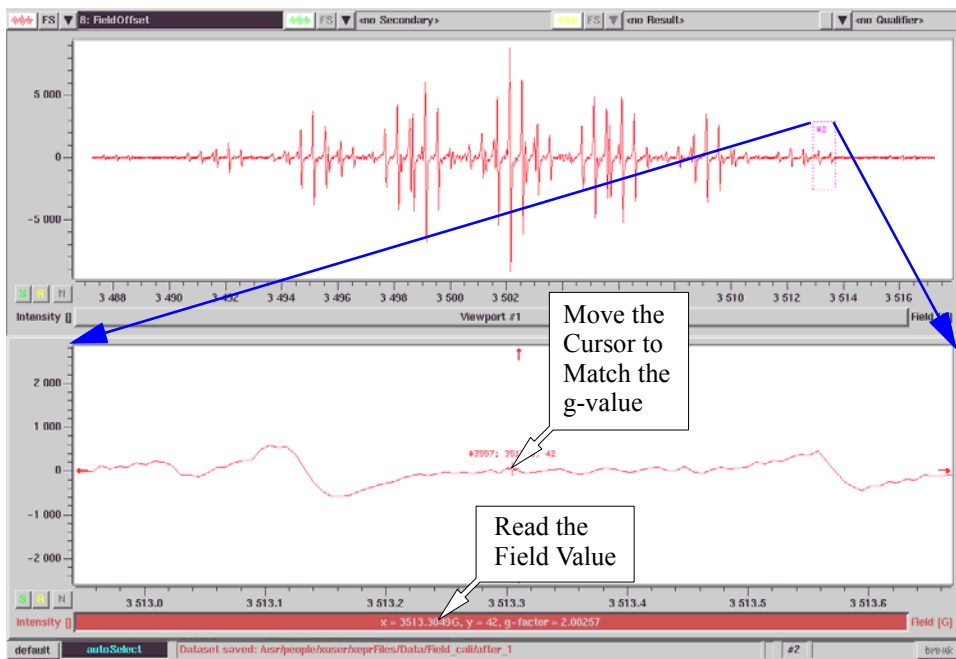


Figure 5-3 Determining the magnetic field value for the known g-value.

10. **Calculate the Field Offset.** The value equals:

$$\text{Field Offset} = B_0 - B_0' \quad [5-2]$$

11. **Set the Field Offset value.** Click Acquisition > Spectrometer Configuration > Field. Enter the value calculated above in the Calibration Offset box. Click Apply and then Close.

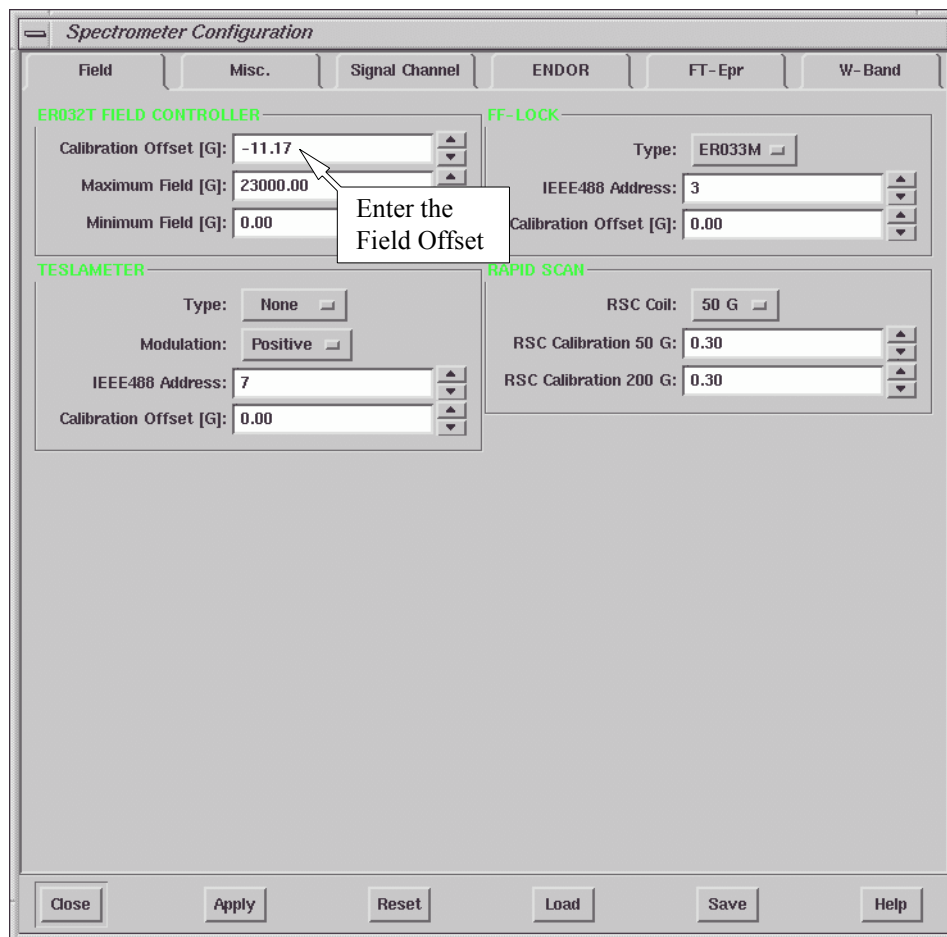


Figure 5-4 Entering the Calibration Offset value.

12. **Re-acquire the spectrum to check the field offset.** Zoom in on the center line and read the g-factor at the zero cross over point. If it matches the given g-factor, the Field Offset has been calibrated successfully. If it does not match the given g-factor, repeat Step 8. through Step 11. Add the result to the Field Offset in the Spectrometer Configuration dialog box. Click Apply and Close. You may need to repeat this procedure several times to get the Field Offset correct.

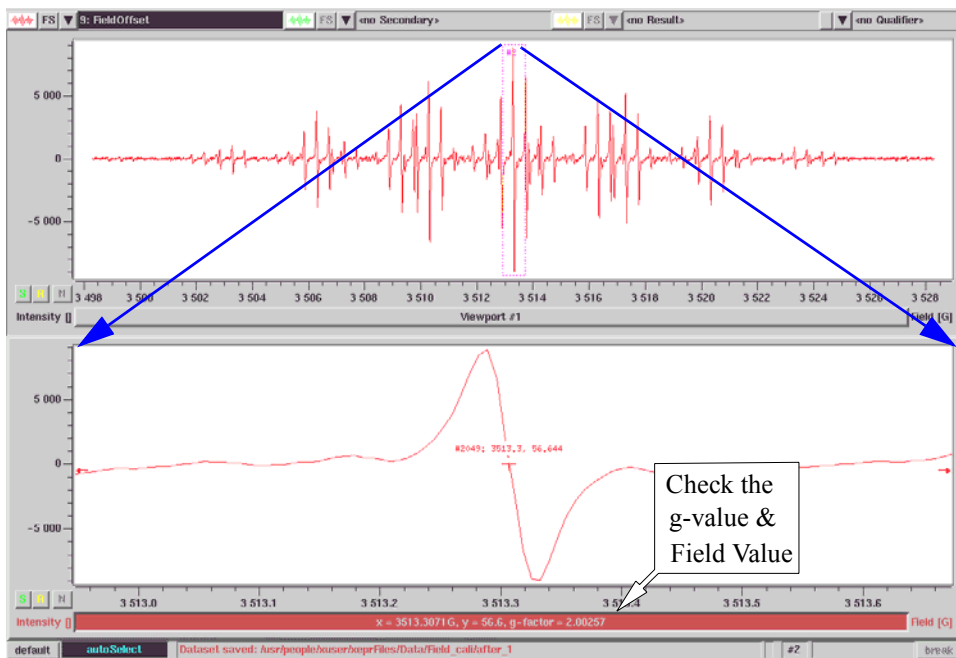


Figure 5-5 Verifying the field offset calibration.

Tips for Measuring the Field Offset

5.2.2



Saturating the magnet only applies to 8" or larger magnet with a 7 kW or greater power supply. For precise measurements of g-values and calibration of the field offset, an 8" or larger magnet and corresponding power supply is recommended.

- **Thermal equilibrium.** The Hall voltage used for field regulation has a temperature coefficient. This effect is compensated by use of a thermostat to control the Hall probe temperature. Very good performance is achieved after about one hour after turning the spectrometer on. Optimal stability is attained after 72 hours. Wait until the spectrometer is warmed up
- **Magnet hysteresis.** Magnet hysteresis affects the accuracy of the field values. You may need to saturate the magnets by setting the magnetic field to $\sim 12,000$ gauss for about one minute before you start measurements. Saturating the magnet first will reduce the hysteresis.
- **Select the proper number of data points.** You may need to select 4096 data points to read the field value precisely. Depending on the sweep width and the line width, more data points usually give you more accuracy. When you are close to the correct field you may need to use narrower sweep width. If the signal is strong you may acquire the second harmonic spectrum to precisely locate the field of the center EPR line.
- **Scan slowly.** If you sweep the field too fast the magnet may not be able to follow. Set the sweep time long enough to avoid any distortion.
- **Avoid saturating the sample.** Too much microwave power not only saturates the sample and causes distortion and broadening of the EPR signal, but also may heat up the sample. The g-value of the sample may be temperature dependent, resulting in an inaccurate calibration. Frequently the EPR absorption signal becomes more dispersive under power saturation, which makes determination of the zero cross over point more difficult and less accurate.

- **Do not over-modulate the signal.** Over-modulation causes signal distortion which can also affect the accuracy of the field offset measurement.
- **How often do I need to calibrate the field offset?** It depends on how precisely you need to know the actual field values. You may need to calibrate the field offset every time you change the cavity or move the bridge if you need to measure your g-values more precisely than 4 figures after the decimal point.

g-Value Measurements

5.3

To precisely determine the g-factor you need to know two values: the magnetic field and the microwave frequency. Then you can calculate the g-value based on the formula:

$$g = \frac{h\nu}{\mu_B B_0}, \quad [5-3]$$

where h is Planck's constant, μ_B is the Bohr magneton, ν is the microwave frequency in GHz and B_0 is the magnetic field in gauss. Alternatively:

$$g = 714.4775 \cdot \frac{\nu}{B_0}. \quad [5-4]$$



A microwave frequency counter is essential to measure the g-value to a precision any greater than two digits after the decimal point.

The precision of the g-factor measurement depends on how precisely you can determine the microwave frequency and the magnetic field. If you are using a microwave frequency counter, the magnetic field uncertainty determines the precision.

Precise g-value measurement requires some care. The tips listed in Section 5.2.2 also hold true for g-value measurements. Con-

tributions to field measurement uncertainties are not necessarily due to instrumental factors. Such factors as line shape, line width, solvent effects, isotropic or anisotropic interactions, sensitivity to concentration, *etc.* must be considered.

If you are using the Hall field controller for field measurement, you can measure g-values to 100 ppm if you have carefully determined the field offset, followed the hints (as described in the previous section), and you have a sufficiently narrow EPR line (< 0.1 G). For more precise g-value determinations, you must use a teslameter. If you have followed the advice of the previous section, have carefully calibrated the teslameter offset, and have a sufficiently narrower line (< 0.1 G) you can measure g-values sometimes down to 100 ppb precision. Consult Section 12.5 for details on teslameter operation.

Xepr can automatically provide the g-factor based on the frequency and field value it has measured. You only need to place the mouse pointer to where you want to know the g-factor and the g-value will be displayed in the selection bar of the viewport. (See Figure 5-2.) Xepr even has a function that can convert the field domain to the g-domain (Processing > Transformation > g-factor).

These methods, can also be used to interpret hyperfine coupling constants and zero field splittings.

The Fast Digitizer & Rapid Scans 6

Many physical and chemical processes occur on time-scales too short for the magnet and the digitizer in the signal channel to follow them. In order to expand the capabilities of the spectrometer to shorter time-scales, the spectrometer is equipped with a fast digitizer and a set of rapid scan coils.

Rapid Time Scan Experiments

6.1

This section describes the use of the fast digitizer for acquiring fast time scans at a constant magnetic field value.

The Fast Digitizer

6.1.1

The ADF board is a fast, two channel digitizer board capable of digitizing at a 2 MHz rate. The first channel (Channel A) is reserved for digitizing EPR signals. The second channel may be used to digitize other signals, such as the output of a photomultiplier tube. The fast digitizer can capture data more quickly than the integrating ADC (Analog to Digital Converter) of the signal channel. It has a fixed 12 bit resolution and therefore yields better y-resolution than the signal channel digitizer when the conversion time is less than 2.56 ms.

The digitizer acquires data at a fixed 2 Msamples/s rate. In order to change the sweep time for an experiment, the number of data accumulations for a specific field or time value is increased or decreased. The fast digitizer contains an on-board accumulator which accumulates several data values (y-value) at each time or field step (x-value). The number of accumulations is set by the software to make maximum use of the selected sweep time.

Performing a Rapid Time Scan Experiment

6.1.2

Before you start, you should have already turned the spectrometer on, inserted your sample, and tuned the spectrometer. Consult the Elexsys E 500 User's Manual: Basic Operations for further details regarding ordinary time scan experiments because both types of experiments share many common operations and parameters.

1. **Create a time scan experiment using the fast digitizer.** Click New Experiment and select the CW sub-folder. In the Abscissa 1 drop-down list, select Time. Select Fast digitizer in the Ordinate drop-down list. Click Create to close the dialog box. (See Figure 6-1.)

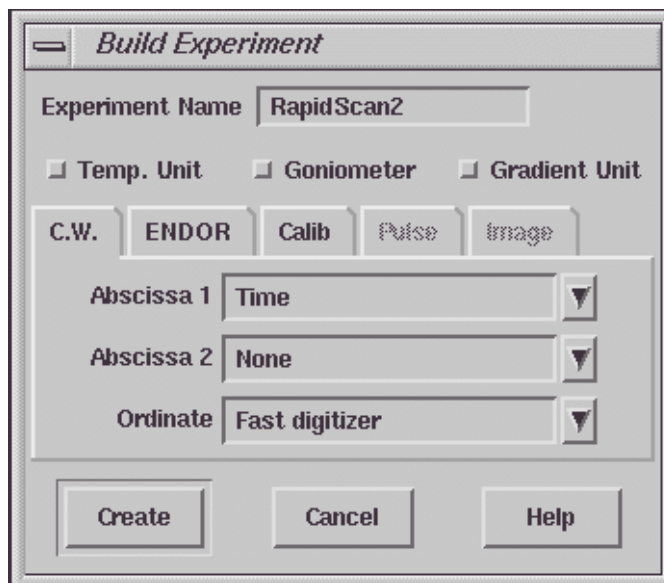


Figure 6-1 Creating a time scan experiment.

2. **Click the Acquisition Parameters button to open the Acquisition Parameters dialog box.**

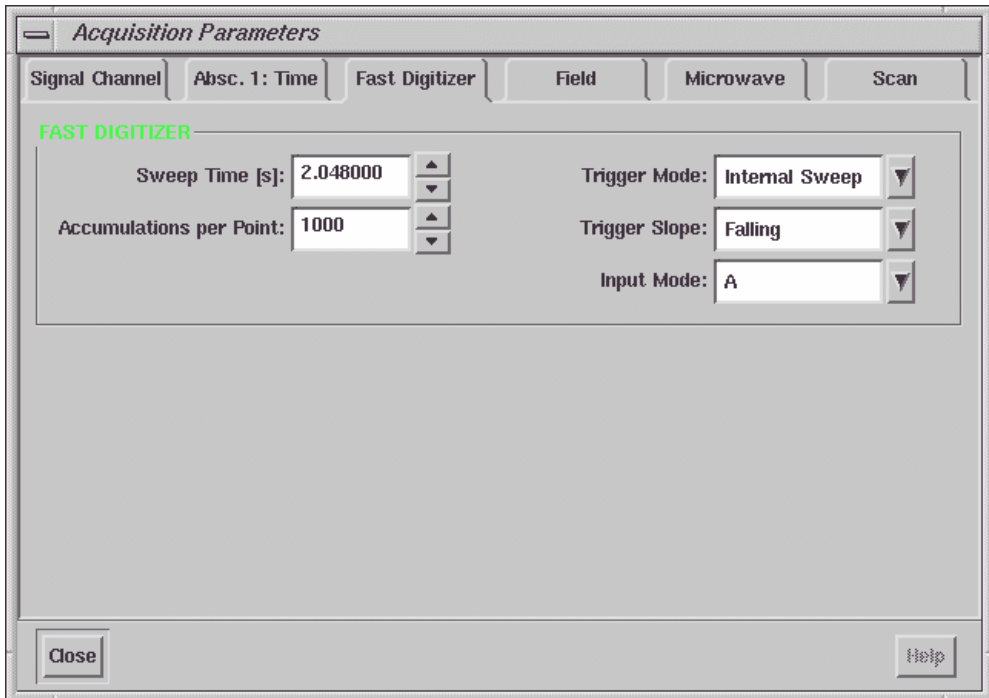


Figure 6-2 The Fast Digitizer subfolder.

3. **Set common EPR parameters.** Set the microwave power, modulation amplitude, and receiver gain to appropriate values.
4. **Set the Static Field.** Set the Static Field in the Field folder so that it coincides with the top of your EPR signal.

5. **Set Auto Offset to Off in the Scan folder.** In a time scan experiment, you are usually interested in the initial and final intensities and this information is lost if Auto Offset is set to On.
6. **Set the Sweep Time.** Change either the Sweep Time or Accumulations per Point in the Fast Digitizer folder. When you change one parameter, the other automatically updates to the proper value.
7. **Set an appropriate time constant.** Because this class of experiments is measuring rapidly changing signals, you must insure your time constant is low enough that it does not distort the acquired signal. A good rule of thumb if you have a strong signal is:

$$\text{Time Constant} = \text{Number of Accumulations} * 500 \text{ ns} / 4. \quad [6-1]$$

If your signal is weak, you may need to compromise and increase the time constant.

8. **Run the experiment.** Click the Run button to acquire the time scan.

Fast Digitizer Parameters

6.1.3

Refer to Figure 6-2 to identify the various Fast Digitizer parameters.

Sweep Time As was mentioned previously, a number of parameters determine the Sweep Time. In Internal Sweep mode:

$$\text{Sweep Time} = \text{Accumulations per Point} \quad [6-2] \\ * \text{Number of points} * 500 \text{ ns.}$$

The maximum Sweep Time is 134 seconds (with the default options) corresponding to 65535 maximum Accumulations per Point.

Accumulations per Point This parameter determines the time resolution and consequently the Sweep Time in Internal Sweep mode.

$$\text{Time Resolution} = \text{Accumulations per Point} * 500 \text{ ns.} \quad [6-3]$$

The minimum Accumulations per Point is 1 and the maximum value is 65535.

Trigger Mode There are four different Trigger Modes. Internal Sweep is the default mode in which the internal clock triggers the beginning of the sweep. Equation [6-2] relates the Accumulations per Point and Sweep Time. In this mode, if one parameter is changed, the other is automatically updated to fulfill Equation [6-2].

The second mode is **Internal Point** mode in which each point is triggered by the internal clock. In this mode, the **Accumulations per Point** becomes more independent than in the first mode. The **Sweep Time** is specified (< 134 seconds) and the **Accumulations per Point** can be set to values:

$$1 \leq \text{Accumulations per Point} \leq \frac{\text{Sweep Time}}{\text{Number of Points}} * 500 \text{ ns.} \quad [6-4]$$

There are two analogous modes, **External Sweep** and **External Point** in which the experiments are synchronized to an external trigger as opposed to the internal clock. The external signal is applied to the cable labeled **Ext. Trigger** that is connected to the **AUX** connector of the **ADF** board. (See Section 14.2.3.)

Trigger Slope If you have selected one of the external trigger modes, you can select whether the **Falling** or **Rising** edge of the external trigger pulse triggers the acquisition.

Input Mode There are two **Fast Digitizer** input channels. Channel **A** is reserved for EPR signals, but channel **B** is available for other signals such as the output of a photomultiplier tube, *etc.* You have the option of choosing to record channel **A**, **B**, or both **A & B** simultaneously in this drop-down list.

The Rapid Field Scan Experiment

6.2

To sweep the magnetic field rapidly we need to employ a special device called the Rapid Scan Controller (RSC). It has a programmable ramp generator which is used to set the shape of the field sweep. It generates very fast field sweep cycles to match the speed of the ADF board. The center field of the rapid field scans is centered about the static field supplied by the main magnet.

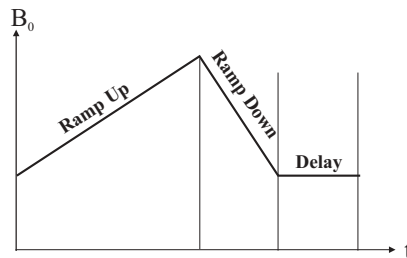


Figure 6-3 The rapid field sweep cycle.

The field sweep cycle is applied to the rapid scan coils. The cycle consists of three distinct regimes. (See Figure 6-3.) The acquisition occurs during the Ramp-up period. The Ramp-down and Delay times are necessary to return to the initial magnetic field value.

Since the inductance of the rapid scan coils is much lower than that of the electromagnet, the field can be swept much faster. There are two rapid scan coils. The internal rapid scan coils are built in some of the Bruker cavities. The external rapid scan coils are mounted on the magnet pole caps. The maximum rapid scan sweep widths are 50 G for internal rapid scan coils and 200 G for external rapid scan coils respectively. You can select the rapid scan coils and adjust the calibration factors of the sweep width in the configuration menu.

Configuring the Rapid Scan Experiment

6.2.1



Not all Bruker cavities are equipped with 50 G rapid scan coils. If the cavity you are using does not have an R.S. connector, you can only use 200 G rapid scan coils.

The ADF board should be already connected when the spectrometer is delivered. You may need to connect the rapid scan coil and configure the Xep program.

1. **Connect the Rapid Scan Coils.** As mentioned before, there are two rapid scan coils; the 200 G rapid scan coils that are mounted to the pole caps of the magnet and the 50 G rapid scan coils that are built in some Bruker cavities such as the standard cavity ER 4102ST. Connect the orange cable from the Modulation Amplifier RS 200 G connector in the console to the connector of the 200 G rapid scan coils on the magnet. Connect the yellow cable from the Modulation Amplifier RS 50 G connector to the left connector (labeled R.S.) of the Bruker cavity.
2. **Configure the spectrometer.** Turn on the spectrometer and connect the workstation to the spectrometer. Select Acquisition > Spectrometer Configurations. Clicking the Field tab, you can find the Rapid Scan section. In this section, select 50 G or 200 G as the rapid scan coils (RSC) depending upon which rapid scan coil you want to use. Click Apply. Underneath, there are calibration factors of the rapid scan width for 50 G and 200 G coils respectively. We will discuss this calibration in Section 6.4.

Defining a Rapid Scan Experiment

6.2.2

After you have configured the rapid scan hardware, you need to define a rapid scan experiments. It is a little different from the regular experiments with the signal channel.

1. **Create a New Experiment.** Click the New Experiment button. Click the CW tab in the dialog box if it is not in front.
2. **Select Field (Rapid Scan).** In the Abscissa 1 drop-down list, select Field (Rapid Scan). (See Figure 6-4.)

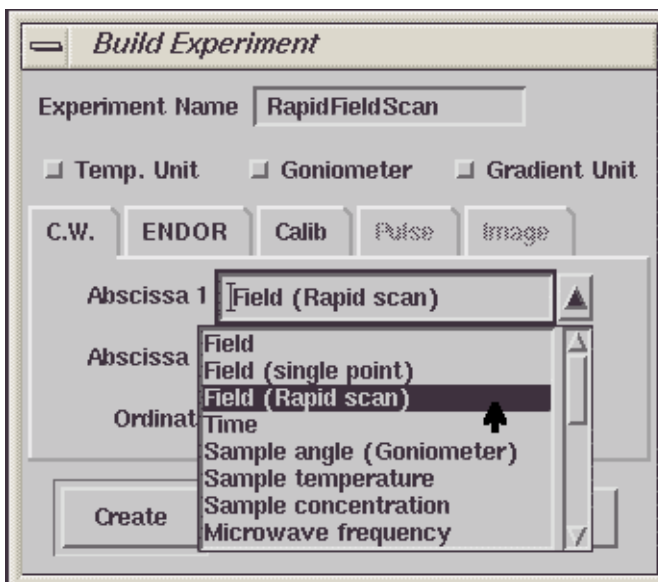


Figure 6-4 Selecting Field (Rapid scan) for Abscissa 1.

3. **Select Fast Digitizer.** In the drop-down list of the Ordinate select Fast digitizer instead of Signal channel. Click Create to close the dialog box. (See Figure 6-5.)

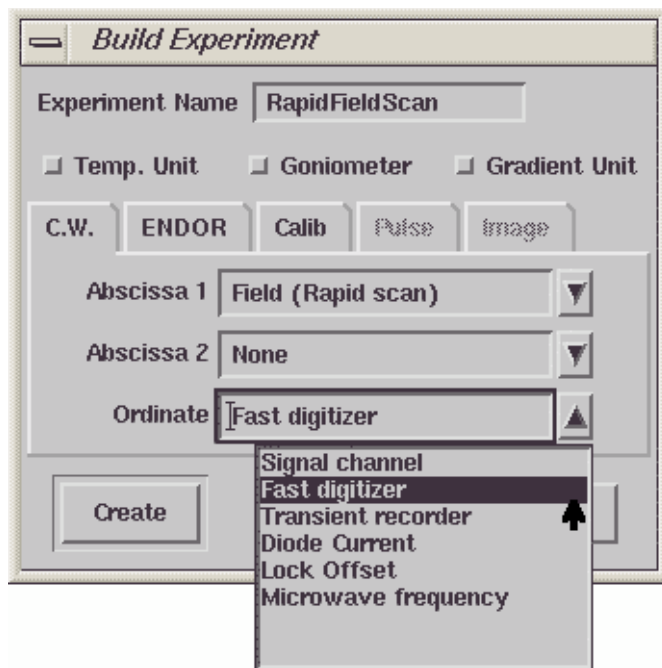


Figure 6-5 Selecting Fast digitizer as Ordinate.

4. **Set common EPR parameters.** Click the Acquisition Parameters button to open the dialog box. Set the microwave power, modulation amplitude, and receiver gain to appropriate values. The Time Constant in the Signal Channel subfolder should be set very short, otherwise distortion may occur.

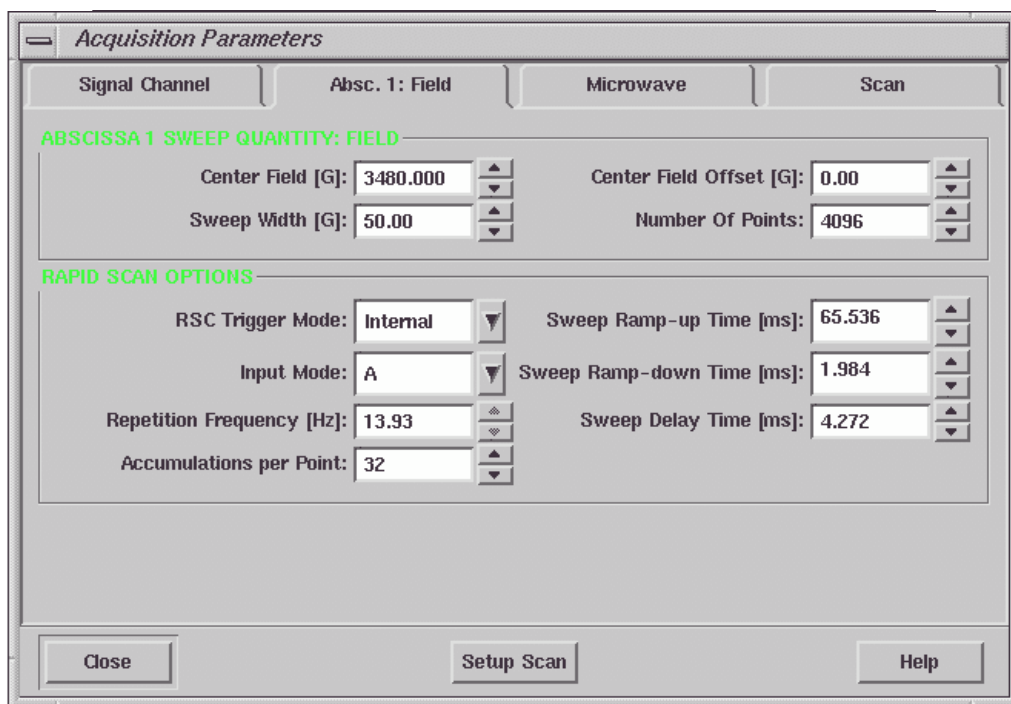


Figure 6-6 Parameters for rapid field scans.

5. **Set Sweep Quantity: Field parameters.** In the Abscissa 1 Sweep Quantity: Field section enter the Center Field value for your experiment. Enter the desired Sweep width. The maximum sweep width depends on the rapid scan coils you are using and selected in the System Configuration folder. By default the Number of Points is set at 4096 which gives you a reasonable resolution in most cases. We shall keep the Center Field Offset value at zero for now. (See Section 6.4 for details on the Center Field Offset parameter.)



The Sweep Ramp-up Time, Number of Points, and the Accumulations per Point are related. More data points usually provide better resolution but longer sweep time or less Accumulations per Point. You need to balance these three parameters to achieve the acceptable distortion, resolution, and acquisition time.

6. **Set the RSC Trigger and Input Mode parameters.** Select Internal as the RSC Trigger Mode. Select A as Input Mode. Channel A is reserved for EPR signals.
7. **Select an appropriate Sweep Ramp-up Time.** Since the minimum signal channel time constant is 0.01 ms, you need to set the Sweep Ramp-up Time long enough so that the time sweeping through the upper peak and the lower peak of an EPR line is at least 0.1 ms (or 10 times the Time Constant) to avoid distortion.
8. **Set the Sweep Ramp-down Time.** The Sweep Ramp-down Time is the time for the field to fly back. The optimal Sweep Ramp-down time depends on your experiment and other settings. The best way to set an optimal Sweep Ramp-down Time is lengthen it until there are no flyback glitches appearing in your spectrum.
9. **Set the Sweep Delay Time.** If you set the scan number to more than 1 in the Scan subfolder you need to set a proper Sweep Delay Time so that the field can settle after fly-back. The optimal Sweep Delay Time depends on your experiment and other settings. The best way to set an optimal Sweep Delay Time is lengthen it until there are no flyback glitches appearing in your spectrum.
10. **Run the experiment.** Click the Run button to acquire the time scan.

Rapid Scan Parameters

6.2.3

Refer to Figure 6-6 to identify the various Fast Digitizer parameters.

- Center Field** The static field about which the magnetic field is rapidly swept.
- Sweep Width** The width of the rapid scan magnetic field sweep. It has a maximum value of 50 G for the 50 G rapid scan coils and 200 G for the 200 G rapid scan coils.
- Center Field Offset** There is a **Center Field Offset** in order to compensate for the field hysteresis caused by the rapid scan. (See Section 6.4.)
- Number of Points** The number of points acquired during the rapid scan experiment.
- Input Mode** There are two **Fast Digitizer** input channels. Channel **A** is reserved for EPR signals, but channel **B** is available for other signals such as the output of a photomultiplier tube, *etc.* You have the option of choosing to record channel **A**, **B**, or both **A & B** simultaneously in this drop-down list.
- RSC Trigger Mode** The acquisition can be triggered in **Internal** mode in which pressing the **Run** button initiates the acquisition. You may also select to trigger the acquisition in **External** mode with an external trigger. (See Section 14.2.4.)

A sweep cycle consists of three sections, the Sweep Ramp-up, Sweep Ramp-down, and Sweep Delay Times. (See Figure 6-7.)

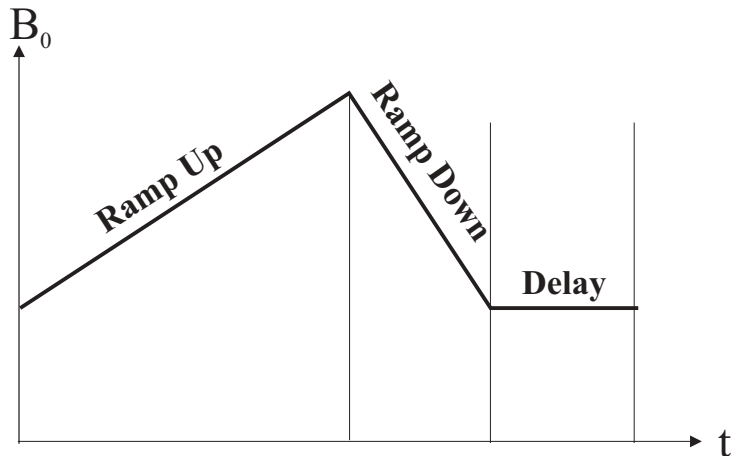


Figure 6-7 The rapid field sweep cycle.

Sweep Ramp-up Time

This is the field sweep during which the actual acquisition takes place. It equals

$$\text{Sweep Ramp-up Time} = \text{Accumulations per Point} [6-5] \\ * \text{Number of points} * 500 \text{ ns.}$$

(Refer to Section 6.1.3.) When you set the Sweep Ramp-up Time and the Number of Points, Xepi will automatically set the Accumulations per Point.

**Sweep
Ramp-down
Time**

This is the “fly-back” time during which the field returns to its starting position. No acquisition is done. It is part of the dead time. If this parameter is set too low, the field is not yet fully back to its starting value when the next acquisition begins and generates glitches caused by the fly-back trace in the next spectrum. The minimum Ramp-down time is 0.1 ms.

**Delay
Time**

After the ramp down time, there is a delay time during which the system can recover from overshoots after the fly-back. This is also part of the dead-time. This is the time delay between successive sweeps when using multiple sweeps. Depending on the sweep width, a sufficiently long delay time is needed to avoid glitches and distortion. The minimum Delay time is 0.1 ms. If the Overload LED on the Field Controller turns red it is an indication that the Sweep Delay Time is too short.

**Repetition
Frequency**

The sweep Repetition Frequency is the reciprocal of the sum of Ramp-up, Ramp-down, and Delay times. It indicates the rate of successive sweeps when multiple scans are acquired.

**Accumulations per
Point**

The value of the Accumulations per Point will be set automatically in accordance with Equation [6-5] when you set the Sweep Ramp-up Time. However, you can also set the Accumulations per Point. If you do so the Sweep Ramp-up Time will be adjusted automatically by the software. It is analogous to Accumulations per Point described in Section 6.1.3.

2D Experiment with Rapid Scan

6.3

You can combine rapid field scans with a time scan into a 2D experiment using the fast digitizer.

1. **Create a 2D experiment.** Click the New Experiment button. Select Field (Rapid scan) as Abscissa 1 and Time as Abscissa 2. Select Fast digitizer as Ordinate. Click the Create button to close the dialog box. (See Figure 6-8.)

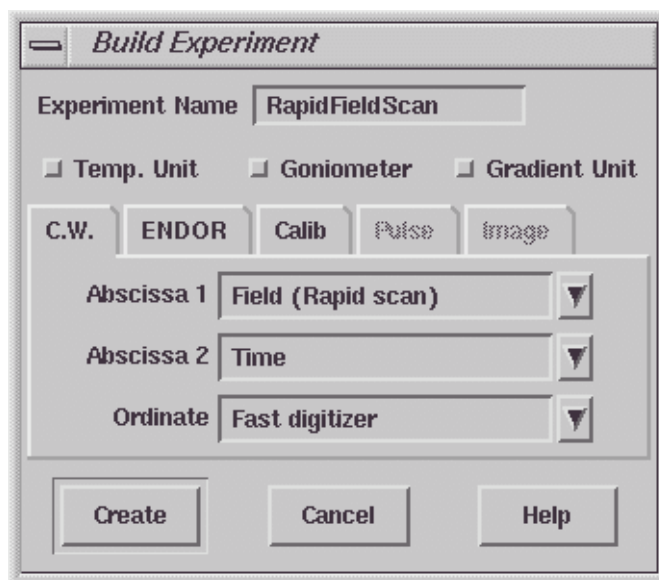


Figure 6-8 Defining a 2D rapid scan experiment.

2. **Select parameters for rapid field scan.** Click the Acquisition Parameters button to open the dialog box. Set the parameters in the Absc.1 Field the same way as for the Rapid Field Scan experiment described in Section 6.2.2.
3. **Set parameters for Absc. 2: Delay.** Click the Absc. 2: Delay tab. Set the Delay time in ms between rapid field scans. Although you can set the Delay time to 0, the minimum delay time is the minimum time necessary for communication and data transfer. The Delay Since Acq. Starts displays the overall time since the acquisition starts. Each slice will be labeled by the time when the acquisition of this slice starts. You need to choose the number of data points or slices on the Y-axis and enter this value into the Number of Points. (See Figure 6-9.)

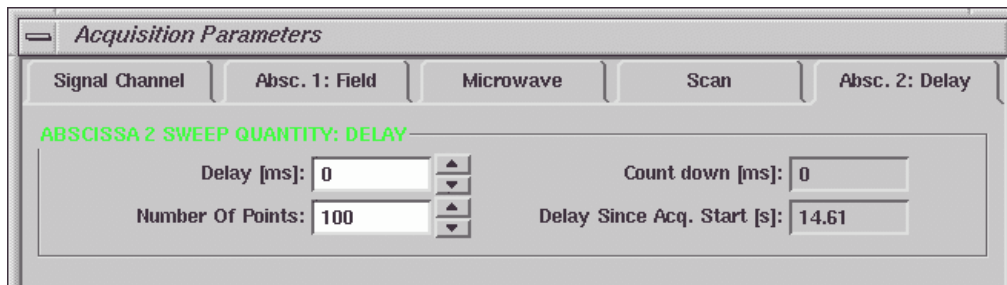


Figure 6-9 Parameters for Absc. 2 Delay.

4. **Run the experiment.** Click the Run button to acquire the 2D experiment.

Field Sweep Calibration

6.4

You need to calibrate the field sweep width for rapid scan. The standard sample for calibration depends on which RSC coil you use. For the 200 G RSC coil you can use a commercially available CaO sample. The Mn^{+2} impurity in CaO is a good standard for the calibration of the 200 G RSC coils. A 1 mM TEMPOL solution can be used for 50 G RSC coil calibration.

1. **Prepare for the calibration.** Insert the appropriate sample in the cavity and tune the system.
2. **Acquire a regular field sweep EPR spectrum.** Create a regular field experiment using the signal channel. Acquire an EPR field sweep spectrum with either 200 G or 50 G sweep width. Center the EPR signal. Measure the field difference between two characteristic EPR lines.
3. **Create a Rapid Scan experiment.** Click New Experiment. Select Field (Rapid scan) in Absc. 1. Select Fast Digitizer in the Ordinate drop-down list. Click OK.
4. **Select parameters.** Click the Activate button and open the parameters dialog box. Select appropriate parameters for the experiment.
5. **Acquire a Rapid Scan spectrum.** Click the Run button.
6. **Adjust the RSC Calibration factor.** Acquire the rapid scan spectrum. Carefully adjust the corresponding RSC Calibration factor in the System Configuration dialog box until the distance between the EPR lines matches the distance you measured in the regular field sweep spectrum using the signal channel. Click Apply each time you change the calibration factor. After you are satisfied with the results, click Save to store the calibration factor.

7. **Set the Center Field Offset.** The center field in a rapid field scan experiment may differ from that in the regular field sweep experiment. Measure the line positions of the EPR signals in the rapid scan and conventional EPR experiments. The offset is equal to:

$$\text{Center Field Offset} = B_{\text{rapid scan}} - B_{\text{conventional}} \quad [6-6]$$

Enter the difference in the **Center Field Offset** in the **Absc. 1 Field** folder of the **Acquisition Parameters** dialog box. The **Center Field Offset** may be different when you change the field sweep width of the rapid field sweep.

So far, all the experiments we have discussed are linear acquisitions, *i.e.* the data points are evenly distributed with a constant step-size between the individual datapoints of the dataset. However, a nonlinear acquisition is more convenient in certain occasions. For example, if we need to scan through base line in larger steps and scan through the signal area in smaller steps to achieve better resolution or we need a coarse scan over a temperature range beyond the phase transition but need a very small increment around the phase transition temperature. Other examples are non-equal increments of sample concentrations, microwave power, sample angles, time intervals, *etc.* The Bruker Elexsys spectrometer provides not only nonlinear field sweeps but also nonlinear scans for a variety of other variables in 2D experiments. In this chapter we introduce the nonlinear scan feature of the Bruker Elexsys spectrometer.

Nonlinear Field Sweeps

7.1

This is an example of a nonlinear field sweep experiment. The sample used in this experiment is a ruby crystal. The EPR spectrum of the ruby crystal at certain orientation spans up to 5000 gauss with relatively narrow linewidths. With a linear field sweep we need to increase the number of data points significantly to achieve reasonable resolution and spend most of the acquisition time measuring the baseline where there is no EPR signal. With the nonlinear acquisition capability of the Elexsys we can sweep the magnetic field near the EPR signal with small field increments and with large steps where there is no signal. Figure 7-1 shows the spectrum of a ruby crystal with a linear field sweep.

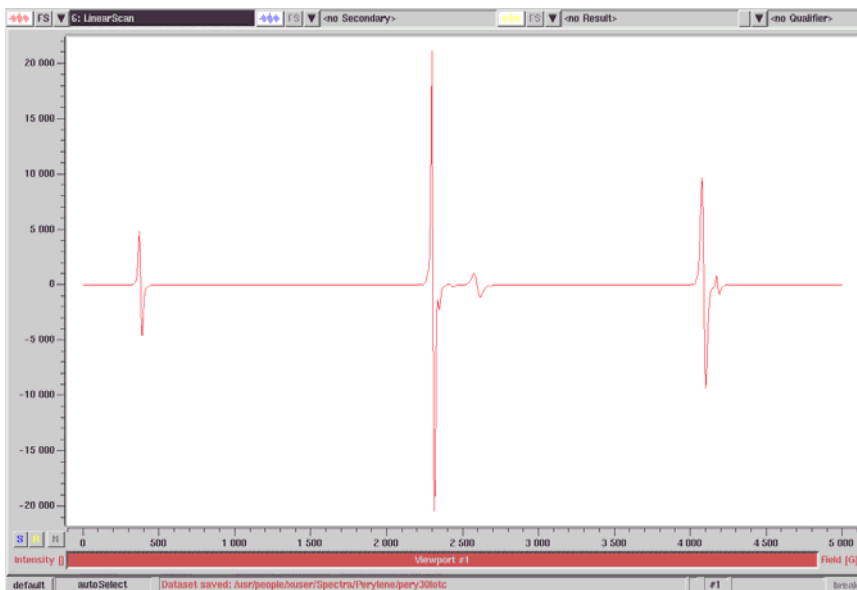


Figure 7-1 A linear field sweep of the ruby crystal.

Performing Nonlinear Field Sweeps

7.1.1

1. **Create a nonlinear field sweep experiment.** Click the New Experiment button. In the CW folder, select Field (single point) as Abscissa 1. Click Create to close the dialog box. (See Figure 7-2.)

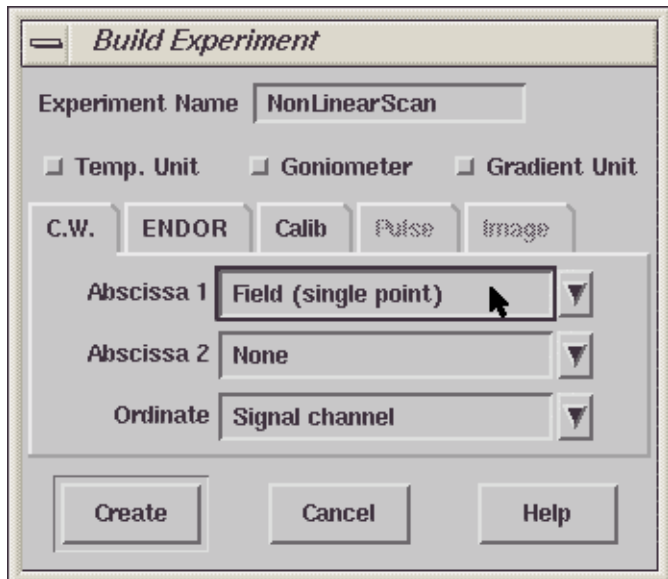


Figure 7-2 Creating a nonlinear field sweep experiment.

2. **Open the Parameter dialog box.** Click the Parameter button. Click the Activate button.
3. **Select a user defined sweep.** Click the tab of the Field folder. Select an appropriate Center Field for your sample. Click the arrow button of Sweep Direction in the Field Sweep Options section. In the drop-down list select User Sweep Up. (See Figure 7-3.)

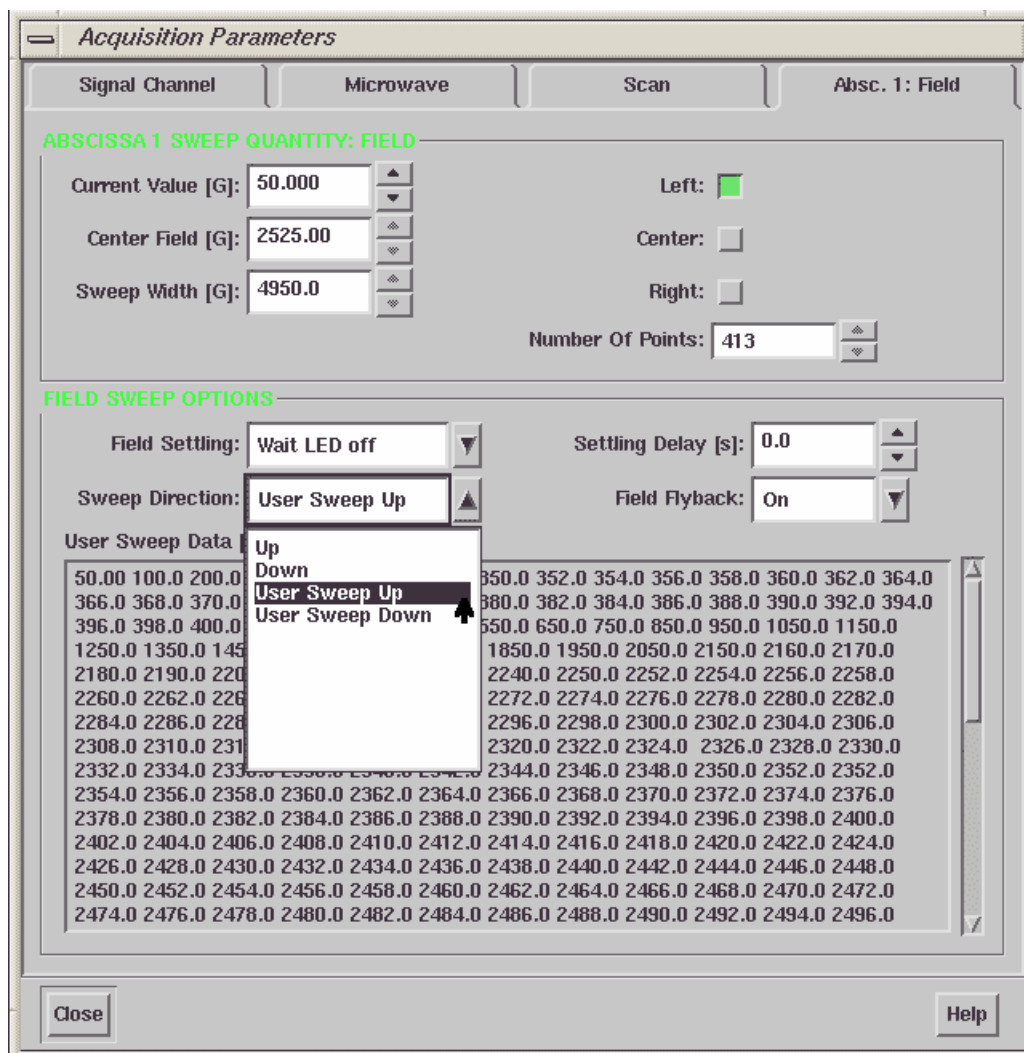


Figure 7-3 Selecting a user defined field sweep.

4. **Set up the field.** Edit the field values you would like to use in the User Sweep Data box. (See Figure 7-4)

Acquisition Parameters

Signal Channel | Microwave | Scan | Absc. 1: Field

ABSCISSA 1 SWEEP QUANTITY: FIELD

Current Value [G]: 50.000 Left:

Center Field [G]: 2525.00 Center:

Sweep Width [G]: 4950.0 Right:

Number Of Points: 413

FIELD SWEEP OPTIONS

Field Settling: Wait LED off Settling Delay [s]: 0.0

Sweep Direction: User Sweep Up Field Flyback: On

User Sweep Data [G]:

50.00	100.0	200.0	300.0	320.0	330.0	340.0	350.0	352.0	354.0	356.0	358.0	360.0	362.0	364.0
366.0	368.0	370.0	372.0	374.0	376.0	378.0	380.0	382.0	384.0	386.0	388.0	390.0	392.0	394.0
396.0	398.0	400.0	410.0	420.0	430.0	450.0	550.0	650.0	750.0	850.0	950.0	1050.0	1150.0	
1250.0	1350.0	1450.0	1550.0	1650.0	1750.0	1850.0	1950.0	2050.0	2150.0	2160.0	2170.0			
2180.0	2190.0	2200.0	2210.0	2220.0	2230.0	2240.0	2250.0	2252.0	2254.0	2256.0	2258.0			
2260.0	2262.0	2264.0	2266.0	2268.0	2270.0	2272.0	2274.0	2276.0	2278.0	2280.0	2282.0			
2284.0	2286.0	2288.0	2290.0	2292.0	2294.0	2296.0	2298.0	2300.0	2302.0	2304.0	2306.0			
2308.0	2310.0	2312.0	2314.0	2316.0	2318.0	2320.0	2322.0	2324.0	2326.0	2328.0	2330.0			
2332.0	2334.0	2336.0	2338.0	2340.0	2342.0	2344.0	2346.0	2348.0	2350.0	2352.0	2352.0			
2354.0	2356.0	2358.0	2360.0	2362.0	2364.0	2366.0	2368.0	2370.0	2372.0	2374.0	2376.0			
2378.0	2380.0	2382.0	2384.0	2386.0	2388.0	2390.0	2392.0	2394.0	2396.0	2398.0	2400.0			
2402.0	2404.0	2406.0	2408.0	2410.0	2412.0	2414.0	2416.0	2418.0	2420.0	2422.0	2424.0			
2426.0	2428.0	2430.0	2432.0	2434.0	2436.0	2438.0	2440.0	2442.0	2444.0	2446.0	2448.0			
2450.0	2452.0	2454.0	2456.0	2458.0	2460.0	2462.0	2464.0	2466.0	2468.0	2470.0	2472.0			
2474.0	2476.0	2478.0	2480.0	2482.0	2484.0	2486.0	2488.0	2490.0	2492.0	2494.0	2496.0			

Close Help

Figure 7-4 Entering the field values for a nonlinear field sweep.



The total number of the field values is 413. The field values you entered are stored in a buffer with limited size. (See the discussion at the end of Section 7.1.2.)

In this example, we use increments as large as 100 G while sweeping through the sections with no EPR signal and 2 G increments while sweeping through the EPR signal area.

5. **Adjust the other parameters.** Select appropriate parameters such as receiver gain, *etc.*, for your sample in the other folders of the **Parameter** window. Click **Close** to exit the **Parameter** dialog box.
6. **Acquire the spectrum.** Click the **Run** button to start the acquisition. The nonlinear field sweep of the ruby crystal is shown in Figure 7-5.

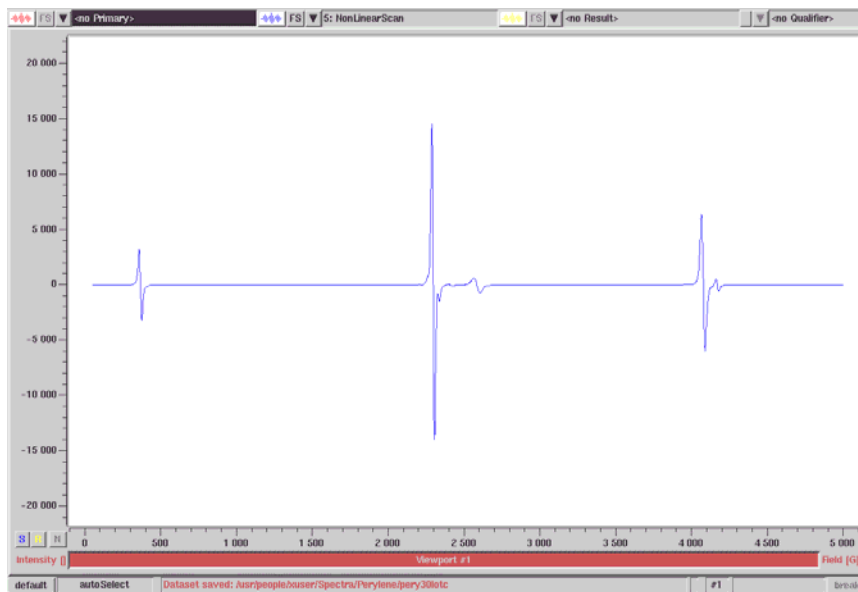


Figure 7-5 A nonlinear field sweep of the ruby crystal.

Comparison of the Results

7.1.2

Figure 7-6 shows the difference between the linear field sweep and the nonlinear field sweep spectra. Curve A is the linear field sweep in which the total number of the data points is 512. Curve B is the nonlinear field sweep in which the total number of data points is 413. Although the overall data points in the nonlinear sweep is less than that in the linear sweep, they are concentrated in the EPR signal areas.

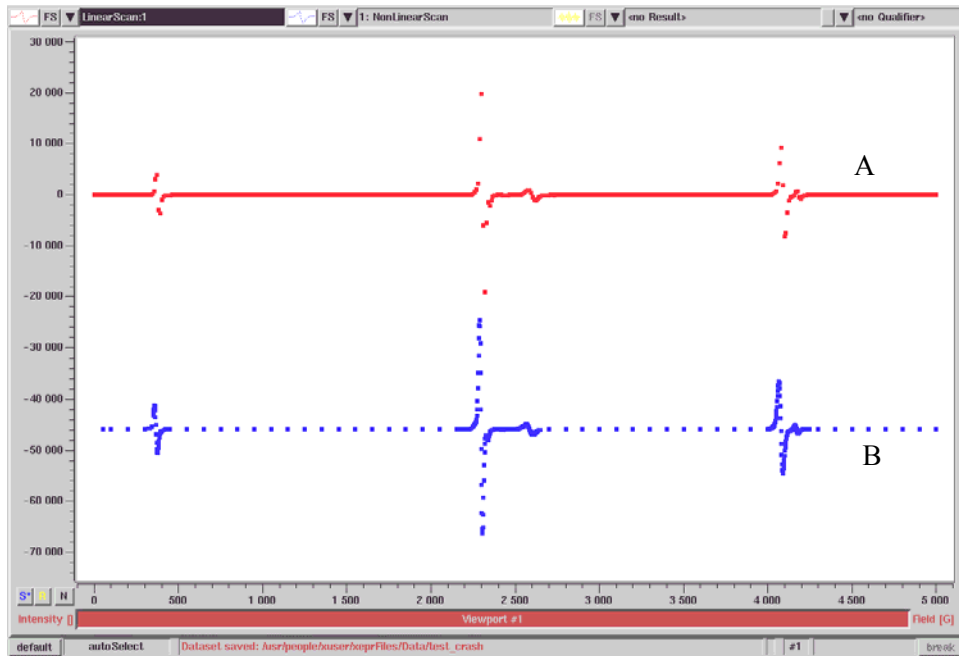


Figure 7-6 A comparison of the linear field sweep (A: 512 points) and the nonlinear field sweep (B: 413 points).

Therefore, the resolution in the signal area is much better in the nonlinear field sweep than that in the regular linear field sweep. (See Figure 7-7.)

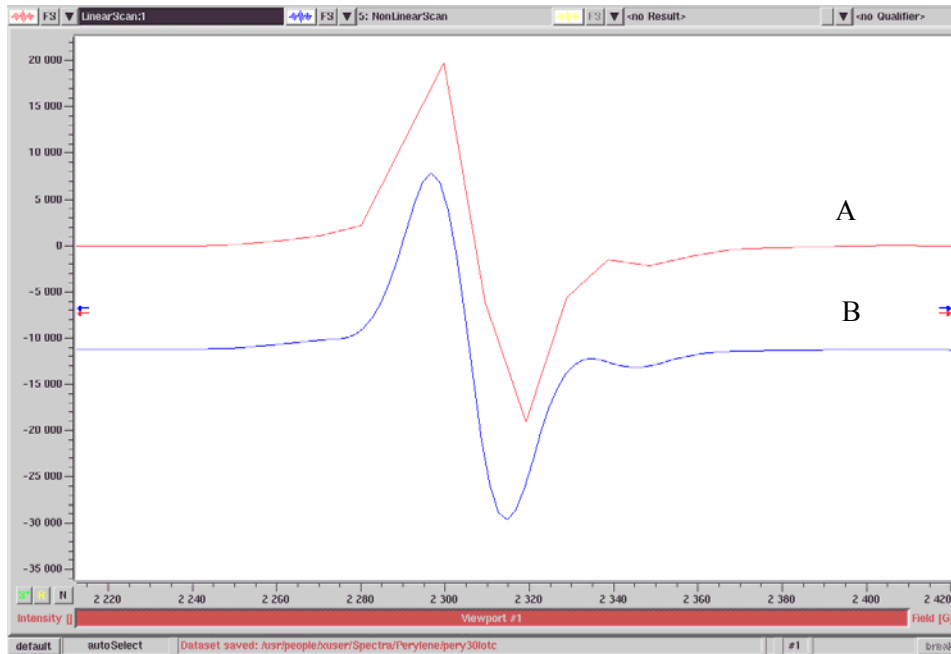


Figure 7-7 An expanded display of the signal region. A: is from the linear field sweep. B: is from the nonlinear sweep.

The field values you entered are sent to a buffer. The size of the buffer is limited. The maximum number is approximately 512. The maximum number of data points may vary depending on how many digits each field value has. After clicking the **Activate** button the actual number of the data points is displayed in the **Number of Points** box. If the number you entered exceeds the size of the buffer it will be automatically truncated to fit the buffer.

Nonlinear Scans in a 2D Experiment

7.2

In a 2D experiment we scan two independent variables. For example we can select the magnetic field as **Abscissa 1** and select the time, temperature, sample concentration, or the microwave power as **Abscissa 2**. We do not have to increment the second variable with a constant step-size. Instead, we can use a nonlinear acquisition to distribute the data points according to our needs. In this example we are going to measure the power saturation behavior of a DDPH sample by creating a 2D field vs. microwave power experiment with a nonlinear increment in the microwave attenuation.

1. **Create a 2D experiment.** Click the New Experiment button. Select Field as **Abscissa 1**. Select Microwave Power as **Abscissa 2**. Click **Create** to close the dialog box. (See Figure 7-8.)

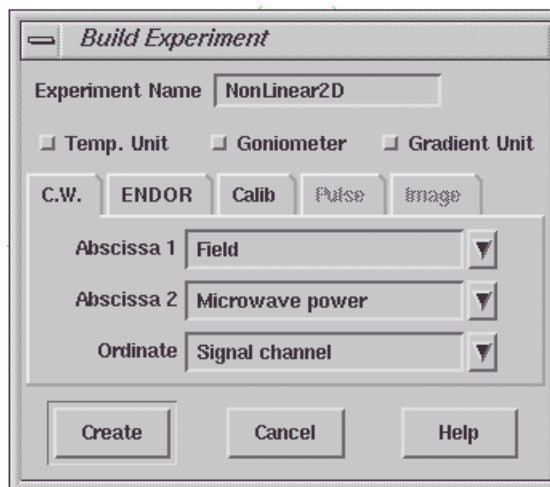


Figure 7-8 Creating a 2D experiment.

2. **Select a user-defined increment.** Click the Parameter button to open the dialog box. Click the Absc. 2 MW Att. folder. Click Sweep Type and select User defined up (or User defined down) in the drop-down list. (See Figure 7-9.)

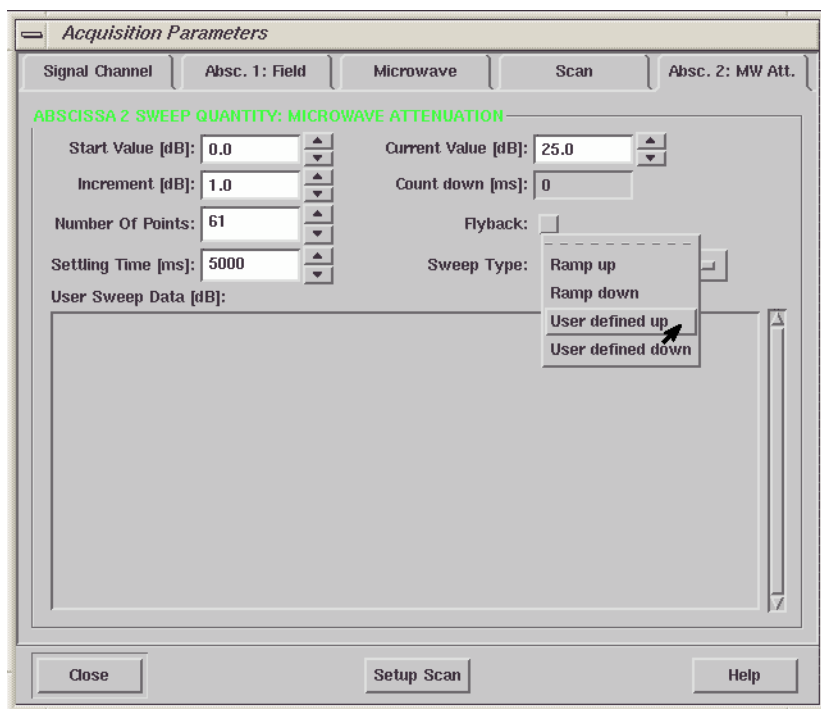


Figure 7-9 Selecting the user-defined scan for microwave attenuation.

3. **Enter the microwave attenuation values.** In the User Sweep Data (dB) box, input the microwave attenuation values manually. By default, the attenuation increment is set at 1 dB and the total number of microwave attenuation settings is 61. This is too fine at low microwave powers since the saturation curve is usually plotted

as peak intensity versus the square root of microwave power (mW). Here we can set the microwave attenuation in a 1 dB increment between 0 dB to 20 dB and 2, 3, or 5 dB increments at the low microwave range. The total number of microwave attenuations is reduced to 33 instead of 61. (See Figure 7-10.)

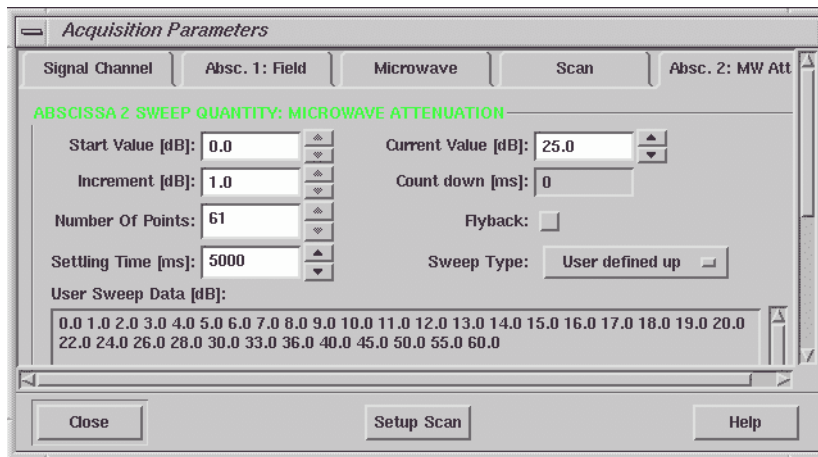


Figure 7-10 Entering the microwave attenuation values in the User Sweep Data box.

4. **Adjust the other parameters.** Select appropriate parameters such as receiver gain, *etc.*, for your sample in the other folders of the Parameter window. Click Close to exit the Parameter dialog box.
5. **Acquire the spectrum.** Click the Run button to acquire the spectrum. Process the 2D dataset. The microwave power saturation curve (peak intensity vs. square root of microwave power) is shown in Figure 7-11.

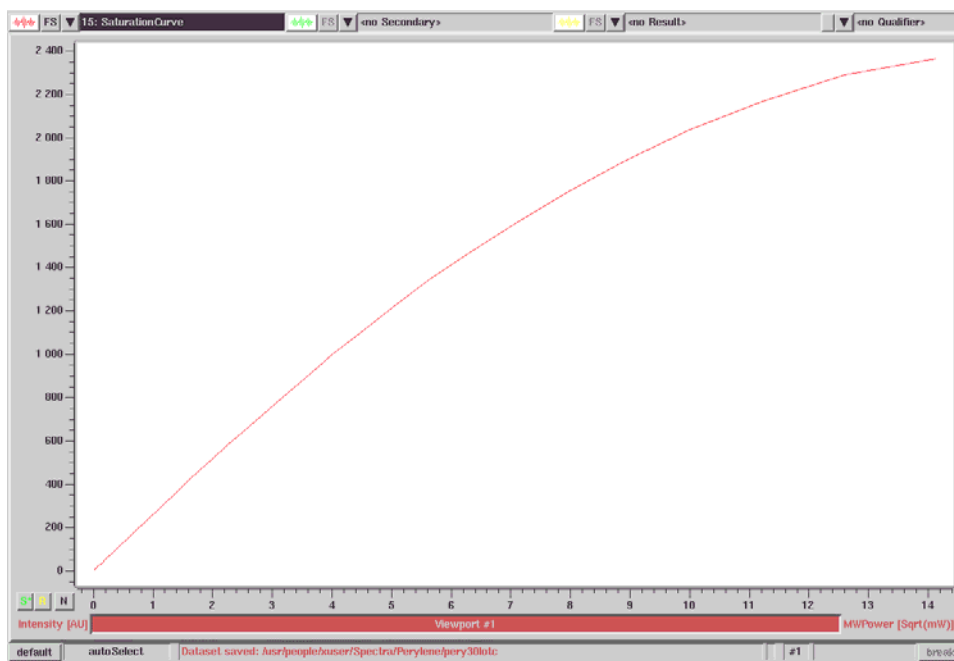


Figure 7-11 The microwave power saturation curve of DPPH sample acquired with a nonlinear 2D acquisition.

You can apply the nonlinear acquisition function to other 2D experiments. The name and the appearance of the second abscissa may be different but you can always select a user defined sweep to achieve a nonlinear acquisition. You can even select both the field sweep and the second variable as user defined sweeps.

We have implemented macros in Xepr to give users more convenience and freedom to customize their software. A macro is a stored list of commands that can be executed in order to automate routine tasks such as data processing or data acquisition. It is especially convenient for routine processing of large amounts of spectra. In the first section we will introduce the automatic recording method to make a macro. You do not need to know any macro commands or terms to create your own macro. The second section will guide you through the basic commands of a macro so you can write your own macro using a text editor. This method allows you to use arguments and create dialog boxes in a macro.

Recording a Macro

8.1

Xepr makes it easy to record a macro and assign a User Button to the macro in order to easily execute the macro. This section guides you, step by step, through recording your macro.

Xepr keeps a history of the commands you have executed. The recording process described here simply takes this history and saves it into a macro file.

1. **Get ready for recording.** That includes, for example, loading a dataset, *etc.* Any procedures that are necessary for the operation but you do not want to include in the macro should be performed before you start.
2. **Open the Command Line.** Click Options > Commands & History. (See Figure 8-1.) This opens a dialog box called Xepr Command Line.

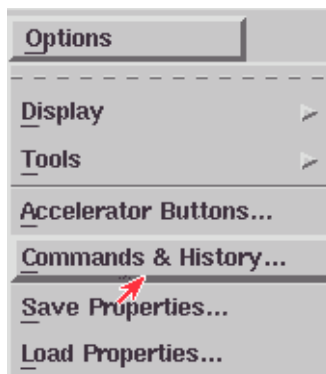


Figure 8-1 Opening the Commands & History dialog box.

3. **Start recording.** Click Options > Record Macro. (See Figure 8-2.)

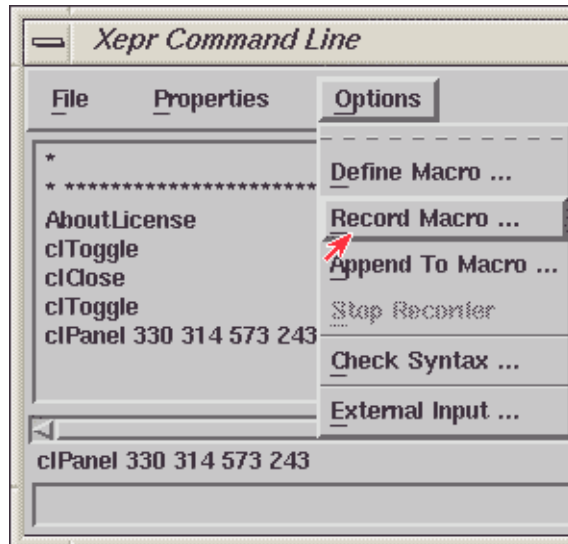


Figure 8-2 Starting to record a macro.

4. **Name your macro.** A dialog box appears, asking for the name you want to call the macro. Type a name in the File box. You can choose into which directory you want to put the macro file. By default it is saved in the ~/xepr-Files/Macros subdirectory under your home directory. The program will automatically add an .xmc extension to the file name.

5. **Record the macro.** Click the Record button at the left bottom corner to start recording the macro. (See Figure 8-3.)

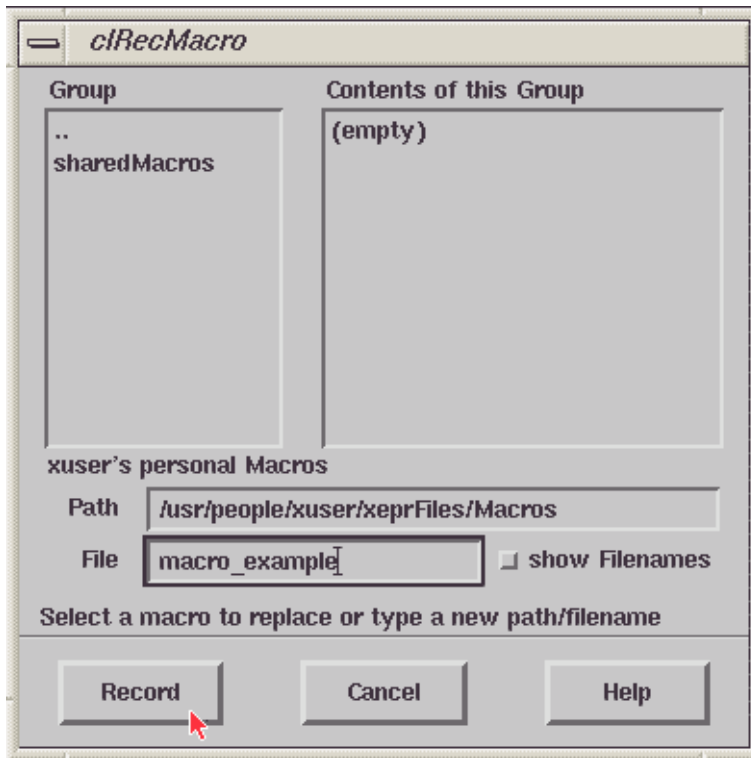


Figure 8-3 Setting a macro name and its path.

6. **Perform the operations.** Perform the operations, in sequence, you wish to record in the macro.

7. **Stop recording.** After you have finished your operations, click Options > Stop Recorder. (See Figure 8-4.)

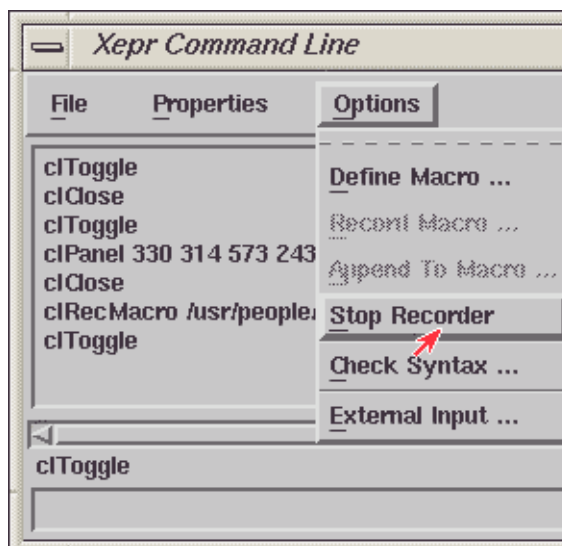


Figure 8-4 Stopping the recording.

8. **View the contents of the macro.** A dialog box containing all the macro commands you just recorded appears. Click on the **Close** button. (See Figure 8-5.)

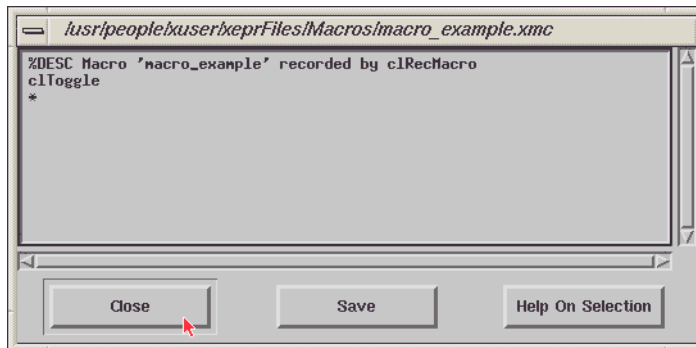


Figure 8-5 A dialog box showing the macro commands you recorded.

9. **Test your macro.** Return to the conditions before you started recording the macro, *e.g.* select the dataset that is displayed in the **Primary**. Type the macro filename (without the *.xmc extension) in the command line followed by <Enter> to execute the macro.

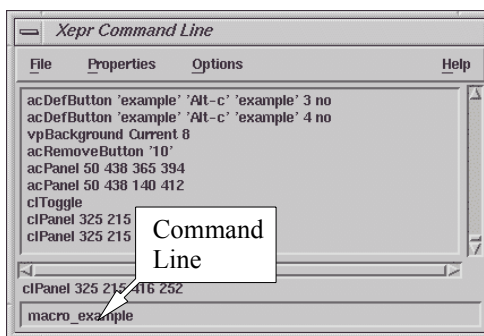


Figure 8-6 Executing the macro from the command line.

Making a Macro User Button

8.2

Typing a macro filename in the Xepr Command Line is not the most convenient means of executing a macro. A **User Button** in the **Tasks** bar makes running a macro as easy as clicking a button.

1. **Load the macro.** First load the macro. Click **Options > Command & History > File > Load macro**. If you have just recorded a macro, you do not need to load that macro.
2. **Open the Xepr Accelerators dialog box.** Click on **User Buttons in Tasks** (See Figure 8-7.) to activate the Xepr Accelerators dialog box. (See Figure 8-8.)

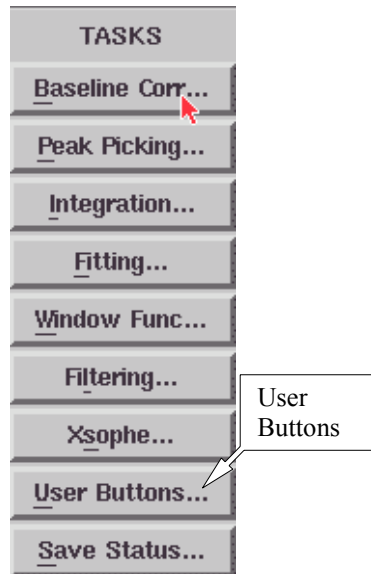


Figure 8-7 The Tasks menu.



Figure 8-8 Defining the user button.

3. **Define the macro.** Click on define button. If you can not find define button you can click File > Define Macro.
4. **Name the user button.** In the acDefButton dialog box type a name for the button. (See Figure 8-9.)

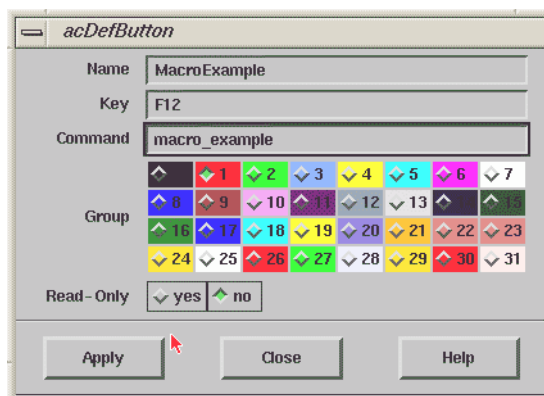


Figure 8-9 Defining the user button properties.

5. **Select a hot key for your macro.** You need to assign a hot key for your macro in the Key column. Special rules apply to assigning a hot key. You can find those rules in the Help menu.

6. **Enter a command line.** Enter the macro filename without the *.xmc extension. (See Figure 8-9.)
7. **Assign a color to the button.** Grouping buttons by color helps organize your macros. To choose a color other than the default color, click the desired Group button.
8. **Finish defining the user button.** Click Apply and then Close to finish making the user button. The newly defined button should appear in the Xepr Accelerators dialog box. (See Figure 8-10.)

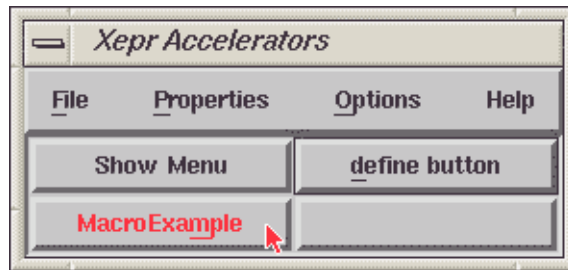


Figure 8-10 The macro buttons in the Xepr Accelerators box.

9. **Test your macro.** Return to the conditions before you started recording the macro, *e.g.* select the dataset that is displayed in the Primary. Click on the user button to execute the macro.

Writing a Macro with a Text Editor

8.3

You can also write a macro using any text editor. Simply save your file with an *.xmc extension for macro files. Writing a macro with this method allows you to use arguments, create a dialog box, direct the macro to terminate upon an error, and choose whether the macro executed commands will be recorded in the data history.

Special Macro Commands

8.3.1

In addition to the normal Xepr commands that are executed by the macro, there are special macro commands that are preceded by a “%” character. These special commands control how the macro executes and allow you to pass data to the various Xepr commands in the macro. All special macro commands must be uppercase.

- %DESC** This command briefly describes the macro. The string after %DESC is similar to the title of a dataset. For example, %DESC Example indicates this macro is an example and the string “Example” will appear in the dialog box as a title when you load the macro. It will also be displayed when you type the macro name into the command line.
- %TERM** If this statement is present, the macro terminates if an internal error occurs. No additional statement is needed.
- %NOHIS** If this options is present, commands in your macro will not generate history output. Instead, only the macro itself will produce a history line like any other Xepr command.

%ARG This statement defines an argument. An argument is the means by which you create a prompt or dialog box to input and pass data to the Xep commands in your macro. Argument syntax is as follows:

`%ARG <name> (<type>) <flag> '<default>'`

- **name.** The argument requires a name so that it can be referenced by the Xep commands of the macro. You can use any alphanumeric characters in the name. The name will appear in prompts or dialog boxes.
- **type.** Type specifies the data type of the argument. To determine the data type required by an Xep command, use the Help system. (See Section 4.7 of the Elexsys E 500 User's Manual: Basic Operations for more information on using the Xep Help system.) For example, Figure 8-11 shows the syntax description for the `clHistory` Xep command. Here we see that this command requires a word and a boolean argument. Note that the datatype must be delimited by parentheses.

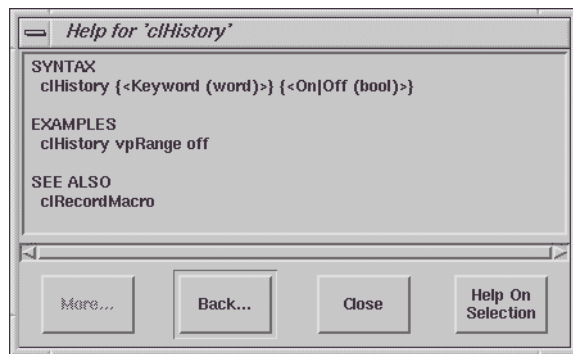


Figure 8-11 Syntax description of the `clHistory` Xep command.

The data type must be one of the following:

Data Type	Definition
INT_ARG	integer
CARD_ARG	cardinal number (integer ≥ 0)
FLOAT_ARG	floating point number
COLOR_ARG	color (used to change background colors, <i>etc.</i>)
WORD_ARG	a single word
TEXT_ARG	text string (can contain spaces and punctuation)
SWITCH_ARG	several strings, only one of which can be selected
BOOL_ARG	logic value with only two possible values, On or Off
PAF_ARG	pointer to a file

Table 8-1 Definition of data types.

- **Flag.** There are three flags available that determine how the argument value will be entered.
 - ! The argument value is required right from the start.
 - # The argument value will be entered in a dialog box.
 - ? The default value will be used if the argument value is not entered.
- **'<default>'** The string enclosed by the apostrophes defines the default value. If no default value is specified, the last value for that argument will be the default value. For switch or boolean arguments, this string defines the allowed values, e.g. '<value1|value2|value3>', where the first one is the default value.

To call an argument in your macro body, simply type `$<name>` in place of using a constant value.

An Argument Example

As an example, the following section of a macro file will produce the following dialog box to prompt you for input. (See Figure 8-12.)

```
%DESC Macro loading & displaying a dataset
%NOHIS
%TERM
%ARG INT_ARG (INT_ARG)# '1'
%ARG CARD_ARG (CARD_ARG)#
%ARG FLOAT_ARG (FLOAT_ARG)#
%ARG COLOR_ARG (COLOR_ARG)#
%ARG WORD_ARG (WORD_ARG)#
%ARG TEXT_ARG (TEXT_ARG)#
%ARG SWITCH_ARG (SWITCH_ARG)# 'Choice1|Choice2|Choice3'
%ARG BOOL_ARG (BOOL_ARG)# 'On|Off'
%ARG PAF_ARG (PAF_ARG)#
```

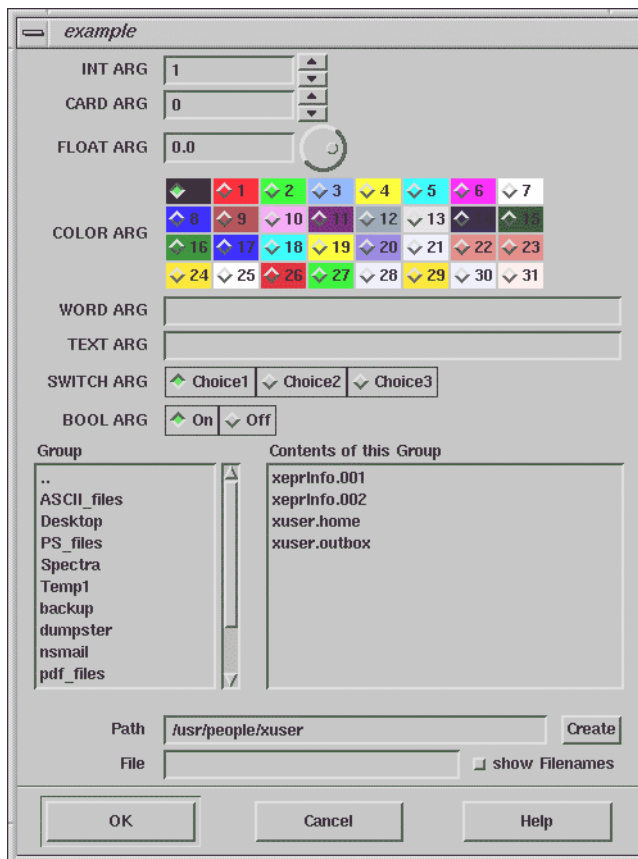


Figure 8-12 A dialog box prompting for input.

The Macro Body

8.3.2

The macro body consists of comments and commands. Lines starting with an asterisk (*) are marked as comments and are ignored by Xepr. Any Xepr command can be a macro command. Refer to the Xepr software manual or **Help** for available Xepr commands. No end statement is necessary.

A Macro Example

8.3.3

The following example macro prompts you for a data filename, a data handle, and whether you want to convert the abscissa type. It then loads the selected data file into Xepr under the specified handle number, displays it in the current **Primary** viewport, and then converts the abscissa to the selected type. The commands in the macro will not appear in the history except the macro itself. It will terminate upon internal error. It contains three arguments: the file name, the data handle number, and the abscissa type.

```
%DESC Macro loading & displaying a dataset
%NOHIS
%TERM
%ARG paF (PAF_ARG) #
%ARG number (CARD_ARG) #
%ARG axis (SWITCH_ARG) # 'indexed|index-gauged|n-tuple'
* Load the dataset
ddLoad $paF $number
* switch off all other datasets in current viewport
vpCurrent -1 Secondary 0
vpCurrent -1 Qualifier 0
prNOOp -1
* now display newly loaded data set
vpCurrent -1 $number
ddAbscType $number $axis
```

Type the above example in a text editor and save it as **example.xmc**. Load the macro and enter **example** in the command line. You can also follow the procedure in Section 8.2 to make it a user button.

The prompt for the macro will look as follows.

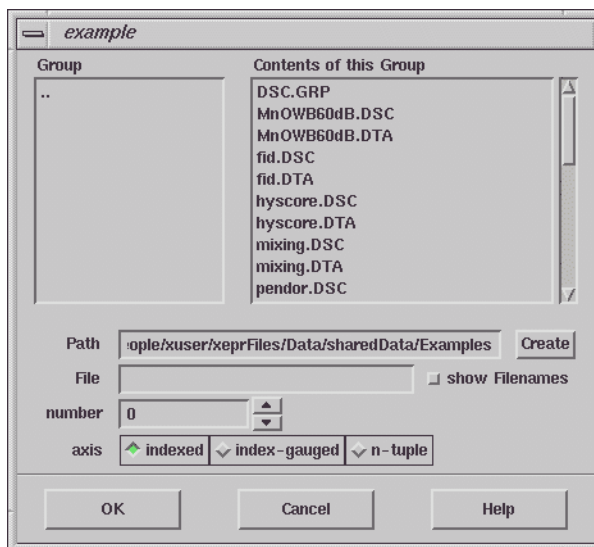


Figure 8-13 An example prompt.

The Xepr software offers an automatic post processing feature. After an acquisition is finished, this feature executes a sequence of commands that you have defined. This feature can combine acquisition, data processing, and data storage operations into one action. It also accepts task-bar routines and user-made macros.

Activating Auto Post-Processing

9.1

Let us start with a simple example: we wish to acquire a spectrum, double integrate the spectrum, and save the original and double integrated spectra automatically.

1. **Open the auto post-processing dialog box.** Click Acquisition > Auto Post-Processing in the menu bar. A dialog box appears.

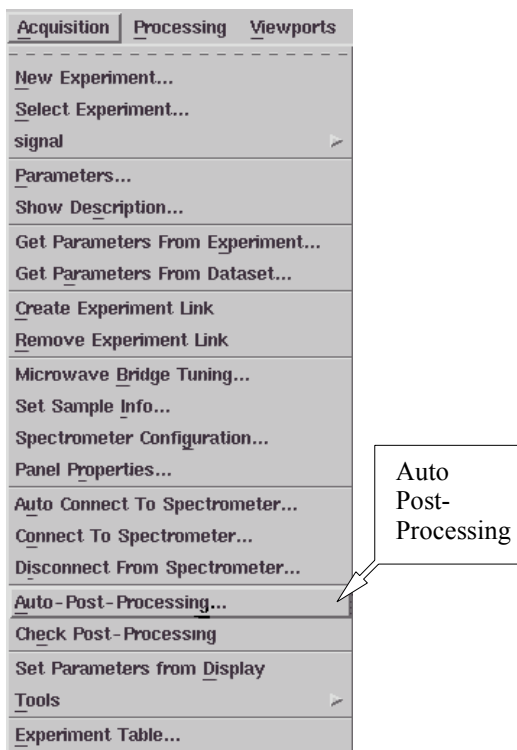


Figure 9-1 Opening the Automatic Define #1 dialog box.

2. **Define the auto post-processing commands and options.** (See Figure 9-2.)

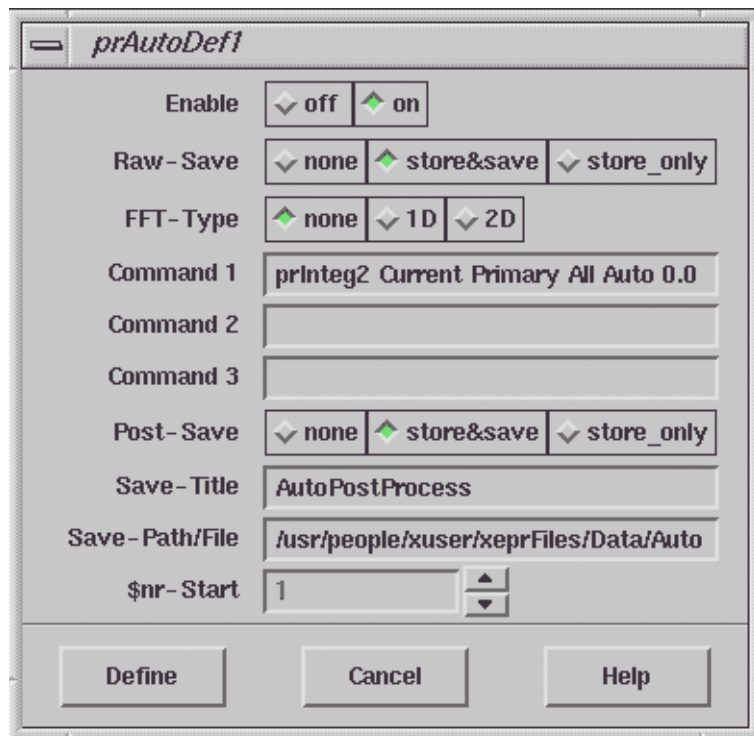


Figure 9-2 The auto post-processing definition dialog box.

Enable Click on to activate auto post-processing.

Raw-Save If you select **store&save** the raw (unprocessed) spectrum will be saved as *filename_R.DSC* and *filename_R.DTA* files where *filename* is defined under **Save-Path/File**. If you select **store_only** the raw spectrum will be stored temporarily under the title defined in **Save-Title** and the handle number indicated

- by **\$nr-Start**. **none** specifies the raw dataset is to be neither saved nor stored.
- FFT-Type** If you want to perform a Fast Fourier Transformation you need to select either **1D** or **2D** for one or two dimensional transforms. Otherwise select **none**.
- Command** **Command 1**, **2**, and **3** define the Xepr commands to be executed. Each entry accepts one command. You may enter as many as three commands in this dialog box. Refer to the next section for entering more than three commands. In this example we use the command for automatic double integration. To find the Xepr command for the function you want to use, execute that function and look at either **Help > Main Help** or **Options > Command & History**. You can also enter the name of a macro or ProDeL program in the command line.
- Post-Save** If you select **store&save** the post-processed spectrum will be saved as *filename_P.DSC* and *filename_P.DTA* files where *filename* is defined under **Save-Path/File**. If you select **store_only** the processed spectrum will be stored temporarily under the title defined in **Save-Title** and the handle number indicated by **\$nr-Start**. **none** specifies the processed dataset is to be neither saved nor stored.
- Save-Title** Enter the title you want to use if you selected **save&store** or **store_only** options.
- Save-Path/File** Enter the path followed by the filename if you selected the **save&store** option in **Raw-Save** or **Post-Save**. Make sure the directory exists and you have permission to write to that directory. You can use a variable, **\$nr** as part of the file name so that the file name automatically increments when multiple acquisitions are set. For example, you can set the file name as **test\$nr** and the saved files will be **test1_R**, **test2_R** ..., if you clicked the **Raw-Save** button and set **\$nr-Start** to 1. (See next item.)

- \$nr-Start** If you choose to save the spectrum and use \$nr in the file name this is the initial value of the \$nr variable. If you choose to store the spectra only, this is the handle number assigned to the data stored in the data table. It will automatically update to the first available handle number if it is set to -1. You can force the handle number to a specific value by entering that number.
3. **Close the dialog box.** Click the Define button to close the dialog box.

4. **Check the auto post-processing routine.** This command allows you to run auto post-processing without acquiring a spectrum.

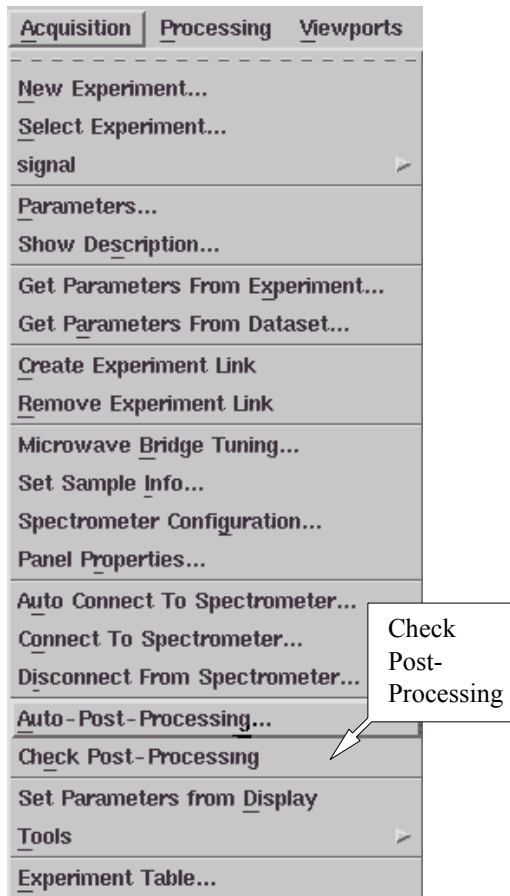


Figure 9-3 Checking auto post-processing.

Load a spectrum in the Primary Dataset and then click Acquisition > Check Post-Processing to check whether the auto post-processing routine operates prop-

erly. If you selected **save&store** in the Auto Post-Processing definition dialog box, you need to check whether the files have been saved and stored. If you do not need these files, delete them. Otherwise you will receive a warning message for overwriting the existing file when you actually execute the auto post-processing routine.

5. **Run auto post-processing.** Create an experiment and set up the parameters. Click **Run** to acquire the spectrum. Upon completion of the acquisition, Xepr executes the auto post-processing routine. Figure 9-4 shows the result of the double integration of the raw spectrum. The original spectrum and the processed dataset have been saved and stored as **AutoPostProcess_R** and **AutoPostProcess_P** files respectively.

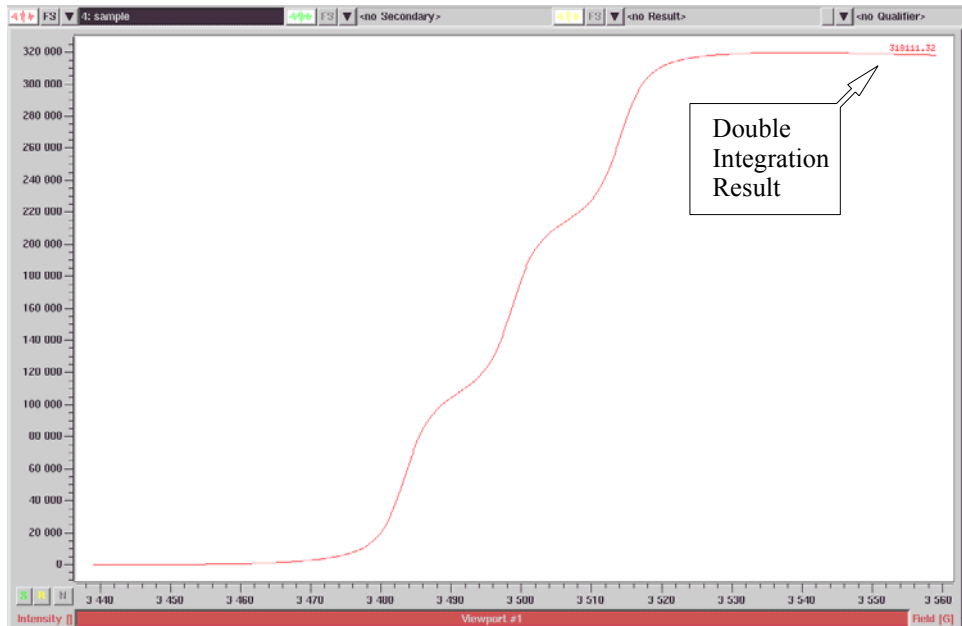


Figure 9-4 Result of auto post-processing.

Multiple Auto Post-Processing Routines 9.2

Only three commands can be executed from one auto post-processing routine. In order to execute more commands, there are two additional post-processing routines. Only the first one (in the Acquisition menu list) can be triggered when the acquisition is finished. If you need to use more than three commands you must activate the other two auto post-processing routines called Auto #2 and Auto #3.

Setting Up More than Three Commands 9.2.1

1. **Call Auto #2 and/or Auto #3.** Click Acquisition > Auto Post-Processing. Enter prAuto2 in one of the command lines of the dialog box.

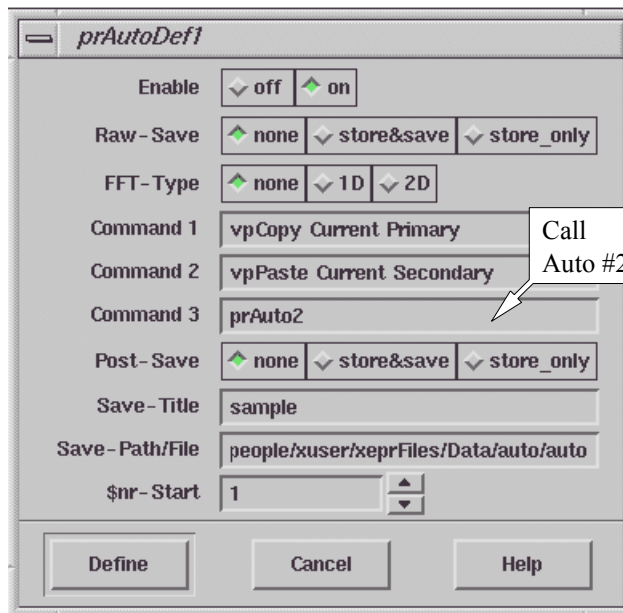


Figure 9-5 Setting up auto post-processing.

You can also enter `prAuto3` if needed and if there is a command entry available. (See Figure 9-5.) In this example, the Primary dataset will be copied and pasted to the Secondary Dataset. This way we will be able to apply different processes to the original dataset. Then it calls the second auto post-processing function **Auto #2**.

2. **Open the second auto post-processing dialog box.** Click Processing > Automatic > Define #2 to open the dialog box. (See Figure 9-6.)

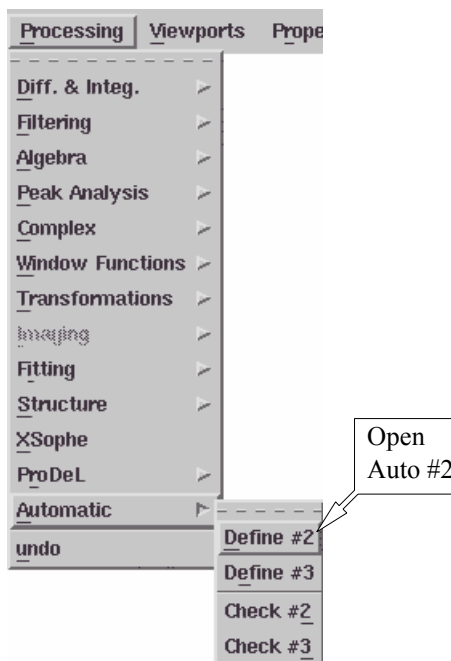


Figure 9-6 Opening the Automatic Define #2 dialog box.

3. **Define Auto #2.** The dialog box for defining this routine is exactly the same as the previous one. You need to **Enable** this function by clicking the **on** button. In this example, we enter the command for double integrating the **Primary** data in **Command 1** and call the third auto post-processing routine in **Command 2**. (See Figure 9-7.)

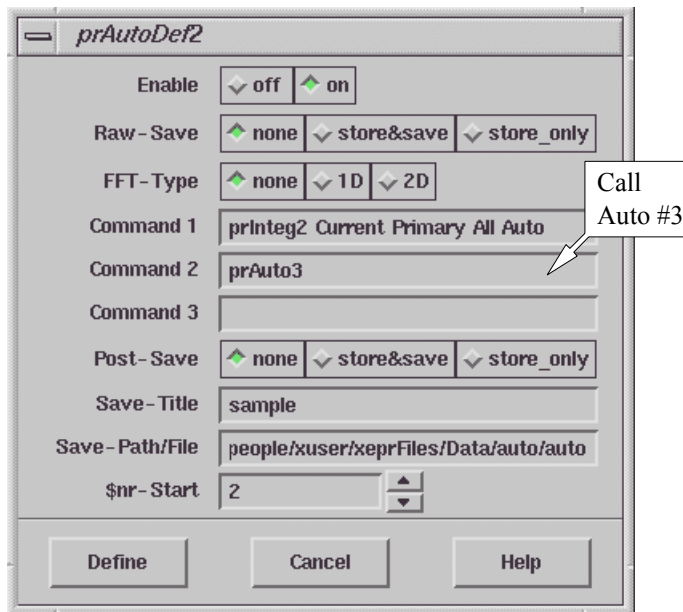


Figure 9-7 Defining the Auto #2 routine.

4. **Open the third auto post-processing dialog box.** Click Processing > Automatic > Define #3 to open the dialog box. (See Figure 9-8.)

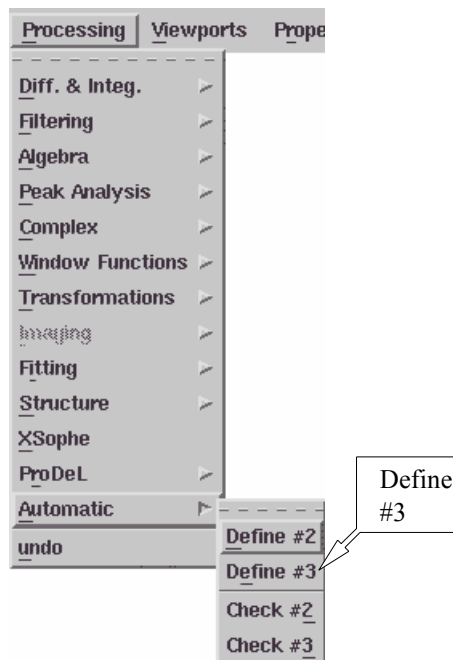


Figure 9-8 Opening the Automatic Define #3 dialog box.

5. **Define Auto #3.** The items in this dialog box are the same as for the other two. Remember to activate this function by clicking the Enable on button. In this example we are going to peak pick the Secondary Dataset.

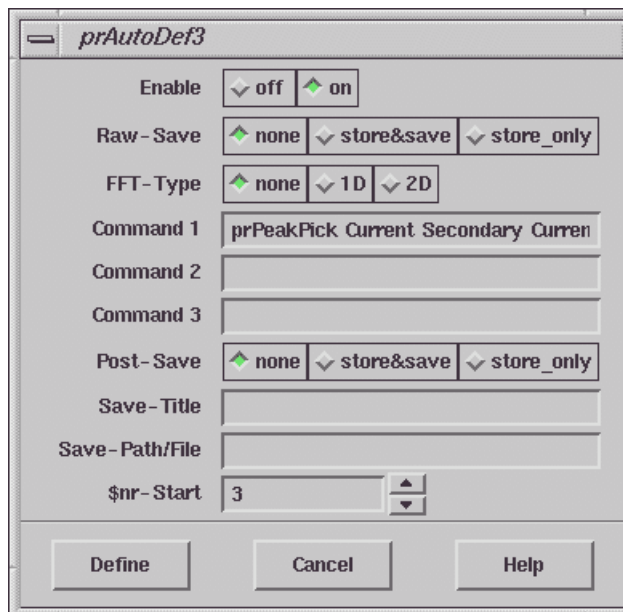


Figure 9-9 Defining the Auto #3 routine.

6. **Check the auto post-processing routines.** Load a spectrum into the Primary Dataset. Click Processing > Automatic > Check #2 or Check #3 to see if the Auto #2 or Auto #3 routines function properly. If you called Auto #3 from Auto #2, both auto post-processing routines will be checked when you click Check #2. Otherwise they must be checked independently. (See Figure 9-10.)

When you activate the chain of auto processing routines, you need to be careful about the sequence in which it executes, especially when you want to automatically store or save the results. You can test the routines with the **Check #1** command to make sure the execution order is correct.

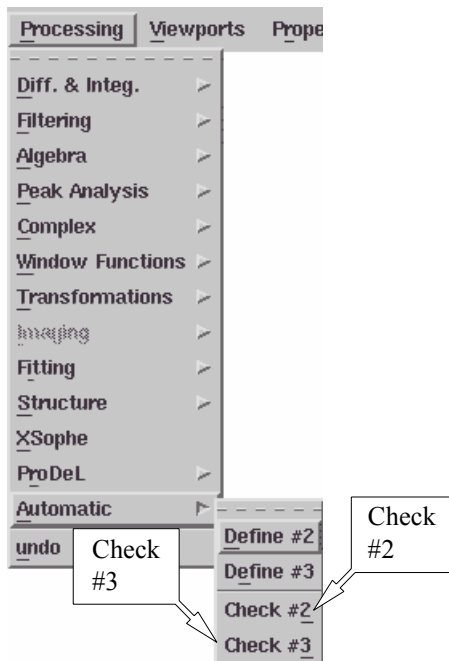


Figure 9-10 Checking the Auto #2 and/or Auto #3 routines.

7. **Execute the multiple auto post-processing routines.** Create or load an experiment and set up the parameters. Click Run to acquire the spectrum. The auto post-processing routines should execute sequentially. In this example the results of the auto-processing are shown in Figure 9-11.

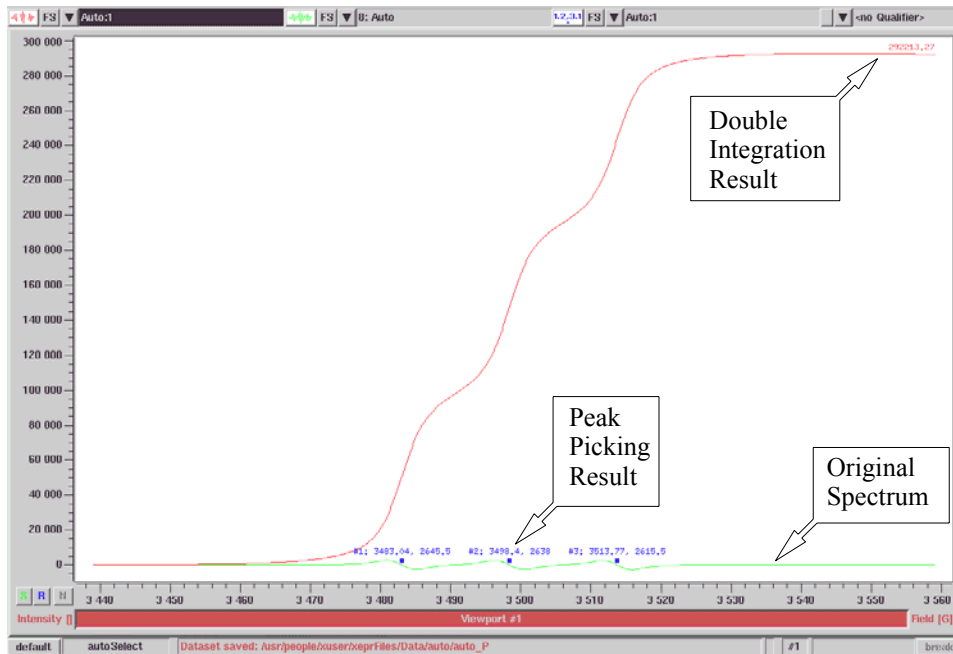


Figure 9-11 Results of a series of auto post-processing routines.

One of the powerful features of the Xepr software is that you can customize your Xepr program by utilizing Xepr macros and ProDeL programs. In order to share these customized routines and functions with your fellow users, they must know how to use them. The Xepr help system allows you to implement your own instructions for these customized functions or routines. The help files can include definitions, descriptions, command and argument syntax, helpful hints, related command, *etc.* This chapter will describe how to implement your own help files in the Xepr help system.

Writing a Help File

10.1

Help File Format

10.1.1

Xepr help files are plain ASCII text files. You can use any text editor to compose the help file. No special format is required. There are two important statements: **DESC** and **MORE**. **DESC** indicates the start of the text to be displayed in the help window. All text preceding the statement is not displayed. If the file is too long to display in one help window, type the **MORE** command into the help file to divide the file into more manageable sections. When viewing the help file, the **More** button in the help dialog box advances you to the next section. You can use **MORE** as many times as you wish if the file is very long. The **Back** button in the help dialog box returns you to the previous section. The following page shows an example help file.

PURPOSE

sets data byte sequence of a Dataset

DESC

DESCRIPTION

Sets the byte sequence used to represent binary values in the current dataset.

MORE

DIALOG PARAMETERS

Binary coordinate values in a BES3T-file can be represented in two different formats:

MSB first: the bytes are saved starting with the most significant byte. This is the format used on SPARC and MOTOROLA microprocessors based machines.

LSB first: the bytes are saved starting with the least significant byte. This is the format used on INTEL microprocessors based machines.

The format thus defined is applied to all binary values of a dataset.

SEE ALSO

BES3T-Documentation

MORE

It is not necessary to change the byte sequence of a dataset if you want to read in dataset files on another machine, because BES3T-compliance requires the ability to read both byte sequences. Changing the byte sequence to that of the target machine just saves the time required to internally swap all data bytes.

SYNTAX

COMMAND LINE USAGE

The dataset indicated by <#> is modified, not the current selection.

EXAMPLES

```
ddByteSeq 4 MSB first
```

SEE ALSO

ddLoad, ddSave

MORE

TECHNICAL ASPECTS

This command changes the BSEQ entry in the BES3T .PAR file.

Changing the format only affects the way data are saved to disk, the memory representation in the Xepr program remains unchanged.

Help Filenames

10.1.2

The help filename for a command must be identical to the command name. No extension is needed. For example, if the macro you made is called eXample and saved as eXample.xmc, the help file for this macro must be named eXample without extension.

Where to Put the Help Files

10.1.3

Access to a help file is determined by the location of the file. If you want every user from all accounts to be able to see a help file you need to save the file in the `/usr/xep/sharedHelp` folder. Note that you need root privileges to do so. If you want to limit access to one user account, *e.g.* `xuser`, save the file in `/usr/people/xuser/xepFiles/Help` on an SGI system running IRIX and only those who log in as `xuser` have access to this help file. For a Linux system, put the file in the `/home/xuser/xepFiles/Help` directory.

Accessing Help

10.2

You can always click the **Help** button in the menu bar and click **Main Help**. Enter the command name in the **Selection** box and click **Keyword**. Click the **Apply** button and a help dialog box appears and displays the content of the help file for that particular command. (See Figure 10-1.)

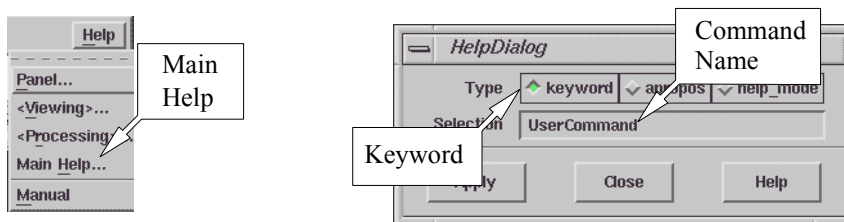


Figure 10-1 Accessing Help.

Alternatively, you can execute the command and then click **Help** > **Main Help** in the menu bar. The command you just executed will appear in the **Selection** box. Click **Keyword** and **Apply** buttons to view the help contents.

If your macro or ProDeL program prompts you for user input, there will be a **Help** button in the dialog box. Click the **Help** button in the dialog box and the contents of the help file for that routine is displayed.

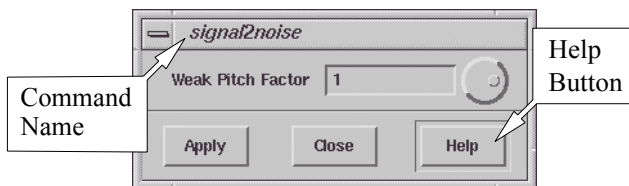


Figure 10-2 The Help button of the prompt dialog box.

Changing Microwave Bridges 11

Removing the Microwave Bridge 11.1

1. **Turn off the spectrometer.** Follow the procedures in Chapter 3 of the Elexsys E 500 User's Manual: Basic Operations to turn off the spectrometer if it is still on. Close the water valves.
2. **Remove the cavity.** Follow the instructions of Section 7.2 of the Elexsys E 500 User's Manual: Basic Operations to remove the cavity. Remove the waveguide extension from the bridge as well.
3. **Disconnect the cooling water.** Not all bridges require water cooling. If the current bridge has water cooling, first close the water valves for the bridge. Disconnect the water hose to and from the bridge. (See Figure 11-1.) Block the water hose to prevent water spillage.

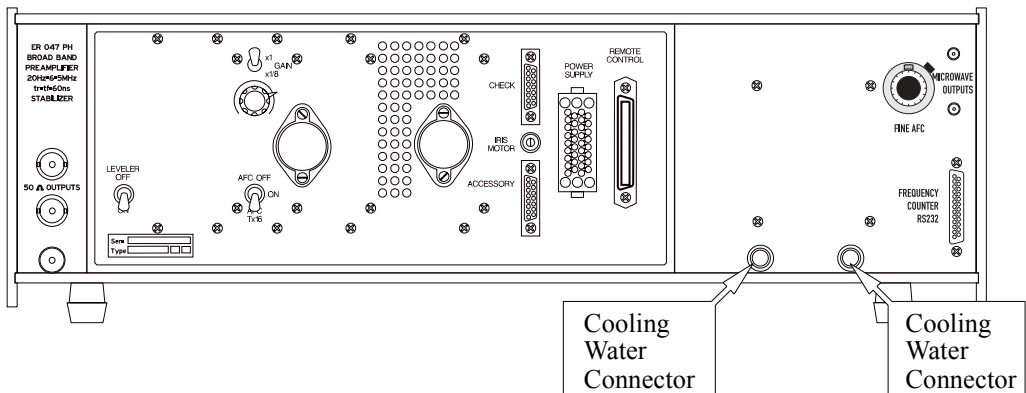


Figure 11-1 The cooling water connectors for the microwave bridge.

4. **Disconnect the power supply cable.** There are two clips used to secure the connection of the power supply cable to the bridge. Press both clips with your thumb and index finger and pull out the power supply cable. (See Figure 11-2.)

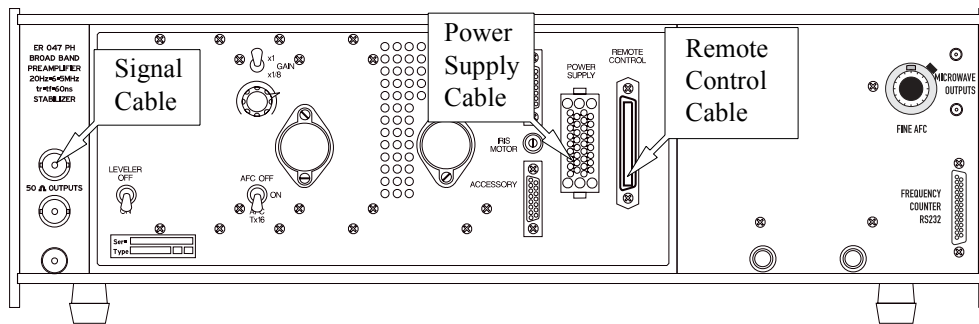


Figure 11-2 The power supply, remote control, and signal cable connectors.

5. **Disconnect the remote control cable.** There are two clips, one on the top and one on the bottom of the remote control cable connector. Flip both clips out against the bridge back panel. Then remove the remote control cable. (See Figure 11-2.)

6. **Disconnect the signal cable.** The signal cable has a BNC connector. Disconnect the signal cable carefully.

7. **Disconnect other connections.** Depending on the bridge, you may have other connections such as an iris motor controller cable, frequency counter cable, FF Lock cable, *etc.* Disconnect all these connections. (See Figure 11-3.)

8. **Remove the bridge from the bridge table.** Unless you need to put the new bridge on top of the old bridge, remove the bridge from the bridge table and place it in a



We strongly recommend that two people move the bridge. It is heavy, fragile, and very expensive. If dropped on someone it can cause serious bodily injuries.

safe place. Cover the waveguide opening on the bridge with the waveguide cover to prevent dust from entering the waveguide.

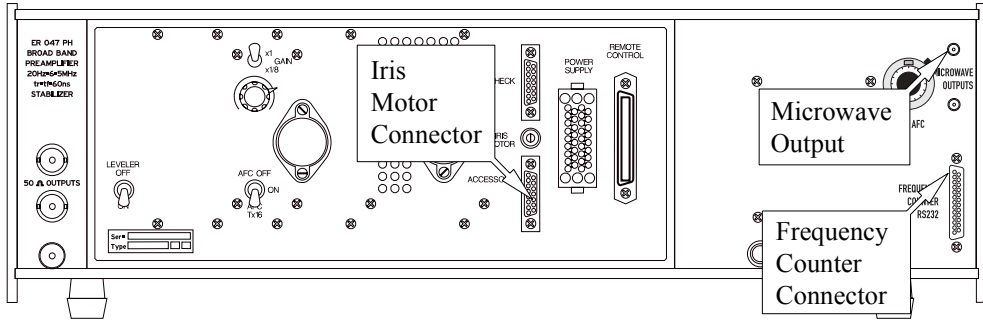


Figure 11-3 Other bridge connections.

Installing a Microwave Bridge

11.2



We strongly recommend that two people move the bridge. It is heavy, fragile, and very expensive. If dropped on someone it can cause serious bodily injuries.

1. **Put the microwave bridge on top of the bridge table.** Some non X-band bridges actually sit on top of the X-band bridge.
2. **Attach the waveguide extension.** For some cavities you need to attach the waveguide extension to the bridge.

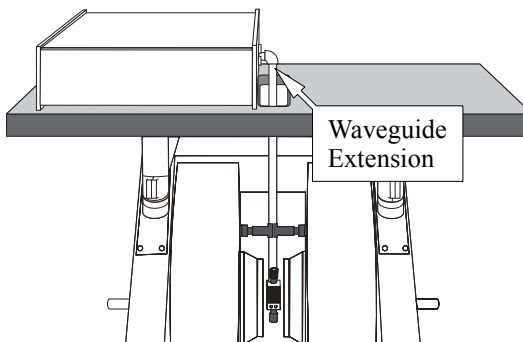


Figure 11-4 The waveguide extension.

Align the waveguide

gasket in the correct direction (Refer to Figure 11-5.) and tighten the waveguide screws.

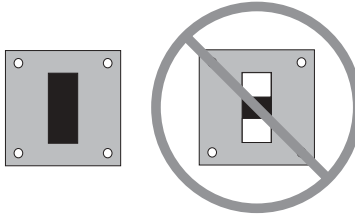


Figure 11-5 Installing the waveguide gasket properly.

3. **Adjust the bridge position.** Adjust the position of the bridge so that the waveguide extension is approximately centered (right to left) in the magnet.
4. **Install the EPR cavity.** Follow the instructions in Section 7.2 of the Eleksys E 500 User's Manual: Basic Operations in order to install the cavity. Adjust the bridge position to center the cavity in the magnet. Secure the cavity position with either the waveguide stabilizers or a cavity mounting device.
5. **Connect the cooling water.** If cooling water is required, connect the cooling water hoses to the connectors and open the water valves for the bridge. Turn on the heat exchanger to test for any water leakage. Make sure there is no leakage from the connections. Turn off the heat exchanger.
6. **Connect the power supply cable.** Line up the pins and push in the bridge power supply cable connector.
7. **Connect the remote control cable.** One side of the connectors is wider and the other side is narrower. Line

them up and push in the connector. Flip the two clips to secure the connection.

8. **Connect the signal cable.** The signal cable has a BNC connector. Push it in and rotate to secure the connection. There are two 50 ohm signal output from the bridge. Connect the signal cable to either one of them.
9. **Connect other accessories.** If you have auto tuning capabilities, connect the iris motor cable. If you have other accessories to connect to the bridge, connect them accordingly.

10. **Set the DIP switches on the microwave bridge controller.** The microwave bridge controller is the top unit of the console. There is a dip switch unit in the microwave bridge controller. The DIP switches need to be set correctly to suit your bridge so that the tuning picture will be displayed correctly. There are four dip switches. The switches are from left to the right, SW1-SW4. The settings for these switches for commonly used bridges are listed in Table 11-1.

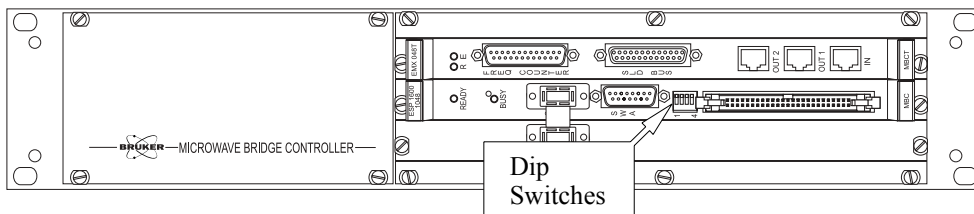


Figure 11-6 The microwave bridge controller.

Bridge Type	SW1	SW2	SW3	SW4
X-, S-, L-, band Gunn bridge, 60 dB attenuator	Down	Down	Up	Up
X-band Klystron bridge, 60 dB attenuator	Up	Up	Up	Up
Q-band Gunn bridge without cooling water	Up	Up	Up	Down
Q- and K-band Gunn bridge with cooling water	Down	Up	Up	Down

Table 11-1 DIP switch settings for commonly used bridges

11. **Turn on the spectrometer.** Follow the instructions in Chapter 3 to turn on the spectrometer. Open the bridge tuning dialog box. Check if the tuning picture is correct. If you see an abnormal tuning picture, you might have wrong DIP switch settings.

Installing & Using Accessories 12

Bruker offers many optional accessories for the Elexsys series spectrometers. In this chapter, we shall concentrate on aspects that directly relate to the use of these products with the Xepr software. Most of the accessories have their own individual manuals: consult their documentation for further information and details.

Frequency Counters

12.1

Precise microwave frequency measurement is crucial for precisely measuring g-values and calibrating magnetic field values. Xepr supports several frequency counters. This section describes how to connect and configure these supported counters correctly. Remember to click the **Apply** and **Save** buttons to activate the changes and save the configuration.

The MBC Board

12.1.1

The MBC (Microwave Bridge Controller) board supplies microwave frequency readings from control voltages in the bridge. If you do not have a frequency counter, this is your only option. Click **Acquisition > Spectrometer Configuration > Misc.** (See Figure 12-1.) Select **MBC** from the drop-down list in the **Frequency Counter** section. (See Figure 12-2.)

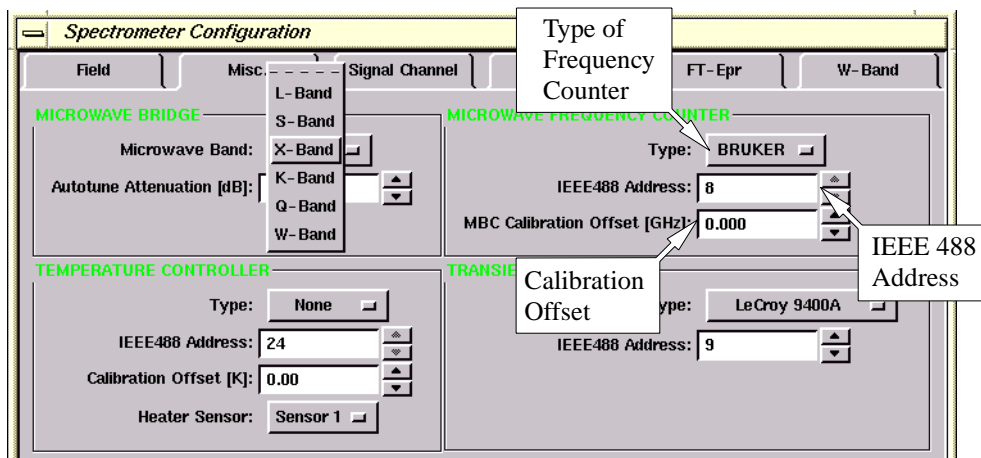


Figure 12-1 Configuring a frequency counter.

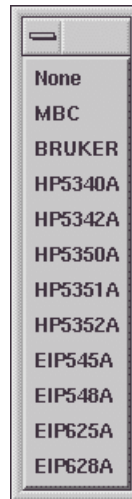
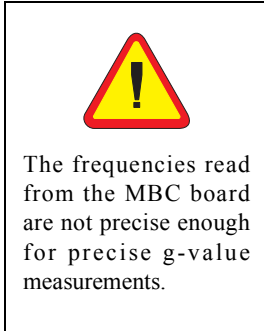


Figure 12-2 Selecting the proper frequency counter.

It is possible to calibrate the frequency displayed by the MBC board if you have a frequency counter available to you. (See Section 12.1.3 to learn how to connect the counter to the bridge.)

1. **Record the frequency reading of the MBC board.**
2. **Record the frequency reading of the frequency counter.**
3. **Calculate the MBC Calibration Offset.** The offset equals:
$$\text{MBC Calibration Offset} = \nu_{\text{Counter}} - \nu_{\text{MBC}} \quad [12-1]$$
4. **Enter the value for the MBC Calibration Offset.** (See Figure 12-1.)

The Bruker Frequency Counter

12.1.2

The Bruker frequency counter is optional for L-, S-, and X-band bridges. The frequency counter is mounted in the front panel of the bridge. It provides more accurate readings of the microwave frequency than the MBC board.

Connect the cable labeled MBCT-MWB 4001589 to the RS232 connector on the back of the bridge. (See Figure 12-3.) Click Acquisition > Spectrometer Configuration > Misc. In the Frequency Counter section select Bruker in the drop-down list. (See Figure 12-1 and Figure 12-2.)

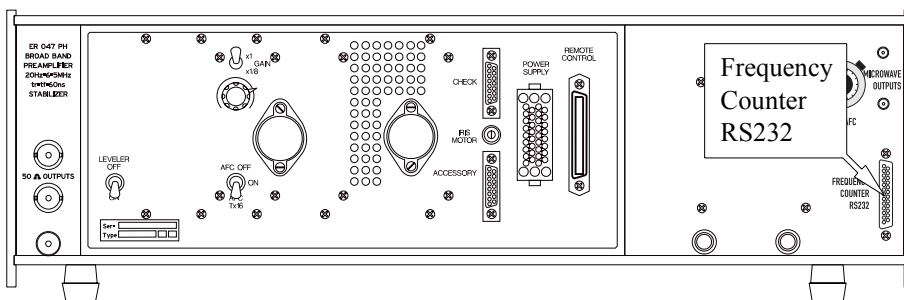


Figure 12-3 The frequency counter RS232 connector on the back of the bridge.

Frequency Counters by Other Vendors

12.1.3

Xepr supports the following frequency counters offered by other vendors:

HP 5340A	EIP 545A
HP 5342A	EIP 548A
HP 5350A	EIP 625A
HP 5352A	EIP 628A
HP 53150A	

You need to set the IEEE address of your frequency counter to 8. Refer to your frequency counter manual for instructions on setting the address. Connect the microwave output on the back of the bridge to the input of the frequency counter with a high quality microwave cable with SMA connectors on both ends. (See Figure 12-4.) Connect the IEEE-488 cable from the back of the Elexsys console to the IEEE connector of your frequency counter. You may have to open the back panel of the console to locate the cable.

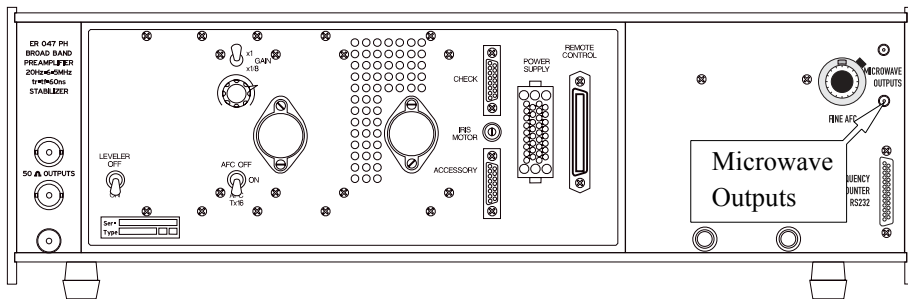


Figure 12-4 Microwave outputs on the back of the microwave bridge.

Turn on the frequency counter and the Elexsys E 500 spectrometer. Click Acquisition > Spectrometer Configuration > Misc. Select the proper frequency counter in the drop-down list. (See Figure 12-1, and Figure 12-2.)

You must set the bridge to **Operate** mode in order to read the frequency from the frequency counter.



When you connect the frequency counter for the first time, you may need to click the **Activate** button to initiate communications between Xpr and the frequency counter.

ER 41X1VT Variable Temperature Systems 12.2



These units are optional accessories and you may or may not have ordered them. If you require an accessory, contact your local Bruker EPR sales representative for purchase information.

These variable temperature systems are used for liquid nitrogen and above room temperature experiments. Refer to the manuals of your variable temperature unit for assembly and operation instructions. In this section we provide instructions for connecting the temperature controller to the Elexsys E 500 spectrometer and configuring the Xepr software.

The X in the model number corresponds to the numbers one, two, or three. Xepr fully supports the newest temperature controller, BVT 3000 (ER 4131 VT). The older BVT 2000 (ER 4121 VT) and BVT 1000 (ER 4111 VT) temperature controllers are only partially supported. The following steps describe connecting and configuring the controller.

1. **Connect the RS 232 cable. (For ER 4131VT and ER 4121VT systems.)** Open the front door of the console. Use the RS 232 cable to connect the RS 232 connector of the BVT temperature controller to the RS2 connector of the ACPU board in the Elexsys console. (See Figure 12-5.)
2. **Connect the Slow Device Bus cable. (For ER 4111VT systems only.)** Open the front door of the console. Connect the COMPUTER connector on the BVT 1000 temperature controller to the SLD connector on the SIP board of the acquisition server using a 25 pin Slow Device Bus cable. (See Figure 12-5.)

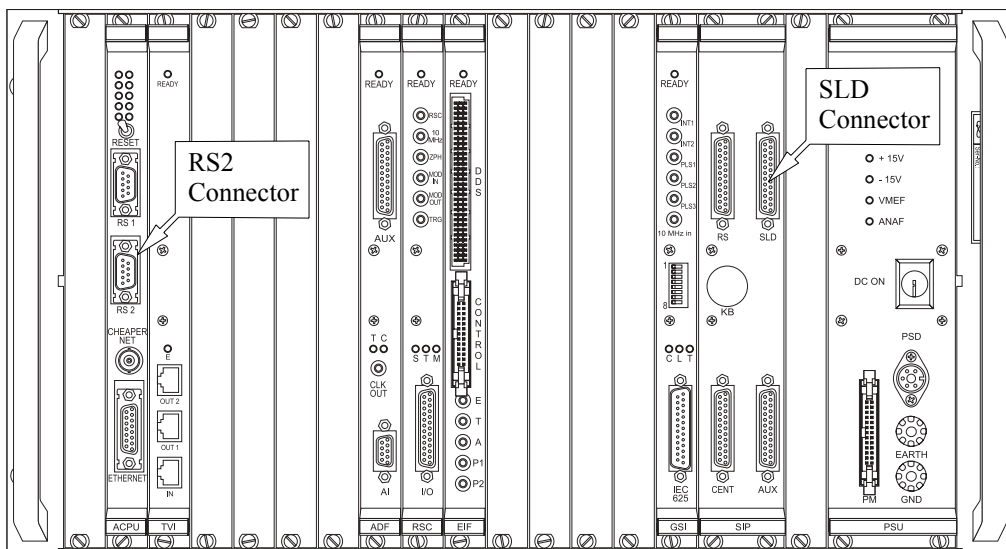


Figure 12-5 Connecting the RS 232 cable to the console.

3. **Configure the temperature controller.** Turn on the temperature controller and the spectrometer. Connect to the spectrometer. Click Acquisition > Spectrometer Configuration > Misc. Select the variable temperature system you are using in the Type drop-down list. Click Apply. If you plan on using the temperature controller in the future, click Save. Then click Close to close the dialog box. (See Figure 12-6.)



Figure 12-6 Setting the Temperature Controller type.



Consult the documentation for the ER 4131VT to learn more details about its operation.

4. **Define an experiment that uses the temperature controller.** Create a new experiment with the Temp. Unit button activated (green). (See Figure 12-7.) Open the Acquisition Parameters dialog box where you will find the VTU folder. This folder allows you to set the temperature after you click the **Activate** button. For the ER 4131 VT controller there is another sub-folder that gives you remote control of many controller parameters such as gas flow and N2 evaporator boiler power. (See the ER 4131 VT User's manual for details.) For the ER 4121 VT and ER 4111 VT controllers you can set the temperature but you need to manually control other controller operating parameters.

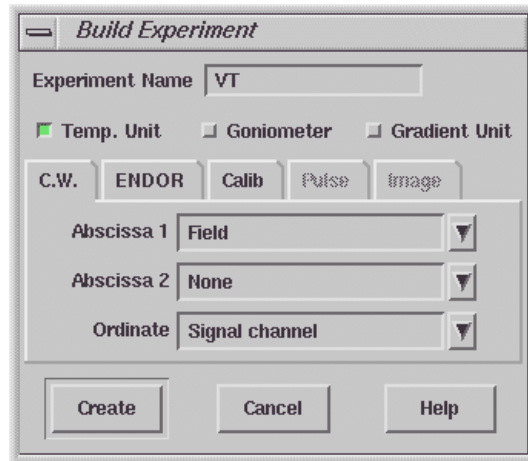


Figure 12-7 Defining an experiment using a temperature controller.

ER 4112HV Variable Temperature Systems 12.3



This unit is an optional accessory and you may or may not have ordered it. If you require an accessory, contact your local Bruker EPR sales representative for purchase information.

The Bruker Elexsys E 500 series spectrometers support the ER 4112HV temperature controller unit for liquid helium. In this section we provide instructions for connecting the ITC 503 temperature controller to the Elexsys E 500 spectrometer and configuring the Xepr software. It is assumed that you are already familiar with the operation of the temperature controller. If not, consult the Oxford Instruments documentation.

Setting the Controller Up

12.3.1



Consult the documentation for the ER 4112HV to learn more details about its assembly and operation.

1. **Connect the controller to the console.** Connect the IEEE 488 cable from the back of the spectrometer console to the IEEE 488 connector of your ITC 503 temperature controller. You may have to open the back panel of the console to locate the cable.
2. **Connect to the spectrometer.** Turn on the spectrometer and the ITC 503 temperature controller. After the acquisition server is booted, connect to the spectrometer.
3. **Set the IEEE address.** Click Acquisition > Spectrometer Configuration > Misc. In the Type drop-down list, select ITC 5 for your temperature controller. The default IEEE address is 24 for both the temperature controller and the Xepr software.
4. **Enter the temperature offset.** If you have carefully calibrated the temperature difference between the sample and the temperature sensor, you can enter this temperature offset in the Calibration Offset box.

The Calibration Offset equals:

$$\text{Calibration Offset} = T_{\text{actual}} - T_{\text{ITC}} \quad [12-2]$$

where T_{actual} is the actual temperature at the sample and T_{ITC} is the temperature measured with the cryostat temperature sensor.

5. **Select a sensor.** In the Heater Sensor section select the temperature sensor you are using on the ITC 503 controller to control the sample temperature. Unless you have multiple helium cryostats in your lab, you usually should select **Sensor 1**. Click **Apply**. If you plan on using the temperature controller in the future, click **Save**. Then click **Close** to close the dialog box. (See Figure 12-8.)

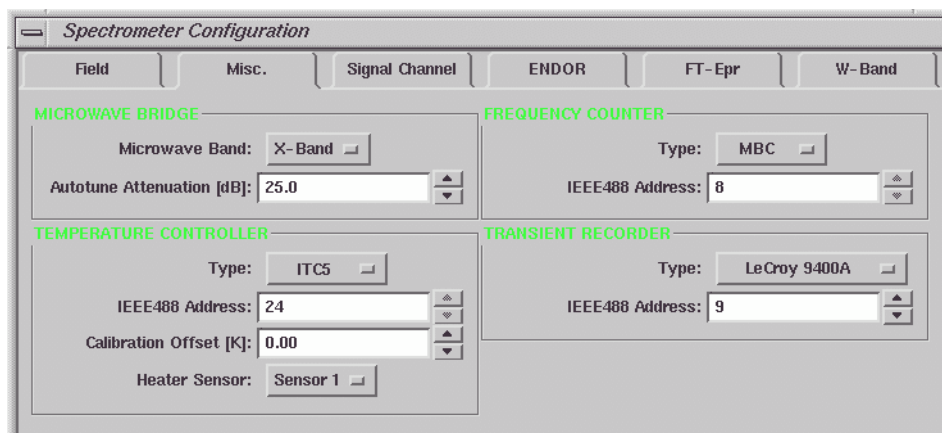


Figure 12-8 Configuring the ITC 503 temperature controller.

6. **Define an experiment that uses the ITC 503.** Create a new experiment with the Temp. Unit button activated (green). Click **Create** to close the dialog box. (See Figure 12-9.)

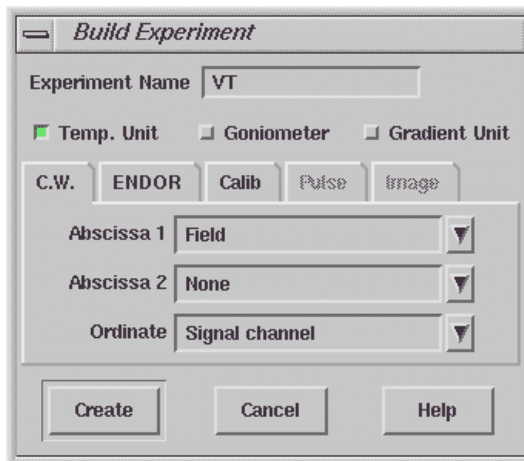


Figure 12-9 Defining an experiment using a temperature controller.

The Temperature Folder

12.3.2

If you click the **Acquisition Parameters** button you will find a new folder labeled VTU. Click the **Activate** button in the main application window to interactively control the ITC 503 unit. There are two sub-folders in the VTU folder. In the **Temperature** folder you find the following parameters:

Temperature [K]

Enter the set temperature here. The set temperature is the desired temperature for the experiment and hence the temperature the variable temperature accessory attempts to obtain and maintain.

Temp Settling Time [s]

You cannot conclude that just because the temperature sensor has attained your set temperature that your sample has attained this temperature as well. The sample temperature will change more slowly because of the larger thermal mass of the sample as well as any possible phase transitions. If you are performing a quick temperature survey, you can set this value to a few seconds. If you are performing a careful study such as a phase transition temperature determination, it is advisable to wait at least 10 min. if you are changing your set temperature by 10 K jumps. Enter the desired delay after the set temperature is reached here.

Tolerance

The Xepr EPR software waits until the set temperature is reached before it acquires an EPR spectrum. In physical measurements there is no such thing as exactly the same temperature. By entering a value for the tolerance, you specify how closely the measured temperature matches the set temperature before the Xepr software acquires an EPR spectrum.

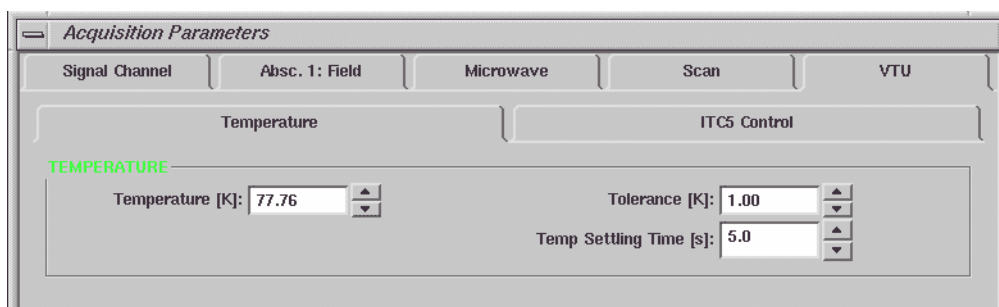


Figure 12-10 The Temperature folder.

The ITC5 Control Folder

12.3.3

In the ITC5 Control folder you can change many ITC 503 parameters remotely. Among your choices and options are:

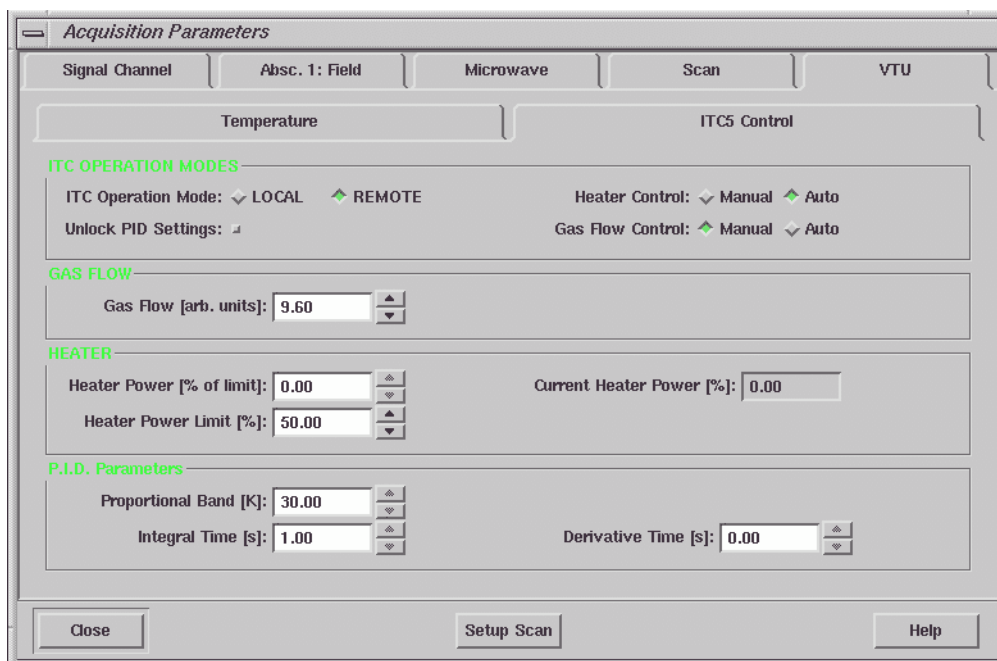


Figure 12-11 The ITC5 Control folder.

ITC Operation Mode

REMOTE specifies that the Xepr software remotely controls the temperature controller. If you must access controls on the front panel of the temperature controller manually, select LOCAL mode.

Unlock PID Settings

If this option is activated (green) you can change the parameters in the P.I.D. Parameters section. If inactive, the temperature controller operates in Auto PID mode.

-
- Heater Control** In Auto mode, the temperature controller stabilizes the temperature at the set temperature by controlling the Heater Power automatically. Under some unusual circumstances you may need to specify a constant Heater Power value. Select Manual mode to manually control the Heater Power.
- Gas Flow Control** If you have a transfer line with a motorized needle valve, the temperature controller can automatically control the cryogen flow when Auto mode is selected. Normally, you should select Manual mode to adjust the Gas Flow manually and let the heater stabilize the temperature first. Once a stable temperature with Auto Heater Control is reached, you can activate Auto Gas Flow Control. If you initially set both Gas Flow Control and Heater Control to Auto mode at the same time, the two regulation loops can fight each other, causing wild temperature oscillations.
- Gas Flow** If you selected Manual Gas Flow Control mode, you can change the needle valve position here. Zero corresponds to fully closed and 99.9 corresponds to fully open. Note that the flow does not vary linearly with needle valve position. If you selected Auto mode, the current needle valve position is displayed.
- Heater Power [% of limit]** If you selected Manual Heater Control mode, you can change the Heater Power here. Zero corresponds to no power and 100 corresponds to full power. If you selected Auto mode, the display is grayed out.
- Heater Power Limit[%]** The Heater Power Limit allows you to limit the amount of power applied to the heater. 100% corresponds to 40 V. This value should be set to 87.5% for all Helium variable temperature controllers in order to operate at the standard 35 V heater limit.
- Current Heater Power[%]** The Current Heater Power is displayed here.

Normally you should run experiments using **Auto PID** mode. Under some circumstances, you may need to change the PID values.

Proportional The **Proportional** term corresponds to the reciprocal of the feedback gain. Consult your cryostat data sheet in the cryostat manual for the appropriate value for your set temperature. You need to activate (green) the **Unlock PID Settings** button in **ITC Operation Modes** to make any changes in this parameter.

Integral The **Integral** term corresponds to the time constant of the feedback loop. Consult your cryostat data sheet in the cryostat manual for the appropriate value for your set temperature. You need to activate (green) the **Unlock PID Settings** button in **ITC Operation Modes** to make any changes in this parameter.

Derivative The **Derivative** term controls the response of the controller to rapid changes in temperature. Consult your cryostat data sheet in the cryostat manual for the appropriate value for your set temperature. You need to activate (green) the **Unlock PID Settings** button in **ITC Operation Modes** to make any changes in this parameter.

There is a **Temperature** box in the **Monitoring Panel** which displays the current temperature. It turns to green when the measured temperature reaches the set temperature within the specified tolerance. There are built-in 2D experiments available in the **New Experiments** for your variable temperature studies.

The E 033M Field Frequency Lock

12.4



This unit is an optional accessory and you may or may not have ordered it. If you require an accessory, contact your local Bruker EPR sales representative for purchase information.

If you have warmed up your spectrometer sufficiently, you can usually perform stable field sweep experiments. Sometimes the microwave frequency can drift owing to a number of external influences. For example, sample heating can occur at high microwave (or RF in ENDOR experiments) power levels. With very long data acquisitions, the room temperature and hence the cavity frequency can change. Frequency drift results in drift of the apparent field for resonance owing to the Zeeman effect.

This drift affects two different classes of experiments. In static magnetic field experiments such as ENDOR or time sweeps in which you set your magnetic field to a specific point in your EPR signal, you will drift away from that specific point, resulting in baseline distortions and changing intensities. In magnetic field sweep experiments requiring signal averaging of weak and particularly narrow signals, the frequency drift produces a broadening or smearing of the EPR lines.

The FF-Lock (Field-Frequency Lock) unit corrects for these drifts by locking to an external $g = 2$ EPR standard. As the microwave frequency changes, the FF-Lock adjusts the magnetic field to keep the EPR standard in resonance. In order to stabilize at fields where $g \neq 2$ or to perform field sweeps, there are a set of offset coils on the FF-Lock probe. By changing the magnetic field offset at the probe, we change the magnetic field value at which the FF-Lock locks the magnetic field. The FF-Lock has a total operating range of 120 Gauss, *i.e.* ± 60 G either side of $g=2.0$.

Installing an FF-Lock

12.4.1

If the FF-Lock is not currently installed in your console, you need to follow the instructions in this section.

1. **Install the FF-Lock probe head.** Follow the instructions of the FF-Lock manual to mount the FF-Lock probe head and connect the semi-rigid cable to the microwave bridge.
2. **Remove the blank panel.** Open the front door of the Eleksys console. Remove the blank panel under the microwave bridge controller of the console. There are two panels. You can use whichever one is available.
3. **Install the FF-Lock unit into the console.** Align the FF-Lock unit to the support brackets in the console and slide it in. Secure the unit with the mounting screws.
4. **Change the ER 032 T cable connections.** Open the back door of the console. There is a cable labeled ER 032 T connected to the 5 pin connector on the back of the field controller. (See the dotted arrow in Figure 12-12.) Disconnect the cable and reconnect it to the MAG. OUT 5 pin connector on the back of the FF-Lock. (See Figure 12-12.)

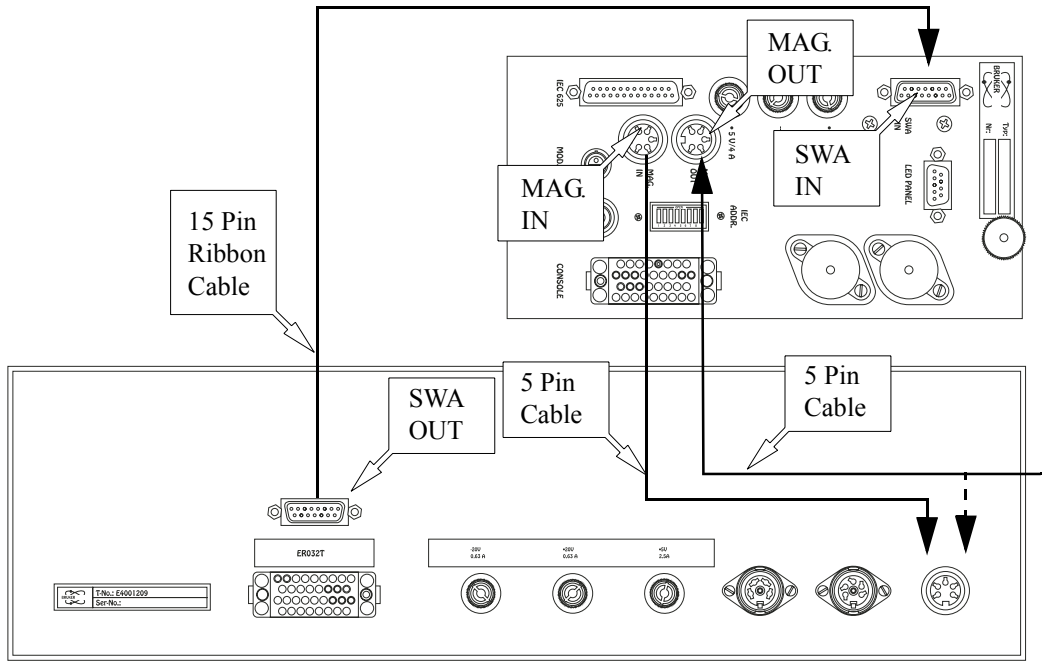


Figure 12-12 Back view of the FF-Lock (above) and field controller (below).

5. **Connect MAG. IN to the field controller.** Using another 5 pin cable connect the MAG. IN signal of the FF-Lock to the 5 pin connector on the back of the field controller where the ER 032 T cable was connected.
6. **Connect the sweep address cable.** Use the 15 pin ribbon cable to connect SWA OUT of the field controller to SWA IN of the FF-Lock. (See Figure 12-12.)

7. **Connect the power supply cable.** Locate the FF-Lock Burndy cable from the power rack on the back of the console. Connect it to the Burndy connector on the back of the FF-Lock. (See Figure 12-13.)

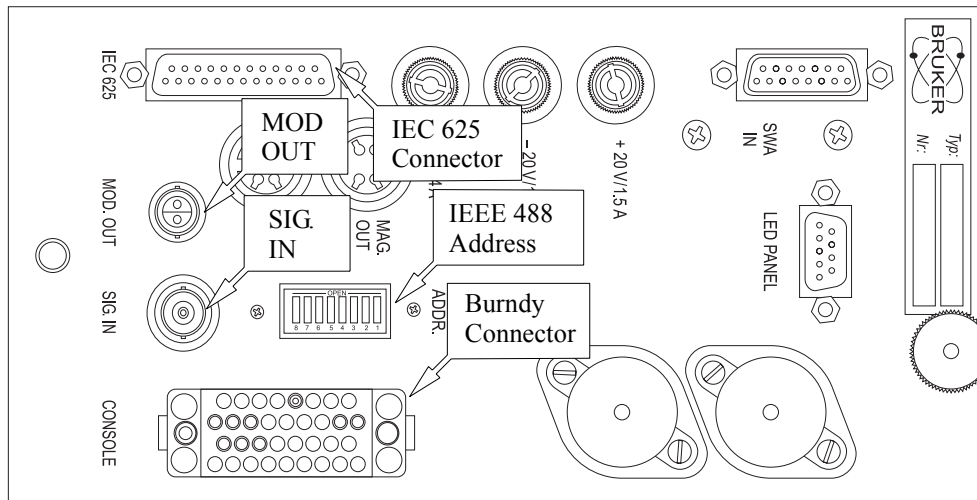


Figure 12-13 Back view of the FF-Lock.



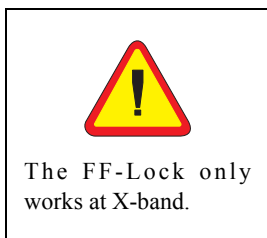
Consult the FF-Lock documentation for further details.

8. **Connect the IEC 625 cable.** There is an IEC 625 cable connected to the front of the GSI board of the acquisition server. Connect the other end to the IEC 625 connector on the back of the FF-Lock. (See Figure 12-13.)
9. **Connect the FF-Lock probe head.** There are two cables coming out of the FF-Lock probe head. The Modulation/Sweep cable (triax) needs to be connected to MOD OUT on the back of the FF-Lock. The Signal cable (coax) needs to be connected to SIG IN on the back of the FF-Lock. (See Figure 12-13.)

10. **Check the IEEE 488 address.** The default IEEE 488 address is three. The DIP switches for setting the address define a binary number. Therefore three corresponds to switches 1 and 2 on. Switches 6 and 7 should be on for proper communication. All other switches should be off. The switches are rocker switches and on corresponds to having the upper part pressed in. (See Figure 12-13.)
11. **Configure the FF-Lock.** Follow the instructions in Section 4.1 to configure the FF-Lock.

Using the FF-Lock in Lock-in Mode

12.4.2



The lock-in mode of the FF-Lock allows you to keep an EPR signal in resonance despite shifts in frequency. It also allows you to perform stable field-sweeps over very long periods of time in which the microwave frequency can drift.

1. **Get ready for using the FF-Lock.** Set the microwave bridge controller to Operate mode.
2. **Define a field sweep experiment.**
3. **Select the center field and sweep width.** As mentioned in the introduction the FF-Lock can lock over a ± 60 G range centered at $g = 2$. Set the center field value to a value that corresponds to $g = 2$ at your microwave frequency. Set the sweep width to less than 120 G. To ensure that we can lock the FF-Lock, set the current magnetic field to the center field value.
4. **Open the FF-Lock/Teslameter dialog box.** Click the FF-Lock/Teslameter button and open the dialog box. (See Figure 12-14.)

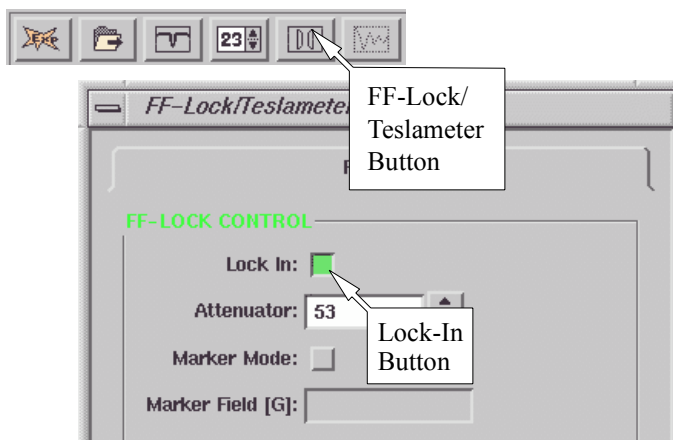


Figure 12-14 The FF-Lock dialog box.

5. **Click the Lock-In button.** In the FF-Lock/Teslameter dialog box click the Lock-In button. The FF-Lock will start to lock onto the current field. This procedure takes approximately 3-5 seconds and the FF-Lock box in the monitoring panel informs you of the progress. (See Figure 12-15.)

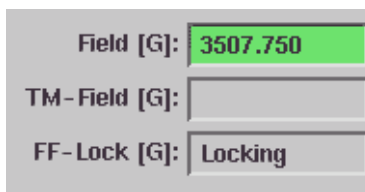


Figure 12-15 Locking the field.

During the locking process the FF-Lock finds the magnetic field offset (produced by the offset coils) required to bring the internal $g=2$ standard in resonance. This value appears in the FF-Lock [G] display of the monitoring panel. It corresponds to the difference between the present magnetic field and the field for resonance of the $g = 2$ standard. This value will change as you sweep the field. (See Figure 12-16.)

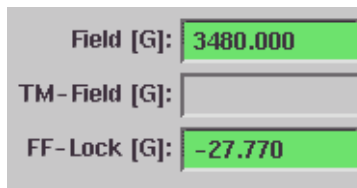


Figure 12-16 The magnetic field offset of the FF-Lock.

6. **Sweep the field with the FF-Lock.** When the Lock-In procedure has been successfully completed, the FF-Lock then takes over control of the field from the Hall field controller after you click the Run button. The FF-Lock now controls both the Center Field and the Sweep Width. Once the FF-Lock is locked, all acquisition parameters, including the field sweep parameters, may be changed without unlocking it first.

Troubleshooting Locking Problems

12.4.3

If the Lock-In procedure is unsuccessful after its fifth attempt, an error message appears in the FF-Lock window. (See Figure 12-17.)

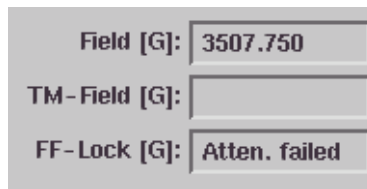


Figure 12-17 Failure to lock the field.

If this happens, check if:

- The magnetic field is in the locking range of the FF-Lock (± 60 G centered at $g = 2$).
- The microwave bridge is not in **STAND BY** mode.
- The SMA connectors on the semi-rigid microwave cable are sufficiently tight. (Do not over-tighten them!)

If for any reason the FF-Lock should lose its lock during an acquisition, the message “Lost Lock” will be displayed in the FF-Lock box of the monitoring panel. (See Figure 12-18.)



Figure 12-18 Lost lock.

Then the field control is automatically transferred back to the Hall field controller until the FF-Lock catches up of its own accord, or is locked in again. If this happens, the acquired data will have to be discarded.

The FF-Lock has a total operating range of 120 Gauss, *i.e.* ± 60 G either side of $g=2.0$. Hence if the field sweep is centered at $g=2.0$, the maximum sweep width is 120 G. The further away the center field is from $g=2.0$, the smaller the maximum possible sweep becomes. The maximum sweep width (in Gauss) is given by the formula:

$$SW_{Max} = 2 * (60 - |OF|) \quad [12-3]$$

where OF is the difference between the center field and the magnetic field where $g=2.0$. If the presently set sweep width and center field exceed this limit, an appropriate error message is displayed.

Marker mode

12.4.4

So far, we have used the FF-Lock in **Lock In** mode. The second mode for the FF-Lock is **Marker Mode**. What this mode does is to supply a marker on your acquired spectrum to indicate where the $g=2$ standard is in resonance. (You are marking where $g=2$ is in your spectrum.) This mode is useful for calibrating the field difference between the FF-Lock probe and the sample.

1. **Activate the Marker Mode.** Open the FF-Lock/Teslameter dialog box. Click the **Marker Mode** button. (See Figure 12-19.) The **Marker Field** box will show **Unknown**.

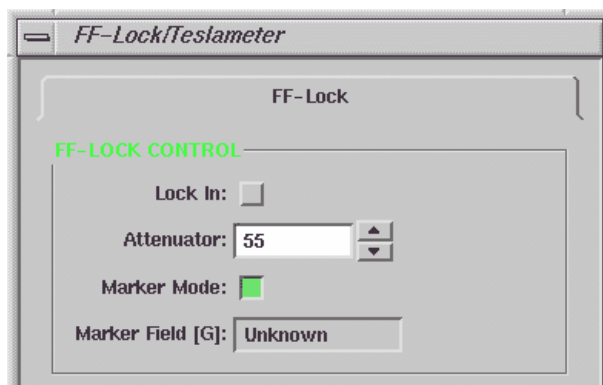


Figure 12-19 Activating the Marker Mode.

2. **Create an FF-Lock field Marker.** Click the X-Marker button to open the dialog box. In the X Parameter drop-down menu, select FFMarkerField. (See Figure 12-20.)

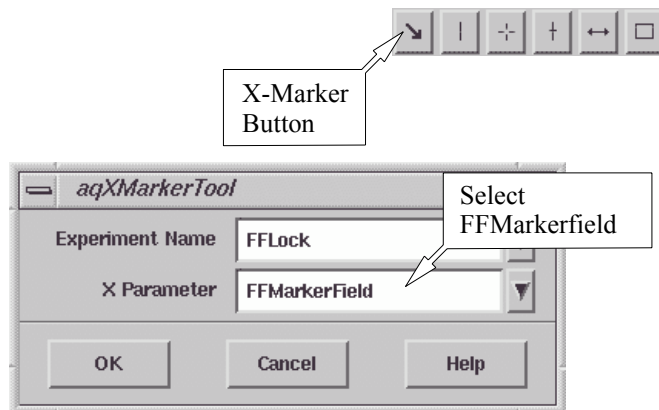


Figure 12-20 Creating an FF-Lock Marker.

3. **Sweep the field.** Click the Run button. After the field sweep finishes the FF-Lock Field Marker will move to the field for resonance of the $g=2$ standard in the FF-Lock probe. A more accurate field value can be found in the FF-Lock/Teslameter dialog box. (See Figure 12-21.)

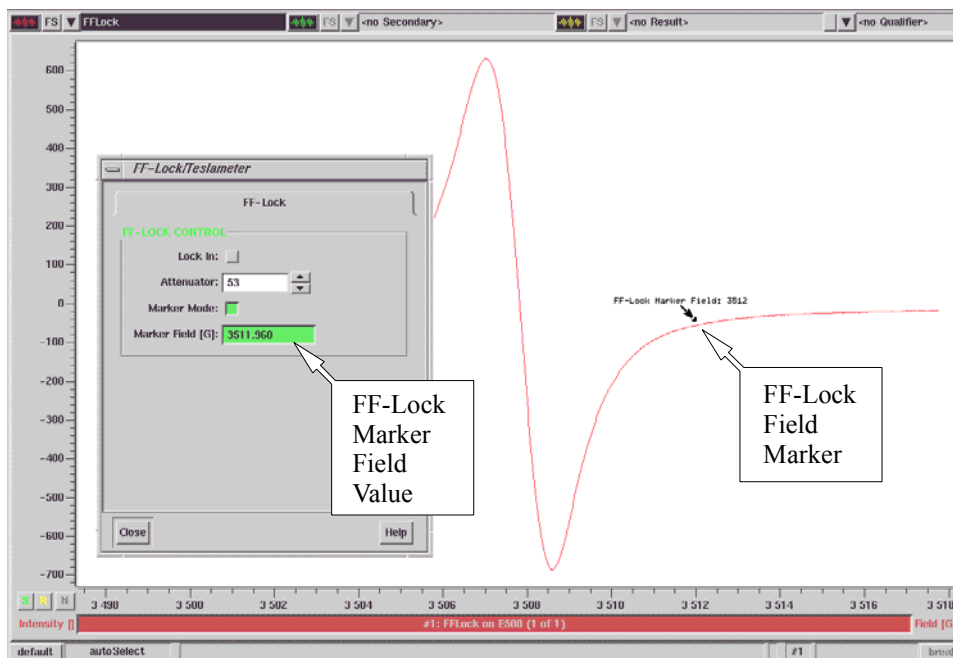


Figure 12-21 The FF-Lock Marker.

4. **Calibrate the field difference.** The FF-Lock probe and the sample are not physically in the same place in the magnet. Therefore there will be a field difference between the sample and FF-Lock probe. To calibrate this difference, insert your Bruker DPPH standard and follow instructions 1-3. Subtract the DPPH field for resonance from the marker field and record this difference.

$$\text{Calibration Offset} = B_{\text{marker}} - B_{\text{DPPH}} \quad [12-4]$$

5. **Enter the Calibration Offset.** Click Acquisition > Spectrometer Configuration. In the Field folder of the dialog box enter the value you measured in the previous step into the Calibration Offset box in the FF-Lock section. (See Figure 12-22.)

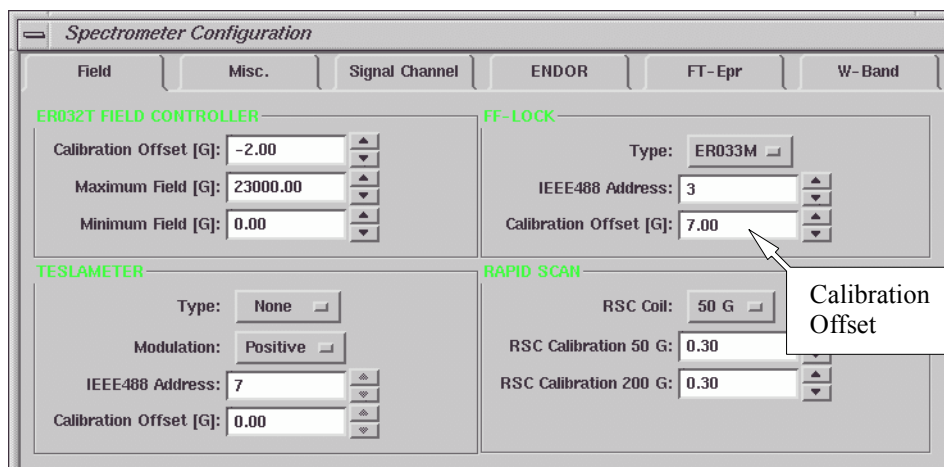


Figure 12-22 Changing the FF-Lock Marker position.

6. **Verify the Calibration Offset.** Reacquire the DPPH spectrum in Marker Mode. The marker position and the DPPH field for resonance should coincide.

Out of Locking Range Errors

12.4.5

If you set the field position or the Calibration Offset out of the locking range you will receive a “No Resonance” error message in the FF-Lock/Teslameter dialog box. (See Figure 12-23.) Reset the magnetic field position to remedy this error.

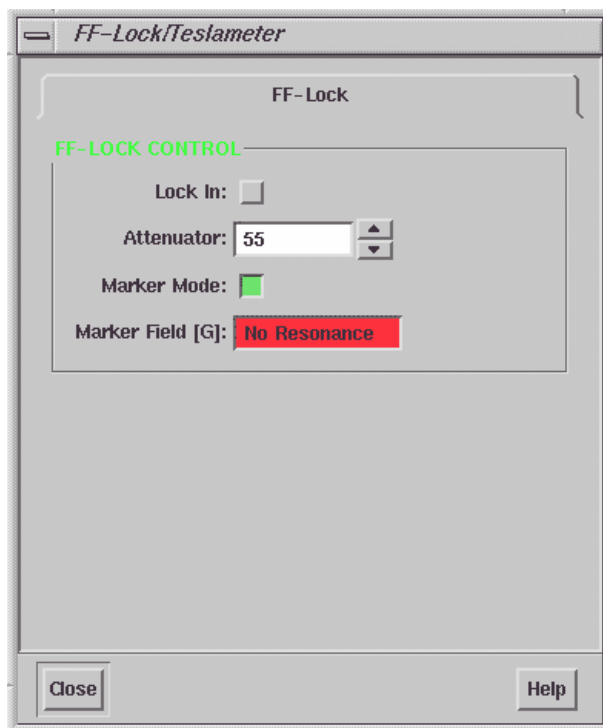


Figure 12-23 The No Resonance error message.

The E 035M Teslameter

12.5



This unit is an optional accessory and you may or may not have ordered it. If you require an accessory, contact your local Bruker EPR sales representative for purchase information.

The Bruker E 035M teslameter (gaussmeter) is designed for precisely measuring the magnetic field B_0 and uses the proton nuclear magnetic resonance frequency to measure the magnetic field. Since the resonant frequency can be measured precisely, the resulting resolution of the magnetic field value can be 1 milligauss. This section describes how to connect it to the Elexsys console as well as how to use it with the Xepr software.

Installing a Teslameter

12.5.1

1. **Mount the teslameter probe.** Follow the instructions of the teslameter manual to mount the teslameter probe. Select the probe that covers the field range at which you are working.
2. **Remove the blank panel.** Open the front door of the Elexsys console. Remove the blank dummy panel under the microwave bridge controller of the console. There are two panels. You can use whichever one is available.
3. **Install the teslameter unit into the console.** Align the teslameter unit to the support brackets in the console and slide it in. Secure the unit with the mounting screws.
4. **Connect the teslameter cable.** Use the connecting cable to connect the teslameter probe to the teslameter cable connector on the rear panel of the teslameter unit. (See Figure 12-24.)



Consult the teslameter documentation for further details.

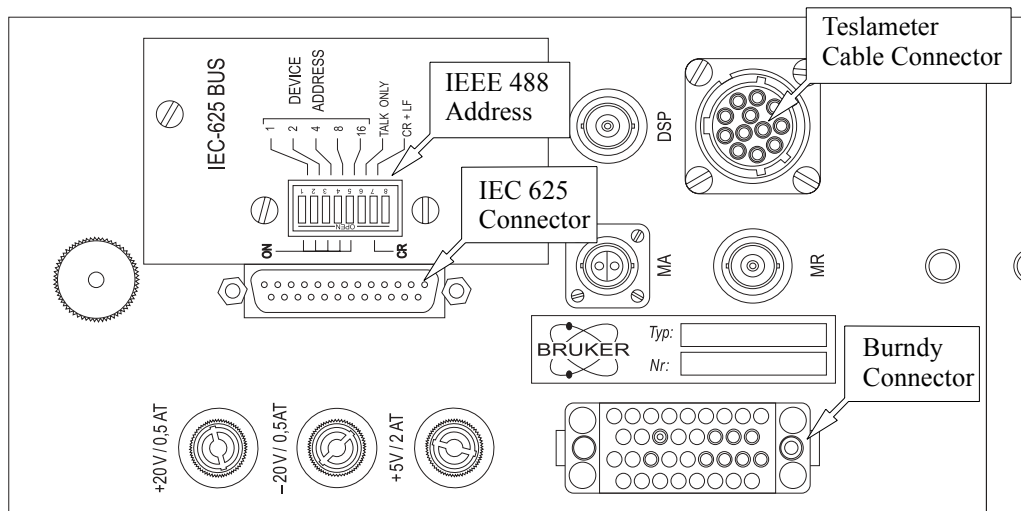


Figure 12-24 Rear view of the teslameter unit.

5. **Connect the power supply cable.** Locate the teslameter Burndy cable from the power rack on the back of the console. Connect it to the Burndy connector on the rear panel of the teslameter unit. (See Figure 12-24.)
6. **Connect the IEC 625 cable.** There is an IEC 625 cable connected to the front of the GSI board of the acquisition server. Connect the other end to the IEC 625 connector on the back of the teslameter. (See Figure 12-24.)
7. **Check the IEEE 488 address.** The default IEEE 488 address is seven. The DIP switches for setting the address define a binary number. Therefore seven corresponds to switches 1, 2 and 3 on. Switches 6 and 7 should be on for proper communication. All other switches should be off. The switches are rocker switches and on corresponds to having the lower part pressed in. (See Figure 12-24.)

8. **Configure the teslameter.** Follow the instructions in Section 4.1 to configure the teslameter.

Using the Teslameter

12.5.2

1. **Define a field sweep experiment.** Create a field sweep experiment. Before you activate the teslameter, acquire a spectrum. Center the spectrum and adjust the parameters.
2. **Open the FF-Lock/Teslameter dialog box.** Click the FF-Lock/Teslameter button in the experiment tool bar to open the dialog box. (See Figure 12-25.)

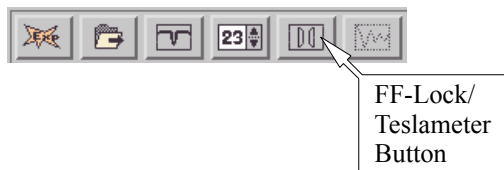


Figure 12-25 The FF-Lock/Teslameter button.

3. **Activate the teslameter.** Click the Standard Control tab. Select the Lock Resolution from the drop-down list. We recommend that you start with low resolution *e.g.* 0.1 G and increase the resolution after the teslameter finds the field. Click the Use Teslameter button to activate it. The teslameter will start to search for the current field position. (See Figure 12-26.)

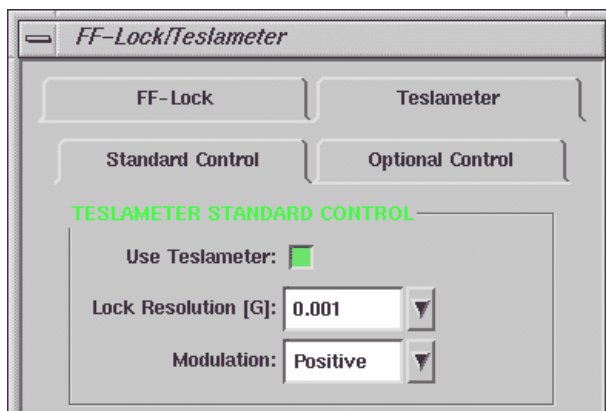


Figure 12-26 The Standard Control folder.

After the teslameter finds the field position it will display the field value in the TM Field box of the monitoring panel. (See Figure 12-27.)

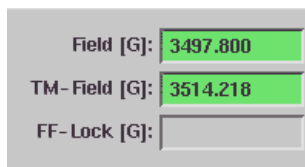


Figure 12-27 Display of the TM Field.

4. **Acquire a spectrum.** Click the Run button to start the field sweep.

The Standard Control Folder

12.5.3

The Standard Control folder contains the following parameters and options:

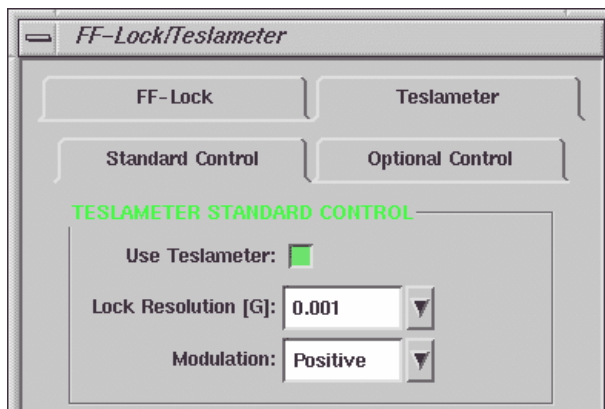


Figure 12-28 The Standard Control folder.

Use Teslameter Click this button to activate (green) the teslameter. Clicking again toggles it off.

Lock Resolution This parameter determines the precision of the field measurement. The choices are 0.1, 0.01, and 0.001 G. As the precision becomes finer, the measurement takes longer and hence requires slower field sweeps.

Modulation The teslameter probe has modulation coils to detect the proton NMR resonance. Depending on the polarity of the magnet or how you installed the teslameter probe, you may have to switch the polarity from positive to negative in order to lock the teslameter.

The Optional Control Folder

12.5.4

The Optional Control folder contains the following parameters and options:

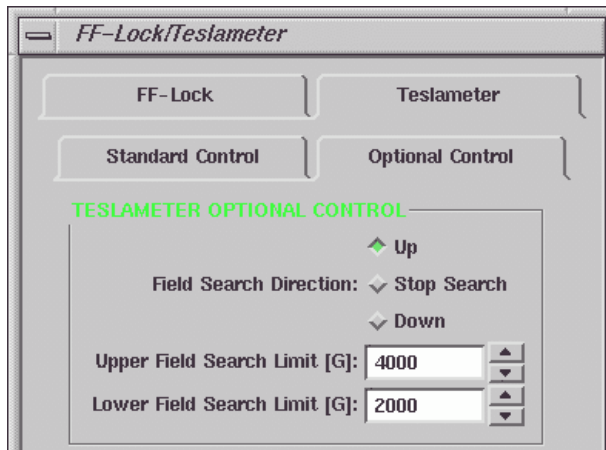


Figure 12-29 The Optional Control folder.

Field Search Direction

Before you can measure magnetic fields with the teslameter, the module must first find the proton resonance. In order to expedite the search for the signal, you can specify the direction of the search. For example, if you were originally at 3000 G and you have changed your magnetic field to 4000, you should choose **Up** as your search direction. Should you want to stop the search for the signal, click the **Stop Search** button.

Field Search Limit [G]

In order to find the proton resonance more quickly, you may narrow the search range. Enter appropriate values for **Upper Field Search Limit** and **Lower Field Search Limit**. Make sure that the search range covers the field your experiment defined and does not exceed the cover range of the probe you use. (See Figure 12-29.)

Calibrating the Field Offset

12.5.5

The teslameter measures the field value where the teslameter probe is located. It could be slightly different from the field that sample experiences because they are not physically in the same position. To correct the field value you can calibrate the teslameter by setting the **Calibration Offset**. There are two ways to calibrate the field. You can either use a field marker or the optional intra-cavity probe (E 035M-1009).

- **Calibrate the field offset using the intra-cavity probe.** Follow the intra-cavity probe user's manual for installation and operation. Record the intra-cavity probe (B_{sample}) and teslameter probe ($B_{\text{teslameter}}$) readings.
- **Calibrate the field offset with a field marker.** Insert a field marker sample to the cavity. Tune and match the cavity. Calculate the field value (B_{sample}) by using the known g-factor of the field marker and the microwave frequency.

$$B_{\text{sample}} = 714.4775 \cdot \frac{\nu}{g} . \quad [12-5]$$

Record this value as well as the field value for the g-value standard read by the teslameter ($B_{\text{teslameter}}$).

The Calibration Offset equals:

$$\text{Calibration Offset} = B_{\text{sample}} - B_{\text{teslameter}} , \quad [12-6]$$

where B_{sample} is the field measured with the intra-cavity probe or g-value standard and $B_{\text{teslameter}}$ is the field read by the teslameter. In the **Teslameter** section of the **Field** folder enter the difference into the **Calibration Offset**. Click **Apply** and close the **Spectrometer Configuration** dialog box.

Open the **Spectrometer Configuration** dialog box.

If you calibrate the field offset of the field controller at the same time and enter the difference in the **Field Offset** in the **Spectrometer Configuration** dialog box, you may use the center field tool with the teslameter activated.

Troubleshooting

12.5.6

If the teslameter cannot find the field position or it keeps searching for a long time the modulation may have the wrong phase. Deactivate the teslameter by clicking the **Stop Search** button in the **Optional Control** folder. Select **Negative** from the **Modulation** drop-down list in the **Standard Control** folder. Press the **Use Teslameter** button again.

The field values of spectra acquired with the teslameter are the teslameter's readings. You may have problems using the acquisition tools such as the **Center Field** tool or the **Center Field & Sweep Width** tool. This is because the tool's readings are those of the teslameter's. What is actually required by the field controller to center the spectrum are the values measured by the hall probe. To solve this problem you can either set the center field before activating the teslameter or calibrate both the teslameter and the **Field Offset** of the field controller. Refer to Chapter 5 and the previous section for the calibration process.

The ER 218PG1 Goniometer

12.6



This unit is an optional accessory and you may or may not have ordered it. If you require an accessory, contact your local Bruker EPR sales representative for purchase information.

A goniometer is a necessity when the EPR signal of a sample is orientationally dependent such as single crystals or other ordered samples. Xepr can control the programmable goniometer from the software. This section provides you step-by-step instructions on how to use a Bruker programmable goniometer with the Eleksys E 500 spectrometer.

Installing a Programmable Goniometer

12.6.1

Please refer to the Programmable Goniometer ER 218PG1 User's Manual for how to install a goniometer. There are two cables you must connect to the Eleksys console.

1. **Connect the Slow Device Bus cable.** Open the front door of the console. Connect the **Slow Device Bus** connector (25 pin) on the goniometer electronic box mounted on the magnet to the SLD connector on the SIP board of the acquisition server using a 25 pin Slow Device Bus cable. (See Figure 12-30.)
2. **Connect the power supply cable.** Locate the Burndy cable labeled **Goniometer** from the power rack on the back of the console. You may have to open the back of the console to locate the cable. Connect it to the power supply Burndy cable of the goniometer control unit

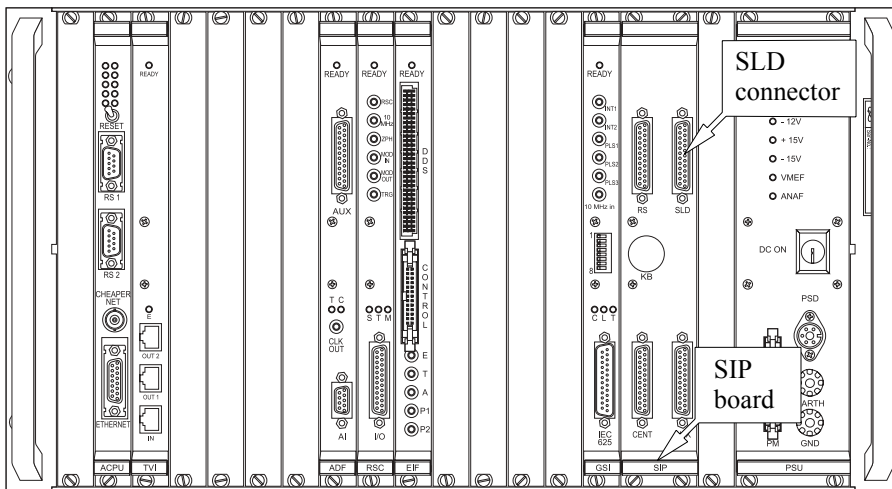


Figure 12-30 Connecting the control cable.

Configuring the Goniometer

12.6.2

1. **Define a goniometer experiment.** Open the New Experiment dialog box. Click the Goniometer button to activate it. If you want to acquire a 2D spectra you can select Sample angle (Goniometer) in the Abscissa 2 drop-down list. Click Create to close the dialog box. (See Figure 12-31.)

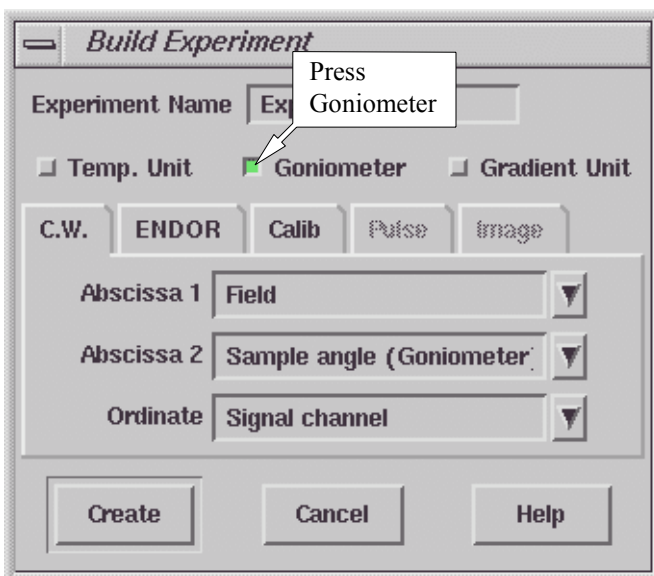


Figure 12-31 Defining a goniometer experiment.

2. **Open the Acquisition Parameters dialog box.**
Click the Goniometer tab. (See Figure 12-32.)

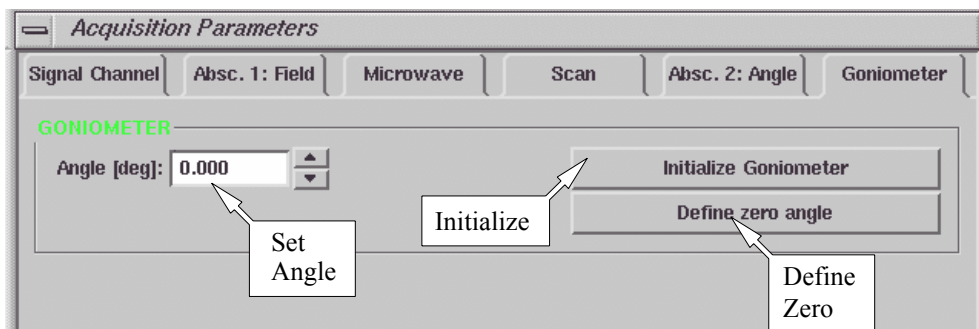


Figure 12-32 The Goniometer folder in the Acquisition Parameter dialog box.

3. **Initialize the goniometer.** Make sure the shaft is in place. Click the **Activate** button. Click the **Initialize Goniometer** button in the **Goniometer** folder. The software will search for the 0 notch on the goniometer and initialize that point as the “home” point. (See Figure 12-32.) Meanwhile the angle display in the monitoring panel will turn from red to green. (See Figure 12-33.)



Figure 12-33 The goniometer angle display.

4. **Define the zero angle.** You can define any angle to be the new zero, regardless of the present goniometer angle. Click the **Define Zero Angle** button to set the current sample angle as the new zero.
5. **Set the angle.** Enter a value in the **Angle** box. The goniometer will turn to that angle.

6. **Set up for a 2D experiment.** If you create a 2D experiment you can set the angle range and angle increment in the Absc. 2: Angle folder. (See Figure 12-34.)

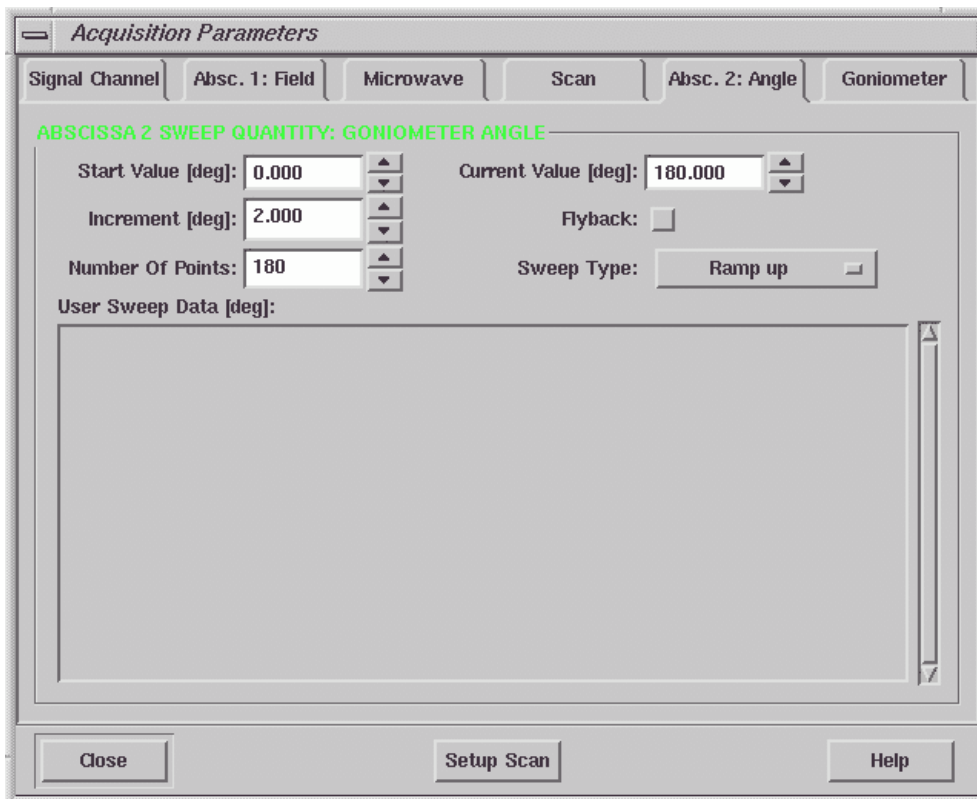


Figure 12-34 Setup for a 2D spectra using the programmable goniometer.

Select Each Slice Scan for Acq Fine Tuning parameter in the Microwave folder. The acquisition software will perform a fine tune before acquiring each slice of the 2D spectrum. This option is particularly important because changing the orientation of the sample with a programma-

ble goniometer causes the frequency and coupling to change.

- 7. **Example.** Figure 12-35 shows an example 2D spectra of a ruby crystal. The angle increment is 1 degree. It is displayed as a contour plot.

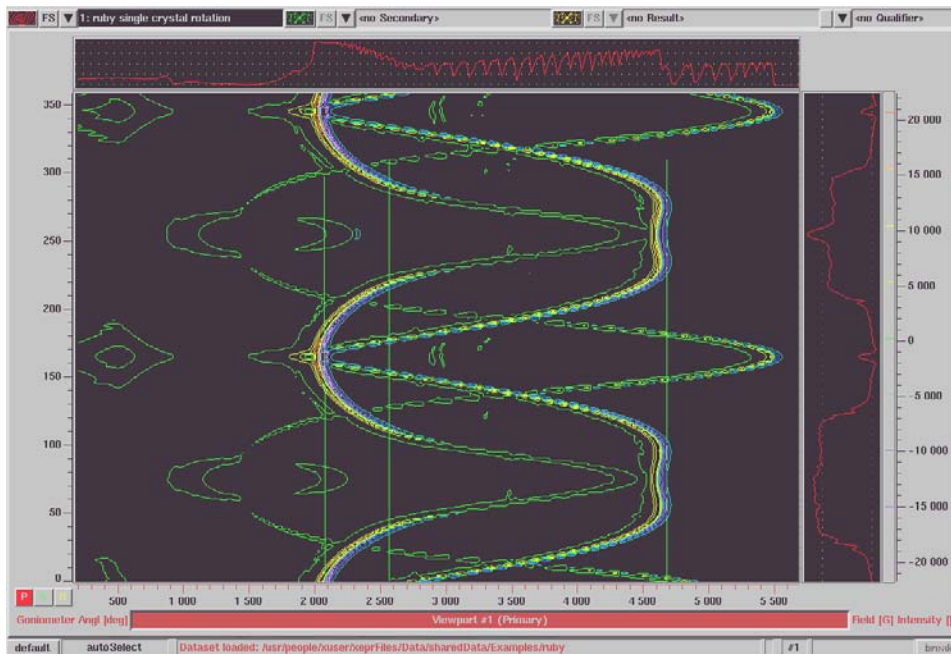


Figure 12-35 Road map of a ruby crystal using the programmable goniometer.

This chapter describes the connectors on the front panel of many of the modules and boards in the console. It also guides you through configuring the signal paths using the software.

Console Module Arrangement

13.1

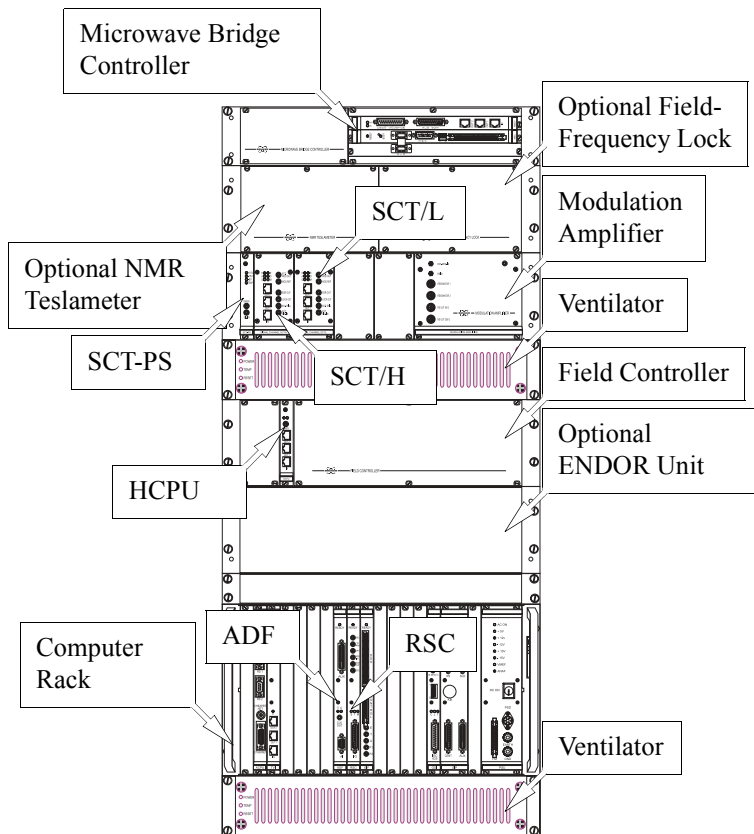


Figure 13-1 Console module arrangement.

Connectors and Indicators in a Nutshell 13.2

This section lists in table format the important information about the connectors and LED indicators of the console modules.

SCT Connectors

13.2.1

Connector	Module	Description	Enabled	Signal
SWADV	SCT-PS	sweep advance signal for Hall field controller	always	TTL output
SIG. IN	SCT-PS	signal input for all signal channels (routed via the backplane)	Signal I/O: Signal Input: Internal	analog input 1 V _{pp} max. 50 Ω
EXT. MOD OUT	SCT/H or L	modulation output signal	Signal I/O: Modulation Output: External	analog output 0-5 V _{pp} sine wave into 50 Ω
MOD REF	SCT/H or L	modulation reference signal	always	digital output ±5V square wave
ESR OUT	SCT/H or L	EPR signal after lock-in, time constant, and offset adjustment	always	analog output 3 V full scale
LOCK OUT	SCT/H or L	EPR signal after lock-in, but before time constant and offset adjustment	always	analog output 150 mV full scale into 50 Ω
EXT. SIG. IN	SCT/H or L	signal input for one signal channel	Signal I/O: Signal Input: External	analog input 1 V _{pp} max. 50 Ω
EXT. TRG ^a	SCT/H or L	external experiment trigger	Signal I/O: External Trigger: On	TTL input

Table 13-1 Connector definitions and descriptions for the SCT modules.

Connector	Module	Description	Enabled	Signal
EXT. TRG ^b	SCT/H or L	external lock-in frequency	Signal I/O: External Lock In: On	TTL input
EXT. MOD. IN	Mod.Amplifier	external modulation input	Signal I/O: Modulation Input: External	analog input 10 V _{pp} max.
RS IN	Mod.Amplifier	rapid scan input	Rapid Scan experiment	analog input ramp 5 V _{pp} max.
RESONA- TOR 1	Mod.Amplifier	1st resonator modulation output	Signal I/O: Resonator: 1	analog output 300 V _{pp} sine wave max.
RESONA- TOR 2	Mod.Amplifier	2nd resonator modulation output	Signal I/O: Resonator: 2	analog output 300 V _{pp} sine wave max.
RS OUT 50 G	Mod.Amplifier	50 G rapid scan coil output	Spectrometer Configurati on: Field: RSC Coil: 50 G & Rapid Scan experiment	analog output 30 V _{pp} ramp max.
RS OUT 200 G	Mod.Amplifier	200 G rapid scan coil output	Spectrometer Configurati on: Field: RSC Coil: 200 G & Rapid Scan experiment	analog output 30 V _{pp} ramp max.

Table 13-1 Connector definitions and descriptions for the SCT modules.

SCT LED Indicators**13.2.2**

Indicator	Module	Color	Indication
+15 V	SCT-PS	green	+15 V present
-15 V	SCT-PS	green	-15 V present
+5 V	SCT-PS	green	+5 V present
TEMP	SCT-PS	red	temperature fault
R	SCT/H or L	green	transputer software loaded
A	SCT/H or L	green	unit is acquiring spectra
Q	SCT/H or L	green	quadrature detection
E	SCT/H or L	red	transputer error
O	SCT/H or L	red	AC overflow
M	SCT/H or L	red	modulation overload

Table 13-2 Indicator definitions and descriptions for the SCT modules.

HCPU Hall Field Controller LED Indicators**13.2.3**

Green LED	Red LED	Indication
off	off	ER032T is not initialized.
on	off	ER032T is operating correctly.
on	on	An input error has occurred. The red LED remains active until the error is cleared by a correct entry. Both LEDs also remain active after booting if no calibration file has been read. The calibration file is read after you have connected to the instrument.
off	on	Software has crashed. Rebooting is required.
on	blinking	A field regulation fault has occurred. The ER032T cannot attain the desired field value.
blinking	off	A temperature regulation fault has occurred. This occurs momentarily during a normal booting process.
blinking	blinking	Both field and temperature regulation faults have occurred.
blinking	on	Both an input error and temperature regulation fault have occurred. The input error indicator (red LED on) will overwrite a field regulation fault indicator (red LED blinking).

Table 13-3 Indicator definitions and descriptions for the Hall field controller

Transputer Connectors

13.2.4

Each module contains one input and two output connectors. In order for a unit to work, it must have at least one cable connected to the input. The output connectors are used for buffering and fanning out the transputer signals to other units. The chain of cables starts at the OUT connectors of the Transputer board next to the Acquisition Server. The black end of the Transputer cable connects to OUT and the red end connects to IN.

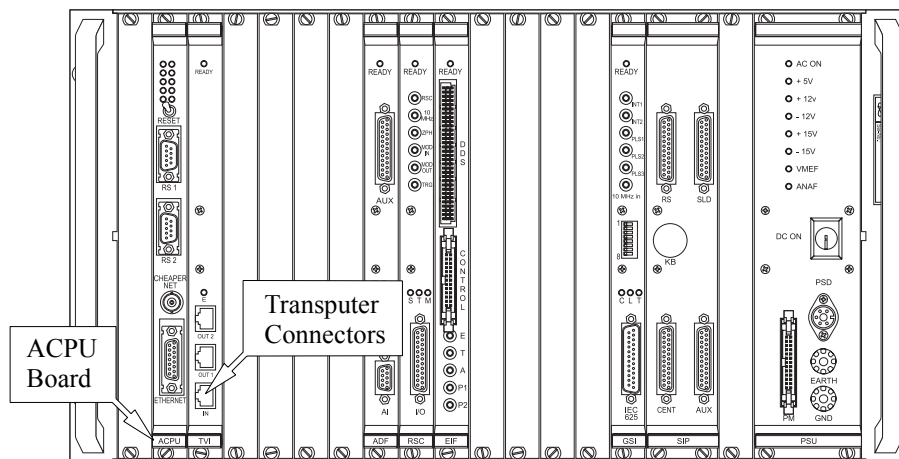


Figure 13-2 The ACPU (Acquisition CPU) rack.

IN **The input connector.** This is the input connector for the transputer cable.

OUT1 & OUT2 **The output connectors.** These are the output connectors for transfer of transputer communications to other units.

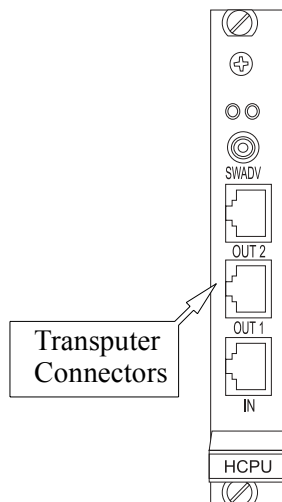


Figure 13-3 The Transputer connectors of the HCPU board.

A typical transputer cable configuration is shown in Figure 13-4.

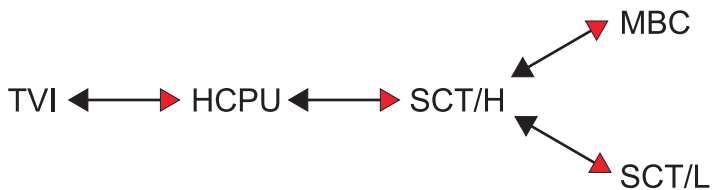


Figure 13-4 Transputer cable configuration for an E 500 spectrometer.

SCT-PS

13.3

The SCT-PS signal channel power supply module has one input, one output, and four LED indicators for the voltages required by the signal channel modules.

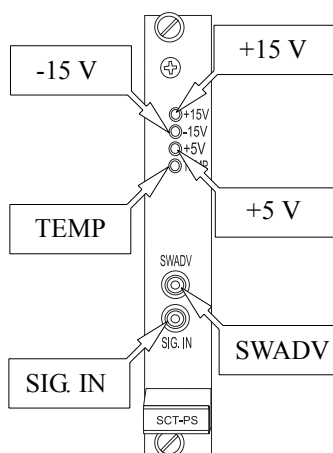


Figure 13-5 The SCT-PS (signal channel power supply) module.

SCT-PS Connectors

13.3.1

SWADV **The sweep advance output.** This TTL signal synchronizes the digitization of the EPR signal with the magnetic field sweep. The signal first goes from the SCT-PS board to the RSC rapid scan board and then further on to this connector on the Hall field controller. These connections are seldom changed.

SIG. IN **The signal input.** The signal from the bridge supplied to this connector is distributed through the back plane of the rack to the individual signal channel modules.

SCT-PS Indicators

13.3.2

- +15 V** **The +15 V indicator.** Normally this indicator is green, meaning +15 volts is supplied to the signal channel.
- 15 V** **The -15 V indicator.** When this indicator is green, -15 volts is supplied to the signal channel.
- +5 V** **The +5 V indicator.** When this indicator is green, +5 volts is supplied to the signal channel.
- TEMP** **The temperature fault indicator.** Under normal conditions this indicator is off. If this indicator turns to red a temperature fault exists. The console may be overheated. Check if the fans on the back panel are on. Check the filters of the ventilator units and clean them if they are dirty. If all else fails, contact your local Bruker EPR service representative for help.

SCT

13.4

There are two types: the high frequency signal channel (SCT/H) and the low frequency signal channel (SCT/L). The connectors and indicators arrangements are identical in both.

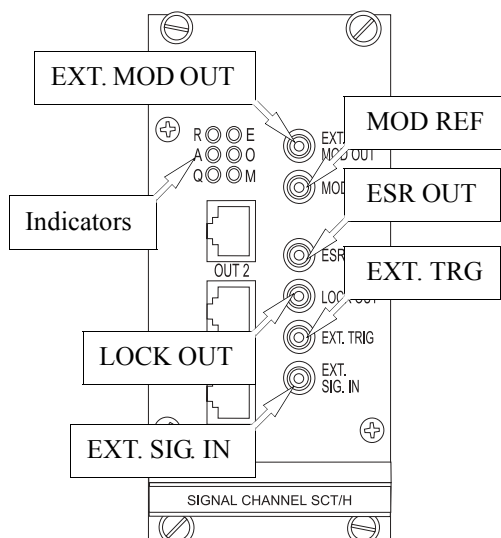


Figure 13-6 The signal channel module.

SCT Connectors

13.4.1

EXT. MOD. OUT **The external modulation output connector.** This connector provides an analog modulation output signal. It is a 0-5 V_{pp} sine wave into 50 Ω. To activate the output, select External for Parameters > Signal Channel > Signal I/O > Modulation Output.

MOD. REF. **The modulation reference output connector.** This connector provides a digital modulation reference output. It is a

± 5 V square wave signal with a frequency equal to the modulation frequency. It is always enabled.

ESR OUT **The EPR signal output connector.** This is an analog EPR signal output. Full scale corresponds to 3 V into 50 Ω . The signal is obtained after the lock-in amplifier, time constant filter, and DC offset adjustment and just before the digitizer. It is always enabled.

LOCK OUT **The unfiltered EPR output connector.** This is also an analog EPR signal output. Full scale corresponds to 150 mV into 50 Ω . LOCK OUT differs from ESR OUT in that the signal is obtained after the lock-in amplifier but before the time constant filter and the DC offset adjustment. It is always enabled. It is most often used in double modulation experiments.

EXT. SIG. IN **The external signal input connector.** Normally, the signal channel obtains its signal from the SIG. IN connector of the SCT-PS via the back plane of the rack. This connector is designed for external signal input into individual signal channels. It accepts a 1 V_{pp} maximum analog signal and the impedance is 50 ohm. To enable external signal input, select **External** for **Parameters > Signal Channel > Signal I/O > Signal Input**.

EXT. TRG **The external trigger input connector.** The signal applied to this connector can perform two different and mutually exclusive functions. The first function of the connector is as an external trigger input to start an acquisition. It requires a TTL level signal (> 2V) to trigger the acquisition. To enable the external trigger function, click the **External Trigger** button (until it is green or active) in **Parameters > Signal Channel > Signal I/O**.

The second function for this input is as an external modulation reference for demodulating the EPR signal. It requires a TTL level square wave signal (> 2V) with the same frequency as the

modulation frequency. To enable the external modulation reference function, click the **External Lock In** button (until it is green or active) in **Parameters > Signal Channel > Signal I/O**.

Transputer Connectors

See Section 13.2.4.

SCT Indicators

13.4.2

- R The ready LED.** The LED blinks when the transputer software is loading. It remains a steady green when the signal channel is ready for acquiring a signal. If this led is not on after you turn on the console there may be a problem with booting the signal channel transputer.
- A The acquisition LED.** When an acquisition with this signal channel is in progress, this LED is lit.
- Q The quadrature LED.** When a quadrature detection acquisition is in progress, this LED is lit, indicating that two signal channels are acquiring in parallel.
- E The error LED.** It indicates a transputer error when lit.
- O The overload LED.** This indicator lights when there is an AC overflow. Turn down the receiver gain, microwave power, or reduce the AFC gain if this occurs.
- M The modulation overload LED.** This indicator lights when the modulation amplifier is overdriven. Turn down the modulation amplitude if this occurs.

Modulation Amplifier

13.5

The Modulation Amplifier module has two SMB input connectors and four twin-ax output connectors.

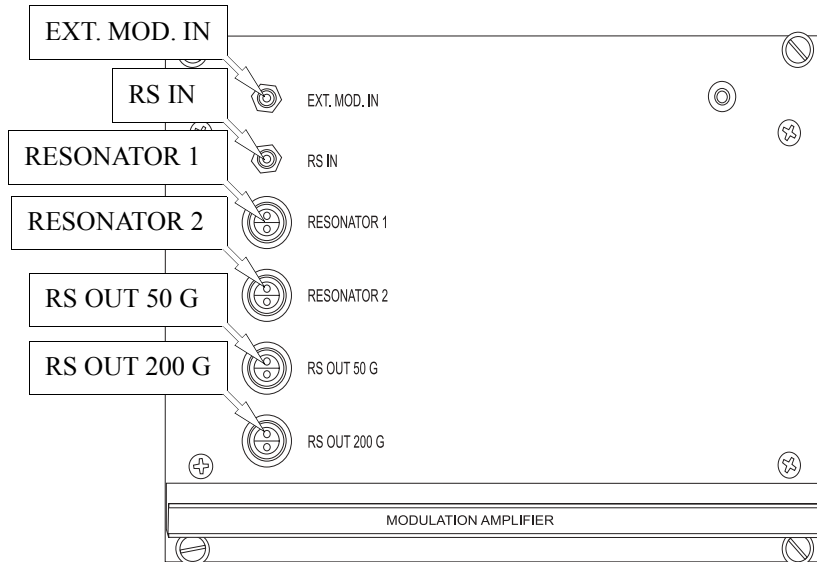


Figure 13-7 The Modulation Amplifier.

Modulation Amplifier Connectors

13.5.1

EXT. MOD. IN **The external modulation input connector.** This connector is for input of an external (*i.e.* not from either signal channel) modulation signal. It accepts an analog input with up to 10 V_{pp}. In order to enable external modulation (and disable internal modulation), select External for Parameters > Signal Channel > Signal I/O > Modulation Input.

- RS IN** **The rapid scan input connector.** This connector is for input of the rapid scan ramp. It accepts an analog input of a maximum 5 volt ramp. It is enabled whenever a rapid scan experiment is active. The RSC cable is normally attached to this connector.
- RESONATOR 1** **The first modulation output connector.** This connector provides the modulation output for the first resonator. It is an analog sine wave output supplying up to 300 V_{pp}. It is activated by selecting 1 for Parameters > Signal Channel > Signal I/O > Resonator.
- RESONATOR 2** **The second modulation output connector.** This connector is similar to RESONATOR 1 except it is for RESONATOR 2 and is used for the second set of modulation coils for the ER 4114DR dual resonator. The two outputs cannot be used simultaneously. To enable RESONATOR 2, select 2 for Parameters > Signal Channel > Signal I/O > Resonator.
- RS OUT 50 G** **The 50 G rapid scan output connector.** This is the rapid scan ramp output for the 50 G rapid scan coil. It supplies an analog ramp output with up to 30 V_{pp}. To activate it, create a rapid scan experiment and select 50 G for Acquisition > Spectrometer Configuration > Field > RSC Coil.
- RS OUT 200 G** **The 200 G rapid scan output connector.** This is the rapid scan ramp output for the 200 G rapid scan coil. It supplies an analog ramp output with up to 30 V_{pp}. To activate it, create a rapid scan experiment and select 200 G for Acquisition > Spectrometer Configuration > Field > RSC Coil.

HCPU

13.6

The HCPU Hall controller power unit has two LEDs, the SWADV connector, and three transputer connectors.

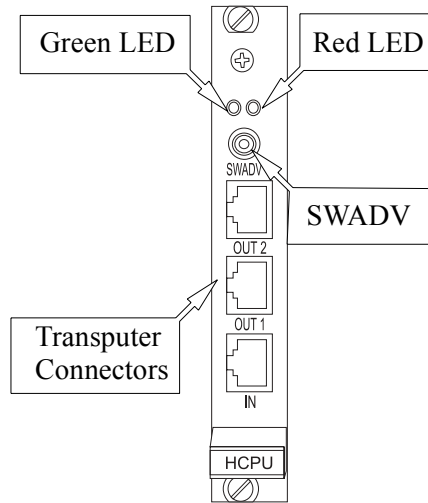


Figure 13-8 The HCPU.

HCPU Indicators

13.6.1

Green & Red LED **The status indicator LEDs.** The meaning of the green and red LEDs is somewhat complicated. Refer to Table 13-3 for complete definitions.

HCPU Connectors

13.6.2

SWADV **The sweep address advance input.** This TTL signal synchronizes the digitization of the EPR signal with the magnetic field sweep. The signal first goes from the SCT-PS board to the

RSC rapid scan board and then further on to this connector on the Hall field controller. These connections are seldom changed.

**Transputer
Connectors**

See Section 13.2.4.

ADF

13.7

The ADF is a fast digitizer board. It has three LED indicators and three connectors.

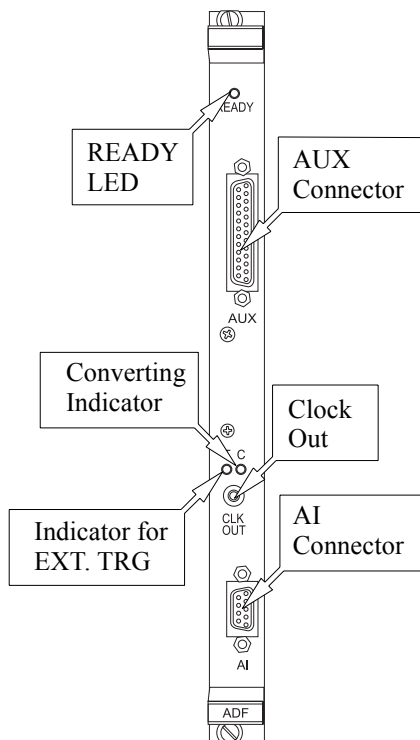


Figure 13-9 The ADF board.

ADF Indicators

13.7.1

READY **The ready LED.** This green LED indicates that the ADF board is ready. When the console is turned on this LED should be lit.

- T The external trigger LED.** When this yellow LED is on it indicates that the ADF is using an external trigger.
- C The converting LED.** This indicates that the ADF is digitizing data.

ADF Connectors

13.7.2

CLK OUT The clock out connector. This connector supplies the TTL clock signal from the ADF.

AUX The auxiliary connector. This is a 25 pin connector. The pin out is shown in Figure 13-10. Most of the pins are specifically reserved for spectrometer operation and are not available for general use. The pins available to users are highlighted in Figure 13-10. The most important is pin 5, the external trigger input. There is a cable labeled TRIGGER connected to this pin. You can use it to externally trigger the ADF with a TTL level pulse. To enable the external trigger, select **External Sweep** for **Parameters > Fast Digitizer > Trigger Mode**. In the **Trigger Slope** drop-down menu in the same menu, you must also select whether the rising or falling edge triggers the acquisition. If your spectrometer is equipped with an optional ENDOR unit, the Trigger cable should be connected to the T connector of the EIF board.

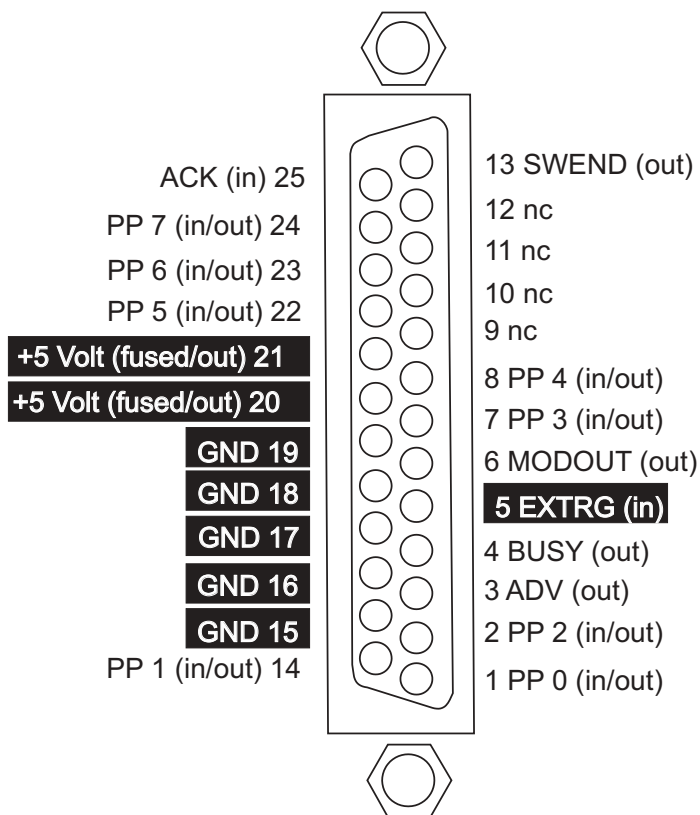


Figure 13-10 The ADF board AUX connector pin layout.

AI The analog input connector. The AI connector has two differential input pairs for the signal to be digitized. The first pair is reserved for digitizing the EPR signal. It connects to the ESR OUT connector of the signal channel in the standard configuration. It is referred to as **Channel A**. The second pair can be used to digitize other signals. There is a cable connected to the second channel (**Channel B**) labeled as ADF ENDOR. If you have an

optional ENDOR unit, it is connected to the ADF connector of the DICE box. The center conductor is the non-inverting input and the shield is the inverting input. The impedance of the inputs is $200\ \Omega$. The voltage range is $\pm 2.5\ \text{V}$ full scale. For a pin-out see Figure 13-11.

There are options for digitizing either Channel A or B or both channels. For a rapid field scan experiment, select A, B or A&B for Parameters > Field > Rapid Scan Options > Input Mode. For a rapid time sweep, select the appropriate values for Parameters > Fast Digitizer > Input Mode.

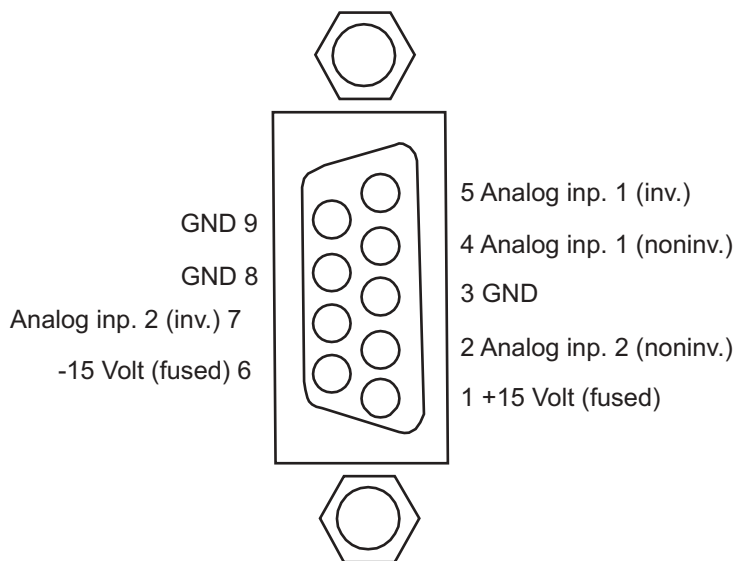


Figure 13-11 The ADF board AI connector pin layout.

RSC

13.8

The RSC (Rapid Scan Controller) board is essentially a programmable ramp generator. It supplies the signal that is amplified by the modulation amplifier. This board is used in conjunction with the ADF board to acquire field sweep signals rapidly. It has four indicators and seven connectors.

RSC Indicators

13.8.1

- READY** **The ready LED.** This LED is the ready indicator for the RSC board. It is lit after the first time you initiate the rapid scan function.
- S** **The sweep LED.** This LED indicates a rapid field sweep is active.
- T** **The trigger LED.** This LED indicates the RSC board is ready for a trigger.
- M** **The modulation overload LED.** This LED indicates that the modulation amplifier (that supplies the current to the rapid scan coils) is overloaded.

RSC Connectors

13.8.2

- RSC** **The rapid scan output.** This is the rapid scan voltage output. It is an analog signal ranging ± 1 V into 50Ω and drives the modulation amplifier through its RS connector.
- 10 MHz** **The external clock input.** This connector allows you to input an external 10 MHz TTL external clock signal.
- ZPH** **The zero phase output.** This connector supplies a $50 \mu\text{s}$ negative TTL pulse upon the start of the rapid scan ramp. This pulse is useful for synchronizing other events with the sweep.

- MOD IN** **The modulation input.** An external modulation signal supplied here simply goes out the MOD OUT connector.
- MOD OUT** **The modulation output.** The signal supplied to the MOD IN connector comes out of this connector.
- TRG** **The trigger input.** The RSC board can be triggered by an external signal (generated by an external event such as a laser pulse) applied to this input. The board is triggered by the falling edge of a TTL level signal. (Note that this can be inverted by using jumper J8 on the RSC board.)
- I/O** **The input/output connector.** This connector is reserved for use by Bruker.

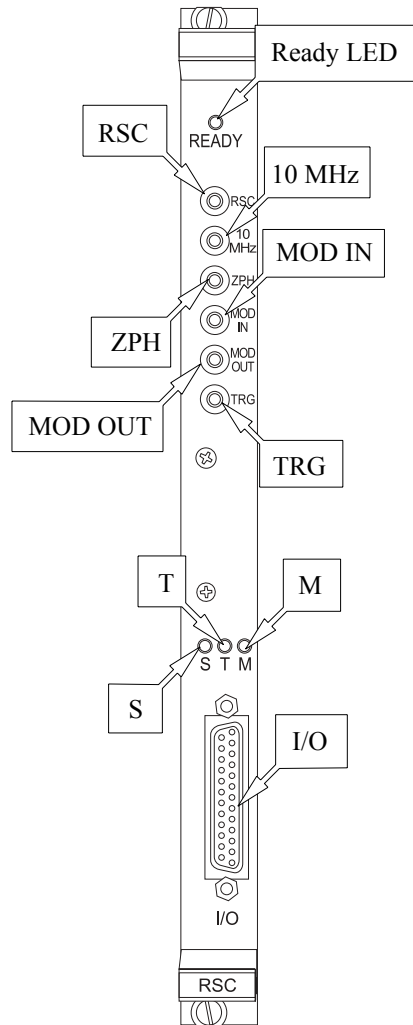


Figure 13-12 The RSC board.

A standard Bruker Elexsys EPR spectrometer is equipped with a high frequency signal channel (6-100 kHz), low frequency signal channel (100-10,000 Hz), and rapid digitizer (ADF) for performing routine field/time scan experiments. By rerouting the signal paths using the inputs and outputs of the front panels of the modules and selecting options in the software, many custom experiments such as double modulation can be implemented. The previous chapter describes the connections, indicators, and parameters available to you for configuring your experiments. This chapter presents how to set up the experiments. Standard Experiments describes configuring experiments that do not require any wiring changes or extra components. Custom Experiments describes experiments that need extra wiring or devices. Figure 14-1 shows the symbols and figure legends used in this chapter.

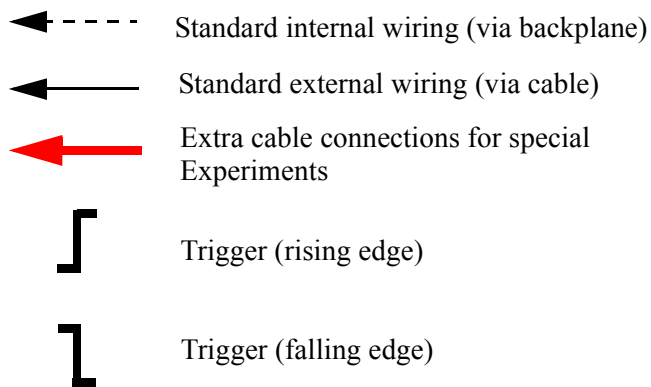


Figure 14-1 A key to the symbol and figure legends.

Standard Experiments

14.1

Field or Time Sweeps with the Signal Channel

14.1.1

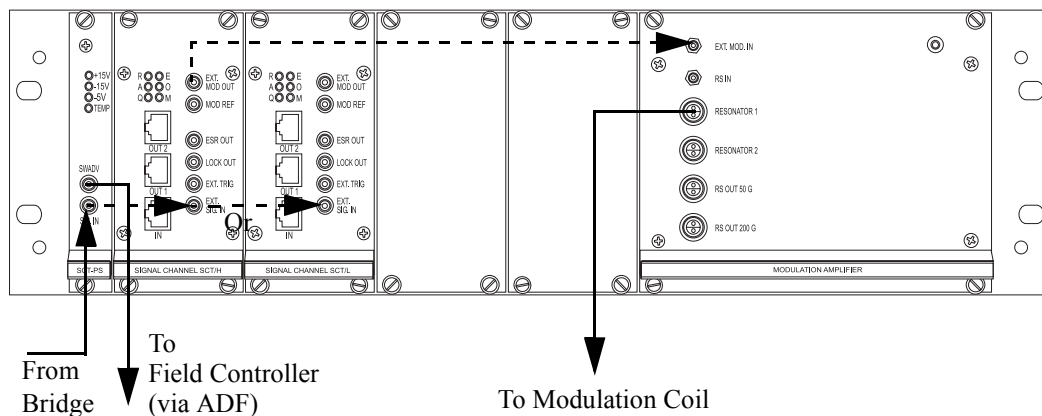


Figure 14-2 Configuration for field or time sweeps with the SCT/H or SCT/L signal channels.

These are the most basic CW EPR experiments. The Elexsys E 500 User's Manual: Basic Operations gives a complete description of these experiments. All connections either through internal back connections or through external cables are standard and ready for use in the default configuration of the spectrometer. Remember, if you use an ER 4105DR double resonator, you need to connect both the Resonator 1 and Resonator 2 cables from the Modulation Amplifier unit to the double resonator.

For modulation frequencies between 100 and 6 kHz, the SCT/H (high frequency) signal channel is automatically selected by the software. If you choose a modulation frequency between 6 kHz and 100 Hz, the SCT/L (low frequency) signal channel is automatically selected.

Field or Time Sweeps with the Fast Digitizer (ADF) 14.1.2

When the acquisition is shorter than 5 seconds, you should use the fast digitizer (ADF) to acquire a field or time scan instead of the signal channel digitizer. A complete description of this experiment is given in Section 6.1 of this manual. All connections either through internal back connections or through external cables are standard and ready for use in the default configuration of the spectrometer.

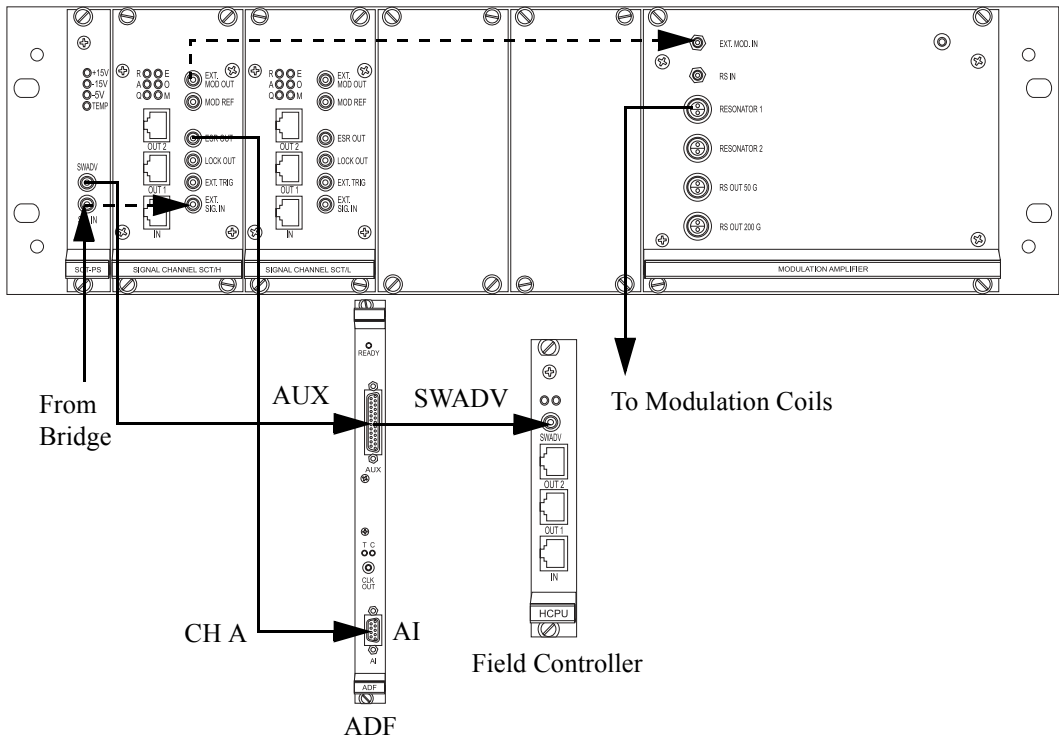


Figure 14-3 Configuration for field or time sweeps with the ADF.

Rapid Field Scans with the Fast Digitizer (ADF)

14.1.3

The combination of the ADF (fast digitizer) and RSC (Rapid Scan Controller) with rapid sweep coils provides a tool to acquire spectra of short lived species. A complete description of this experiment is given in Section 6.2 of this manual. All connections either through internal back connections or through external cables are standard and ready for use in the default configuration of the spectrometer.

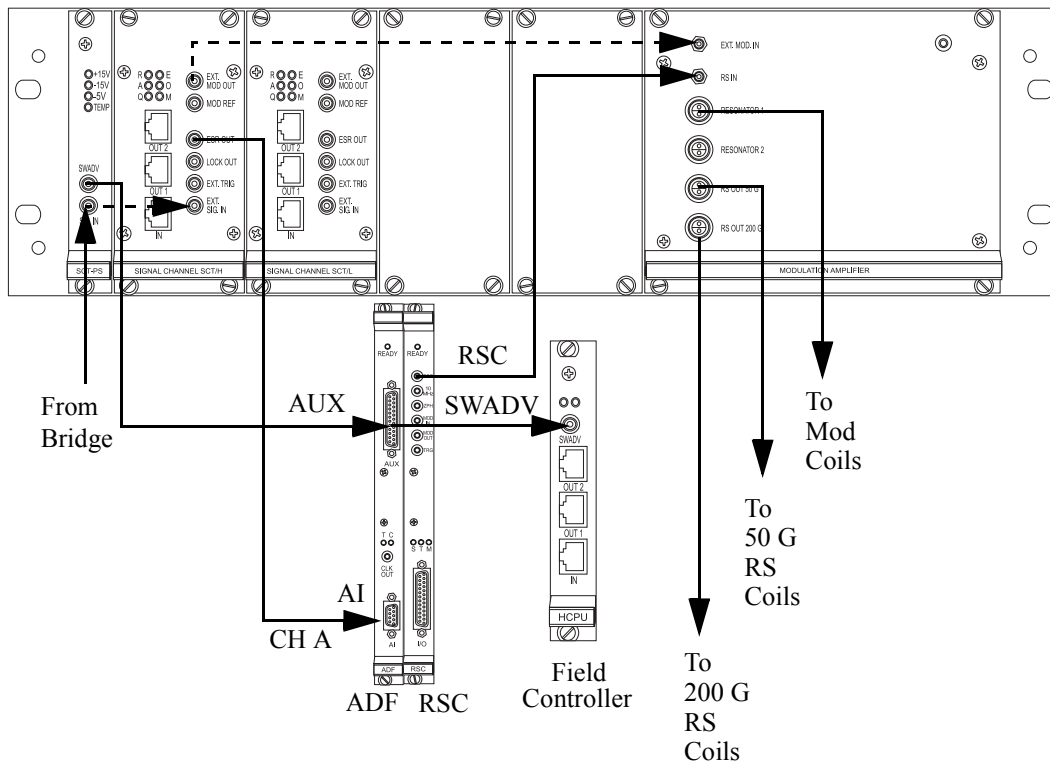


Figure 14-4 Configuration for rapid field scans with the ADF.

Custom Experiments

14.2

This section provides instructions on configuring custom experiments. You need to attach external cable(s), have special optional Bruker equipment, or supply your own external equipment to set these experiments up.

Quadrature Detection

14.2.1

Quadrature detection simply means acquiring both the in-phase and phase-quadrature components of the field modulated EPR signal. This detection scheme proves very useful in saturation-transfer experiments. The acquisition requires two signal channels with a 90° mutual phase difference to acquire the EPR signal. Most consoles are delivered with one SCT/H (high frequency) and one SCT/L (low frequency) signal channel. In order to perform this experiment, you must purchase a second SCT/H (or SCT/L) signal channel.

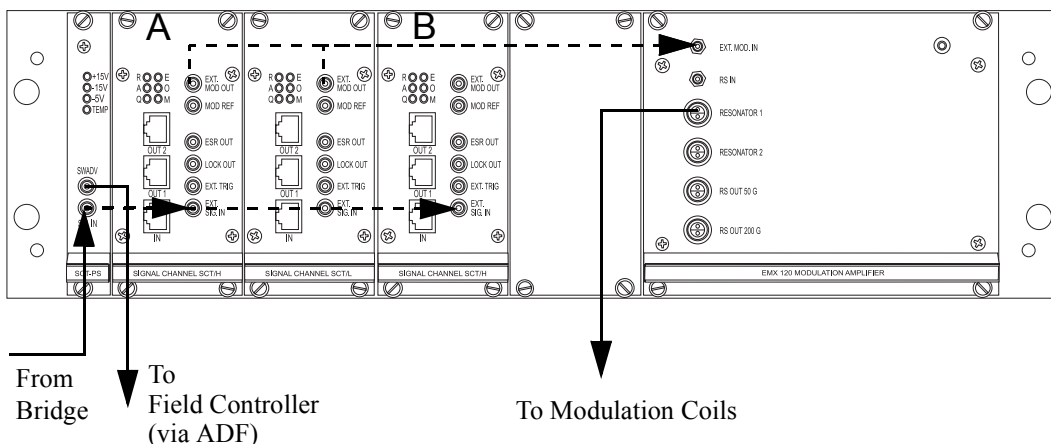


Figure 14-5 Configuration for quadrature detection.



These instructions are for modulation frequencies greater than 6 kHz. For lower frequencies, simply substitute SCT/L for SCT/H.

It is assumed that you have already created a standard field or time sweep experiment.

1. **Install the second signal channel.** Turn off the spectrometer. Install a second SCT/H (high frequency) signal channel, B, next to the SCT/L (low frequency) signal channel. Connect a transputer cable from OUT2 of signal channel A to IN on signal channel B. (See Figure 14-5.) Turn on the console and connect the workstation to the spectrometer.
2. **Set up for quadrature detection.** Open the Parameter dialog box and select the Signal Channel folder. Click the Detection tab and press the Quadrature Detection button. (See Figure 14-6.) Press the Activate button.

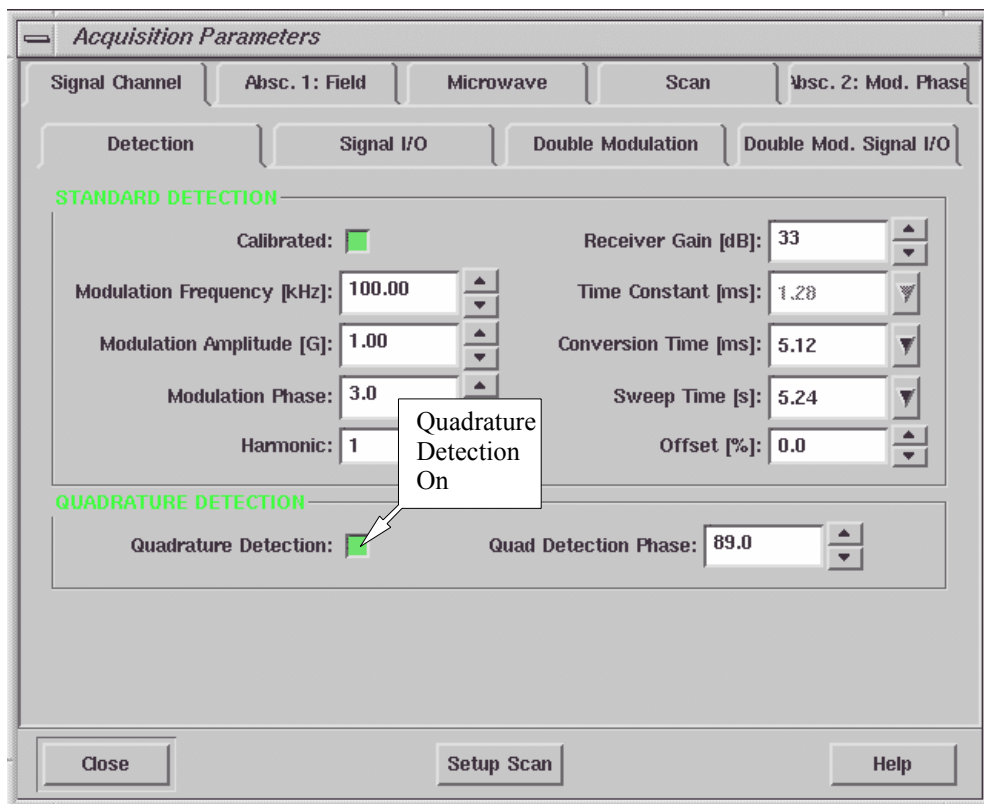


Figure 14-6 The Quadrature Detection button.



Pressing the <Ctrl> key and clicking the up or down arrow key changes the phase by 90°.

3. **Check the phase of signal channel A.** Although the phase of the signal channel A has been calibrated during the signal channel calibration it still could be a few degrees off. Moving a modulation cable can produce phase shifts of a few degrees. Use a strong sample such as DPPH to check the phase. Change the **Modulation Phase** from 0° to 90° in the **Signal Channel Detection** folder. Adjust the **Modulation Phase** to minimize the signal. It is convenient to monitor the signal with the **Setup Scan** feature. When the signal is minimized, shift the **Modulation Phase** by -90° to maximize the signal.



Figure 14-7 Calibrating the phase of signal channel A.



When you calibrate the phase difference, use low microwave power to avoid saturating the EPR signal.

4. **Calibrate the phase of signal channel B.** The phase of the two signal channels might be slightly different. Therefore the phase difference may not be exactly 90° when you set the Quad Detection Phase to 90° . To calibrate the difference, set the Quad Detection Phase to 90° . Check if the signal from signal channel B is minimized. Adjust the Quad Detection Phase until the signal from signal channel B is minimized. Using the **Setup Scan** function helps you to interactively optimize the phase. You need to select **A&B** in the **Setup Scan SCT** drop-down list to view signals from both signal channels simultaneously.

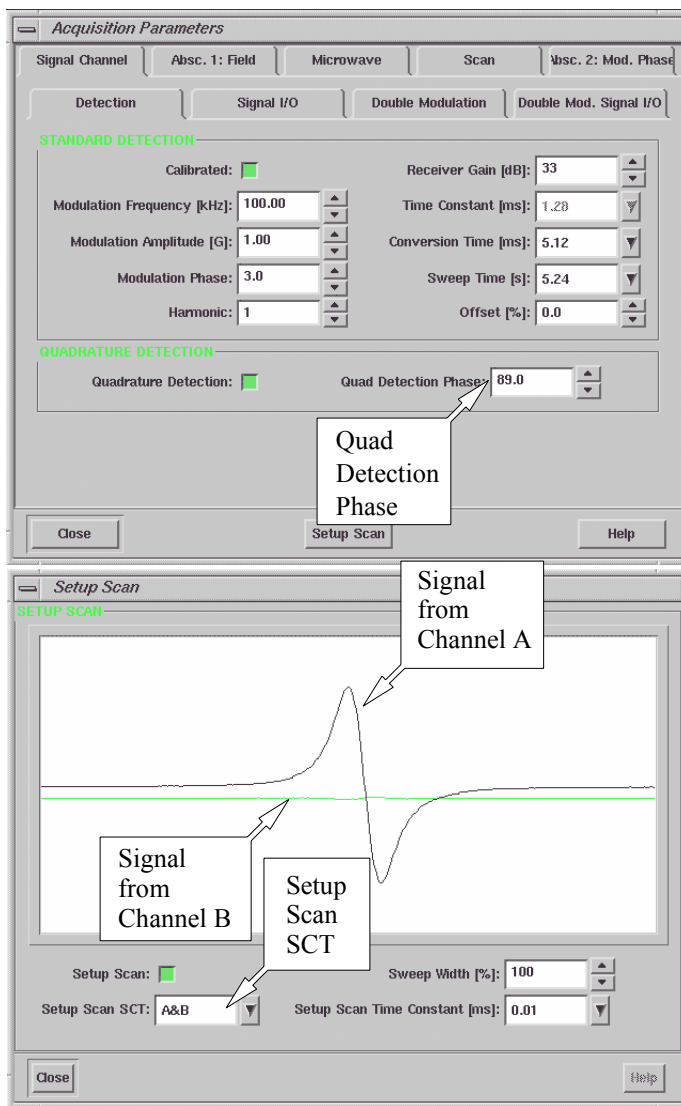


Figure 14-8 Calibrating the phase of signal channel B.

5. **Acquire a quadrature spectrum.** Click the Run button to acquire a quadrature spectrum. The Q LED indicators on both signal channels should light up.
6. **View the signals from both channels.** The resultant spectrum is complex in which the real part is the signal detected by the signal channel A and the imaginary part is the signal detected by the signal channel B. Click the Re/Im button in the Tool bar to display the two signals respectively. (See Figure 14-9.)

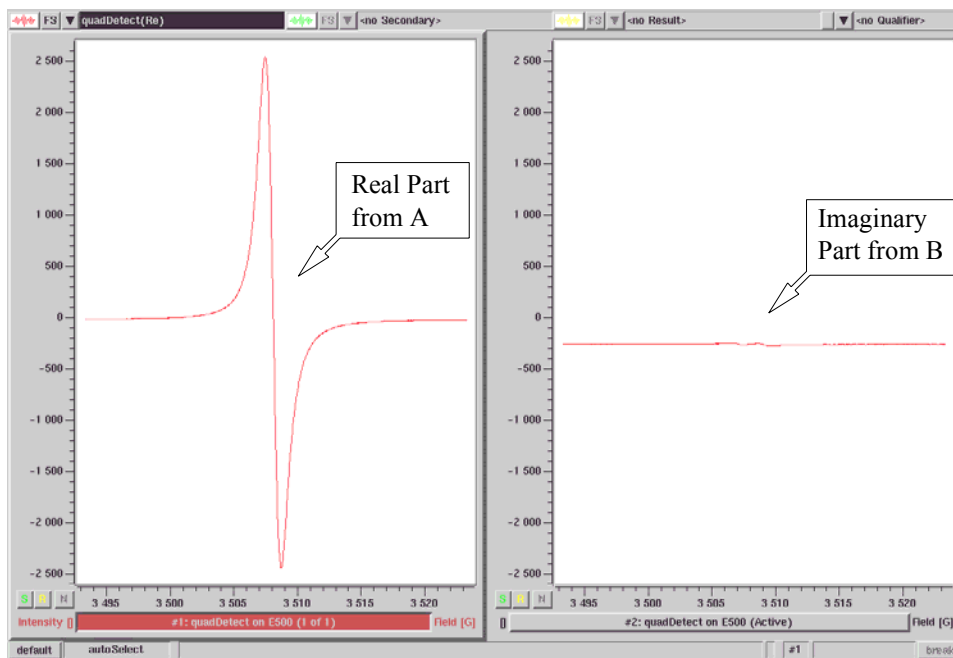


Figure 14-9 Viewing the signals from both signal channels.

Externally Triggered Sweeps with the Signal Channel 14.2.2

If you need to synchronize an external event such as a lamp flash with your EPR acquisition, you can trigger the acquisition externally. This section describes how to trigger a field or time sweep using the signal channel. It is assumed that you have already created a standard field or time sweep experiment.

1. **Provide an external trigger.** Connect an external cable carrying a TTL (5 volt) positive-going trigger pulse to the EXT. TRIG connector of the signal channel. Take care about the impedance, *i.e.* make sure the trigger voltage is high enough after the cable is connected. Also note that the acquisition is triggered by a rising slope.

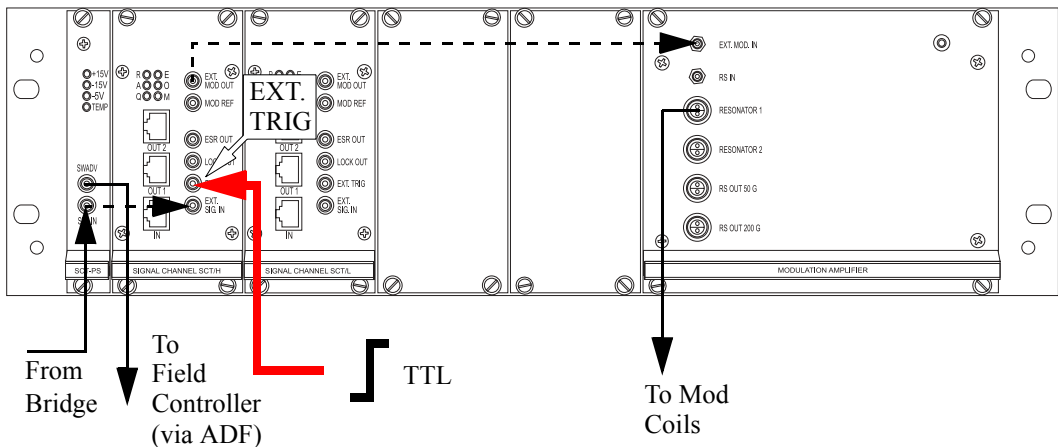


Figure 14-10 Configuration for externally triggered sweeps with the signal channel.

2. **Enable the external trigger in the software.** Open the Parameter dialog box and click the Signal Channel tab. Click the Signal I/O tab and then the External Trigger button to activate it (green).

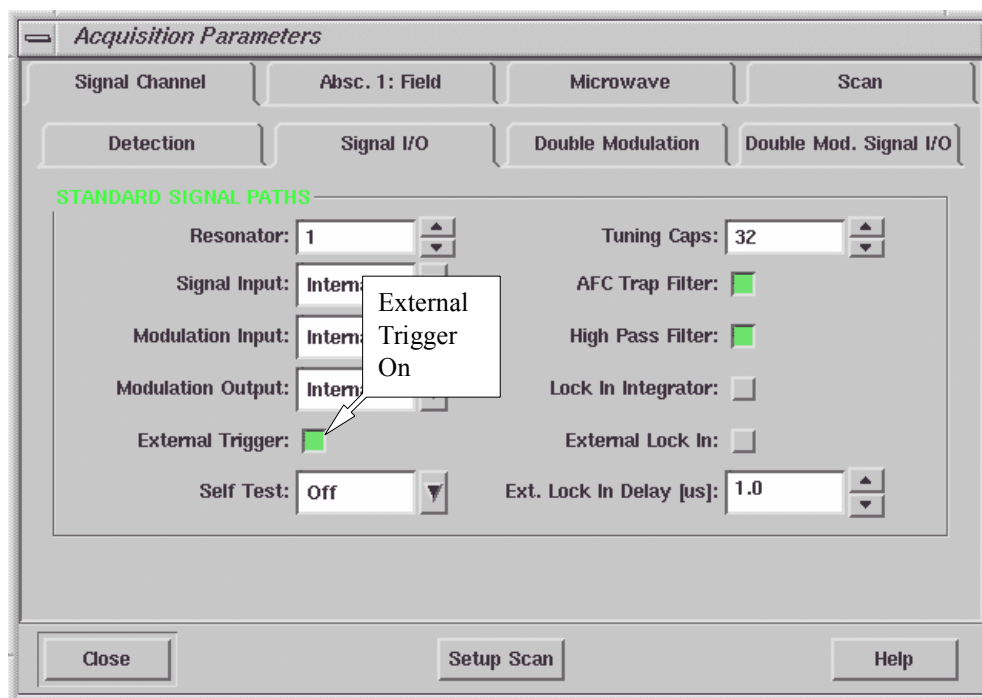


Figure 14-11 Enabling the External Trigger.

3. **Acquire a spectrum.** After clicking the Run button, the spectrometer waits for the external trigger to start the acquisition.

Externally Triggered Sweeps with the ADF

14.2.3

You can also use the ADF for externally triggered acquisitions. It is assumed that you have already created a field or time sweep experiment with Fast Digitizer as the Ordinate as described in Section 6.1.

1. **Provide an external trigger for the ADF.** Connect a TTL (5 volt) trigger pulse to the cable labeled Ext. Trigger from the AUX connector of the ADF board. Take care about the impedance, *i.e.* make sure the trigger voltage is high enough after the cable is connected.

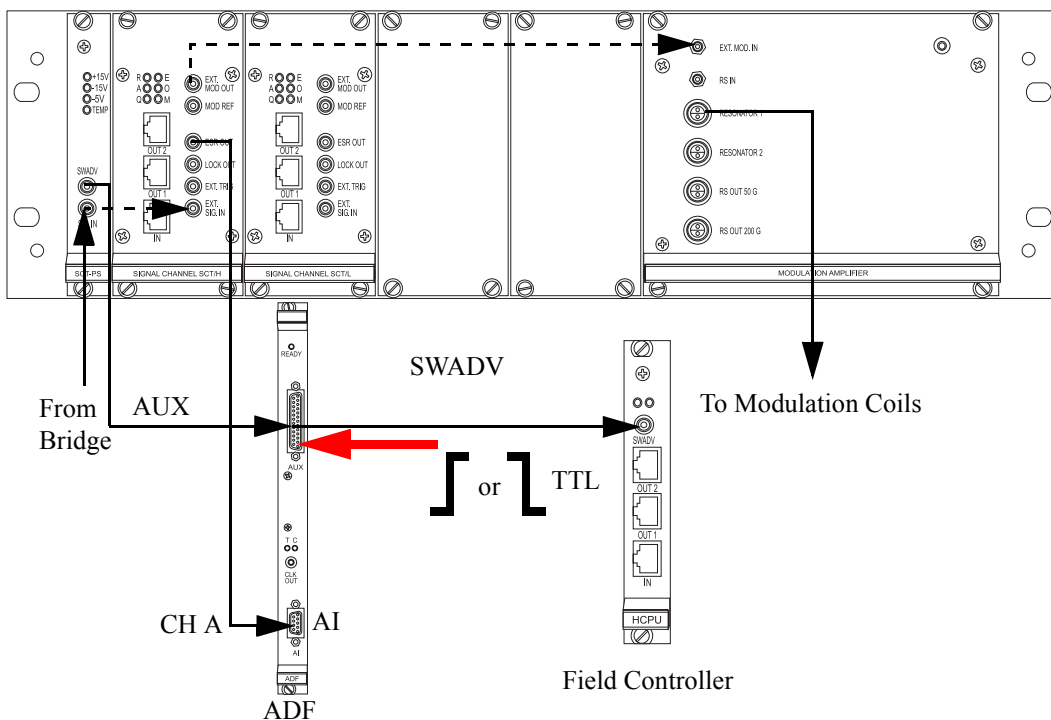


Figure 14-12 Configuration for externally triggered sweeps with the ADF.

2. **Enable the External Trigger.** Open the Parameter dialog box and click the Fast Digitizer folder. Select External Sweep or External Point from the Trigger Mode drop-down list. (See Figure 14-13.)

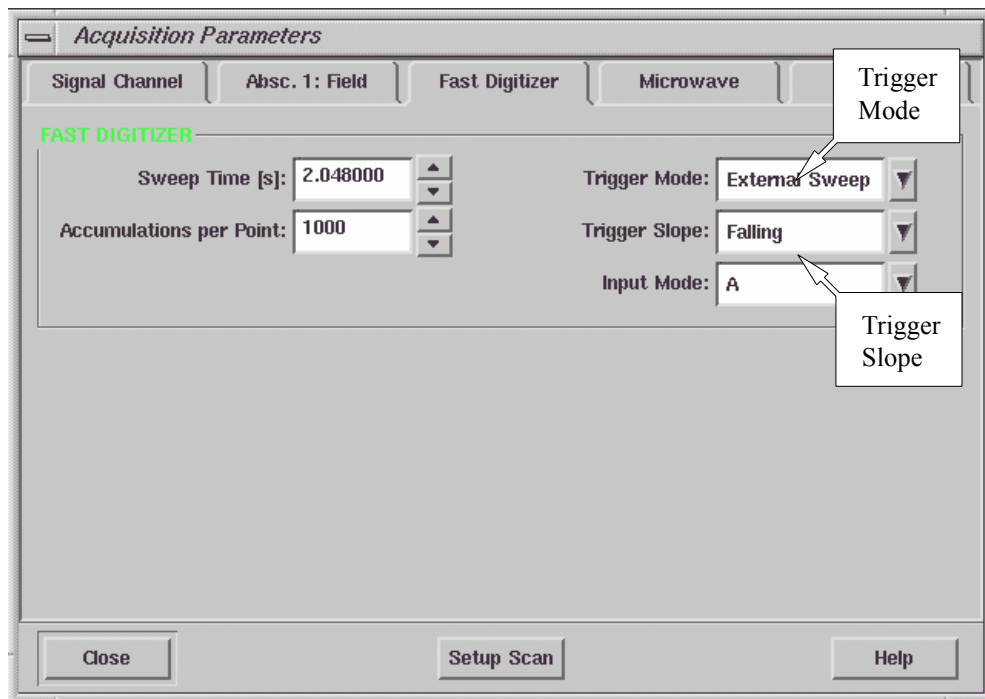


Figure 14-13 Defining the external trigger.

3. **Define the Trigger Slope.** In the Fast Digitizer folder, select Rising or Falling from the Trigger Slope drop-down list. (See Figure 14-13.) Either the rising or falling edge can be used to trigger the acquisition.
4. **Acquire a spectrum.** After clicking the Run button, the spectrometer waits for the external trigger to start the acquisition.

Externally Triggered Rapid Field Sweeps

14.2.4

You can use an external trigger to start a rapid field sweep using the ADF and RSC. It is assumed that you have already created a rapid field sweep experiment as described in Section 6.2.

1. **Provide an external trigger.** Connect an external cable carrying a TTL (5 volt) positive-going trigger pulse to the TRG connector of the RSC board. (See Figure 14-14.) Take care about the impedance, *i.e.* make sure the trigger voltage is high enough after the cable is connected.

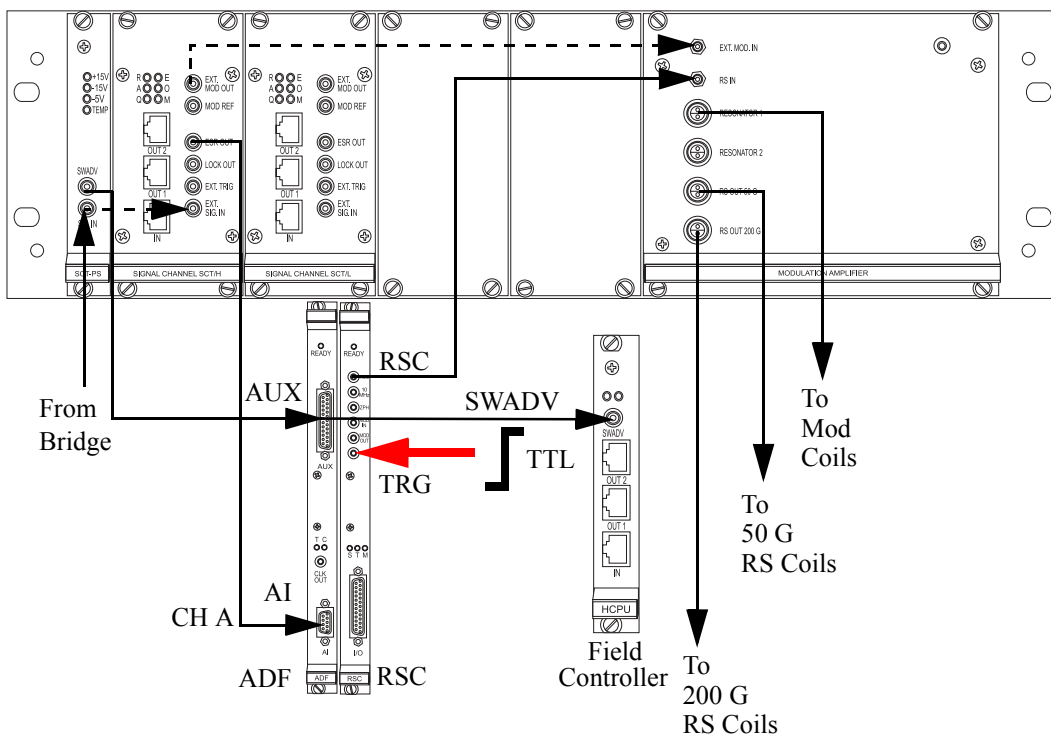


Figure 14-14 Configuration for externally triggered rapid field sweeps.

2. **Enable the External Trigger for the RSC.** Open the Parameter dialog box and click the Absc. 1: Field folder. Select External from the RSC Trigger Mode drop-down list. (See Figure 14-15)

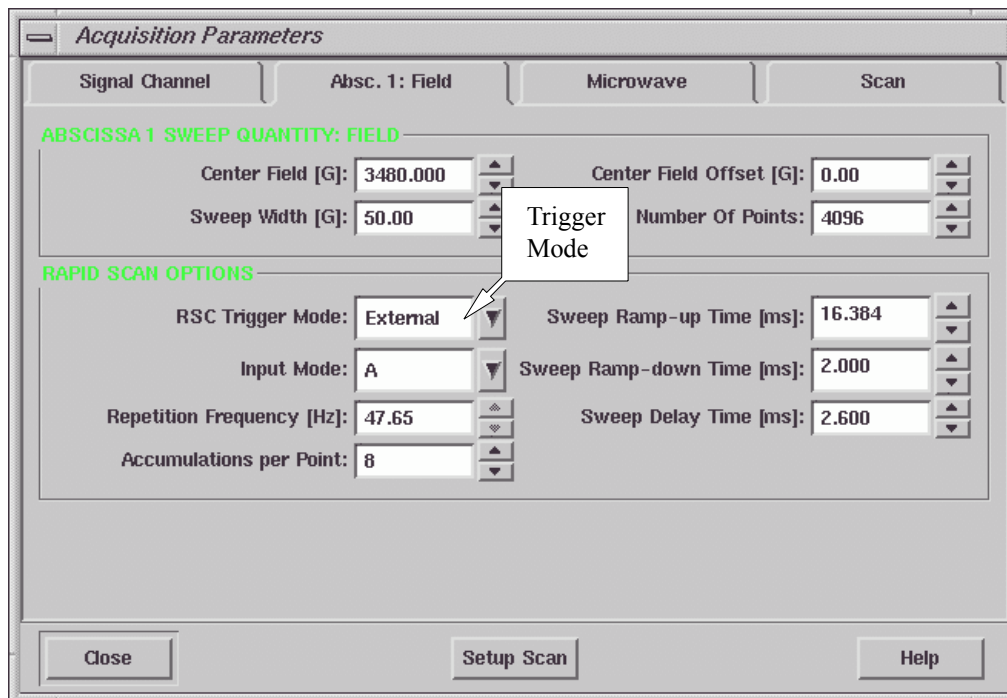


Figure 14-15 Enabling the RSC Trigger Mode.

3. **Acquire a spectrum.** After clicking the Run button, the spectrometer waits for the external trigger to start the acquisition.

External Field Modulation

14.2.5



Connect the cable to EXT. TRIG on the SCT/L signal channel if the frequency is less than 6 kHz.

The Bruker Elexsys spectrometer provides a high frequency signal channel and a low frequency signal channel. They cover a frequency range from 100 kHz to 100 Hz modulation frequency. However if you want to use your own field modulation, you can do so by connecting your modulation source (*e.g.* a function generator) to the console. It is assumed that you have already created a standard field or time sweep experiment.

1. **Cable connections.** Connect the output from the function generator to EXT. MOD. IN on the Modulation Amplifier board and EXT. TRIG on the signal channel. You may need a phase shifter to adjust the modulation phase.

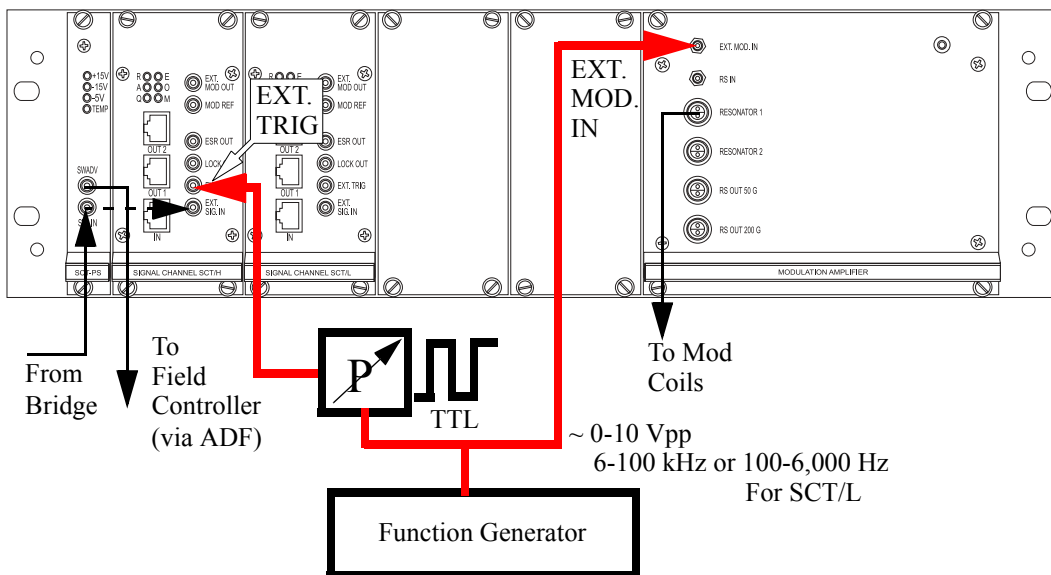


Figure 14-16 Configuration for external field modulation.



If you use a modulation frequency lower than 6 kHz, select a modulation frequency lower than 6 kHz in the Detection folder. This forces the software to use the SCT/L (low frequency) signal channel.

2. **Enable external modulation.** Open the Parameter dialog box and click the Signal I/O tab of the Signal Channel folder. (See Figure 14-17.) Select External from the Modulation Input drop-down list. Click External Lock In to activate it (green). You must change the modulation phase by using an external phase shifter. The Ext. Lock In Delay does not change the phase in this case as it does for double modulation.
3. **Acquire a spectrum.** Click the Run button.

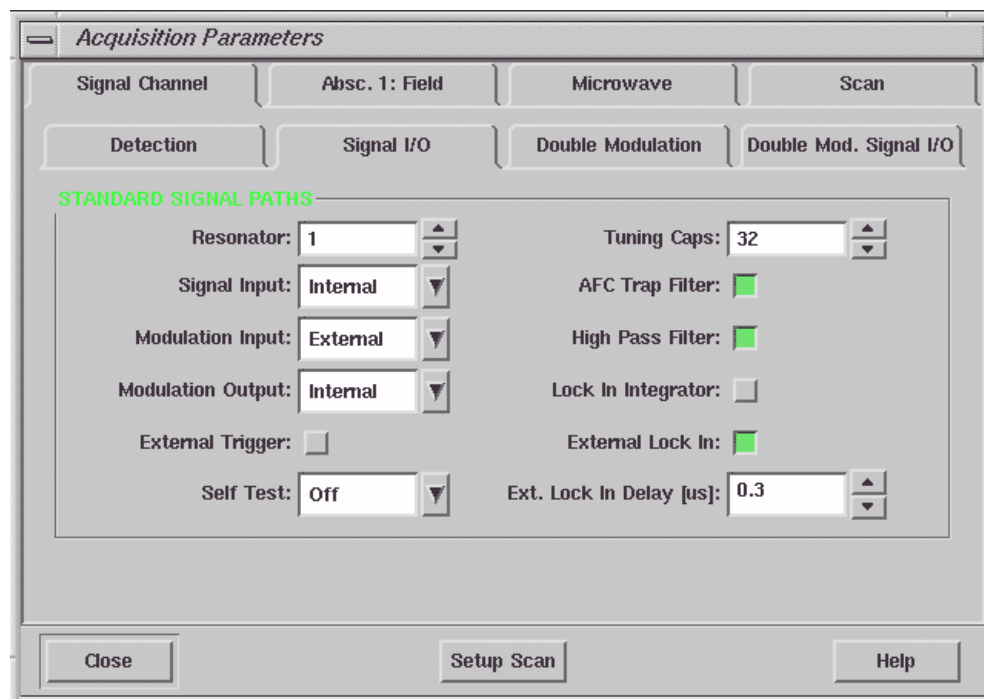


Figure 14-17 Parameter setup for external modulation.

Other Forms of Modulation

14.2.6



This section describes a low frequency (<6 kHz) experiment. For higher frequencies, simply substitute SCT/H for SCT/L and enter a modulation frequency of 100 kHz in the Detection folder.

Field modulation is not the only means to detect your EPR signal. For example, you can use a light chopper to modulate the sample illumination and thereby selectively detect the EPR signals from transient species. It is assumed that you have already created a standard field or time sweep experiment.

1. **Connect the modulation source to the console.** Most light choppers supply a train of TTL pulses synchronized with the light chopping. Other modulation devices also often have a TTL synchronization signal. Connect a cable from the modulation device to the EXT. TRIG of the low frequency signal channel (SCT/L) to supply a reference signal.

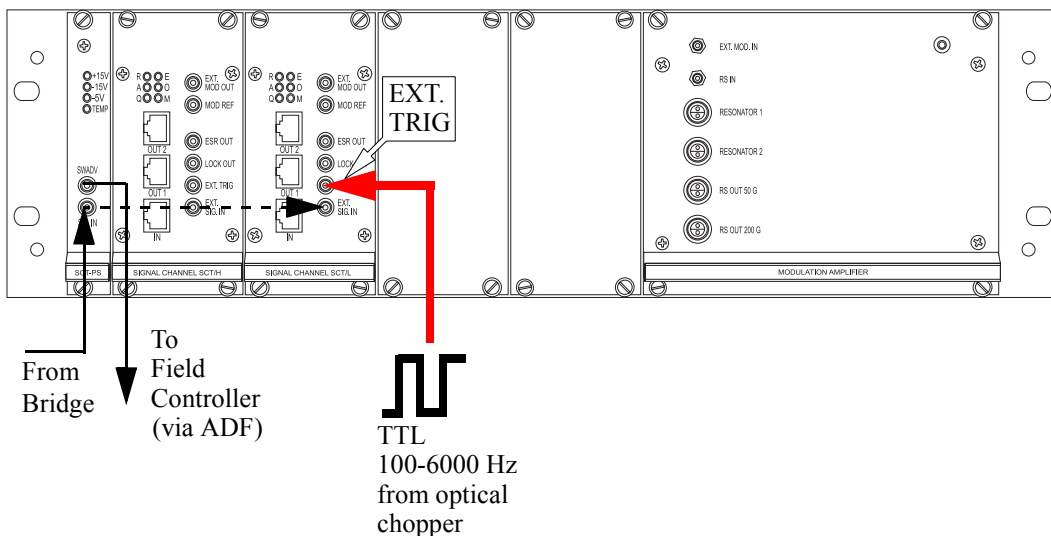


Figure 14-18 Configuration for external modulation using the SCT/L signal channel.

2. **Enable external modulation.** Open the Parameter dialog box and click the Signal I/O tab of the Signal Channel folder. (See Figure 14-19.) Click External Lock In to activate it (green). You must change the modulation phase by using an external phase shifter. The Ext. Lock In Delay does not change the phase in this case.

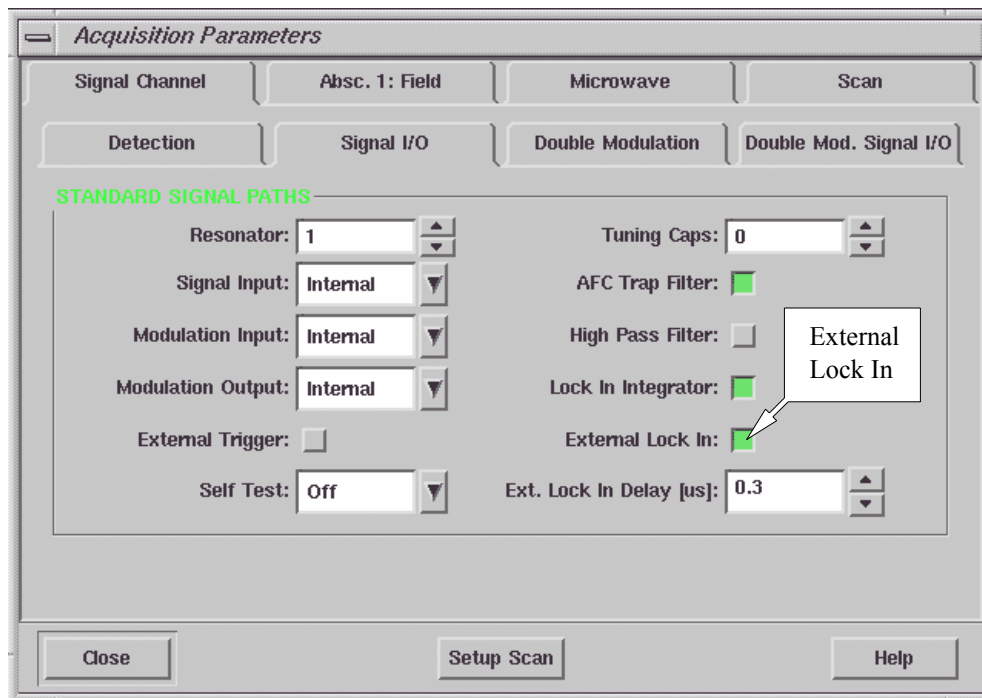


Figure 14-19 Parameter setup for the optical modulation.

3. **Set more parameters.** Since this is a low frequency modulation experiment, we recommend deactivating (not green) the **High Pass Filter** button and selecting the **Lock In Integrator** option to reduce the noise. (See Figure 14-19.) Make sure you enter a modulation frequency value below 6 kHz in the **Modulation Frequency** of the **Detection** folder. This forces the acquisition software to use the low frequency signal channel.
4. **Acquire a spectrum.** Click the **Run** button.

Sweeps with Double Modulation

14.2.7

Because light choppers are limited to low modulation frequencies, high frequency (100 kHz) field modulation is often used together with light chopping to improve the sensitivity. The field modulated signal is first demodulated by the SCT/H high frequency signal channel. The unfiltered (*i.e.* before the time constant filter) output signal is fed into the SCT/L low frequency signal channel where the light-chopped signal is demodulated. It is assumed that you have already created a standard field or time sweep experiment.

1. **Connect the signal cable to the SCT/H signal channel.** Disconnect the signal cable connected to Sig. In of the SCT-PS board. Connect it to EXT. SIG. IN of the high frequency signal channel (SCT/H). (See Figure 14-20.)

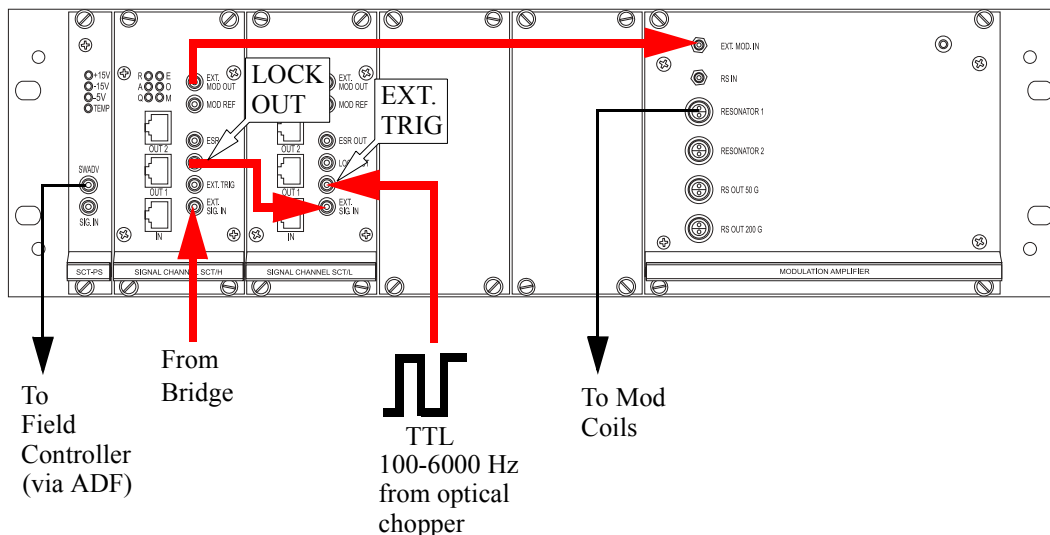


Figure 14-20 Configuration for double modulation (field and optical) experiments.

2. **Connect LOCK OUT to EXT. SIG. IN.** Connect LOCK OUT of the high frequency signal channel (SCT/H) to EXT. SIG. IN of the low frequency signal channel (SCT/L) with an external cable. (See Figure 14-20.)
3. **Connect EXT. MOD OUT to EXT MOD. IN.** Connect EXT. MOD OUT of the high frequency signal channel (SCT/H) to EXT. MOD. IN of the Modulation Amplifier with an external cable. (See Figure 14-20.)
4. **Provide a TTL reference to EXT. TRIG.** Connect the cable carrying the pulses from the light chopper or other low frequency modulation supplier to EXT. TRIG of the low frequency signal channel (SCT/L). (See Figure 14-20.)

5. **Enable Double Modulation.** Open the Parameter dialog box and click the Signal Channel folder. Click the Double Modulation tab. Activate (green) Double Modulation. Select Second in the Detecting SCT drop-down list. Select First in the Field Modulation SCT drop-down list. You may need to reduce Receiver Gain values for both signal channels. Note that the Time Constant in this folder controls the time constant for the low frequency (Detecting SCT) signal channel. (See Figure 14-21.)

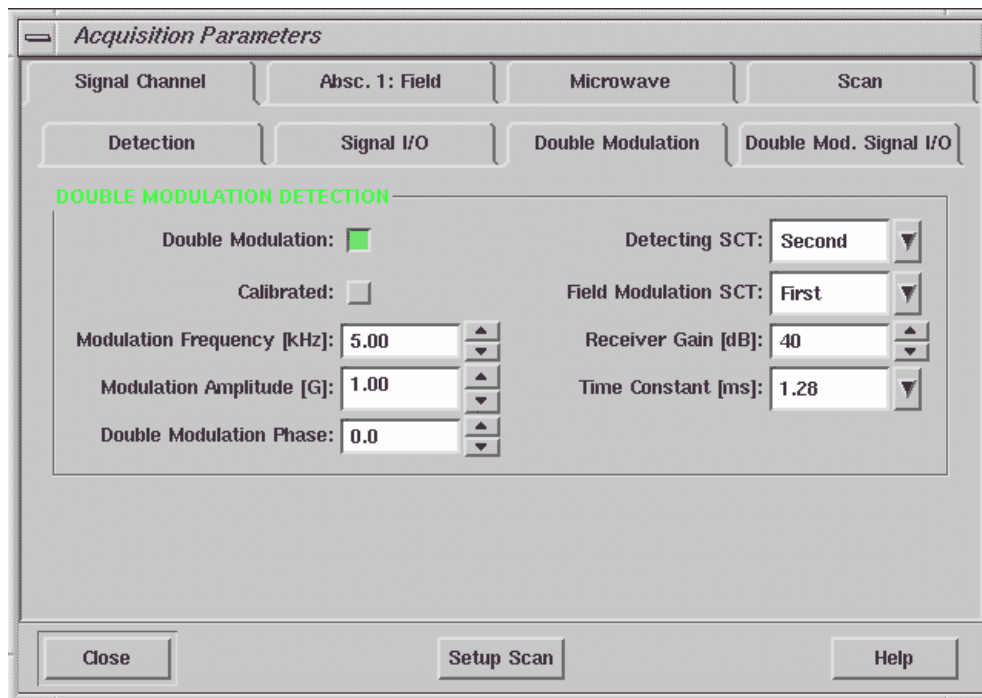


Figure 14-21 Enabling Double Modulation.

6. **Set up the Signal I/O.** Click the Signal I/O tab. Select External for Signal Input, Modulation Input, and Modulation Output. (See Figure 14-22.)

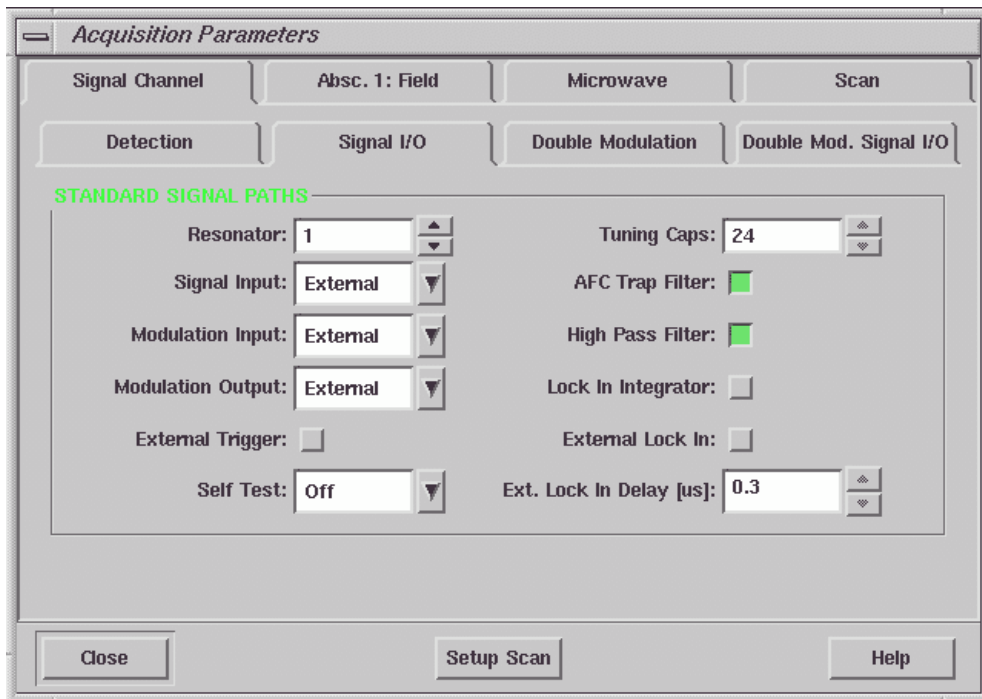


Figure 14-22 Enabling external inputs and outputs in Signal I/O.

7. **Configure Double Mod. Signal I/O.** Click the Double Mod. Signal I/O tab. Select External for Signal Input. You may need to activate the High Pass Filter and the Lock In Integrator to reduce the noise. Click External Lock In to activate it (green). (See Figure 14-23.)

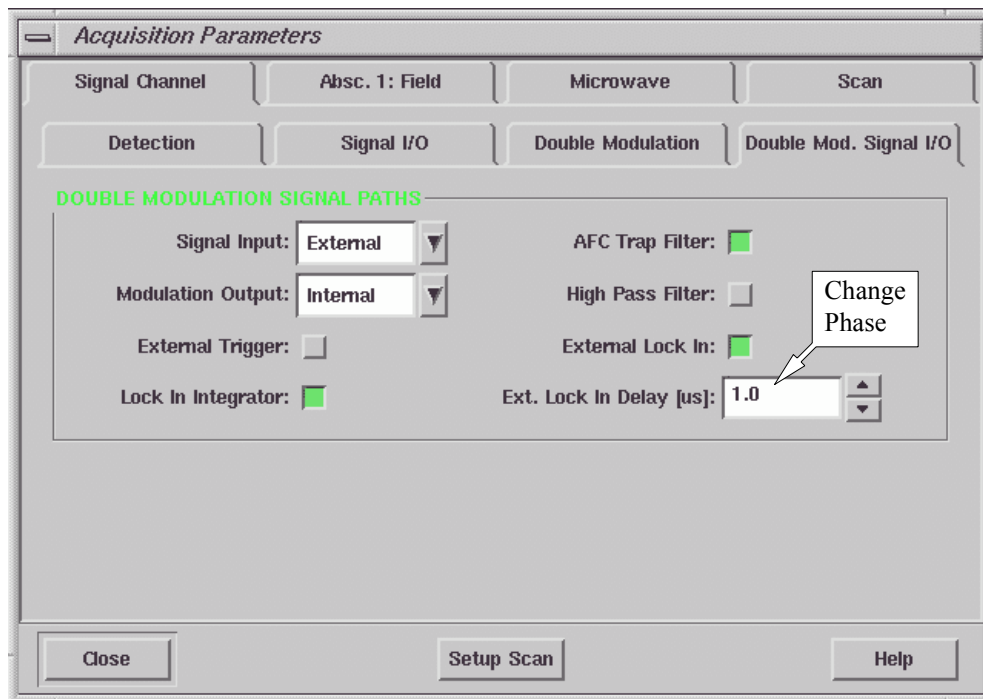


Figure 14-23 Configuring Double Mod. Signal I/O.

8. **Adjust the low frequency modulation phase.** You can adjust the phase of the low frequency modulation. Convert the phase value in degrees you want to adjust to delay time (in μs) based on the following formula:

$$\text{Delay}(\mu\text{s}) = \frac{\text{Phase} \cdot 1000}{360 \cdot \text{Modulation Frequency}(\text{kHz})} \quad [14-1]$$

Enter the value in the Ext. Lock In Delay box. (See Figure 14-23.)

9. **Acquire a spectrum.** Click the Run button.

Triggered Quadrature Detection with the ADF

14.2.8

We described quadrature detection with two signal channels in Section 14.2.1. We can also perform this experiment with the fast digitizer (ADF) instead of the signal channel digitizer. First follow the instructions in Section 14.2.1. It is assumed that you have already created a field or time sweep experiment with Fast Digitizer as the Ordinate.

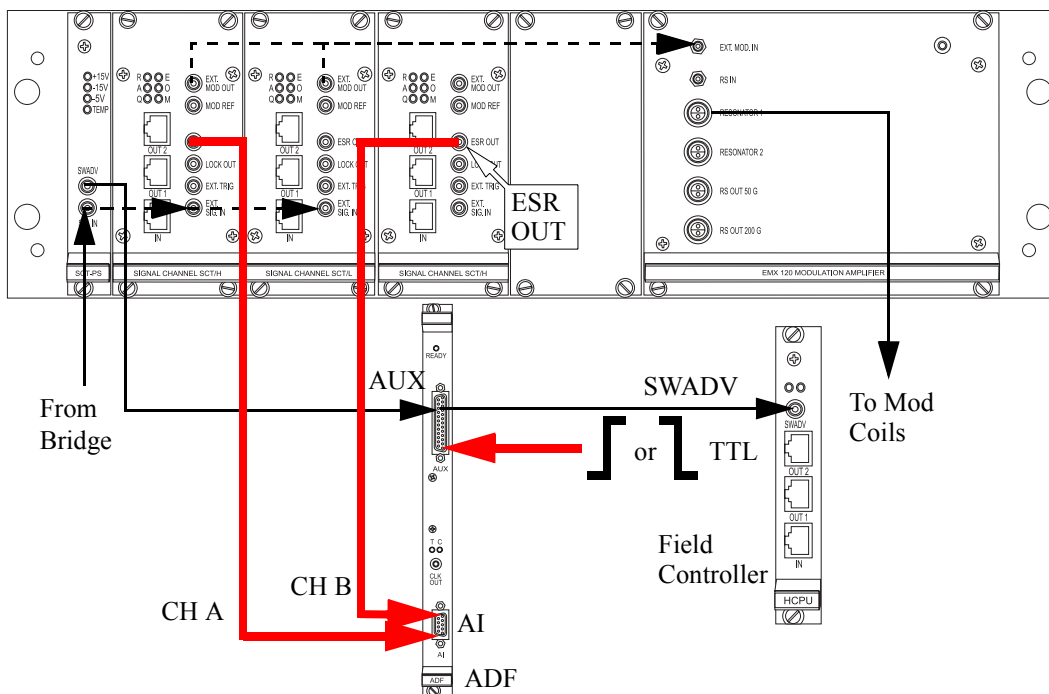


Figure 14-24 Configuration for triggered sweeps with quadrature detection using the ADF.

1. **Cable connections.** Connect the two ESR OUT signals from the two identical signal channels to Channel A and Channel B of the AI connectors on the ADF board. (See Section 13.7.2 for the pin lay out of the AI connector of the ADF board.) You can use the cable labeled ADF ENDOR on the AI connector as Channel B. You also need to send a TTL trigger pulse to the cable labeled Ext. Trigger from the AUX connector on the ADF board. (See Figure 14-24.)
2. **Set up the parameters.** Open the Parameter dialog box and click the Fast Digitizer tab. Select External Sweep or External Point in the Trigger Mode drop-down list. Select the appropriate Trigger Slope for your trigger pulse. Select A&B in the Input Mode. (See Figure 14-25.) The signal from the Channel A is stored as the real part of the spectrum while the signal from Channel B is stored as the imaginary part of the spectrum. You can view each of them by clicking the Re/Im button in the Tool bar.

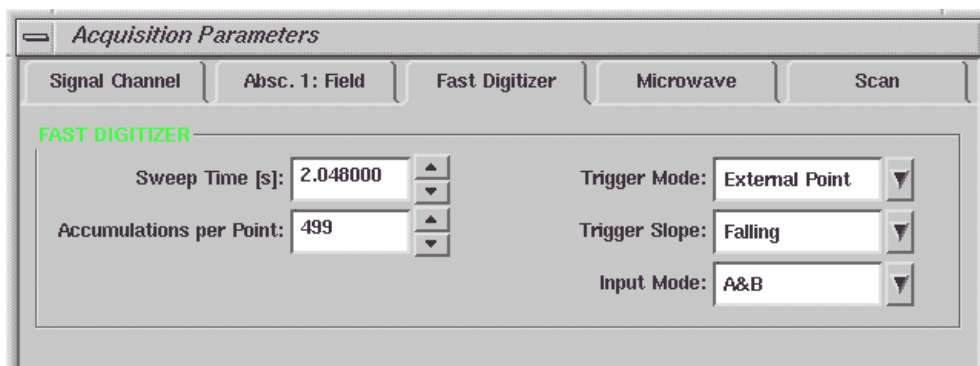


Figure 14-25 Setting up the ADF for quadrature detection.

3. **Acquire a spectrum.** Click the Run button.

Transient Quadrature Signal Capture with the ADF 14.2.9

You may need to digitize transient signals that are not detected by the signal channels. An example of such a direct-detected signal would be the quadrature signals from a transient bridge. Connect the two transient signal outputs, *e.g.* I and Q to Channel A and Channel B of the AI connector of the ADF board respectively. You can use the cables labeled SCT and ADF ENDOR. Send a TTL trigger pulse to the external trigger of the AUX connector of the ADF board. The parameter settings are similar to those in the previous section.

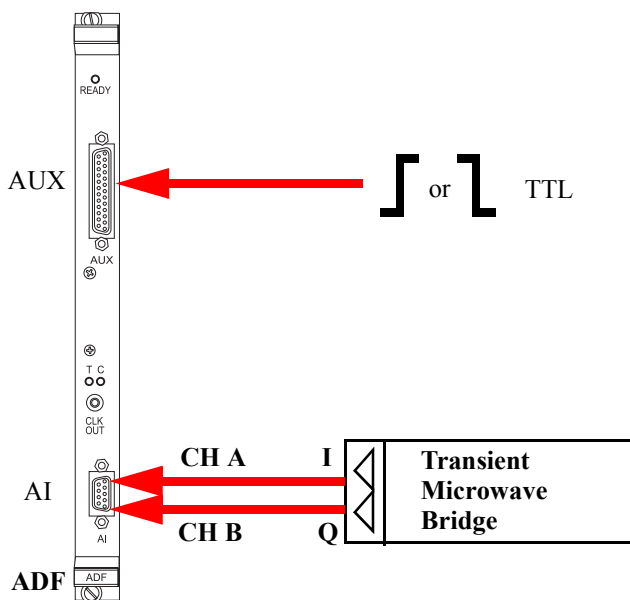


Figure 14-26 Configuration for transient quadrature signal capture using the ADF.

Externally Controlled Field Sweep with the ADF 14.2.10

If you have your own field sweep device you can direct the field sweep pulses to the external trigger input of the AUX connector on the ADF board and the SWADV (sweep address advance) on the field controller board.

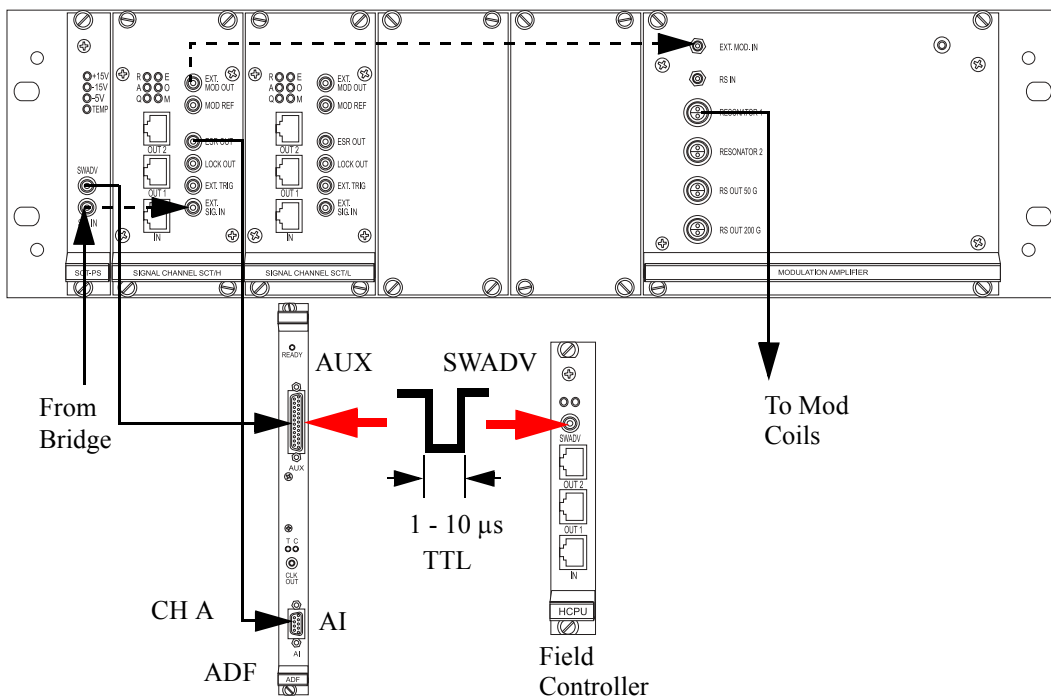


Figure 14-27 Externally controlled field sweeps using the ADF.

You must enable external point triggering and define the slope in the digitizer parameter menu. Table 14-1 shows the sweep

address advances per pulse (field advances on the negative edge of the trigger).

X-resolution	steps/pulse
512	8
1024	4
2048	2
4096	1
8192	1/2

Table 14-1 Sweep address advances per pulse

Bruker delivers either SGI O2 or Linux workstations for the user interface of the Elexsys series EPR spectrometers. This chapter describes various system administration issues related to the Xepr program for the SGI O2 workstation. The names or terms described in this chapter may differ slightly for the Linux system, but the principles are the same. Refer to Chapter 16 for details if you have a Linux workstation

Most of the commands and operations in this chapter require root or super user privileges. In addition, the commands also require at least some basic knowledge of the UNIX operating system. This chapter assumes that you already are familiar with general administration of the UNIX operating system. Be cautious when making changes to the workstation configuration: some mistakes can lead to an unusable workstation. Consult your local Bruker EPR service representative if you are unsure of the changes you are making.

General Information

15.1

The SGI O2 workstation comes with two Ethernet cards and the IRIX 6.5 UNIX operating system. IRIX 6.3 is no longer supported, although Xepr still functions under 6.3.

If you wish to check the hardware inventory (system ID, RAM, CPU, IP address, *etc.*) for your workstation, use the shell command `hinv`. If you prefer to use a GUI (Graphical User Interface) tool, select **Toolchest > System > System Manager > About This System** to check the hardware inventory.

The system as delivered by Bruker has two visible accounts: the **root** account with the password `xepr@sgi` and the **xuser** account with the password `user@xepr`. For your security, change the root password immediately after the system has been installed.

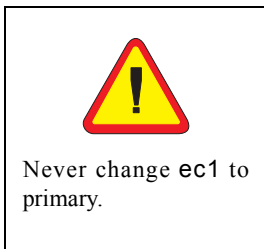
Most of the Xepr files reside in the `/usr/xepr` folder.

Network Settings

15.2

ec0 and ec1

15.2.1



The two Ethernet cards in the workstation are labeled **ec0** and **ec1**. **ec0** is used for connecting to the outside world and is configured as the primary interface. **ec1** is devoted to communications between the workstation and the acquisition server (OS9) of the spectrometer and is configured as the secondary interface. When the workstation is delivered, the hostname and IP address of **ec0** are set to default values. You need to ask your local network administrator to assign a hostname and an IP address for your workstation. Change the hostname and IP address of **ec0** only. Never change the hostname and IP address of **ec1**.

NFS (Network File System)

15.2.2

Xepr uses NFS to transfer files between the UNIX workstation and the acquisition server. Never turn off or deactivate NFS.

UNS (Unified Name Service)

15.2.3

UNS allows you to use easily remembered host names instead of easily forgotten IP addresses. The most common services are files, DNS (Domain Name Service), and NIS (Network Information Service).

Files This is the most controllable way to provide network information. Entries in the `/etc/hosts` file connect an IP address with a corresponding host name. You can simply edit this file to define the names and IP addresses of the computers in your local network as well as on the Internet. The hosts file must contain the local host.

The format is:

IP address	hostname.subdomain.domain	hostname
127.0.0.1	localhost	
123.123.123	Elexsys.university.edu	Elexsys
192.168.99.1	Xepr	
192.168.99.5	E500	

DNS Your local network usually has one or more DNS servers. You can get the IP addresses of the DNS servers from your local network administrator.

NIS Although NIS offers many benefits, the complexity and integration with other products can lead to problems. Do not activate or use NIS on the EPR workstation.

To configure UNS, you can either modify the `/etc/nsswitch.conf` file or select **Toolchest > System > System Manager > Networking and Connectivity > Modify Networking Settings**. We recommend the following configuration:

```
files none none
```

If you really need to use a DNS server, you can configure it as follows:

```
files DNS none
```

Routing

15.2.4

Although the EPR workstation has two Ethernet cards and is able to function as a router, doing so will jeopardize the communication between the workstation and the EPR acquisition server. Modification of the routing table could also direct network traffic to `ec1`, which would slow down the response of the Xepr program or even cause the system to hang up.

UNIX Workstation Configuration

15.3

Flags

15.3.1

Flags are used to activate and deactivate various services that the workstation can offer. Here is the standard configuration for an SGI IRIX workstation as delivered by Bruker. You can check the configuration by entering `chkconfig` in a UNIX shell. The `network` and `nfs` services must be on to allow the workstation to communicate with the acquisition server properly. Although you can turn on more services than the basic configuration, keep in mind that the more services you turn on, especially network related services, the slower the workstation responds.

Flag	Status	Flag	Status
appletalk	off	privileges	on
array	off	proclaim_relayagent	off
autoconfig_ipaddress	off	proclaim_server	off
autofs	on	proxymngr	off
automount	off	rarpd	off
bsdldr	on	routed	off
desktop	on	rsvpd	off
fontserver	off	rtmond	on
gated	off	rwhod	off
ipaliases	on	sar	off
isdnd	off	savecore	on
lockd	on	sdpd	on

Table 15-1 Standard configuration for workstation services.

Flag	Status	Flag	Status
lp	on	sendmail	on
mediad	on	snetd	on
mrouted	off	soundscheme	on
named	off	timed	on
nds	off	timeslave	off
network	on	verbose	on
netwr_client	off	visuallogin	on
nfs	on	vswap	on
noiconlogin	off	webface	off
nostickytmp	off	windowsystem	on
ns_admin	off	xdm	on
ns_fasttrack	on	yp	off
nsd	on	ypmaster	off
pmcd	on	ypserv	off

Table 15-1 Standard configuration for workstation services.

To turn a flag on or off, type

```
chkconfig <flag> <on|off>
```

where <flag> is the service you want to turn on or off followed by either **on** or **off**. Rebooting the Unix workstation is the easiest way to ensure the newly selected service is properly started.

Swap Space

15.3.2

The best way to determine the optimal swap space size is to try several sizes. We recommend setting the swap space size to four times the RAM size. When the swap space is not large enough there will be an error message in the `/var/adm/SYSLOG` file or in the console window. If you routinely process several large 2D datasets in Xepr you may need to increase your swap space. There are two types of swap space: the hard disk swap space and the swap file. The swap file is slower than the hard disk swap space. To increase the hard disk swap space you need to repartition the hard drive which wipes out the contents of the hard disk. The fastest swap space configuration is to have swap spaces on both the system hard disk and the secondary hard disk.

Security

15.3.3

Functionality, accessibility, and convenience may compromise the system security. Balancing these considerations is not a simple job. Refer to the SGI workstation administrative manual for security settings that best suit you. We suggest the following.

Lock accounts There are a few accounts utilized by Xepr such as the `guest` and `lp` accounts. Never delete the `guest` and `lp` accounts. Instead, lock these accounts and other “open to the public” accounts such as `demos`, `EZsetup`, `OutOfBox`, *etc.*

Root and Super User Avoid logging in as root or super user. Only do so when you must perform system administration tasks. Xepr should be run out of your personal user account or `xuser` account.

Transitive Trust The UNIX operating system allows users to set host-to-host equivalency (in the `/etc/hosts.equiv` file), user-to-user equivalency (in the `~/.rhosts` file), and ftp user equivalency (in the `~/.netrc` file). In high security environments, the `/etc/hosts.equiv` file is blank and the `~/.rhosts` and `~/.netrc` files in the user accounts are usually removed.

Reinstalling or Upgrading Xepr 15.4

List of Important Files 15.4.1

The following files are very important and must be backed up before you reinstall the operating system or the Xepr software.

License File	The file <code>license.dat</code> can be found in three possible locations: <code>/usr/xepr/sharedSetup/</code> <code>/usr/Bruker/flexIm/licenses/</code> <code>/usr/local/flexIm/Bruker/licenses/</code>
Hall Probe Calibration File	<code>/usr/xepr/AcquisitionServer/OS9/Servers/FUSETUP/FUEr032t/hall#####.cal</code> and <code>hall#####.dat</code> (where ##### is the serial number.)
ACPU Information	<code>/usr/xepr/AcquisitionServer/admin/ServerSetup</code>
Signal Channel Calibration Files	<code>/usr/xepr/AcquisitionServer/OS9/Servers/<Server name: e.g. E500, E580, etc.>/FUSETUP/FUSct/CalibData/*.cal</code>
Spectrometer.ini File	<code>/usr/xepr/AcquisitionServer/OS9/Servers/<Server name>/Spectrometer.ini</code>
Pulse System Configuration Files	If you have an E 580 or E 680: <code>/usr/xepr/AcquisitionServer/OS9/Servers/<Server name>/FUSETUP/FUFTEpr/XBand (and/or WBand)/*.cfg</code>

Performing an Installation or Upgrade

15.4.2

1. **Check the current version of Xepr.** Use the shell command `Xepr -i` to determine the version of the currently installed Xepr software.
2. **Follow the instructions in the release note.** Login as root to install Xepr. Read the release note on the Xepr CD first. Follow the instructions for installing Xepr. Use the default installation settings to avoid any unforeseen problems. Sometimes you may need to delete the `/usr/xep` folder before you install the Xepr software.
3. **Restore the backed up files.** Put the files you backed up in Section 15.4.1 into the folders from which they came.
4. **Install the acquisition server.** Each time you reinstall Xepr or upgrade the acquisition package you must reinstall the acquisition server. The acquisition server installation script is located in the folder `/usr/xep/AcquisitionServer/admin/`. Make sure you have the correct `ServerSetup` file. Change to the above directory and type `./installServer` to install the acquisition server.
5. **Reboot the UNIX workstation.** If you do not, the upgrade or installation will not work properly.

Initial Setup of Xepr

15.5

Normally users have their own account. If you do not have an account yet ask the system administrator to create one for you. If you are responsible for the system and want to create a user's account, refer to the SGI workstation manual or help to find out how to create a user's account. A user's account has a user's account name, user ID, and a password to log into the account. After you log into your account, you can configure your desktop, windows, UNIX shell type, and other features. You can also change your password. Refer to the Toolchest help menu for configuration of your account. When you run Xepr for the first time in your own account, Xepr will automatically configure your home directory to setup Xepr.

Setting Up Xepr for Your Account

15.5.1

1. **Find the Xepr icon.** Click the Toolchest > Find > Icon Catalog > Applications. A window of application program icons will appear. (See Figure 15-1.)

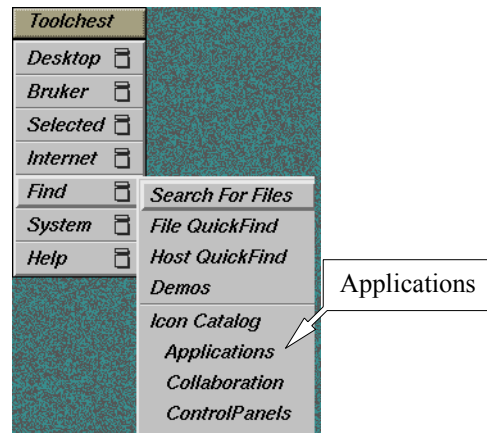


Figure 15-1 Opening the Applications icon window.

2. **Setup a desktop icon for Xepr.** In the **Applications** icon window you can find the Xepr icon. (See Figure 15-2.) Drag the icon onto to the desktop. Now you can launch the Xepr software from the Desktop.

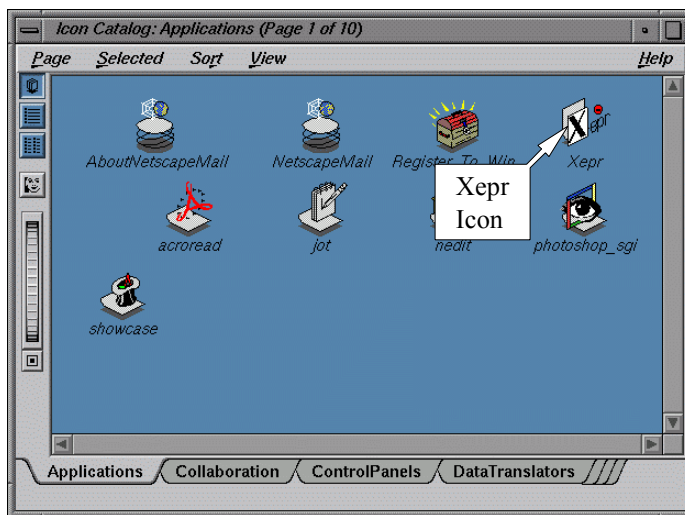


Figure 15-2 The Xepr icon.

3. **Start Xepr program for the first time.** Double click the Xepr icon. The Xepr program will automatically configure your account to run Xepr and launch the Xepr program.

Setting Up the Properties of the Xepr Software 15.5.2

1. **Maximize the Xepr window.** Since Xepr has so many components, a full screen window is recommended. Click the maximize button on the right top corner.
2. **Select the monitoring panel position.** First make sure the spectrometer is on and booted. You can choose either to put the monitoring panel above or below the Viewport. Click the Properties button in the menu bar, and then Panel Properties. A dialog window will appear. Click either Top or Bottom and then OK. (See Figure 15-3.)

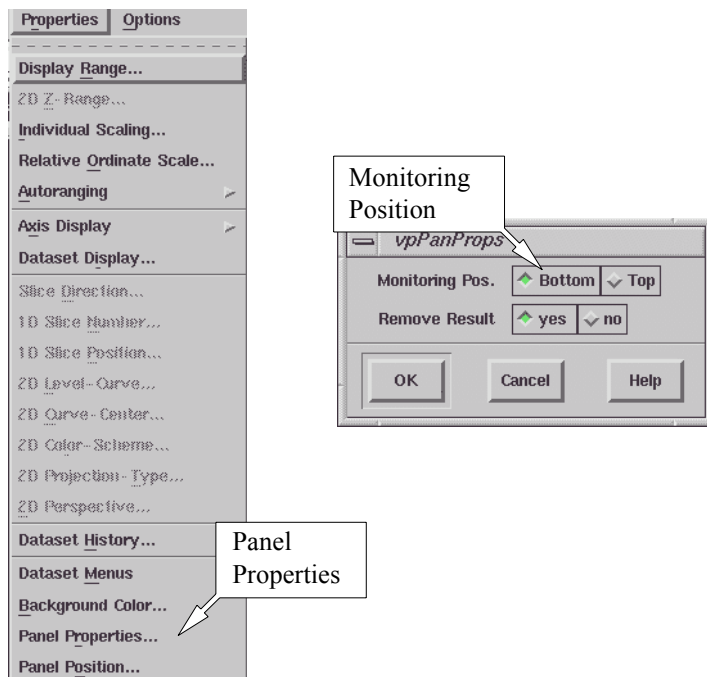


Figure 15-3 Selecting the monitoring panel position.

3. **Connect to spectrometer.** Click Acquisition in the menu bar and then Connect To Spectrometer. A dialog box will appear prompting for the Server Name. Enter the acquisition server's name in the blank box and click OK. (See Figure 15-4.) If you do not know the acquisition server's name, ask the system administrator. The typical servers names are e500, e580, e600, or e680, depending on the type of spectrometer.

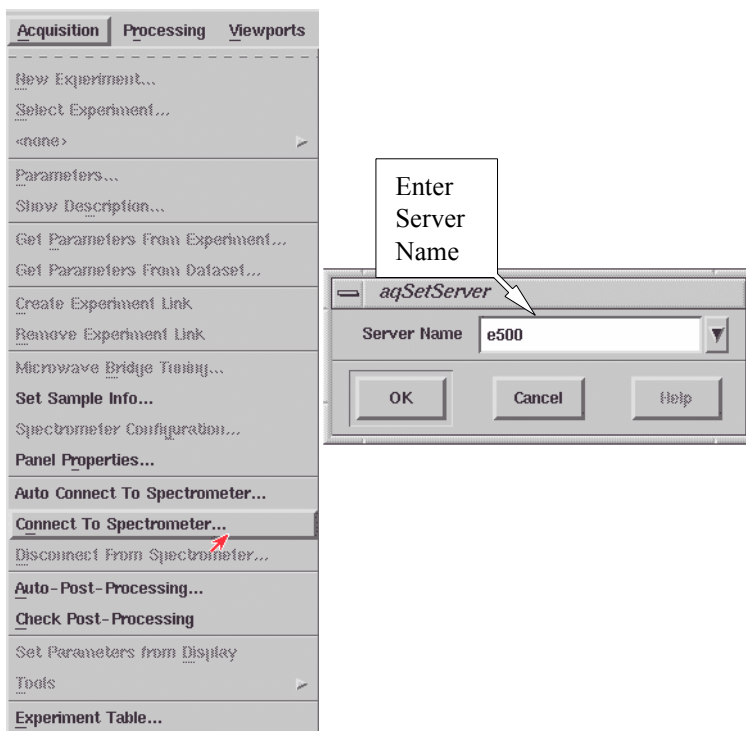


Figure 15-4 Connecting to the spectrometer.

4. **Set up the printer.** Click **File** in the menu bar and then **Setup Printer**. A dialog box appears. Click the arrow button next to the **Printer Type**. A drop-down list of printers will appear. Select the correct **Printer Type** for your printer. The **Printer Command** has been set by default as `lp -c -dXeprPrinter`, where `XeprPrinter` is the default name of the Xepr printer. Do not change this command unless you have changed the printer name. The **Preview Command** should be set to `xpsview -or landscape`. Now that the critical settings are selected, you can choose the other options which are available in the dialog box. Click **Set** to exit this dialog box. (See Figure 15-5.)

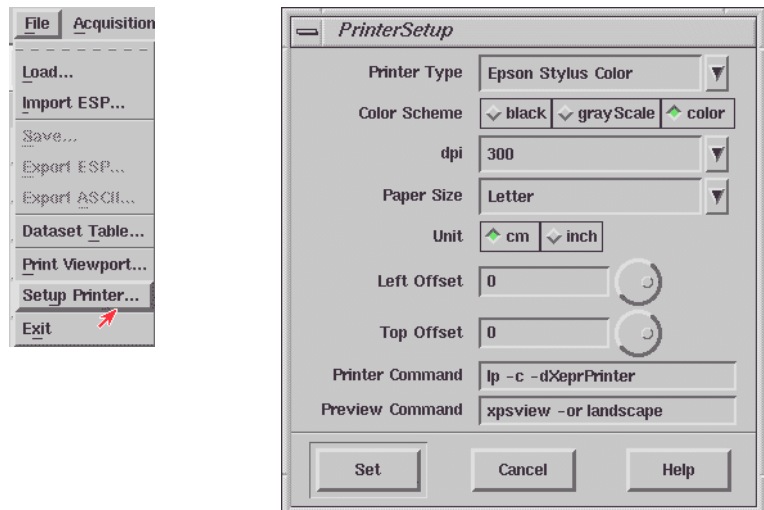


Figure 15-5 Setting up the printer.

5. **Test the printer.** Load an EPR spectrum. Display the spectrum in the viewport. Click Print in the File drop-down menu. A dialog box appears. Select the proper Orientation, Width, Height, and other parameters. Click printer as the Destination. Click the Print button. (See Figure 15-6.)

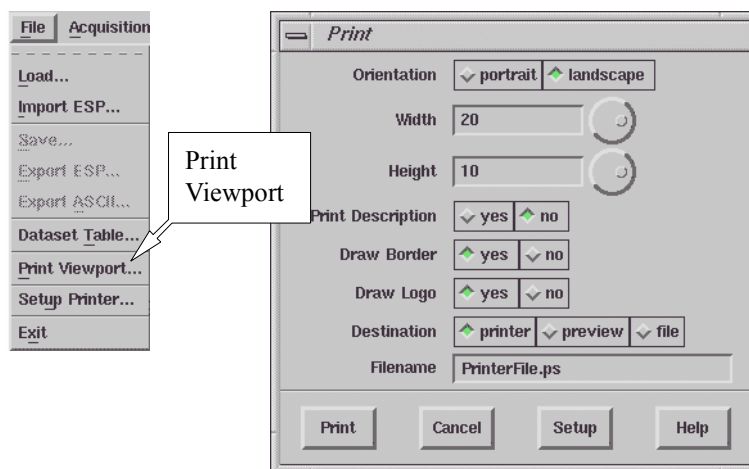
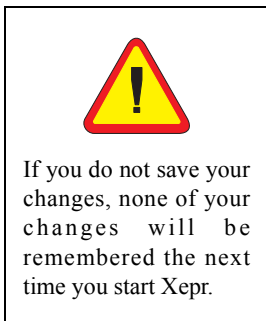


Figure 15-6 Testing the printer.

6. **Disconnect from the spectrometer.** Click Acquisition > Disconnect From Spectrometer. The monitoring panel will disappear.



7. **Exit Xepr.** Click **File** and then **Exit** in the menu bar. A dialog box appears asking you if you want to save the changes. Click **Yes** and then **OK** to exit Xepr program. (See Figure 15-7.) Next time you launch the Xepr software, you will find the **Server Name** appearing in the **Connect to Spectrometer** dialog box. The printer will be ready to print.

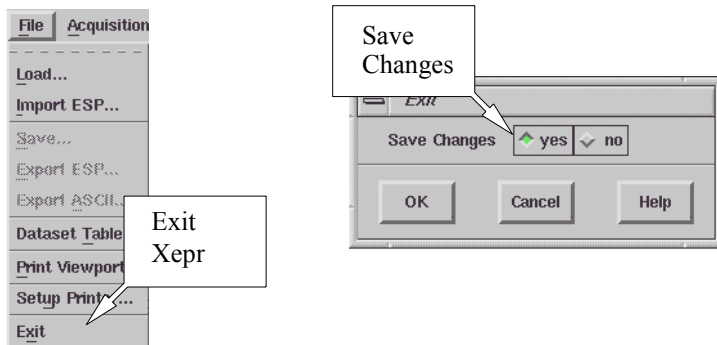


Figure 15-7 Saving the changes and exiting.

8. **Logout from your account.** It is always a good idea to logout from your account when you finish your work.

How to Start and Stop Xepr

15.6

This section describes different ways to start the Xepr program. It also describes how to abort the Xepr program when things go wrong and the system hangs up.

Starting Xepr from a UNIX shell

15.6.1

There are two basic ways to launch the Xepr program. One is to double click the Xepr icon. You can find the Xepr icon on the Desktop or in the Icon Catalog: Applications window. The alternative way to start Xepr is to launch Xepr from a UNIX shell.

1. **Open a UNIX shell.** Click Desktop > Toolchest > Open UNIX Shell. A shell window will open.

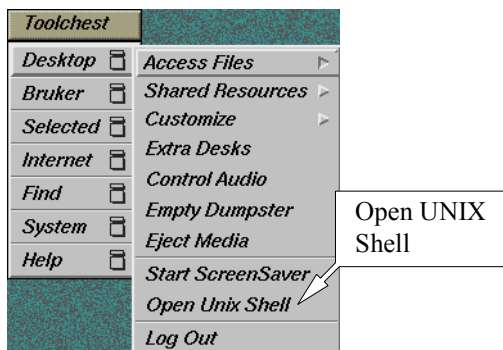


Figure 15-8 Opening a UNIX shell.



UNIX is case sensitive. You need to capitalize the X when you type the shell command Xepr.

2. **Start Xepr with a shell command.** Move the mouse pointer inside the shell window, type `Xepr`, and then press the **Enter** key. The Xepr program will start. You can leave the UNIX shell window open for now. That gives you an option to abort Xepr program. (See the next section.)



Figure 15-9 Launching the Xepr program from a UNIX shell.

Aborting Xepr

15.7

Normally you need to exit Xepr by clicking **File** in the menu bar, and then clicking **Exit** to terminate the Xepr program. However, when something goes wrong or the system hangs up, you may not be able to exit Xepr by normal methods. Keep in mind, sometimes Xepr needs more time to execute certain functions especially the communications between the system and slow devices. It may not respond to your command instantly. Give Xepr some extra time. Avoid continuously clicking the mouse button: it only make the system even busier. When memory is running too low or there is too much network traffic, Xepr might hang up. Keep clearing up the **Result Dataset**. Avoid loading too many large data files into Xepr. Reduce network activities particularly when you connect to the spectrometer. These things will help you reduce the possibility of hanging up. If you fail to close Xepr in the normal way, we suggest the following. First try the normal procedure to exit Xepr. If that fails, try aborting by the methods described in this section. After you abort Xepr, turn off the console and turn on the console again before you reconnect to spectrometer.

Logout from the Current Account.

15.7.1

This is the easiest way to abort Xepr. Simply follow the logout procedure described in Appendix A of the Eleksys E 500 User's Manual: Basic Operations and the Xepr program will be terminated.

The Ctrl-C command

15.7.2

If you start the Xepr program from a UNIX shell, you can also use the **Ctrl-C** command to abort Xepr program. This is a graceful way to terminate the Xepr. Minimize (iconize) the Xepr window. Move the mouse pointer inside the UNIX shell window from which you launched Xepr program. Press the **<Ctrl>** key

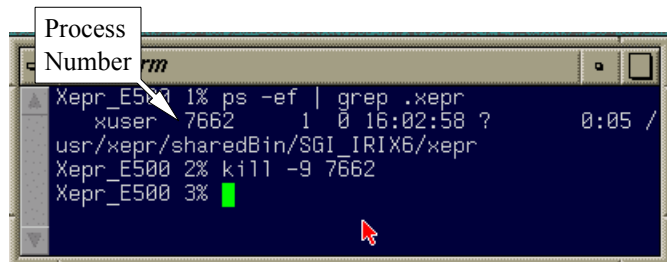
and the **C** key simultaneously. The Xepr program will be terminated. If you started Xepr by clicking the icon, the **Ctrl-C** command will not terminate the Xepr program.

If you start Xepr as a background process by using the shell command **Xepr &**, you need to use the command **jobs** to find out the job number of Xepr. Type **kill %<job number>** to terminate the Xepr program, where **<job number>** is the number you found with the **jobs** command.

The Kill Command

15.7.3

Alternatively you can use the **kill** command to abort Xepr without logging out of your account. You need to open a UNIX shell. Enter **ps -ef | grep .xepr**. This command will display the process number of Xepr. Enter **kill -9 <process#>** where **<process#>** is the number the **ps** command displayed. The Xepr program will be terminated.

A terminal window titled "rm" with a dark blue background and white text. The window shows the execution of three commands: 1. "ps -ef | grep .xepr" which outputs "Xepr_E500 1% ps -ef | grep .xepr", "xuser 7662 1 0 16:02:58 ?", and "usr/xepr/sharedBin/SGI_IRIX6/xepr". A white callout box with a black border and the text "Process Number" has a pointer to the "7662" in the second line of output. 2. "kill -9 7662" which outputs "Xepr_E500 2% kill -9 7662". 3. A green progress bar followed by "Xepr_E500 3%". A red mouse cursor is visible at the bottom right of the terminal window.

```
Xepr_E500 1% ps -ef | grep .xepr
xuser 7662 1 0 16:02:58 ?
usr/xepr/sharedBin/SGI_IRIX6/xepr
Xepr_E500 2% kill -9 7662
Xepr_E500 3% █
```

Figure 15-10 “Killing” Xepr.

The Xepr Printer

15.8

Reinstalling the Xepr Printer

15.8.1



Check if your printer is connected, turned on, and on-line.

The Xepr installation script will install the printer drivers for you. If you have printing problems with the Xepr program, first check that the printer is turned on, connected, and on line. Next, check the printer setup as described in Step 4. of Section 15.5.2. If the problem still exists, follow these instructions to reinstall the printer.

1. **Launch the Printer Manager.** Click the System menu of the Toolchest, and then the Printer Manager command. (See Figure 15-11.)

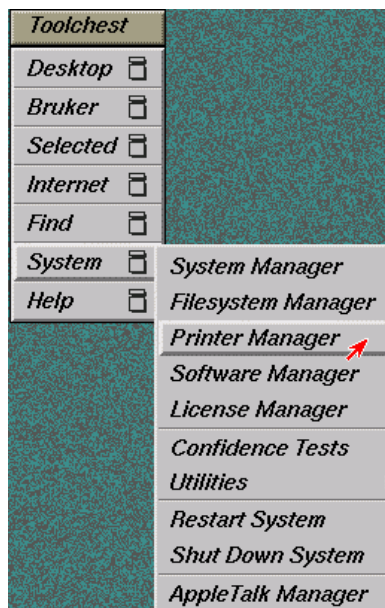


Figure 15-11 Starting the printer manager.

2. **Delete the printer that does not work.** If there is a listed XeprPrinter that does not work, select it by clicking its icon with the left mouse button and then click the Delete button. (See Figure 15-12.) If you did not log in as root or a privileged user, you will be requested to enter the root password.

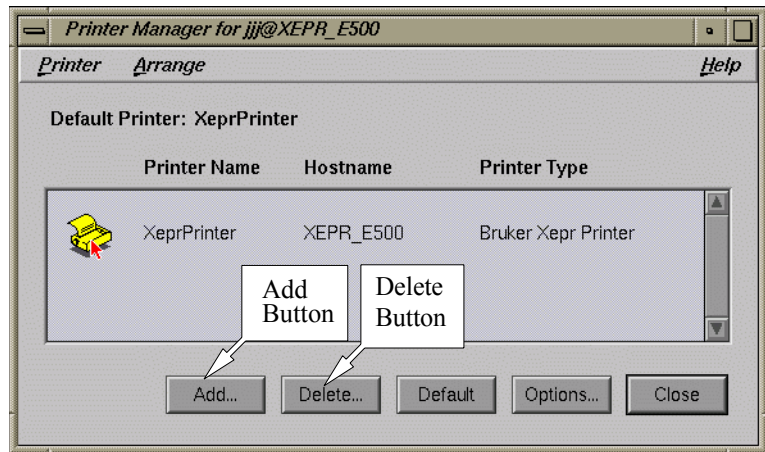


Figure 15-12 The Printer Manager dialog box.

3. **Add a new printer.** Click the Add button in the dialog box and a dialog box for installing a new printer appears. (See Figure 15-13.) Type XeprPrinter in the New Printer Name box. Select Local Host if the printer is connected directly to the UNIX workstation. You can enter some information about the location of this printer in case you want to share this printer with others. Choose Bruker as the Vendor and Bruker Xepr Printer as the Printer Type. If the printer is connected to the parallel port click Parallel and in the Parallel Ports box the words Built in will be highlighted. The printer that Bruker provides with the spectrometer is connected to the parallel

port. Click **Apply** and then **OK** to close the Printer Manager window.

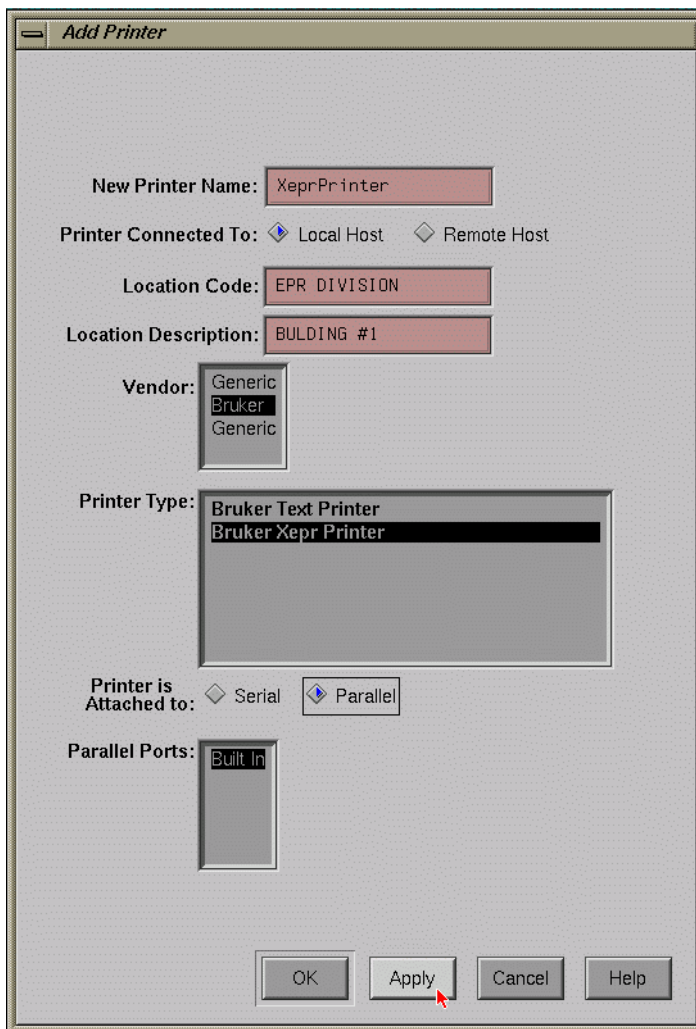


Figure 15-13 Adding a new printer.

4. **Set up the printer for Xepr.** Follow the instructions in Step 4. of Section 15.5.2 to set up and test the printer.

Printing a Spectrum to a File

15.8.2

Xepr not only supports printers from various vendors but can also print the spectrum to a graphics file such as a postscript, pdf, jpeg, tiff file, *etc.*

1. **Set the printer up.** Click the **Printer Setup** command in the **File** menu. Select the file type in the **Printer Type** drop-down list in the **PrinterSetup** dialog box. In our example, we are selecting a jpeg file. Then click **Set** to apply the changes.

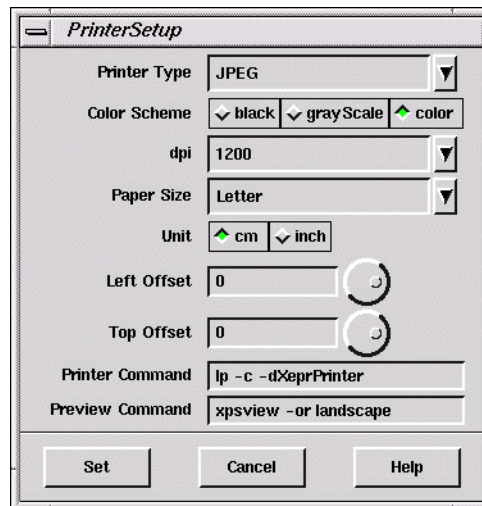


Figure 15-14 Setup for printing to a file.

2. **Print to a file.** Click Print Viewport in the File menu. Click File in the Destination section. Enter a file name and select the desired options for the data set plot. (See Figure 15-15.) Click Print and the file will be saved into your home directory.

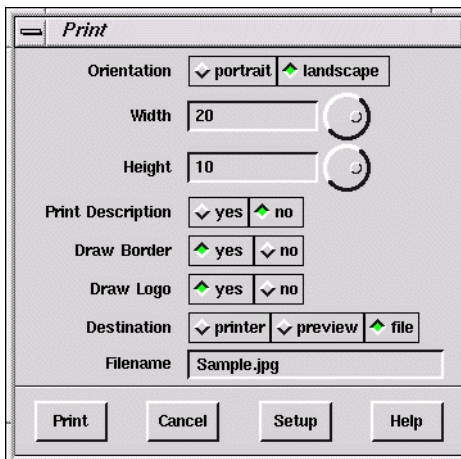


Figure 15-15 Printing to a file.

When Printing Goes Wrong

15.8.3

Sometimes, printing from the Xepr can go wrong. For example, if you choose the wrong printer by mistake in the **Printer Setup**, the printer spills out many pages of useless printing code. If that happens we recommend the following:

1. **Turn off the printer.** Turn off the printer immediately.
2. **Open the printer manager.** Click Toolchest > System > Printer Manager. (See Figure 15-16.)

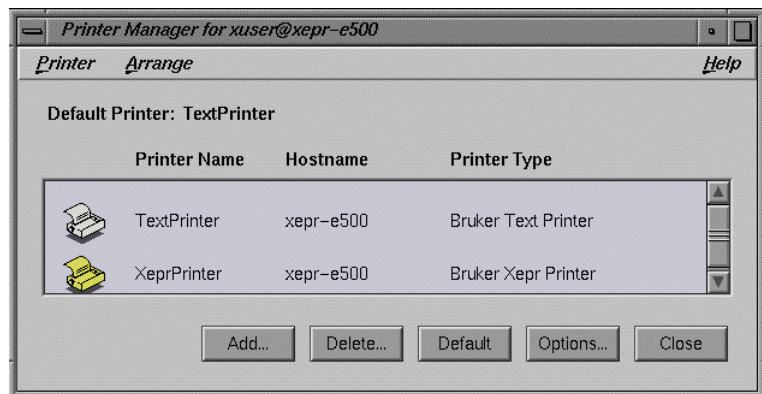


Figure 15-16 The Printer Manager dialog box.

3. **Check the printer status.** Double click the XeprPrinter icon in the Printer Manager dialog box.

4. **Remove all print jobs.** Highlight the job in the Printer Request Queue box and click Queue > Cancel in the menu bar to remove the print job from the queue. You need to remove all the jobs in the queue. (See Figure 15-17.)

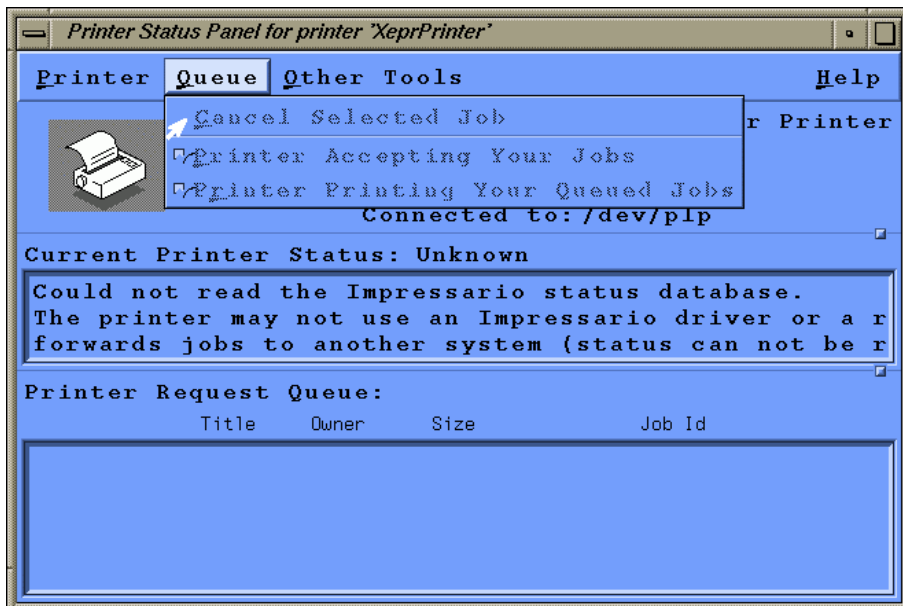


Figure 15-17 Removing print jobs.



Make sure you have selected the correct printer in the Printer Setup of Xepx.

5. **Check the printer setup.** Carefully follow the instructions described in Section 15.5.2, Step 4. to make sure the printer is set up correctly. You may need to reinstall the printer. (See Section 15.8.1.)
6. **Stop printing the many pages.** You may be able to stop the flood of paper with useless code by disconnecting the printer cable from the workstation, turning on the printer, and then reconnecting the printer cable.

Networking & Remote Operation

15.9

The Elexsys E 500 series EPR spectrometer is designed to operate in a networked environment. The workstation is equipped with two Ethernet cards. The primary network adapter (**ec0**) connects the workstation to the outside network. The secondary network adapter (**ec1**) is solely dedicated to communication between the workstation and the acquisition server of the EPR spectrometer. This client/server architecture resolves the conflict between multi-user/multi-tasking on the one hand and real-time functionality on the other hand. Therefore, multiple users from different locations are able to access Xepr remotely. However, excessive communication between the workstation and the outside world not only slows down the Xepr program but also opens the door for unauthorized access. Although the workstation is capable of functioning as a general network server it is highly recommended to use the Elexsys EPR workstation for acquiring EPR spectra only. The administrator of the workstation needs to carefully balance the security of the Elexsys workstation and public network access. This section discusses data transfer through the network and remote login to the workstation.

Remote Access to Xepr

15.9.1

You can log onto the workstation remotely, allowing multiple users access to the Xepr software at the same time. Each user remotely logs into the workstation and launches the Xepr software. In the following section we refer to the workstation directly connected to the Elexsys spectrometer as the EPR server and the other computers connected to the EPR server through network as clients.

Remote Logon to the EPR Server

15.9.2

The client and the EPR server should be assigned appropriate IP addresses and connected to the same network. The primary network connection (**ec0**) of the EPR server should be used to connect to the network. In order to display the Xepr application window on the client's monitor, you must either activate remote display on the client computer (if the client has a UNIX operating system) or use special software, such as PC-Xware (if the operating system is Windows). You also need a valid user account on the EPR server.

From a UNIX System

On the client computer, remote display is enabled by executing the shell command,

```
xhost + <EPR server's IP address>
```

In the shell window, enter

```
telnet <EPR server IP address>
```

Enter the user's name and password when you are prompted. After logging into the EPR server, type **Xepr** to launch the Xepr program.

From a Windows System

Refer to the documentation for your X-terminal application for details on enabling remote display.

After you remotely launch the Xepr program you can execute all the Xepr functions.

Remote Control of the Spectrometer

15.9.3

You can also control the EPR spectrometer remotely. Make sure there are no other Xepr users connected to the spectrometer. Click **Acquisition > Connect to Spectrometer** after you launch the Xepr program remotely. In the dialog box, enter the name of the spectrometer and then click **Connect**. Spectrometer control operations including tuning, acquiring spectra, *etc.* are now available to you through the Xepr software. Note that only one instance of the Xepr application can connect to the spectrometer at a time. After one connection is established, all other connections are blocked.

Data Transfer Via the Network

15.9.4

There are several ways to transfer data between the workstation and other computers connected to the network.

FTP FTP (File Transfer Protocol) is one of the most commonly used methods for data transfer. Elexsys data files are in BES³T[®] format. Each EPR spectrum consists of a binary data file with a *.DTA extension and an ASCII description file with a *.DSC extension. You must select binary transfer mode to transfer *.DTA files properly.

SAMBA SAMBA is an option for data sharing with the Microsoft Windows and other platforms.

NFS NFS (Network File System) can be configured on your workstation for data sharing with other UNIX workstations. There are also versions of NFS that run on Windows and Mac platforms.

Bruker delivers either SGI O2 or Linux workstations for the user interface of the Elexsys series EPR spectrometers. This chapter describes various system administration issues related to the Xepr program for the Linux workstations. The names, terms, or methods described in this chapter may be slightly different for the SGI O2 system, but the principles are the same. Refer to Chapter 15 and SGI documentations for details if you have an SGI O2 workstation.

Most of the commands and operations in this chapter require root or super user privileges. In addition, the commands also require at least some basic knowledge of the UNIX operating system. This chapter assumes that you already are familiar with general administration of the Linux operating system. There are many web sites and documentation describing Linux administration. Be cautious when making changes to the workstation configuration: some mistakes can lead to an unusable workstation. Consult your local Bruker EPR service representative if you are unsure of the changes you are making.

General Information

16.1

The Linux workstation comes with two Ethernet cards. Most of the GUI (Graphical User Interface) operations in this chapter are based on GNOME, the default desktop manager. If you prefer to use other GUIs refer to the corresponding manuals and documentation. Much of the configuration is based on Red Hat Linux 7.0. If your workstation is not running under Red Hat Linux 7.0 you may find the settings or commands are slightly different, but the essentials are the same.

If you wish to check the system information for your workstation, click **Main Menu > System > System Info**. To check the information and configuration of the network cards use the `/sbin/ifconfig` command in a shell window.

You must have X-windows running to launch the Xepr program.

The system as delivered by Bruker has two visible accounts: the **root** account with the password `xepr@linux` and the **xuser** account with the password `user@xepr`. For your security, change the root password immediately after the system has been installed.

Most of the Xepr files reside in the `/usr/xepr` folder. These files belong to root. Please do not alter those files unless it is absolutely necessary. Each user should have links to some of the files related to Xepr. Shared directories and files can be read or executed but not written/rewritten by the users.

Network Settings

16.2

eth0 and eth1

16.2.1

The two Ethernet cards in the workstation are labeled **eth0** and **eth1**. **eth0** is used for connecting to the outside world and is configured as the primary interface. **eth1** is devoted to communications between the workstation and the acquisition server (OS9) of the spectrometer and is configured as the secondary interface. When the workstation is delivered, the hostname and IP address of **eth0** are set to default values. You need to ask your local network administrator to assign a hostname and an IP address for your workstation. Change the hostname and IP address of **eth0** only. Never change the hostname and IP address of **eth1**. The shell command `/sbin/ifconfig` displays the details of all the Ethernet cards settings.

NFS (Network File System)

16.2.2

Xepr uses NFS to transfer files between the UNIX workstation and the acquisition server. Never turn off or deactivate NFS.

Name Server

16.2.3

Some name services allow you to use easily remembered host names instead of easily forgotten IP addresses. The most common services are files, DNS (Domain Name Service), and NIS (Network Information Service).

Files This is the most controllable way to provide network information. Entries in the `/etc/hosts` file connect an IP address with a corresponding host name. You can simply edit this file to define the names and IP addresses of the computers in your local network as well as on the Internet. The hosts file must contain the local host.

The format is:

IP address	hostname.subdomain.domain	hostname
127.0.0.1	localhost	
123.123.123	Elexsys.university.edu	Elexsys
192.168.99.1	Xepr	
192.168.99.5	E500	

DNS Your local network usually has one or more DNS servers. You can get the IP addresses of the DNS servers from your local network administrator.

NIS Although NIS offers many benefits, the complexity and integration with other products can lead to problems. Do not activate or use NIS on the EPR workstation.

You need to setup a proper lookup order for these servers. Login as root. Click Main Menu > Program > System > Control Panel.

Scroll down the panel and click the System Configuration button. (See Figure 16-1.)

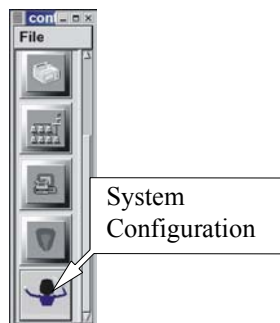


Figure 16-1 The System Configuration button.

On the left part of the window click Networking > Client tasks > Host name search path. The Name service access panel appears on the right side of the window. Select hosts, dns or hosts if you do not have a DNS server. (See Figure 16-2.) Click the Accept button at the bottom of the window and then click Act/Changes on the left. Click Quit to exit the System Configuration window.

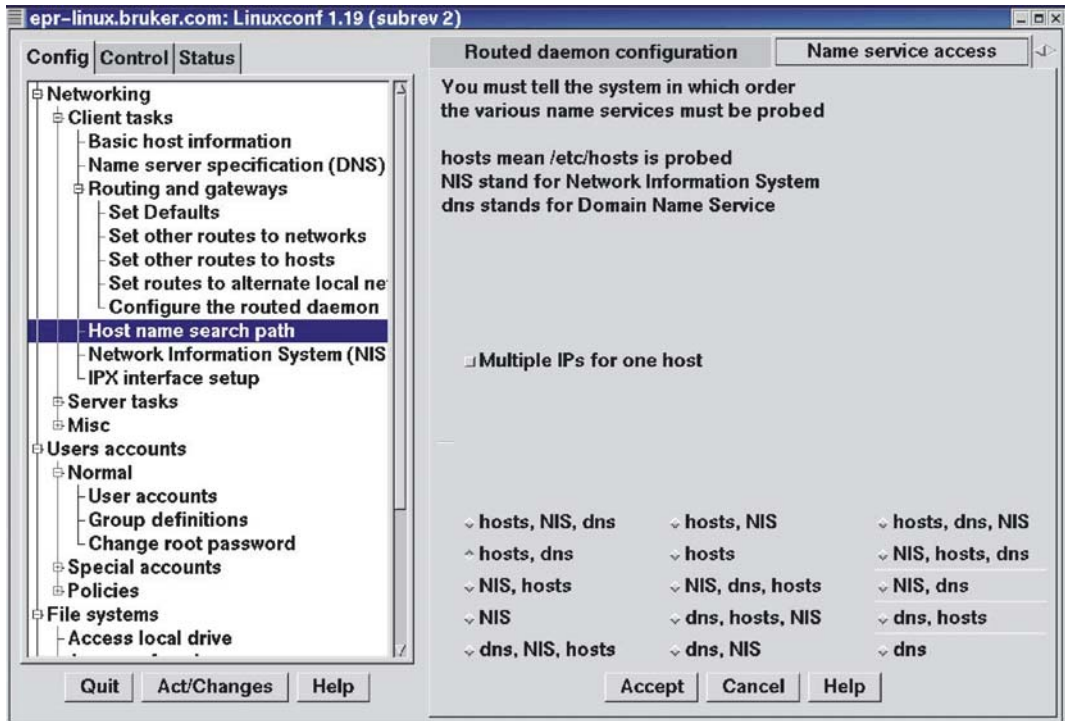


Figure 16-2 Name service access.

Routing

16.2.4

Although the EPR workstation has two Ethernet cards and is able to function as a router, doing so will jeopardize the communication between the workstation and the EPR acquisition server. Modification of the routing table could also direct network traffic to `eth1`, which would slow down the response of the Xepr program or even cause the Xepr program to hang up.

Linux Workstation Service Configuration 16.3

Flags

16.3.1

Flags are used to activate and deactivate various services that the workstation can offer. Here is the default configuration of the Linux workstation as delivered by Bruker. You can check the configuration by typing `/sbin/chkconfig --list` in a shell window. The command will list all the flag statuses under all running levels. Check the flag statuses under level five. `tftp` and `bootps` under `xinetd based services`, `network`, and `nfs` services must be on to allow the workstation to boot and communicate with the acquisition server properly. Although you can turn on more services than the default configuration, keep in mind that the more services you turn on, especially network related services, the slower the workstation responds. You may also need to turn off some services to increase the security. Table 16-1 shows a default service configuration for Red Hat Linux 7.0. The `nameserv` and `sophed` flags are for the XSophe program. If you do not have XSophe installed you may not have these services.

Flag	Status	Flag	Status
syslog	5:on	anacron	5:on
cron	5:on	httpd	5:on
netfs	5:on	apmd	5:on
network	5:on	arpwatch	5:off
random	5:on	atd	5:on
rawdevices	5:on	named	5:off
xf	5:on	keytable	5:on

Table 16-1 Standard configuration for workstation services running under level five.

Flag	Status	Flag	Status
amd	5:off	gpm	5:on
xinetd	5:on	innd	5:off
reconfig	5:on	ipchains	5:on
irda	5:off	snmpd	5:off
isdn	5:on	rhnsd	5:on
pcmcia	5:on	ypbind	5:off
kdcrotate	5:off	yppasswdd	5:off
kudzu	5:on	ypserv	5:off
linuxconf	5:on	autofs	5:on
lpd	5:on	bootparamd	5:off
mars-nwe	5:off	ciped	5:off
nfs	5:on	dhcpcd	5:off
nfslock	5:on	gated	5:off
sshd	5:on	kadmin	5:off
identd	5:on	kprop	5:off
portmap	5:on	krb524	5:off
postgresql	5:off	krb5kdc	5:off
pppoe	5:off	mcserv	5:off
rstatd	5:off	mysqld	5:off
usersd	5:off	nscd	5:off
rwalld	5:off	ntpd	5:off
rwhod	5:off	ups	5:off
smb	5:off	pxe	5:off

Table 16-1 Standard configuration for workstation services running under level five.

Flag	Status	Flag	Status
sendmail	5:on	rarpd	5:off
routed	5:off	nameserv	5:on
squid	5:off	sophed	5:on
vncserver	5:off		
xinetd based services:			
amandaidx:	off	comsat:	off
amidxtape:	off	imap:	off
finger:	on	imaps	off
linuxconf-web:	off	ipop2	off
rexec:	off	ipop3	off
rlogin:	on	pop3s	off
rsh:	on	eklogin:	off
swat:	off	gssftp:	off
ntalk:	off	klogin:	off
talk:	off	krb5-telnet:	off
telnet:	on	kshell:	off
tftp:	on	bootps:	on
wu-ftpd:	on		

Table 16-1 Standard configuration for workstation services running under level five.

Type `chkconfig --add <name>` to turn the service on where `<name>` is the service name. Type `chkconfig --del <name>` to turn off the service. You may need to indicate the run level by using the shell command:

```
chkconfig [--level <levels>] <name> <on|off|reset>.
```

Activate the service by the shell command:

```
service <name> start
```

or

```
service <name> restart
```

or rebooting the workstation.

Swap Space

16.3.2

The best way to determine the optimal swap space size is to try several sizes. The swap space size we recommend is four times the RAM size. When the swap space is not large enough there will be an error message in the `/var/log/messages` file. If you routinely process several large 2D datasets in Xepr you may need to increase your swap space. There are two types of swap space: the swap partition and the swap file. The swap file is slower than the swap partition. To increase the swap partition size you need to repartition the hard drive which wipes out the contents of the hard disk.

Security

16.3.3

Functionality, accessibility, and convenience may compromise the system security. Balancing these considerations is not a simple job. Refer to the workstation administrative manual for security settings that best suit you. We suggest the following.

Lock accounts

There are a few accounts such as the `nobody` and `lp` accounts utilized by Xepr and other application programs. Never delete the `nobody` and `lp` accounts. Instead, lock these accounts and other “open to the public” accounts.

Root and Super User

Avoid logging in as root or super user. Only do so when you must perform system administration tasks. Most tasks can be performed by logging in as a normal user and then switching to root by using the `su - shell` command. You should always run Xepr from a normal user account except for testing or diagnosis.

Service Access Control

You can control network access to tighten security. However, you need to be very careful not to block access for the acquisition server. Here is an extreme case where most services are blocked to all clients except the acquisition server. Modify the file `/etc/xinetd.conf` by adding the following line to the `default {}` section:

`only_from = 192.168.99.0/24`

where 192.168.99.0 is the subnet address assigned to the ACPU when the acquisition server was installed. If a different subnet address is set in the **ServerSetup** file you need to use it to replace the above IP address. 24 is the number of bits for the network and subnetwork. However, if you make the above change, you must append the following line to the file `/etc/xinetd.d/bootps`:

`only_from = 0.0.0.0/0`

Otherwise, the acquisition server will not boot.

You can put addresses of any trusted hosts/subnet in this entry separated by space so that these hosts can access the services controlled by xinetd.

There are many ways to tighten security. Bear in mind that any security settings must allow the acquisition server to access **bootp**, **fttp**, **NFS**, and other services.

Reinstalling or Upgrading Xepr 16.4

List of Important Files 16.4.1

The following files are very important and must be backed up before you reinstall the operating system or the Xepr software.

License File	<code>/usr/xepr/sharedSetup/license.dat</code> or <code>/usr/Bruker/flexIm/licenses/license.dat</code>
Hall Probe Calibration File	<code>/usr/xepr/AcquisitionServer/OS9/Servers/FUSETUP/FUEr032t/hall#####.cal</code> and <code>hall#####.dat</code> (where ##### is the serial number.)
ACPU Information	<code>/usr/xepr/AcquisitionServer/admin/ServerSetup</code>
Signal Channel Calibration Files	<code>/usr/xepr/AcquisitionServer/OS9/Servers/<Server name: e.g. E500, E580, etc.>/FUSETUP/FUSct/CalibData/*.cal</code>
Spectrometer.ini File	<code>/usr/xepr/AcquisitionServer/OS9/Servers/<Server name>/Spectrometer.ini</code>
Pulse System Configuration Files	If you have an E 580 or E 680: <code>/usr/xepr/AcquisitionServer/OS9/Servers/<Server name>/FUSETUP/FUFTEpr/XBand (and/or WBand)/*.cfg</code>

Performing an Installation or Upgrade

16.4.2

1. **Check the current version of Xepr.** Use the shell command `Xepr -i` to determine the version of the currently installed Xepr software.
2. **Follow the instructions in the release note.** Login as root to install Xepr. Read the release note on the Xepr CD first. Follow the instructions for installing Xepr. Use the default installation settings to avoid any unforeseen problems. Sometimes you may need to delete the `/usr/xep` folder before you install the Xepr software.
3. **Restore the backed up files.** Put the files you backed up in Section 16.4.1 into the folders from which they came.
4. **Install the acquisition server.** Each time you reinstall Xepr or upgrade the acquisition package you must reinstall the acquisition server. The acquisition server installation script is located in the folder `/usr/xep/AcquisitionServer/admin/`. Make sure you have the correct `ServerSetup` file. Change to the above directory and type `./installServer` to install the acquisition server.
5. **Reboot the UNIX workstation.** If you do not, the upgrade or installation will not work properly.

Initial Setup of Xepr

16.5

Creating a New User Account

16.5.1

Normally users have their own accounts. A user's account has a user's account name, user ID, and a password to log into the account. As `root` you can create a new user account by typing `useradd <newUserName>; passwd <newUserName>` in a shell window. It will prompt you for the password. Enter and confirm the temporary password for the new user. Linux creates a private group for the new user account. You can add the new user to the default `users` group by typing `usermod -G users <newUserName>` in a shell window. We recommend adding all users to the `users` group so that they can share their files. If you prefer to use a GUI refer to the corresponding manual to find out how to create a new account. Make sure the new account is in the `users` group. After new users log into their account, they can customize the desktop, windows, UNIX shell type, and other features. They should change their password the first time login. Refer to the **Integrated Help System** for details on configuring the account. When running Xepr for the first time in the new account, Xepr will automatically configure its home directory to set up Xepr.

Setting Up Xepr for a New Account

16.5.2



Linux is case sensitive. You need to capitalize the X when you type the command Xepr.

1. **Start the application launcher.** Click Main menu > Panel > Add to panel > Launcher.
2. **Set up the Xepr launcher.** In the Create launcher applet dialog box enter Xepr for the program name. In the Command line enter Xepr. Click the Icon button. (See Figure 16-3.)

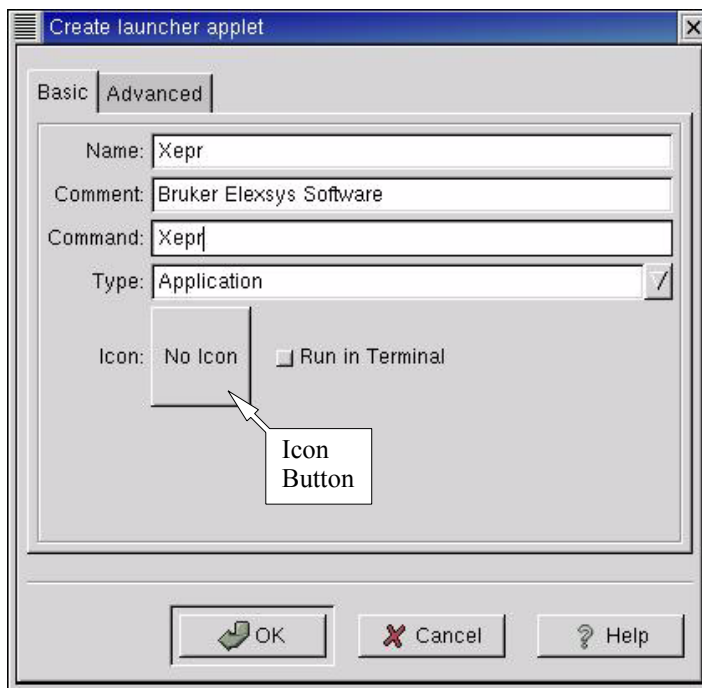


Figure 16-3 Setting up the Xepr launcher applet.

3. **Select the Xepr icon.** In the Choose an icon window scroll down and click on xepriicon.png to highlight it. Click the OK button to exit. (See Figure 16-4.)

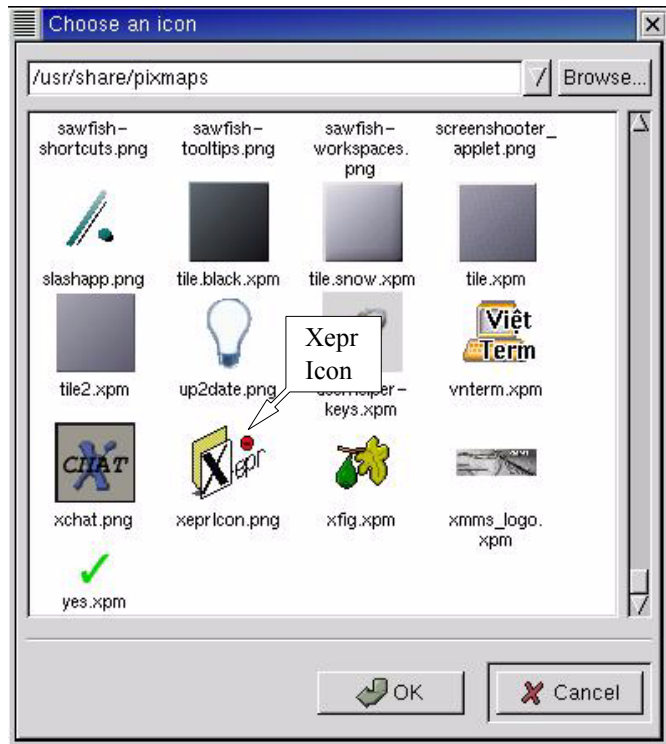


Figure 16-4 Selecting the Xepr icon.

4. **Exit the launcher applet creator.** After selecting the Xepr icon, the icon will appear next to Icon in the Create launcher applet window. Click OK to exit. A launcher with the Xepr icon will appear in the bottom panel. You can drag the icon onto to the desktop if you want to launch the Xepr software from the Desktop.



Linux is case sensitive.
You need to capitalize
the X when you type
the shell command
Xepr.

5. **Start the Xepr application for the first time.** Single click the Xepr icon in the bottom panel or double click the Xepr icon on the desktop. The Xepr program will automatically configure your account to run Xepr and launch the Xepr program. You can perform the same operation directly from a shell. Simply type Xepr in a shell.

Setting Up the Properties of the Xepr Software

16.5.3

1. **Maximize the Xepr window.** Since Xepr has so many components, a full screen window is recommended. Click the maximize button on the right top corner.
2. **Select the monitoring panel position.** First make sure the spectrometer is on and booted. You can choose either to put the monitoring panel above or below the Viewport. Click the Properties button in the menu bar, and then Panel Properties. A dialog window will appear. Click either Top or Bottom and then OK. (See Figure 16-5.)

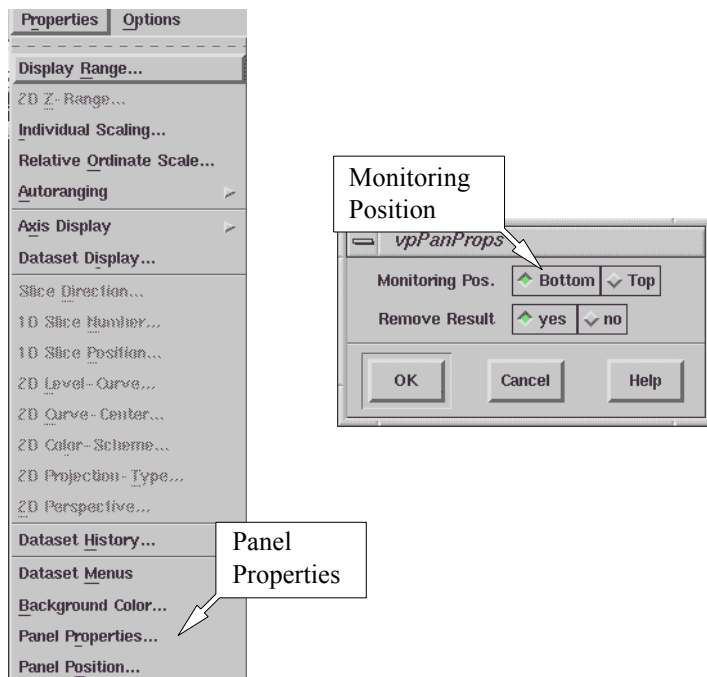


Figure 16-5 Selecting the monitoring panel position.

3. **Connect to spectrometer.** Click Acquisition in the menu bar and then Connect To Spectrometer. A dialog box will appear prompting for the Server Name. Enter the acquisition server's name in the blank box and click OK. (See Figure 16-6.) If you do not know the acquisition server's name, ask the system administrator. The typical servers names are e500, e580, e600, or e680, depending on the type of spectrometer.

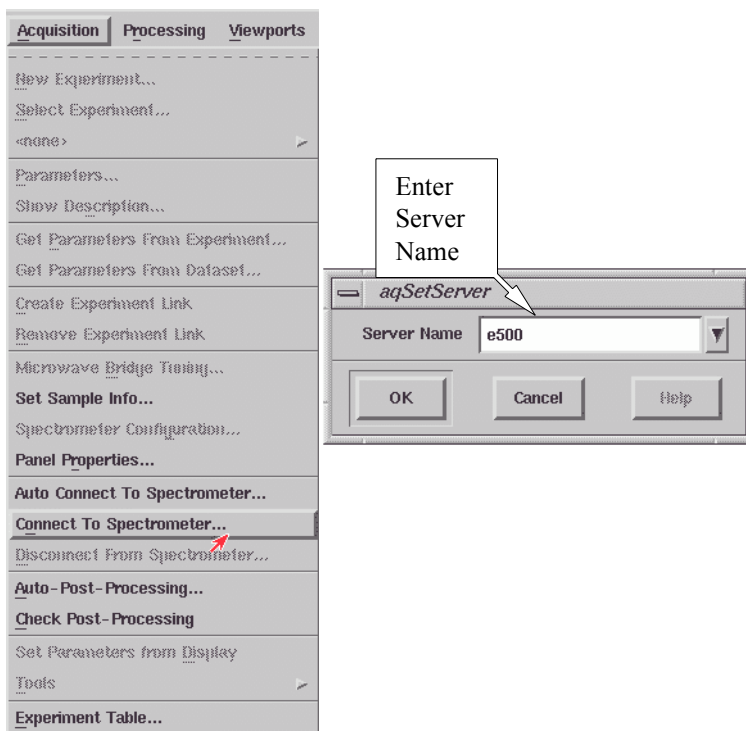


Figure 16-6 Connecting to the spectrometer.

4. **Set up the printer.** Click File in the menu bar and then Setup Printer. A dialog box appears. Click the arrow button next to the Printer Type. A drop-down list of printers will appear. Select the correct Printer Type for your printer. The Printer Command should be set as `lpr -P <printername>`, where <printername> should be replaced by the name of the printer. You can also enter `lpr` which will print to the default printer. The Preview Command should be set to `gv -landscape -media letter` (or A4). Now that the critical settings are selected, you can choose the other options which are available in the dialog box. Click Set to exit this dialog box. (See Figure 16-7.)

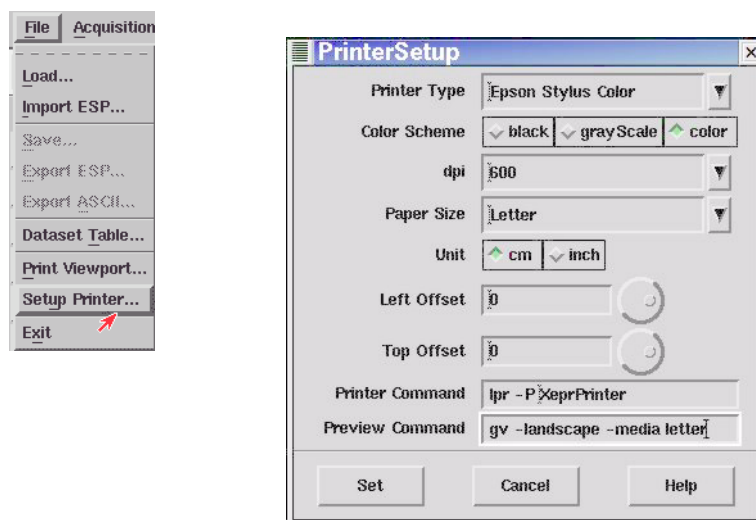


Figure 16-7 Setting up the printer.

5. **Test the printer.** Load an EPR spectrum. Display the spectrum in the viewport. Click Print in the File drop-down menu. A dialog box appears. Select the proper Orientation, Width, Height, and other parameters. Click printer as the Destination. Click the Print button. (See Figure 16-8.)

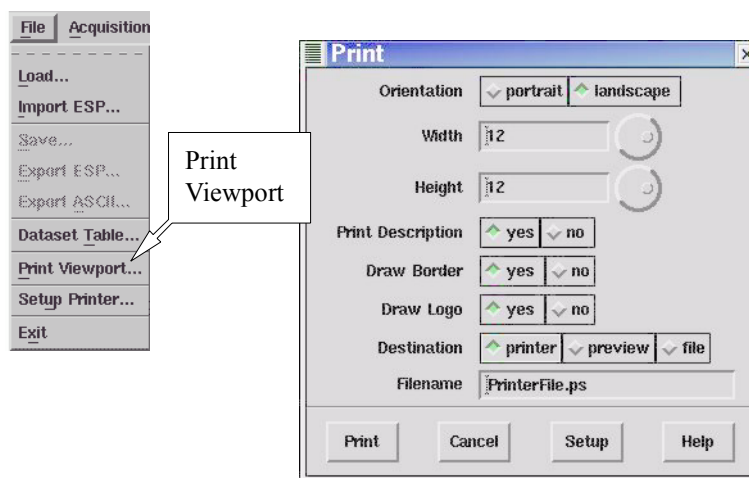
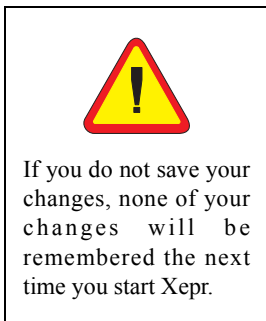


Figure 16-8 Testing the printer.

6. **Disconnect from the spectrometer.** Click Acquisition > Disconnect From Spectrometer. The monitoring panel will disappear.



7. **Exit Xepr.** Click **File** and then **Exit** in the menu bar. A dialog box appears asking you if you want to save the changes. Click **Yes** and then **OK** to exit Xepr program. (See Figure 16-9.) Next time you launch the Xepr software, you will find the **Server Name** appearing in the **Connect to Spectrometer** dialog box. The printer will be ready to print.

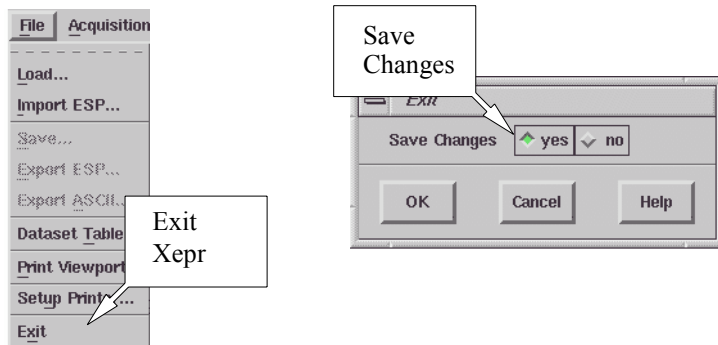


Figure 16-9 Saving the changes and exiting.

8. **Logout from your account.** It is always a good idea to logout from your account when you finish your work.

How to Start and Stop Xepr

16.6

This section describes different ways to start the Xepr program. It also describes how to abort the Xepr program when things go wrong and the system hangs up.

Starting Xepr from a UNIX shell

16.6.1

There are two basic ways to launch the Xepr program. One is to double-click the Xepr icon on the desktop or single-click it in the bottom panel. The alternative way to start Xepr is to launch Xepr from a UNIX shell.

1. **Open a UNIX shell.** Click the right mouse button to open a menu and then left click on **New > Terminal**. You can also click the **Terminal** icon in the bottom panel.

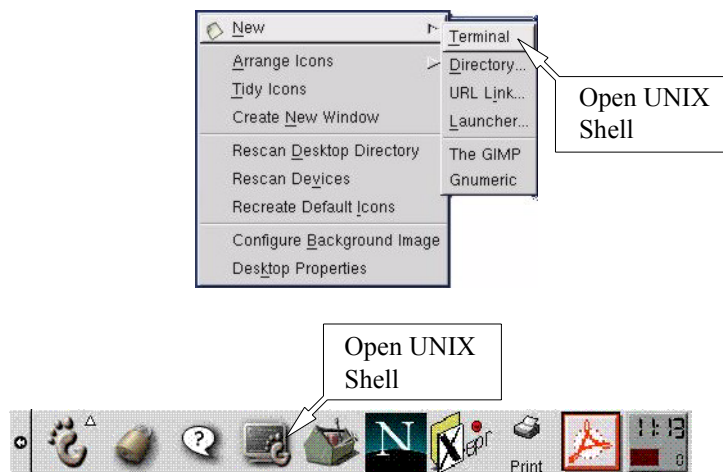


Figure 16-10 Opening a UNIX shell.



UNIX is case sensitive. You need to capitalize the X when you type the shell command Xepr.

2. **Start Xepr with a shell command.** Type Xepr, and then press the Enter key. The Xepr program will start. You can leave the UNIX shell window open for now. That gives you an option to abort Xepr program. (See the next section.)

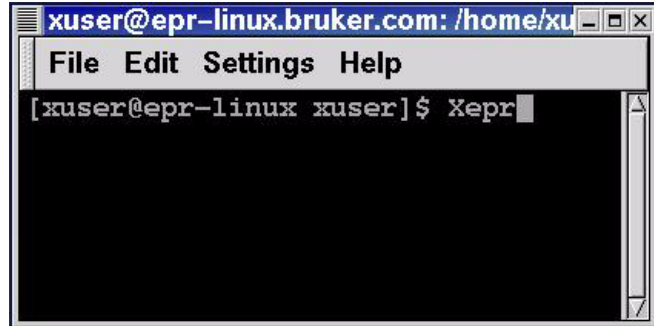


Figure 16-11 Launching the Xepr program from a UNIX shell.

Aborting Xepr

16.7

Normally you need to exit Xepr by clicking **File** in the menu bar, and then clicking **Exit** to terminate the Xepr program. However, when something goes wrong or the system hangs up, you may not be able to exit Xepr by normal methods. Keep in mind, sometimes Xepr needs more time to execute certain functions especially the communications between the system and slow devices. It may not respond to your command instantly. Give Xepr some extra time. Avoid continuously clicking the mouse button: it only make the system even busier. When memory is running too low or there is too much network traffic, Xepr might hang up. Keep clearing up the **Result Dataset**. Avoid loading too many large data files into Xepr. Reduce network activities particularly when you connect to the spectrometer. These things will help you reduce the possibility of hanging up. If you fail to close Xepr in the normal way, we suggest the following. First try the normal procedure to exit Xepr. If that fails, try aborting by the methods described in this section. After you abort Xepr, turn off the console and turn on the console again before you reconnect to spectrometer.

Logout from the Current Account.

16.7.1

This is the easiest way to abort Xepr. Simply follow the logout procedure described in Appendix B of the Eleksys User's Manual: Basic Operations and the Xepr program will be terminated.

The Ctrl-C command

16.7.2

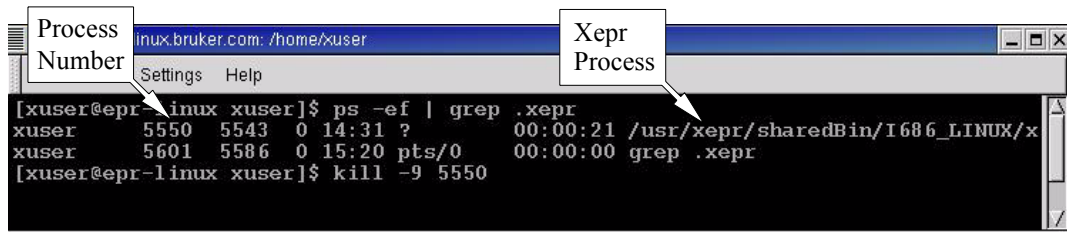
If you start the Xepr program from a UNIX shell, you can also use the **Ctrl-C** command to abort Xepr program. Minimize (iconize) the Xepr window. Move the mouse pointer inside the UNIX shell window from which you launched Xepr program. Press the **<Ctrl>** key and the **c** key simultaneously. The Xepr

program will be terminated. If you started Xepr by clicking the icon, the Ctrl-C command will not terminate the Xepr program.

The Kill Command

16.7.3

Alternatively you can use the kill command to abort Xepr without logging out of your account. You need to open a UNIX shell. Enter `ps -ef | grep .xepr`. This command will display the process number and the process name (`/usr/xexpr/sharedBin/I686_LINUX/xexpr`) of Xepr. Enter `kill -9 <process#>` where the `<process#>` is the process number that the `ps` command displayed. (See Figure 16-12.) The Xepr program will be terminated. Identify the correct process for Xepr. The `ps` command may pick up the `grep .xepr` process which is not the process you want to terminate.



The image shows a terminal window titled "linux.brucker.com: /home/xuser". The terminal displays the following commands and output:

```
[xuser@epr-linux xuser]$ ps -ef | grep .xepr
xuser      5550  5543  0 14:31 ?        00:00:21 /usr/xexpr/sharedBin/I686_LINUX/x
xuser      5601  5586  0 15:20 pts/0    00:00:00 grep  .xepr
[xuser@epr-linux xuser]$ kill -9 5550
```

Two callout boxes are present: "Process Number" pointing to the first column of the `ps` output, and "Xepr Process" pointing to the `/usr/xexpr/sharedBin/I686_LINUX/x` path in the same line.

Figure 16-12 “Killing” Xepr.

The Xepr Printer

16.8

Installing a Printer

16.8.1



Check if your printer is connected, turned on, and on-line.

The printer coming with the spectrometer should already be installed and configured when the Elexsys spectrometer was installed. If you have printing problems with the Xepr program, first check the printer setup described in Section 16.5.3, Step 4. You may also consult in the Linux documentations about the printer. If the problem still exists you can follow these instructions to reinstall the printer. Make sure the printer is physically connected before you start.

1. **Launch the Print System Manager.** Login as root. Click Main Menu > Programs > System > Control Panel, and then the Print System Manager button. (See Figure 16-13.)

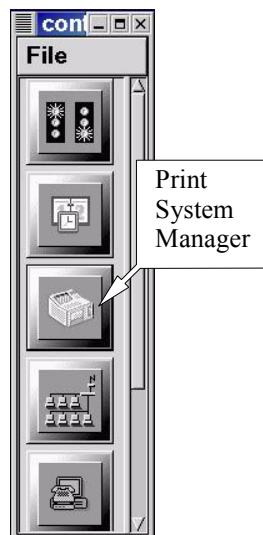


Figure 16-13 Starting the print system manager.

2. **Delete the printer that does not work.** If the listed printer does not work, select it by clicking its entry with the left mouse button and then click the Delete button. (See Figure 16-14.).

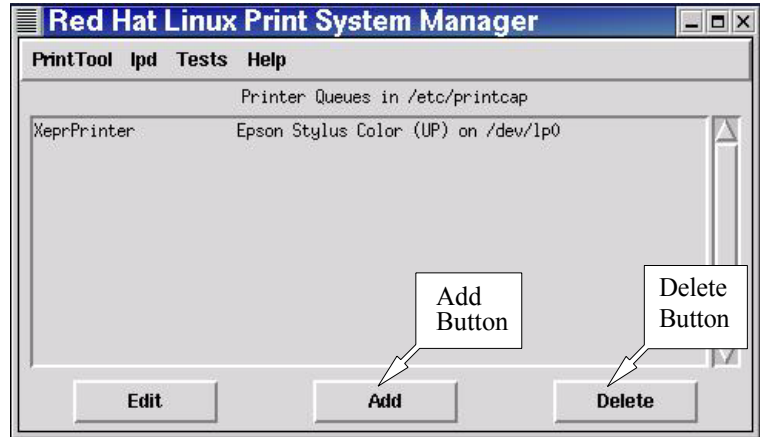


Figure 16-14 The Print System Manager dialog box.

3. **Add a new printer.** Click the Add button in the dialog box and a dialog box for adding a new printer appears. (See Figure 16-15.) Select Local Printer and click OK.

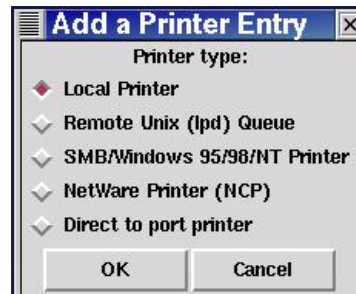


Figure 16-15 Adding a new printer.

4. **Identify the printing device.** An info box appears telling you what printing devices are detected. If no device is detected, check the hardware and its connections. Otherwise, click OK to close this window. (See Figure 16-16.)



Figure 16-16 Info window identifying printing devices.

5. **Name the printer.** Type in a name for the printer in the Names box. (See Figure 16-17.) Avoid using spaces in the name. Change the Spool Directory to `/var/spool/lpd/<printerName>`. The Printer Device should match the detected device in the info box. Do not close this window yet. Instead, click the **Select** button next to Input Filter.

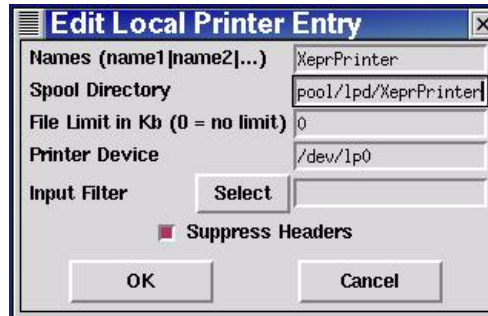


Figure 16-17 Naming the printer.

6. **Configure the filter.** In the Configure Filter window select the proper printer model. On the right side of the window, select the desired Paper Size and Color Depth/Uniprint Mode. Also make your Printing Options choices. These options may need to be set differently for different printers. Usually choosing all three options works. After you finish configuring the filter, click OK to exit. (See Figure 16-18.)

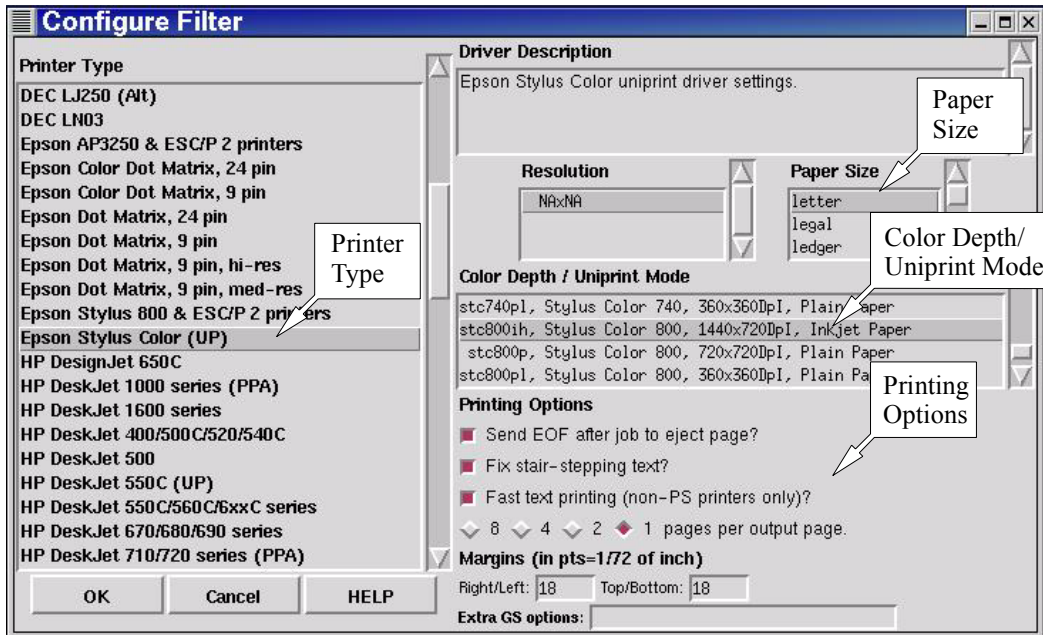


Figure 16-18 Configuring the filter.

7. **Exit Local Printer Entry.** The filter you configured will automatically fill in the Input Filter box in the Local Printer Entry window. Click OK button to exit. (See Figure 16-19.)



Figure 16-19 Closing the Local Printer Entry window.

8. **Restart lpd.** In the Print System Manager window click lpd > Restart lpd. (See Figure 16-20.)



Figure 16-20 Restarting lpd.

9. **Test the printer.** Select the printer by left clicking on the printer entry. Click **Tests > Print ASCII test page** to verify the text printer. (See Figure 16-21.) Click **Tests > Print Postscript test page** to verify that Postscript printing is working properly. If the test page is printed out correctly close this window and the **Control Panel** window. If there is a problem follow the instructions on the test page or consult the Linux documentations.

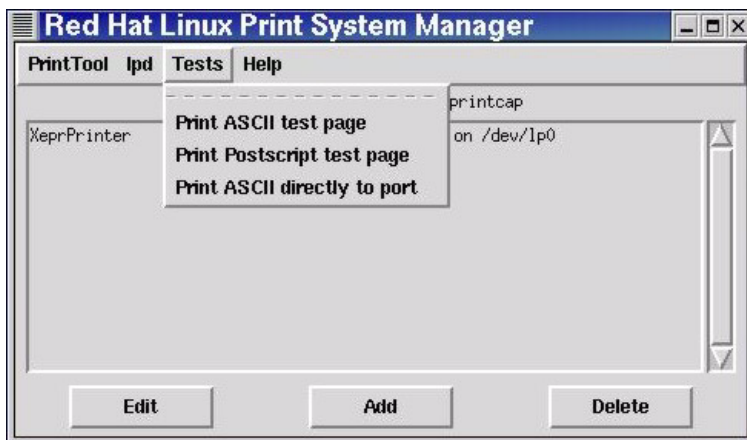


Figure 16-21 Testing the printer.

10. **Set up the printer for Xepr.** Follow the instruction in Section 16.5.3, Step 4. to set up the printer for Xepr and test the printer.

Printing a Spectrum to a File

16.8.2

Xepr can print a spectrum to a graphics file such as a postscript, pdf, jpeg, tiff file, *etc.*

1. **Set the printer up.** Click the Printer Setup command in the File menu. Select the file type in the Printer Type drop-down list in the PrinterSetup dialog box. In our example, we are selecting a jpeg file. Then click Set to apply the changes.

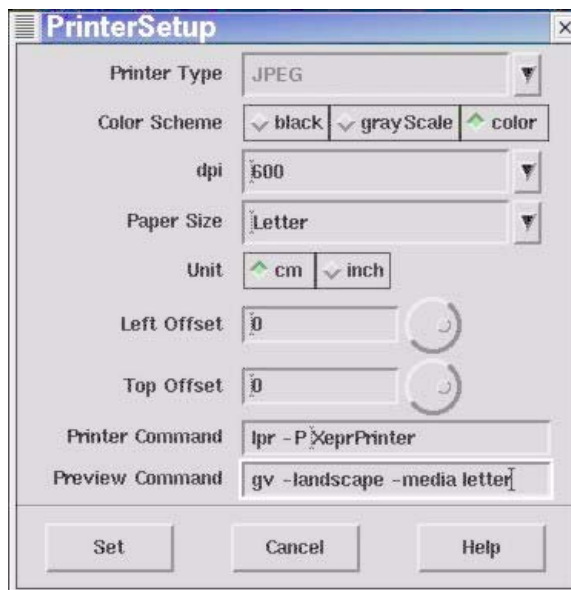


Figure 16-22 Setup for printing to a file.

2. **Print to a file.** Click Print Viewport in the File menu. Click File in the Destination section. Enter a file name and select the desired options for the data set plot. (See Figure 16-23.) Click Print and the file will be saved into your home directory.

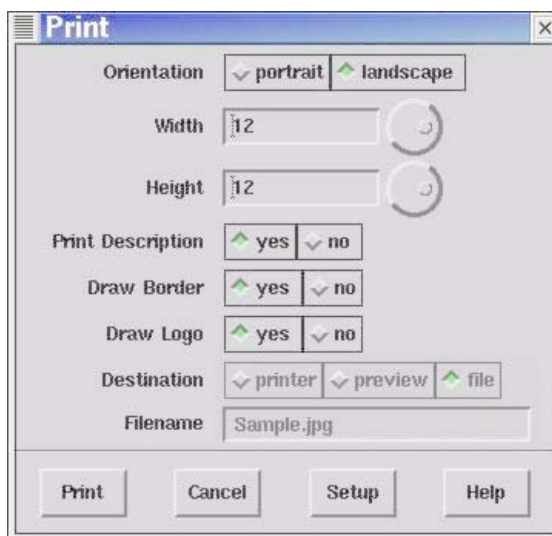


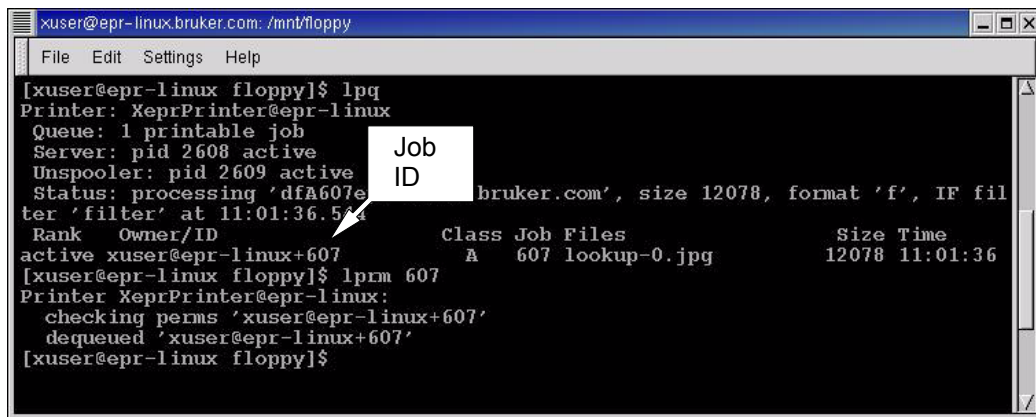
Figure 16-23 Printing to a file.

When Printing Goes Wrong

16.8.4

Sometimes, printing from the Xepr can go wrong. For example, if you choose the wrong printer by mistake in the Printer Setup, the printer spills out many pages of useless printing code. If that happens we recommend the following:

1. **Turn off the printer.** Turn off the printer immediately.
2. **Check the print queue.** Use the `lpq` command to check the print queue of the default printer. The `-P<printerName>` option allows you to specify the printer if your printer is not the default printer. You will find the print job ID numbers from the response of this command. (See Figure 16-25.)
3. **Remove all print jobs.** Remove all the print jobs using the shell command `lprm <jobID>`. (See Figure 16-25.) You may need root privilege to remove any printing jobs that belong to someone else.



```
xuser@epr-linux.bruker.com: /mnt/floppy
File Edit Settings Help
[xuser@epr-linux floppy]$ lpq
Printer: XeprPrinter@epr-linux
Queue: 1 printable job
Server: pid 2608 active
Unspooler: pid 2609 active
Status: processing 'dfA607e... brucker.com', size 12078, format 'f', IF fil
ter 'filter' at 11:01:36.544
Rank  Owner/ID                Class Job Files                Size Time
active xuser@epr-linux+607    A    607 lookup-0.jpg                12078 11:01:36
[xuser@epr-linux floppy]$ lprm 607
Printer XeprPrinter@epr-linux:
  checking perms 'xuser@epr-linux+607'
  dequeued 'xuser@epr-linux+607'
[xuser@epr-linux floppy]$
```

Figure 16-25 Removing a print job.



Make sure you have selected the correct printer in the **Printer Setup** of Xepr.

4. **Check the printer setup.** Carefully follow the instructions described in Section 16.5.3, Step 4. to make sure the printer is set up correctly. You may need to reinstall the printer. (See Section 16.8.1.)
5. **Stop printing the many pages.** You may be able to stop the flood of paper with useless code by disconnecting the printer cable from the workstation, turning on the printer, and then reconnecting the printer cable.

Networking & Remote Operation

16.9

The Eleksys E 500 series EPR spectrometer is designed to operate in a networked environment. The workstation is equipped with two Ethernet cards. The primary network adapter (`eth0`) connects the workstation to the outside network. The secondary network adapter (`eth1`) is solely dedicated to communication between the workstation and the acquisition server of the EPR spectrometer. This client/server architecture resolves the conflict between multi-user/multi-tasking on the one hand and real-time functionality on the other hand. Therefore, multiple users from different locations are able to access Xepr remotely. However, excessive communication between the workstation and the outside world not only slows down the Xepr program but also opens the door for unauthorized access. Although the workstation is capable of functioning as a general network server it is highly recommended to use the Eleksys EPR workstation for acquiring EPR spectra only. The administrator of the workstation needs to carefully balance the security of the Eleksys workstation and public network access. This section discusses data transfer through the network and remote login to the workstation.

Remote Access to Xepr

16.9.1

You can log onto the workstation remotely, allowing multiple users access to the Xepr software at the same time. Each user remotely logs into the workstation and launches the Xepr software. In the following section we refer to the workstation directly connected to the Eleksys spectrometer as the EPR server and the other computers connected to the EPR server through network as clients.

Remote Logon to the EPR Server

16.9.2

The client and the EPR server should be assigned appropriate IP addresses and connected to the same network. The primary network connection (**eth0**) of the EPR server should be used to connect to the network. In order to display the Xepr application window on the client's monitor, you must either activate remote display on the client computer (if the client has a UNIX operating system) or use special software, such as PC-Xware (if the operating system is Windows). You also need a valid user account on the EPR server.

From a UNIX System

On the client computer, remote display is enabled by executing the shell command,

```
xhost + <EPR server's IP address>
```

In the shell window, enter

```
telnet <EPR server IP address>
```

Enter the user's name and password when you are prompted. After logging into the EPR server, type **Xepr** to launch the Xepr program.

From a Windows System

Refer to the documentation for your X-terminal application for details on enabling remote display.

After you remotely launch the Xepr program you can execute all the Xepr functions.

Remote Control of the Spectrometer

16.9.3

You can also control the EPR spectrometer remotely. Make sure there are no other Xepr users connected to the spectrometer. Click **Acquisition > Connect to Spectrometer** after you launch the Xepr program remotely. In the dialog box, enter the name of the spectrometer and then click **Connect**. Spectrometer control operations including tuning, acquiring spectra, *etc.* are now available to you through the Xepr software. Note that only one instance of the Xepr application can connect to the spectrometer at a time. After one connection is established, all other connections are blocked.

Data Transfer Via the Network

16.9.4

There are several ways to transfer data between the workstation and other computers connected to the network.

FTP FTP (File Transfer Protocol) is one of the most commonly used methods for data transfer. Elexsys data files are in BES³T[®] format. Each EPR spectrum consists of a binary data file with a *.DTA extension and an ASCII description file with a *.DSC extension. You must select binary transfer mode to transfer *.DTA files properly.

SAMBA SAMBA is an option for data sharing with the Microsoft Windows and other platforms.

NFS NFS (Network File System) can be configured on your workstation for data sharing with other UNIX workstations. There are also versions of NFS that run on Windows and Mac platforms.

Acquisition Server Diagnostics 17

The Elexsys E 500 series EPR spectrometer is a multi-computer system. It consists of a UNIX workstation, an OS9 acquisition server CPU (ACPU), and a transputer for each transputer controlled device. Normally, when you turn the console on, everything boots up and initializes in an orderly fashion. This chapter describes what to do in the unlikely event that it does not.

What Is Normal?

17.1

This section describes the connections, indicators, and booting procedures for a normal spectrometer booting process.

Connections

17.1.1

Figure 17-1 shows the important connectors and indicators of the ACPU.

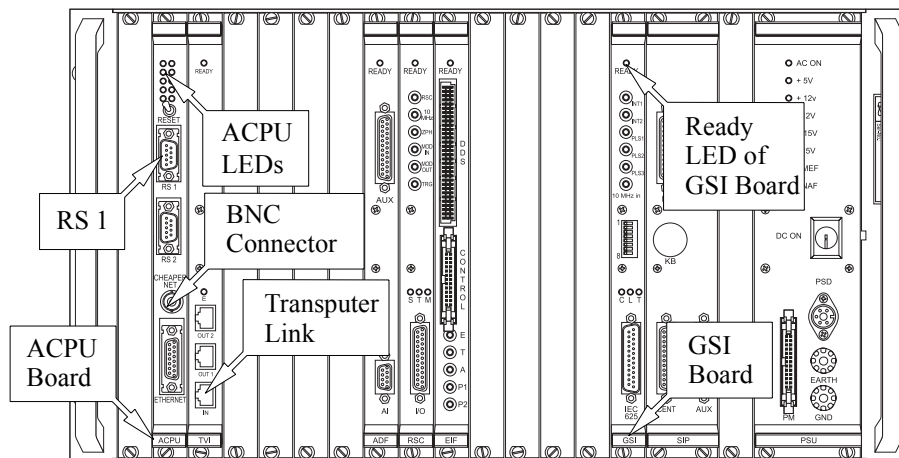



Figure 17-1 The ACPU rack.

The ACPU is connected to the UNIX workstation via a thinnet cable (RG-58 coaxial cable), hub, and a RJ-45 (twisted pair) cable. Figure 17-2 shows how all these components are connected together.



Note that both ends of the thinnet cable must be terminated with 50 Ω loads.

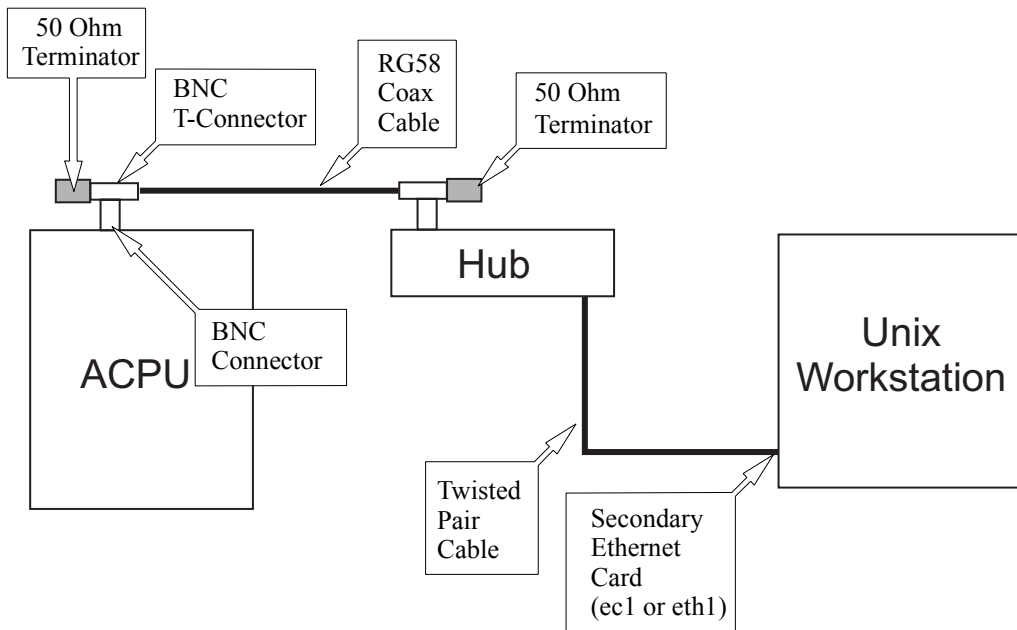


Figure 17-2 The Ethernet connections.

See Section 13.2.4 for information regarding the transputer connections.

A Normal Booting Process

17.1.2

A normal booting process proceeds as follows.

1. **Turn on the hub's power.**
2. **Boot up the UNIX workstation.** If the workstation is not already on, you must turn on the workstation. The UNIX workstation needs to be fully booted up and running before you turn the spectrometer console on.
3. **Turn on the Eleksys console.** Press the ON button of the Eleksys console. The ACPU should start booting. The Acquisition Server CPU (ACPU) board has two red and eight green LEDs on its front panel. To judge whether the ACPU boots properly watch these LEDs closely during booting. When the ACPU boots, LEDs A and B should blink in the first few seconds after power up. (See Figure 17-3.) This may take a minute. (See Figure 17-1.) The following indicates the function of some of the LEDs on the ACPU board.

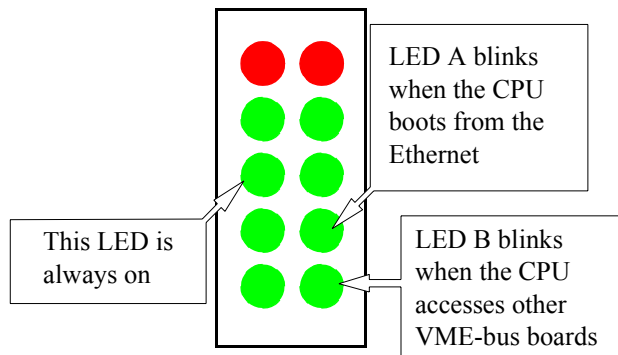
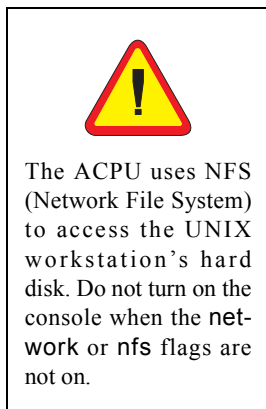


Figure 17-3 The LEDs on ACPU board.

4. **Boot up the transputer devices.** After the ACPU boots up from the UNIX workstation, it immediately begins to boot the transputer devices. As each device is identified its R LED will blink once briefly. When the transputer device is booting, the R LED blinks rapidly about 10 times and then stays on. Upon successful booting of the ACPU and the transputer devices, the GSI board's Ready LED will light. (See Figure 17-1.)
5. **Connect to the spectrometer.** Select the Connect to Spectrometer command in the Acquisition menu.

Trouble Shooting

17.2



In most cases, the server name is the same as the spectrometer type *e.g.*, E 500.

If you were not able to connect to the spectrometer in the previous section, follow the primary diagnostics chart to determine the source of the problem.

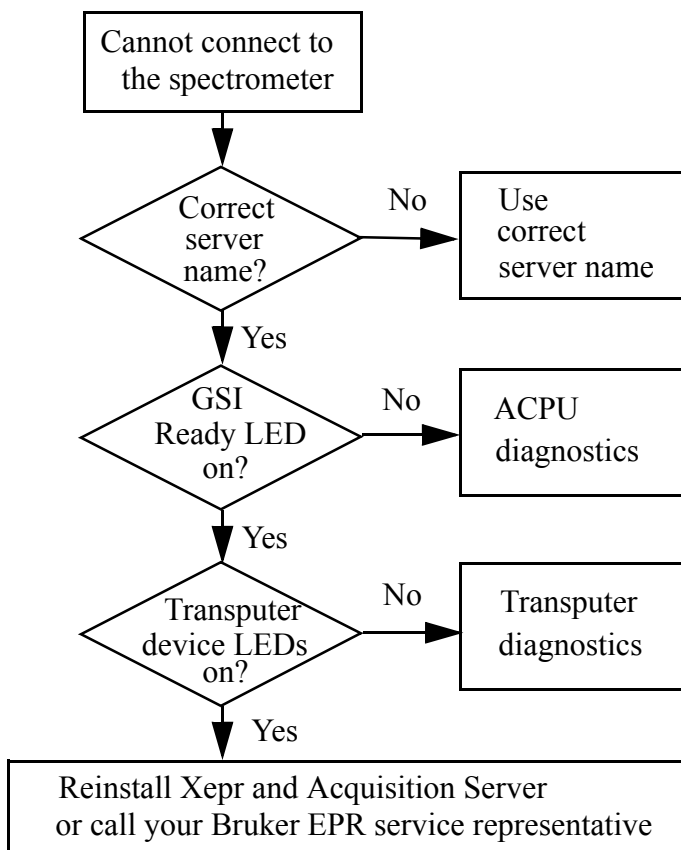


Figure 17-4 The primary diagnostics chart.

ACPU Diagnostics

17.2.1

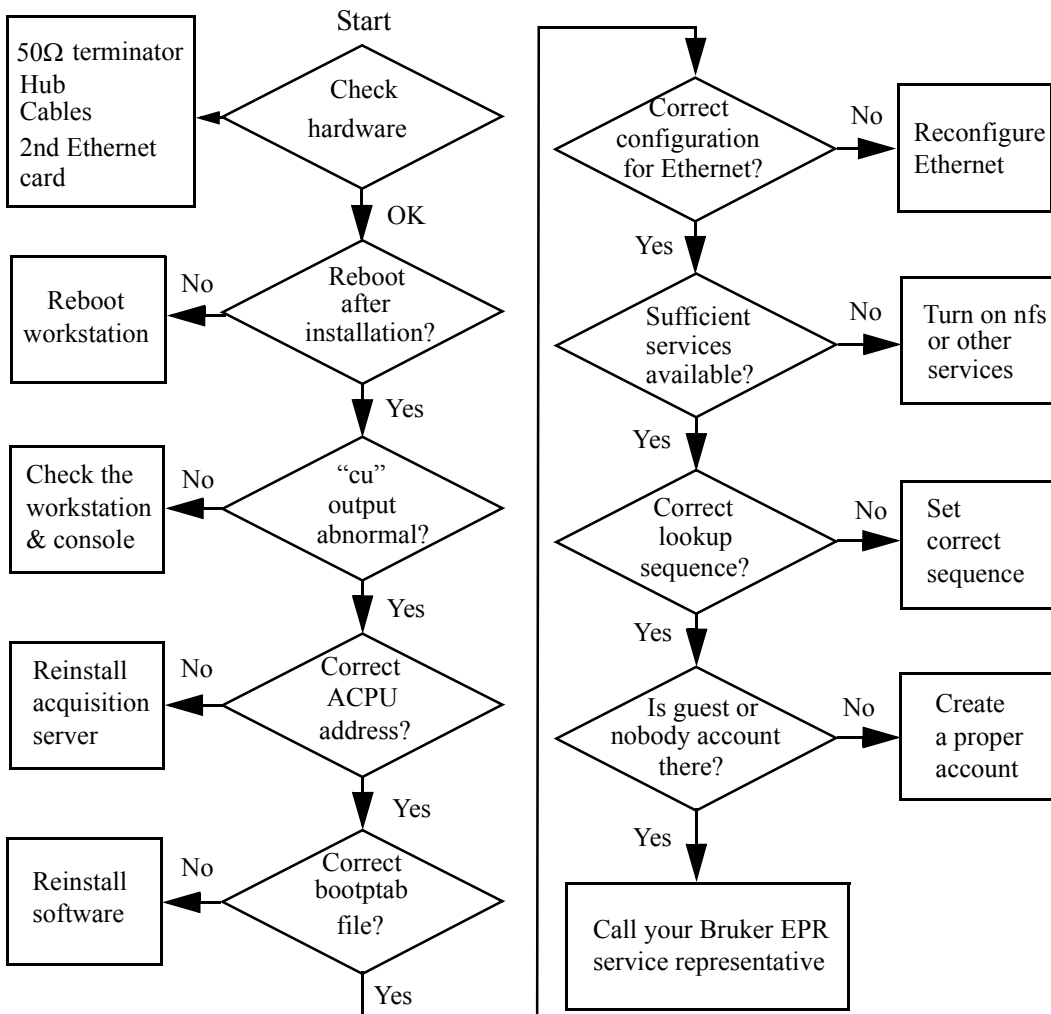


Figure 17-5 The ACPU diagnostics chart.

If the LED of the GSI board is not lit, it is likely that the ACPU is not booted correctly. Follow the flow chart of the ACPU diagnostics to locate the problem.

1. **Check the hardware connections.**
 - Check the console computer rack power supply.
 - Make sure both ends of the thinnet connection are terminated with 50 Ω loads.
 - Check the power and the connection of the hub. Do not use uplink.
 - Carefully inspect all the network cables and their connectors.
 - Make sure the second ethernet card is functioning properly.
2. **Reboot the workstation after installation of the Xepr software and the Acquisition Server.** You must restart the UNIX workstation after you install Xepr and the Acquisition Server in order for the spectrometer to work properly.
3. **Check the booting process.** You can use the cu command to check the booting process of the ACPU. Connect a terminal cable (part number E5002111) from RS1 on the ACPU to port 2 of the SGI O2 workstation (connector closest to the edge) or the first (1) RS232 port of the Linux workstation. In a UNIX shell window type `cu -l /dev/ttyd2` for an SGI O2 workstation or `cu -l /dev/ttyS0` for a Linux workstation. The shell will respond “connected”. Reboot the acquisition server by either pressing the **Reset** switch to the left or switch the console power off and on. If the ACPU boots correctly the display in the shell window of the workstation monitor should look similar to what follows.



Use the letter “e1” not the number “one” with the cu command.

```

eleXsys ~> cu -l /dev/ttyd2
Connected

P4X: SELF-TEST
Duart1      test: completed
Mpu         test: MC68040 completed
Clock       test: completed
Mmu         test: completed
Calender    test: completed
Centronics  test: not ready
Fpu         test: completed
Ethernet    test: completed
Checking Memory: 0008 Mbytes RAM detected

OS-9/68K System Bootstrap

Will try to Boot from network
To get the boot menu press Esc

Now trying to Boot from network.
inetboot: using P4x (MC68360) Ethernet @ 0xFFD80C00 MTU=1500
bootp: 0:40:98:0:0:c5 broadcasting for server
bootp: recv waiting 50 ticks
GOT BOOTP RESPONSE from server 192.168.99.1!
My IP address will be: 192.168.99.5
My      tftp      bootfile      is:      /usr/xexpr/Acquisition
Server/OS9/CPU/P4X/BOOTP/os9boot
My bootfile size is: 1380 (512-byte) blocks
My subnet mask is: 255.255.255.0
<<no timeoffset tag>>
tftp: fetch 1380 blocks
tftp: server response 192.168.99.1 port 1044
tftp: expected 1380: received 1379:76 blocks (690Kb)
A valid OS-9 bootfile was found.
-----
CPU Board Name: P4X
-----
starting ISP subsystem ...
+4

IP Routing Table:

Add gateway = 0.0.0.0 gateway = 192.168.99.1 to NET routing list
+8
-----
mounting '/xacq' ...

```

```
mounting '/dd' ...
mounting '/r0' ...
-----
Mount Status      13:17:06
OS-9              Remote      Gid/Uid      Mount
Device           System      Map          Point
-----
/xacq            BootWS      yes          /usr/xexpr/AcquisitionServer/OS9
/dd              BootWS      yes          /usr/xexpr/Acquisition
Server/OS9/CPU/P4X
/r0              BootWS      yes          /usr/xexpr/Acquisition
Server/OS9/Servers/E500/r0
-----

PROMPT=E500#
PATH=/dd/CMD5
HOME=/r0
AcqSvCPURootPath=/dd
AcqSvRootPath=/xacq/Servers
AcqSvCPUName=P4X
AcqSvHostName=E500
+21

AcqSvG: 'AcqServerD' started ...
-----
E500#
```

If the ACPU is not booted correctly there will be error messages. The following demonstrates what happens when the ACPU hardware address is incorrect or the ACPU is not connected to the UNIX workstation correctly.

```

Now trying to Boot from network.
inetboot: using P4x (MC68360) Ethernet @ 0xFFD80C00 MTU=1500
bootp: 0:40:98:0:0:c5 broadcasting for server
bootp: rcv waiting 50 ticks
bootp: retry 1 with new timeout=0
bootp: 0:40:98:0:0:c5 broadcasting for server
bootp: rcv waiting 50 ticks
bootp: retry 2 with new timeout=0
bootp: 0:40:98:0:0:c5 broadcasting for server
bootp: rcv waiting 50 ticks
bootp: retry 3 with new timeout=0
bootp: 0:40:98:0:0:c5 broadcasting for server
bootp: rcv waiting 50 ticks
bootp: retry 4 with new timeout=30
Exiting after 5 retry
This error occurred during the Internet boot boot driver!
The OS-9 error code is: #007:025.

```

Now trying to Reconfigure the boot system.

BOOT SYSTEM RECONFIGURATION

Boot drivers available:

- 1 - Boot from Teac SCSI floppy drive
- 2 - Boot from OMTI 5400 SCSI floppy drive
- 3 - Boot from SCSI hard drive
- 4 - Boot from SCSI tape drive
- 5 - Boot from ROM
- 6 - Boot from network

The priority of these boot drivers can be set below and will remain set even when power to your system is turned off. This priority determines the order that the boot drivers will be selected when your system is configured to boot automatically. It also determines the order that they appear in the "boot driver menu" as well.

The current system boot driver priority is: 6

Enter the numbers of the boot drivers in the order that you want them prioritized. ->
that you want them prioritized. ->

4. **Find the correct ACPU address.** A common error is an incorrect ACPU address. This address is the hardware address of the ACPU's ethernet adapter, not the ACPU's IP address. There are several ways to find the correct ACPU hardware address.

- From the output of the `cu` command:

```
bootp: 0:40:98:0:0:c5 broadcasting for server
```

where 0:40:98:0:0:c5 is the hardware address. However, you need to convert to the following format, 0x0040980000c5, when you edit the ServerSetup file.

- From the SYSLOG or messages file. In a UNIX shell window type `tail -f /dev/adm/SYSLOG` for an SGI O2 workstation or `tail -f /var/log/messages` for a Linux workstation. Then reset the ACPU. The screen display will indicate the correct ACPU hardware address.

```
eleXsys /> tail -f /var/adm/SYSLOG
Aug  8 22:15:44 6B:eleXsys Xsession: xuser: login
Aug  8 22:17:21 6D:eleXsys bootpd[673]: reading new "/etc/bootptab"
Aug  8 22:17:21 6D:eleXsys bootpd[673]: read 4 entries (1 hosts) from "/etc/bootptab"
Aug  8 22:17:21 5D:eleXsys bootpd[673]: Ethernet address not found: 0040980000C5
Aug  8 22:17:32 5D:eleXsys bootpd[673]: Ethernet address not found: 0040980000C5
```

For a Linux system the error message is slightly different.

```
Aug  8 22:17:32 epr-linux bootpd[5504]: unknown client Ethernet address 00:40:98:00:00:C5
```

- From the ACPU board label. The ethernet adapter has a label attached to it indicating the adapter hardware address.

5. **Check the bootptab file.** Open the `/etc/bootptab` file. The file contains the information for the ACPU to boot. (See Figure 17-6.)

```
# First, we define a global entry which specifies the stuff every
# host uses.
#
# the bs tag is required for OS-9 BOOTP clients
# bf is set to os9boot to cause the bootfile.host lookup action
# hn is set so that the client host name is sent in the vendor
# specific area
#
alloS9PECO43:sm=255.255.255.0:td=/:hd=/usr/xexpr/AcquisitionServer
/OS9/CPU/PECO43/BOOTP:bs=auto:hn:bf=os9boot:
alloS9P4X:sm=255.255.255.0:td=/:hd=/usr/xexpr/AcquisitionServer/OS9
/CPU/P4X/BOOTP:bs=auto:hn:bf=os9boot:
alloS9MVME162:sm=255.255.255.0:td=/:hd=/usr/xexpr/AcquisitionServer
/OS9/CPU/MVME162/BOOTP:bs=auto:hn:bf=os9boot:
#
# individual hosts
#
E500:tc=alloS9P4X:ht=ethernet:ha=0x0040980000c5:ip=192.168.99.5
```



Figure 17-6 The contents of the bootptab file.

6. **Check the Ethernet configuration (IRIX).** The `ec0` ethernet adapter should be set as primary while `ec1` should be set as secondary with the host name of Xexpr. If you reconfigure the ethernet settings you need to reboot the workstation. Instead of rebooting the workstation you can recycle the network script if you are a UNIX expert.

7. **Check the Ethernet configuration (Linux).** Type `/sbin/ifconfig` in a terminal window and you will receive information regarding your network settings. For example:

```
eth0      Link encap:Ethernet  HWaddr 10:00:00:42:54:01
          inet addr:137.1.7.27  Bcast:137.1.255.255  Mask:255.255.0.0
          UP BROADCAST RUNNING MULTICAST  MTU:1500  Metric:1
          RX packets:5298627 errors:0 dropped:0 overruns:0 frame:0
          TX packets:24 errors:0 dropped:0 overruns:0 carrier:0
          collisions:0 txqueuelen:100
          Interrupt:19 Base address:0x1080

eth1      Link encap:Ethernet  HWaddr 10:00:00:42:54:02
          inet addr:192.168.99.1  Bcast:192.168.99.255  Mask:255.255.255.0
          UP BROADCAST RUNNING MULTICAST  MTU:1500  Metric:1
          RX packets:248023 errors:0 dropped:0 overruns:0 frame:0
          TX packets:180600 errors:0 dropped:0 overruns:0 carrier:0
          collisions:0 txqueuelen:100
          Interrupt:19 Base address:0x1000

lo        Link encap:Local Loopback
          inet addr:127.0.0.1  Mask:255.0.0.0
          UP LOOPBACK RUNNING  MTU:3924  Metric:1
          RX packets:139 errors:0 dropped:0 overruns:0 frame:0
          TX packets:139 errors:0 dropped:0 overruns:0 carrier:0
          collisions:0 txqueuelen:0
```

Check that the following is set for `eth1`:

inet addr: 192.168.99.1

Bcast: 192.168.99.255

Mask: 255.255.255.0

8. **Check the services.** It is crucial for the acquisition server to be able to access some critical services such as `bootp`, `tftp`, and `nfs`. If a booting problem only exists after you configure the services or turn off some flags, you may have blocked critical services from the acquisition server. Reconfigure the services or turn the flags on. The `nfs` flag must be `on` to allow `Xepr` to work properly. Use `chkconfig` to check the flags. Make sure the `nfs` and network flags

are **on**. If they are **off** you need to turn them on and restart the workstation. (See Section 15.3.1 or Section 16.3.1.)

9. **Check the lookup sequence.** Set **files** as the first choice for the lookup sequence. Setting the NIS or DNS servers prior to **files** in the lookup sequence may cause ACPU booting problem. (See Section 15.2.3 or Section 16.2.3.)
10. **Check the guest or nobody account.** Although the guest or nobody account has been locked and hidden, Xepr uses this account to transfer files. Do not delete this account. If the guest or nobody account does not exist you need to create one.
11. **If problems still exist.** You may need to reinstall the Xepr software and acquisition server. Sometimes, deleting the existing Xepr folder (`/usr/xepr`) before reinstallation may help you to get rid of problems. (See Section 15.4 or Section 16.4.) If problems still persist call your Bruker EPR service representative.



Xepr running on an SGI IRIX workstation uses the guest account. Xepr running on a Linux workstation uses the nobody account.

Transputer Diagnostics

17.2.2

If the ACPU is booted but the transputer device LEDs are not lit, the transputer devices are not booted correctly. Follow the chart to locate the problem.

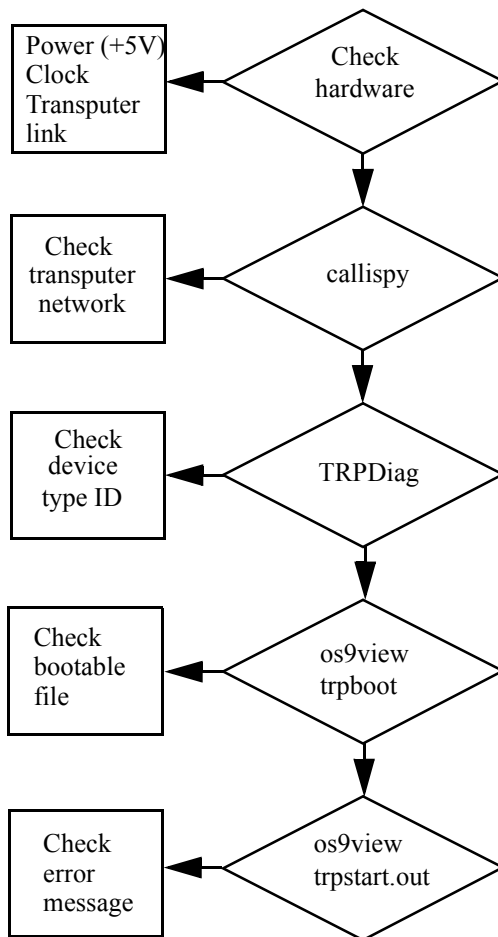


Figure 17-7 The transputer device diagnostics chart.

1. **Check the hardware.** Check the Burndy cable for each transputer device. The power (+5 V) and the clock (10 MHz) are supplied through the Burndy cable. Check the transputer links. The black end connects to OUT and the red end connects to IN. Check if the links are valid. (See Section 13.2.4.)
2. **Check the transputer network.** You can use a command called `callispy` to check the transputer network.
 - If the `cu` command is still active you will find a `#` prompt sign after the server name when the ACPU boots properly. Typing `callispy` followed by the `Enter` key will launch the diagnostics program.
 - You can also open a UNIX shell window and type `telnet server name`. Use `root` as the user name and `root!` as the password. After you login you can enter `callispy` when the prompt sign `#` appears.

The output of the `callispy` command displays information about the transputer network. The example in Figure 17-8 shows a typical Elexsys system with a TVI transputer board, a field controller, a high frequency signal channel, a low frequency channel, and a bridge controller.

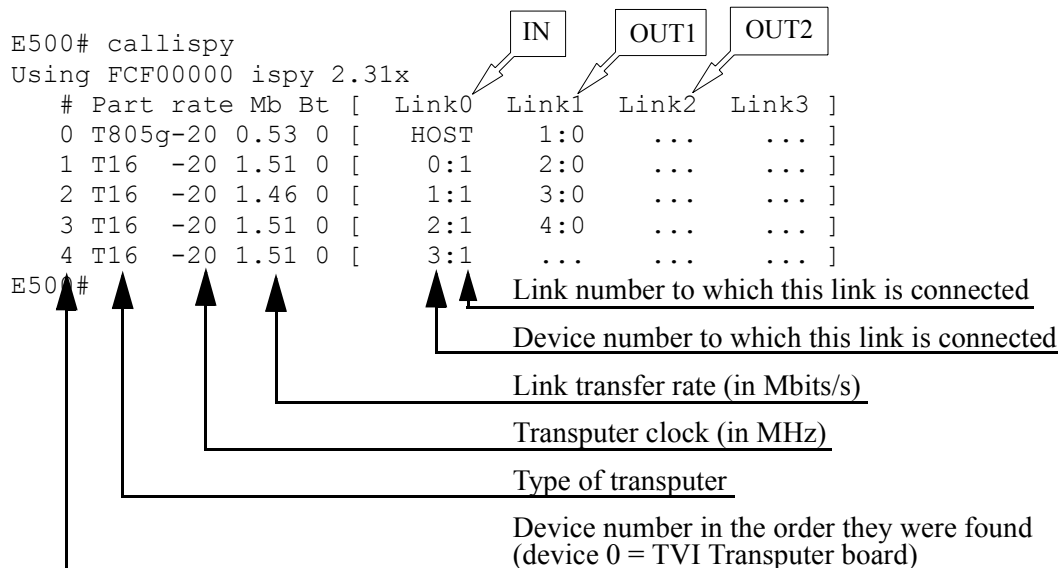


Figure 17-8 The output of the callispy command.



The TRPDiag command does not work immediately after the command `callispy`. Reboot the acquisition server before you start the TRPDiag command.

3. **Check the device type ID.** In order to boot the transputer devices, the server needs to know not only how the devices are connected but also which devices they are. Each device is identified by a Bruker Board Identification System (BBIS) device type ID number that is contained in an EEPROM. Upon the acquisition server prompt `#` sign, use the command `TRPDiag`. Typical output is as follows.

```
E500# TRPDiag
Following Transputer Bootfile Version have been installed:
HALL V2.20
MBCT V2.25
SCT V2.22
SCTL V2.22
```

```
Display BBIS Information ? ( Y/N )
```

All 4 transputer devices (the Hall field controller, the microwave bridge controller, the high frequency signal channel, and the low frequency signal channel) are properly identified and the bootfiles have been installed. If you answer `Y` to the question `Display BBIS Information? (Y/N)` the detailed information about each device such as the serial number, EC-level, BBIS version, device type, number of links, *etc.*, will be displayed.

4. **Check the transputer booting files.** The server needs to know where to find the correct bootable files for each device. A file called `trpboot` lists the device type ID, the corresponding bootable file, and the path of the file. You can check the `trpboot` file in a UNIX shell window. Change to the `/usr/xexpr/AcquisitionServer/OS9/Servers/<Server name: e.g. E500, E580, etc.>/r0` directory and then type `os9view trpboot`. The contents of the `trpboot` file looks as follows.

```

eleXsys /usr/xexpr/AcquisitionServer/OS9/Servers/E500/r0> os9view trpboot
*****
*
*           BOOT FILE FOR THE TRANSPUTER SYSTEM
*
* The instructions and comments.
*
*****

#DEVICE:          /trp/root          * name of ROOT transputer

#BOOTFILES

    0      0      /xacq/Servers/TRPRTL/ident16.btl      * Initial Loading File
    1      0      /xacq/Servers/TRPRTL/ident32.btl      * Initial Loading File
    4      0      /xacq/Servers/TRPRTL/er032t_win.btl    * ER032T Field Controller
    5      0      /xacq/Servers/TRPRTL/mbct_win.btl      * Microwave Bridge Controller
    6      0      /xacq/Servers/TRPRTL/sct_win.btl      * High Frequency Signal Channel
    7      0      /xacq/Servers/TRPRTL/mwb_win.btl      * Microwave Bridge Controller
   10      0      /xacq/Servers/TRPRTL/sctl_win.btl     * Low Frequency Signal Channel
   11      0      /xacq/Servers/TRPRTL/ppu.btl          * Pulse Programmer
   12      0      /xacq/Servers/TRPRTL/tsa_win.btl      * Transient recorder

#DUMMYFILES

    0      0      /xacq/Servers/TRPRTL/dummy16.btl     * Dummy Loading File
    1      0      /xacq/Servers/TRPRTL/dummy32.btl     * Dummy Loading File

```


5. **Check for transputer device booting errors.** If the bootable file for the device type ID is missing, file name/extension is incorrect, or the file is in a wrong directory the transputer device will abort the booting procedure. The error messages are stored in the file called **trpstart.out** in the same directory as the **trpboot** file. You can view this file by typing `os9view trpstart.out`.

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