

## Why Do We Use Resonators?

## 5.1

In optical spectroscopy, spectra are usually acquired by simply shining light through the sample and detecting how much light gets through. (See Figure 5-1.) This technique is unfortunately not very effective for EPR spectroscopy. The reason is that an optical absorption spectrum is much stronger than an EPR absorption spectrum. We need a little help to see our weak EPR signals. Optical absorptions are stronger for two reasons:

1. Population differences between the two energy levels involved in the transition are greater for optical spectroscopy, resulting in more intense absorptions. The greater population difference is a consequence of the Boltzmann population distribution of the energy levels.
2. Optical absorptions are usually electric dipole transitions and EPR spectra are usually magnetic dipole transitions. The magnetic dipole transitions usually have a smaller transition moment (*i.e.* are weaker) than electric dipole transitions.

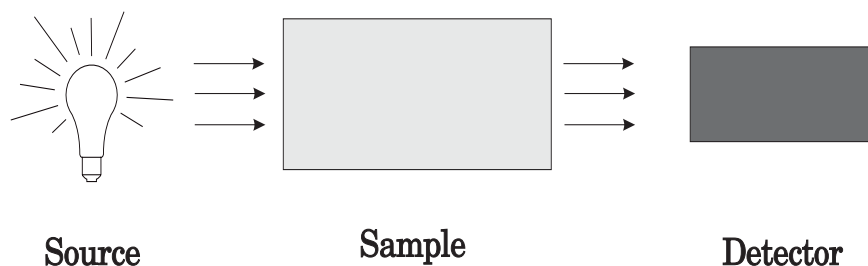


Figure 5-1 A simple optical spectrometer.

In order to enhance sensitivity, we use microwave resonators. These resonators are usually cavities. (Note: all cavities are resonators but not all resonators are cavities, for example, split ring and dielectric resonators.) A cavity is a metal box in which the microwaves can bounce around. A resonator helps increase sensitivity by “focusing” or “concentrating” the microwave power at the sample and storing the microwave energy. The next section explains how this happens as well as some of the basic ideas needed to troubleshoot EPR resonators.

## Basic Concepts of EPR Resonators

## 5.2

In this section, we shall discuss the properties of EPR cavities and how changes in these properties due to absorption result in an EPR signal. A microwave cavity is simply a metal box with a rectangular or cylindrical shape which resonates with microwaves much as an organ pipe resonates with sound waves. Resonance means that the cavity stores the microwave energy; therefore, at the resonance frequency of the cavity, no microwaves will be reflected back, but will remain inside the cavity. (See Figure 5-2.)

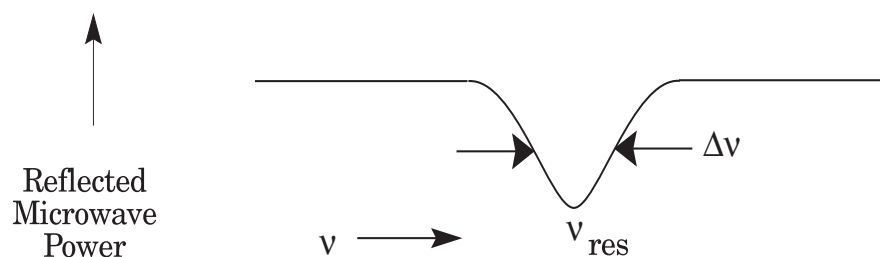


Figure 5-2 Reflected microwave power from a resonant cavity.

Cavities are characterized by their  $Q$  or quality factor, which indicates how efficiently the cavity stores microwave energy. As  $Q$  increases, the sensitivity of the spectrometer increases. The  $Q$  factor is defined as:

$$Q = \frac{2\pi(\text{energy stored})}{\text{energy dissipated per cycle}}, \quad [5-1]$$

where the energy dissipated per cycle is the amount of energy lost during one microwave period. Energy can be lost to the side walls of the cavity because the microwaves generate electrical currents in the side walls of the cavity which in turn generates heat. We can measure  $Q$  factors easily because there is another way of expressing  $Q$ :

$$Q = \frac{\nu_{res}}{\Delta\nu}, \quad [5-2]$$

where  $\nu_{res}$  is the resonant frequency of the cavity and  $\Delta\nu$  is the width at half height of the resonance.

A consequence of resonance is that there will be a standing wave inside the cavity. Standing electromagnetic waves have their electric and magnetic field components exactly out of phase, *i.e.* where the magnetic field is maximum, the electric field is minimum and *vice versa*. The spatial distribution of the amplitudes of the electric and magnetic fields in a commonly used EPR cavity is shown in Figure 5-3. We can use the spatial separation of the electric and magnetic fields in a cavity to great advantage. Most samples have non-resonant absorption of the microwaves via the electric field (this is how a microwave oven works) and the  $Q$  will be degraded by an increase in the dissipated energy. It is the magnetic field that drives the absorption in EPR. Therefore, if we place our sample in the electric field minimum and the mag-

netic field maximum, we obtain the biggest signals and the highest sensitivity. The cavities are designed for optimal placement of the sample.

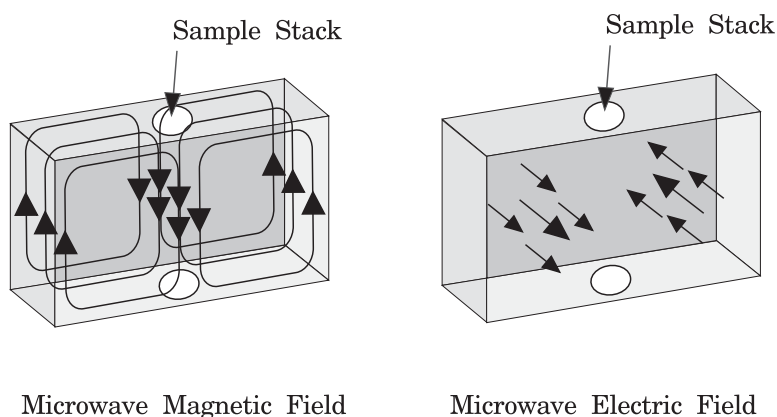


Figure 5-3 Magnetic and electric field patterns in a standard EPR cavity.

We couple the microwaves into the cavity via a hole called an iris. The size of the iris controls the amount of microwaves which will be reflected back from the cavity and how much will enter the cavity. The iris accomplishes this by carefully matching or transforming the impedances (the resistance to the waves) of the cavity and the waveguide (a rectangular pipe used to carry microwaves). There is an iris screw in front of the iris which allows us to adjust the “matching”. This adjustment can be visualized by noting that as the screw moves up and down, it effectively changes the size of the iris. (See Figure 5-4.)

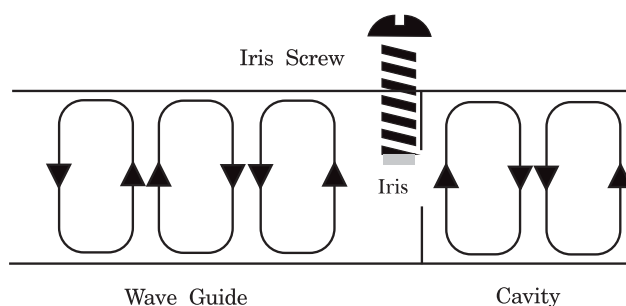


Figure 5-4 The matching of a microwave cavity to a waveguide.

How do all of these properties of a cavity give rise to an EPR signal? When the sample absorbs the microwave energy, the Q is lowered because of the increased losses and the coupling changes because the absorbing sample changes the impedance of the cavity. The cavity is therefore no longer critically coupled and microwave will be reflected back to the bridge, resulting in an EPR signal.

## A More Technical Description

## 5.3

A more detailed explanation now follows for those who are more technically minded. An equivalent circuit diagram for a reflection cavity is shown in Figure 5-5. L,C, and r form the equivalent circuit for the resonator. L and C determine the resonant frequency and r (the losses in the walls and the sample) determines the unloaded Q:

$$Q_u = \frac{\omega L}{r} \tag{5-3}$$

The transformer (with n = turns ratio) represents the iris and iris screw; it transforms the impedance of the microwave source, R<sub>0</sub> to the impedance of the cavity r. Moving the iris screw (with the metal stub on the end) downwards effectively increases the turn ratio in the equivalent circuit.

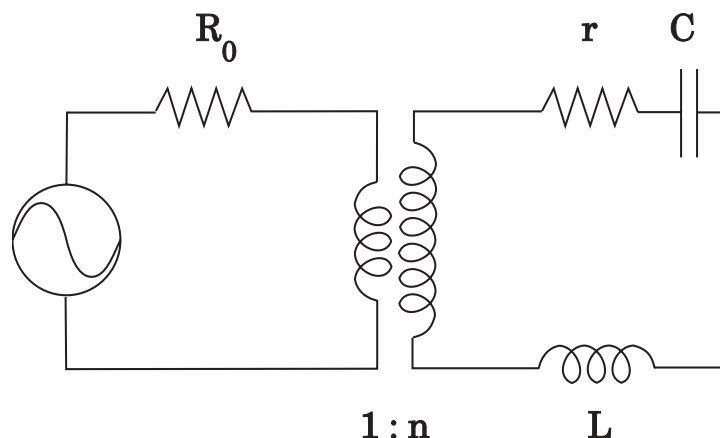


Figure 5-5 The equivalent circuit of a reflection cavity.

The iris lets microwaves enter the cavity but also lets microwaves leak out as well. The power leaking out is proportional to R<sub>0</sub>n<sup>2</sup>. Combining the two losses (losses in the cavity as well as leakage out of the cavity), a new Q, called the loaded Q, can be defined:

$$Q_L = \frac{\omega L}{R_0 n^2 + r} \tag{5-4}$$

It is useful to define yet another parameter, the coupling coefficient:

$$\beta = \frac{R_0 n^2}{r} \tag{5-5}$$

The coupling coefficient conveniently defines three conditions. β = 1 corresponds to a critically coupled or “matched” cavity. This corresponds to maximum power transfer from the microwave source to the cavity. Maximum EPR sensitivity is achieved in this condition. The VSWR (Voltage Standing

Wave Ratio) is equal to 1, which corresponds to a reflection coefficient  $\Gamma$ , of zero where

$$\Gamma = \frac{VSWR-1}{VSWR+1}. \quad [5-6]$$

This means when we are matched, no microwaves are reflected from the cavity. It also means (See Equation [5-4].) that the loaded Q is half the unloaded Q.  $\beta < 1$  corresponds to an undercoupled cavity with  $VSWR = 1/\beta$  and  $\Gamma > 0$ . This means that microwaves are reflected from the cavity. Equation [5-4] also tells us that the Q is somewhat higher than for a matched cavity.  $\beta > 1$  corresponds to an overcoupled cavity with  $VSWR = \beta$  and  $\Gamma < 0$ . Microwaves are reflected from the cavity with a 180 degree phase shift. Equation [5-4] predicts that the Q is lower than for a matched cavity.

Armed with the above equations, we can now investigate what happens when we observe an EPR spectrum. We start with a matched cavity. When the sample absorbs microwaves, it effectively increases  $r$ , the equivalent resistance of the cavity. From Equation [5-5] and Equation [5-6], it is apparent that the cavity becomes undercoupled and reflects microwaves back to the bridge. The reflected microwaves are the EPR spectra observed with the spectrometer.

The preceding equations also help us understand what we are doing while matching the cavity. We usually start with an undercoupled cavity. The mode picture on the spectrometer (with the reference arm turned off) looks similar to Figure 5-6.

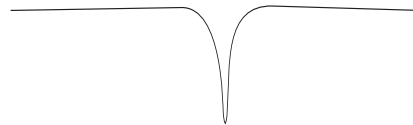


Figure 5-6 The mode picture of an undercoupled cavity.

The coupling coefficient is not 1, therefore some microwaves are reflected back from the cavity. (See Equation [5-5] and Equation [5-6].) As we lower the iris screw, the dip will drop towards the baseline and eventually meet it (See Figure 5-7.), indicating critical coupling (*i.e.* no microwaves reflected back.).

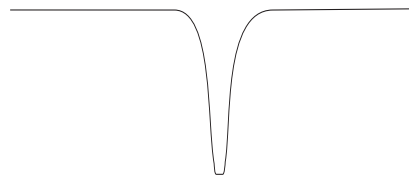


Figure 5-7 The mode picture of a matched cavity.

Lowering the iris screw corresponds to increasing the turns ratio  $n$  in the equivalent circuit shown in Figure 5-5. If the iris screw is still further lowered, the cavity becomes overcoupled. The  $Q$  therefore decreases and microwaves are reflected back. (See Equation [5-4] through Equation [5-6] and Figure 5-8.) The lower  $Q$  can be seen from the width of the cavity resonance.

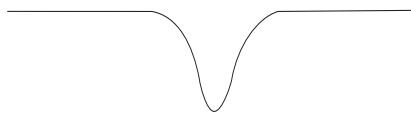


Figure 5-8 The mode picture of an overcoupled cavity.

Sometimes, if the iris is even further lowered, we can once again match and undercouple the cavity. This is not particularly mysterious if you study Figure 5-9.

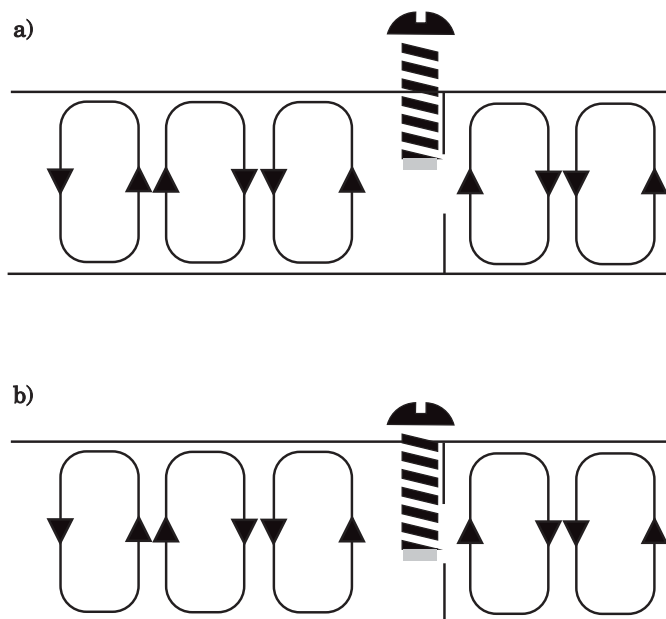


Figure 5-9 Two equivalent positions of an iris screw.

The metal stub on the end of the iris screw is in equivalent positions for a high and low iris screw position. The disadvantage of the lower position is that the microwaves have to pass through the lossy plastic of the iris screw. Figure 5-9 also suggests a solution if you are having problems matching your cavity. If you use a larger iris tip, you can change the effective size of the iris more, resulting in a greater coupling range.

Larger iris tips are particularly useful for lossy samples. Lossy samples (usually) interact with the electric field of the microwaves to heat the sample. This effect is similar to heating food in a microwave oven. Examples of lossy samples are polar liquids such as water, sulfuric acid, chloroform, and methanol. Sometimes, semiconductor samples exhibit such effects. This non-resonant

absorption of microwaves increases  $r$ . The  $Q$  of the cavity therefore drops, thereby decreasing the sensitivity of the spectrometer. The increased value of  $r$  also makes it more difficult to match the cavity. A larger  $n$ , the turns ratio in the equivalent circuit, is required to match the cavity. The larger turn ratio corresponds to moving the iris screw downwards.

In order to avoid placing our samples in the electric field of the cavity, we need to know the field distributions inside the cavity. The distributions depend on the shape and the “mode” of the different cavities. These properties determine the utility of a particular cavity for a specific sample.

The most commonly used cavity in EPR has been the rectangular  $TE_{102}$  cavity. The Bruker ER 4102ST is such a cavity. The squares of the microwave magnetic and electric fields are displayed in Figure 5-10. The electric and magnetic fields are constant across the cavity. Note that the maxima in the peaks of the electric and magnetic fields are out of phase with each other. We therefore can place our sample simultaneously in a magnetic field maximum and an electric field minimum. The sample holders on the ER 4102ST are designed such that the sample and sample tube lies in the “valley” between the two electric field “mountains”. Note that if you have a small sample, moving the sample up and down will change the microwave magnetic field seen by the sample. It is therefore important, particularly for small samples, to center the sample in the cavity at the magnetic field maximum. The ER 4102ST cavity is a good “universal” resonator. It is easy to use with cryogenics. Large modulation amplitudes can be obtained.

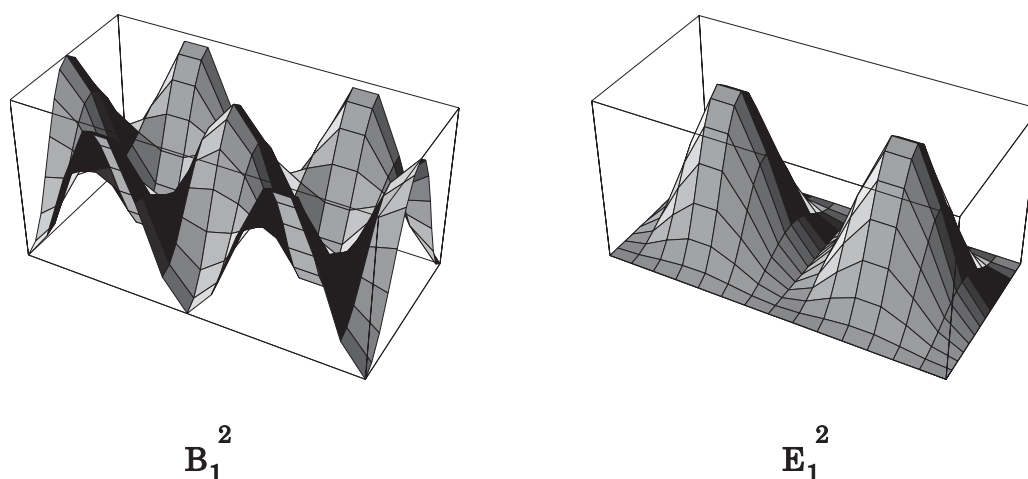


Figure 5-10 The field distribution in a  $TE_{102}$  rectangular cavity.

The second most popular cavity has been the cylindrical  $TM_{110}$  cavity. The corresponding cavities in the Bruker product line are the ER 4103TM and ER4108 TMH cavities. The squares of the microwave magnetic and electric fields are displayed in Figure 5-11. The electric and magnetic fields are constant across the cavity. The sample holders on both cavities are designed such that the sample and sample tube lies in the “valley” between the two electric field “mountains”. The ER 4103TM cavity is used predominately for flat cell work. A flat cell is a thin and flat sample tube for working with lossy liquids such as water. The advantage of this cavity over the ER 4102ST for such

samples is that much more sample can be put in the peak magnetic field parts of the cavity and the Q is higher. The ER 4108TMH is a thinner version of the ER 4103TM with no irradiation grids and smaller openings for sample insertion. It has a higher Q than the ER 4103TM, making it more sensitive. It also has a larger filling factor. The filling factor is an indication of the volume of the sample in the magnetic field peaks compared to the volume of the cavity. The higher it is, the more sensitive the cavity becomes. The disadvantage of the ER 4108TMH is that the smaller openings make cryogenic work more problematic.

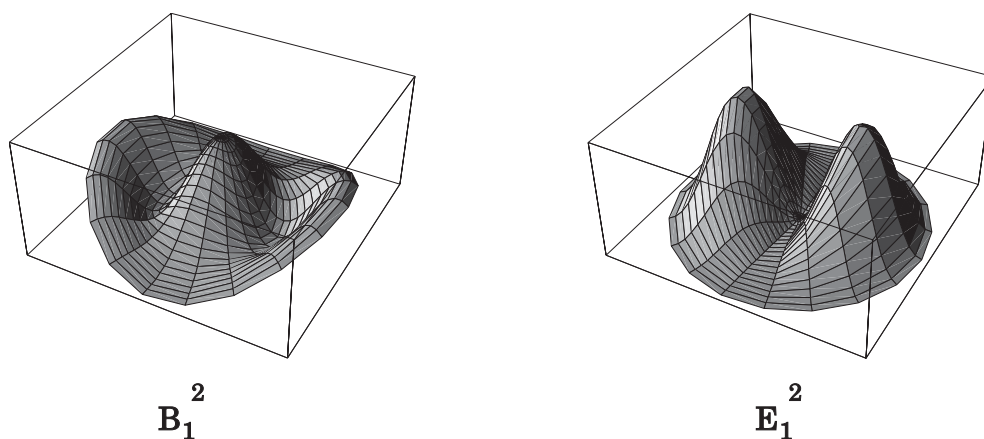


Figure 5-11 The field distribution in a  $TM_{110}$  cylindrical cavity.

The other type of cavity offered by Bruker is the cylindrical  $TE_{011}$  cavity. There are many special cavities offered by Bruker which have this mode. Among them are the ER 4106 ZR, ER 4107WZ, and ER 4115OD resonators. The ER 4119HS and ER 4122SHQE cavities have a similar mode, but with some proprietary modifications. These cavities have very high Q's and hence are very sensitive cavities for low loss samples. Also, if you use proper aqueous sample cells such as AquaX, very high sensitivity can be attained for lossy samples. This is why the ER 4119HS and ER 4122SHQE cavities are currently the standard resonators for the EMX and Elexsys spectrometers respectively.

The squares of the microwave magnetic and electric fields of the  $TE_{011}$  mode are displayed in Figure 5-12. The sample holders on both cavities are designed such that the sample and sample tube lies in the “valley” between the two electric field “mountains”. The dielectric resonator can be considered a special case of this type of cavity. It consists of some high dielectric constant material, which concentrates the microwave fields in a much smaller volume. This results in a much better filling factor.



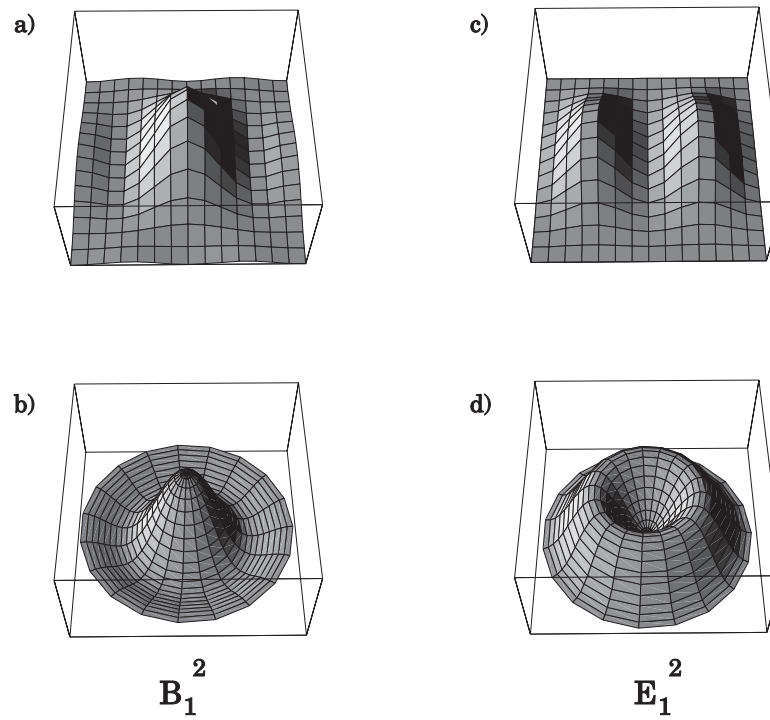


Figure 5-12 The field distribution in a  $TE_{011}$  cylindrical cavity.

- a) Side profile of magnetic field.
- b) Axial profile of magnetic field in center of cavity.
- c) Side profile of electric field.
- d) Axial profile of electric field in center of cavity.

## Common Problems

## 5.4

### No Cavity Dip

#### 5.4.1

1. **Waveguide gasket installed improperly.** See Figure 5-13 for the proper orientation of the gasket.

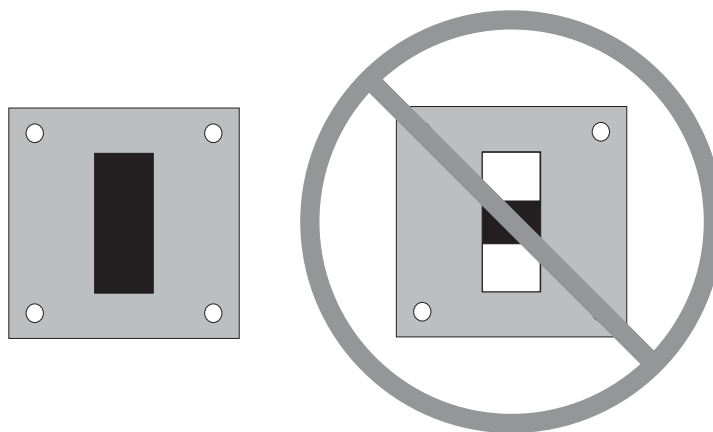


Figure 5-13 Correct and incorrect installation of waveguide gasket.

2. **Cavity undercoupled or overcoupled.** First, look at the microwave frequency where you normally expect the cavity to resonate and then adjust the iris screw for better coupling. This can occur when working with lossy samples such as aqueous solutions in flat cells or capillaries.
3. **You need more microwave power.** If you are using insufficient microwave power, it can be difficult to see the cavity dip. We recommend setting the microwave attenuator to at least 35 dB for the best visibility.

### Unable to Critically Couple Cavity

#### 5.4.2

1. **Sample position.** If too much of a lossy sample is in the microwave electric field in the cavity, you will not be able to critically couple the cavity. Move the sample until the coupling becomes better. This is particularly important for flat cells and capillaries.
2. **Microwave reference phase.** If the microwave reference phase is not set properly, you will not be able to critically couple the cavity. Retune the microwave bridge properly.
3. **Iris tip size.** When working with lossy samples, it is advisable to use a larger iris tip to increase the coupling range of the cavity. This is particularly important when working with flat cells or capillaries.
4. **Condensation on the cryostat.** Make sure that you purge the cavity with dry Nitrogen if you are operating with a cryostat at temperatures lower than room temperatures.

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## Baseline Distortion

### 5.4.3

1. **Linear baseline drifts.** The use of very large modulation fields can produce large eddy currents in the cavity side walls. These currents can interact with the magnetic field to produce a torque on the cavity and create a resonant frequency shift. A linear field dependent or modulation amplitude dependent baseline is indicative of such an effect. This phenomenon should not be observed if the cavity end plates are properly fitted and torqued. Do not attempt to adjust the torque on the plates. Contact your local Bruker EPR service representative.
2. **Slowly and randomly varying baseline.** The use of high microwave power or large modulation fields can heat the cavity and the sample. The ensuing thermal drifts in the coupling of the cavity, as well as the frequency of the cavity, can result in a fluctuating offset in the signal. Allow the tuned cavity and sample to come to thermal equilibrium before performing the final tuning of the cavity. Once the cavity is equilibrated and properly tuned under the equilibrated condition, you can start acquiring a spectrum. Avoid air drafts around the cavity, as they can randomly change the temperature of the cavity and sample and hence the baseline of the spectrum.
3. **Variable temperature operation.** Cavity frequency and coupling instability may be induced during variable temperature operation, especially at very low or very high temperatures. Increase the flow rate of the cavity and waveguide purging gas as the operating temperature departs further from room temperature. Wait for the cavity to stabilize at each new operating temperature before recording the spectrum. Retune the cavity to compensate for any frequency shift and re-establish critical coupling at each temperature.
4. **Background signal.** Your cavity, cryostat, sample tube, or sample may be contaminated. (See Section 5.5 to learn how to clean cavities.)

## Excessive Noise Output

### 5.4.4

1. **Microphonic generated noise.** Secure the waveguide and cavity assembly by using the plastic waveguide stabilizers. Secure the sample firmly in the collet. If you use a cryostat, make sure that the cryostat sits firmly in the cavity. Make sure that an excessive flow rate through the cryostat does not vibrate the sample.
2. **Worn iris screw.** Check for a worn iris coupling screw. An iris screw that does not fit snugly in the waveguide may generate noise by modulating the cavity coupling. Replace the worn iris screw with a new one.
3. **Boiling liquids.** If you are using a dewar with a boiling refrigerant such as liquid nitrogen, you must increase the AFC modulation level.

## Poor Sensitivity

### 5.4.5

1. **Wrong cavity type for sample.** The type of cavity you use for a particular sample can make a large difference in sensitivity. Consult the Bruker literature on the full line of EPR cavities to determine which one is best for your samples.
2. **Low cavity Q.** The cavity Q can be degraded because of improper sample positioning. Sample in the microwave electric field will reduce the sensitivity by degrading the cavity Q, especially for high dielectric loss samples. This can happen if you are using flat cells or capillaries.
3. **Cavity not critically coupled.** Maximum power is transferred between the cavity and waveguide when the cavity properly matches the impedance of the waveguide, *i.e.*, is critically coupled. A drastically undercoupled iris will not transmit power to the cavity and so will not excite EPR transitions. A drastically overcoupled cavity will have a lower Q, resulting in lower sensitivity. These effects can happen when using lossy samples such as aqueous solutions or conducting samples.
4. **Water condensation.** During low temperature operation, water can condense inside the cavity. Water, being a high dielectric loss material, will destroy the cavity Q and absorb the microwave power in the cavity. Avoid condensation by using a purging nitrogen gas flow through the cavity.
5. **Sample not properly positioned.** Make sure that your sample is properly positioned in the cavity. The sample should be at the center of the cavity.

## Lineshape Distortion

### 5.4.6

1. **High conductivity.** High conductivity exhibited by samples with mobile electrons will result in asymmetric lines known as Dysonian lineshapes. This results from a mixing of the absorption and dispersion signals in these samples.
2. **Lossy samples.** If you put large lossy samples in a cavity, you can also obtain Dysonian lineshapes. Use progressively smaller capillaries until you obtain a symmetric lineshape.
3. **Background signal.** A strong background signal from contamination of the EPR cavity or the sample can produce distortions.

## No Signal When Everything Works

### 5.4.7

1. **Check cables.** Make sure that all the cables are connected. Check the modulation cable and the preamplifier cable.
2. **Sample position.** If you have a small sample, make sure that the sample is centered in the cavity.
3. **Magnetic field values.** Are you using the correct field values to see your EPR signal? If you are using a cryostat, remember that the microwave frequency drops and hence the field for resonance will also be lower.

## Cavity Cleaning

## 5.5



Cleaning instructions are specific for a cavity type. If your cavity is not listed in the cleaning instructions, do not use the cleaning techniques described. You may damage your cavity. When in doubt, contact your local Bruker EPR service representative.

Sometimes a sample may break in the cavity and contaminate the cavity. The adage “An ounce of prevention is worth a pound of cure.” is very appropriate here. Take care when inserting and removing sample tubes. Make sure you clean the outside of your sample tubes before insertion. Use a nitrogen cryostat; that way, the easily cleaned cryostat is contaminated instead of the cavity. Contaminants may also come from the lab as well. Maintain the cleanliness of your lab. Avoid smoking near the cavity. Keep the cavity sealed up with the solid collet plugs and the irradiation grid cover plate when it is not in use.

To test if your cavity background complies with the specifications, consult Chapter 13 to learn how to run a background test.

Cavities can be cleaned in some cases. Care must be taken because some of the solvents can damage paint on the waveguide or damage the cavity itself. It is easiest to clean the cavity if you know what the contaminant is and in what it is soluble. This knowledge is easily available if you are the one who actually contaminated the cavity. Alas, your colleagues usually will not admit to breaking any sample tubes in the cavity. Therefore there are instructions for washing away many different types of contaminants. If the cleaning procedures do not succeed, contact your local Bruker EPR service representative for advice.

### ER 4102ST Style Resonators

### 5.5.1

ER 4102ST, ER 4103TM, ER 4108TMH, ER 4116DM, and ER 4105DR can be cleaned with the following procedure.



Wear safety glasses when cleaning your cavity. Always wash the cavity with sufficient ventilation *i.e.* a hood. Wear gloves and avoid contact with the solvents because they are toxic and irritating. Dispose of the solvents properly after cleaning the cavity.

1. **Blow the cavity out.** Remove the collet nuts and collets as well as any irradiation grid cover. Use a stream of clean high pressure nitrogen to dislodge or blow out any contamination. Direct the gas stream down the collet stacks and through the irradiation grid (if present). Do not use compressed air because it can often be contaminated with oil, water and other substances. Run another background test as described in Chapter 13. If the background is not within specification after the treatment, proceed to the next step.
2. **Wash the cavity out with toluene.** Place a solid collet piece on the bottom of the cavity and tighten the collet nut. Put the irradiation grid cover plate on the front of the cavity. Fill the cavity with clean toluene. (Be careful as the toluene can strip the paint off the waveguide.) Shake the cavity for a few minutes, taking care not to spill the solvent. Empty the cavity of the solvent. This should wash away the most nonpolar contaminants. The theory is to use a series of solvents of increasing polarity. One of the solvents has a good chance of dissolving the dirt.
3. **Wash the cavity out with acetone.** Repeat the above procedure with clean acetone. (Be careful as the acetone can strip the paint off the waveguide.)

4. **Wash the cavity out with methanol.** Repeat the above procedure with clean methanol. (Be careful as the methanol can strip the paint off the waveguide.)
5. **Wash the cavity out with distilled or deionized water.** Repeat the above procedure with distilled or deionized water.
6. **Dry the cavity.** Dry the cavity with a stream of dry clean Nitrogen or let it dry overnight in a clean dry place.
7. **Cleaning metal contamination.** If you are certain that the contaminant is a metal ion, a quick rinse with 0.1 M disodium EDTA followed by a thorough rinse of distilled or deionized water sometimes removes the contaminant. Dry the cavity as described above.

## ER 4119HS Style Resonators

### 5.5.2

The ER 4119HS and ER 4122SHQE resonators are cleaned in a similar manner. It does differ in one respect; the cavity must **not** be filled. Remove both the upper and lower collets. Turn the cavity upside down. Flow the solvents through the bottom collet stack using a pipette or wash bottle. Then dry the cavity with a stream of dry clean Nitrogen or let it dry overnight in a clean dry place.

## Changing EPR Cavities

## 5.6

### Getting Ready (Elexsys)

### 5.6.1

We assume that the spectrometer is turned on, connected, and an experiment loaded before performing the following operations.

1. **Click the Activate button.** If this button is not already activated, click its button in the Acquisition Control Tool section. (See Figure 5-14.) This button allows you to interactively control the spectrometer when it is activated.
2. **Click the Parameter button.** Click this button to open the Acquisition Parameter dialog box. (See Figure 5-14.)

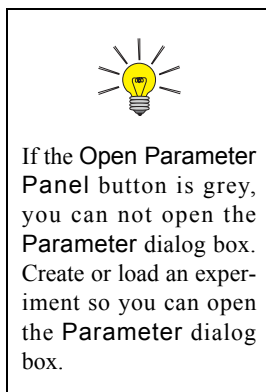


Figure 5-14 The Activate and Parameter buttons.

3. **Set the modulation amplitude to zero.** Click the Signal Channel tab. Enter a value of 0.00 in the Modulation Amplitude box. (See Figure 5-15.)

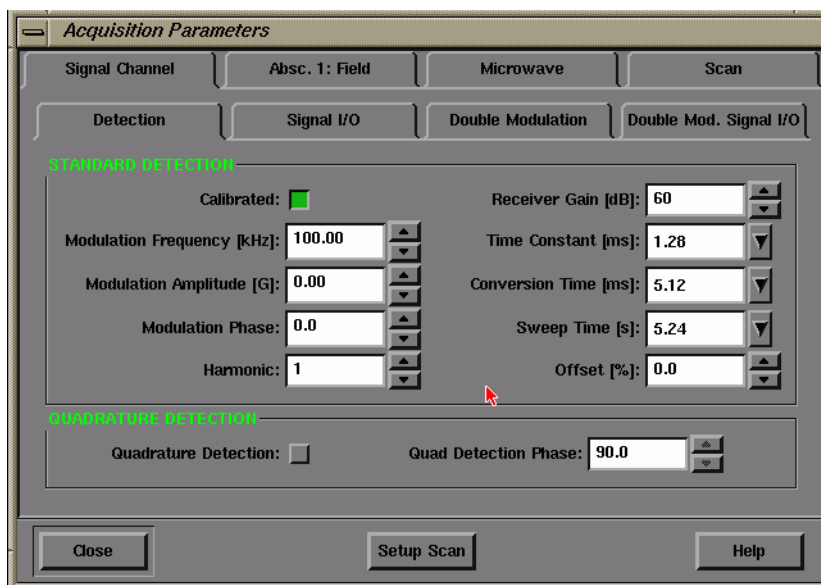
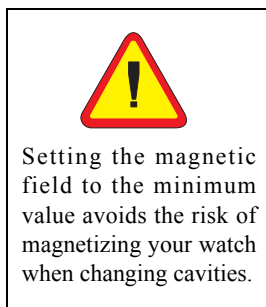


Figure 5-15 Set the Modulation Amplitude to zero.



4. **Set the magnetic field to the minimal value.** Click the Absc. 1:Field tab. Enter in a value of 0.00 in the Sweep Width box and then a value of 0.00 in the Center Field box.

5. **Close the Acquisition Parameter dialog box.** Click the Close button of the Acquisition Parameter dialog box. Click the Activate button again to exit the interactive control mode.
6. **Open the Microwave Bridge Tuning dialog box.** If this window is not already open, click its button in the monitoring panel. The button toggles the dialog box open and closed. The Microwave Bridge Tuning dialog box will then appear. (See Figure 5-16.)

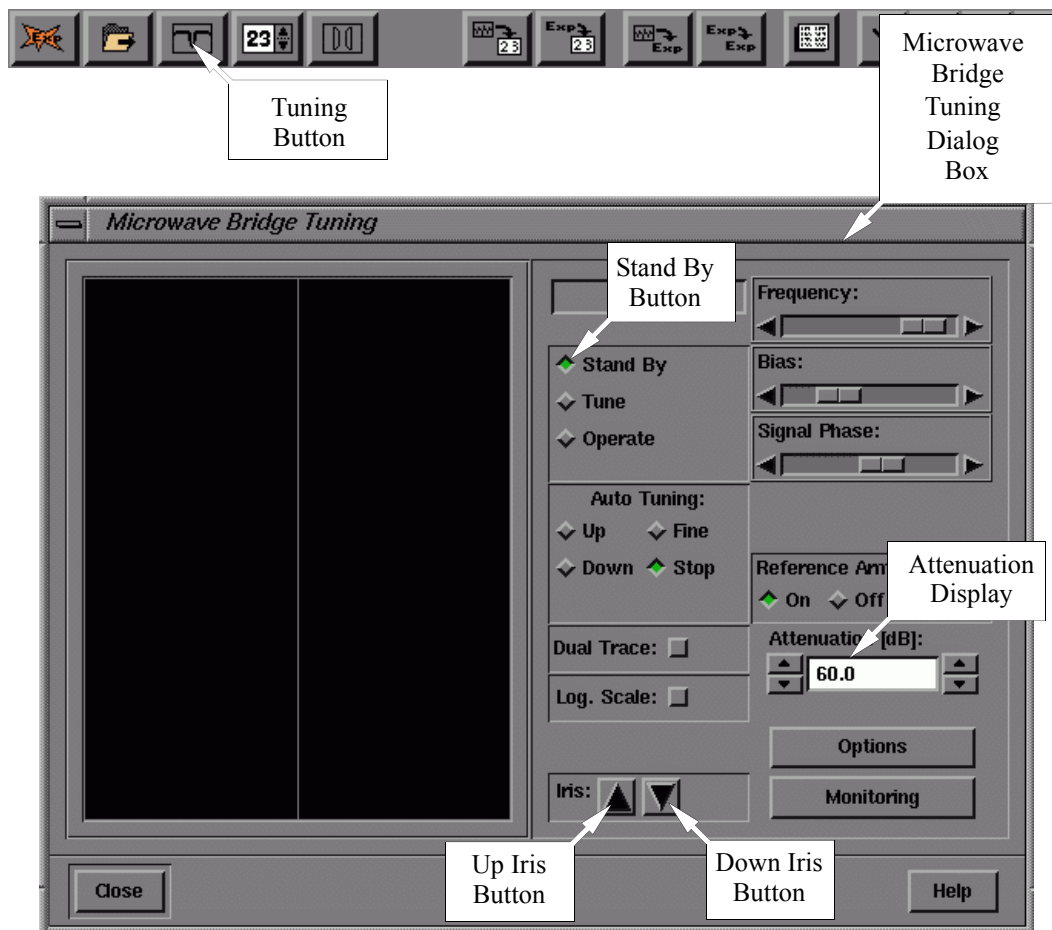


Figure 5-16 The Microwave Bridge Tuning dialog box.

7. **Switch the microwave bridge to Stand By mode.** (See Figure 5-16.) Click the Stand By button in the dialog box to change to the Stand By mode.



## Getting Ready (EMX)

## 5.6.2

1. **Open the Interactive Spectrometer Control dialog box.** If this window is not already open, click its button (See Figure 5-17.) in the tool bar. The button toggles the dialog box open and closed. The Interactive Spectrometer Control dialog box will then appear. (See Figure 5-18.)

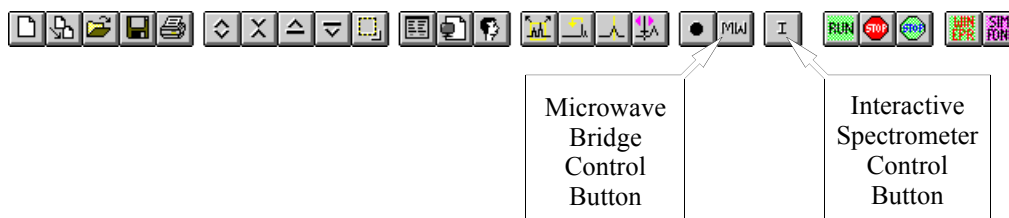


Figure 5-17 The Interactive Spectrometer Control button.

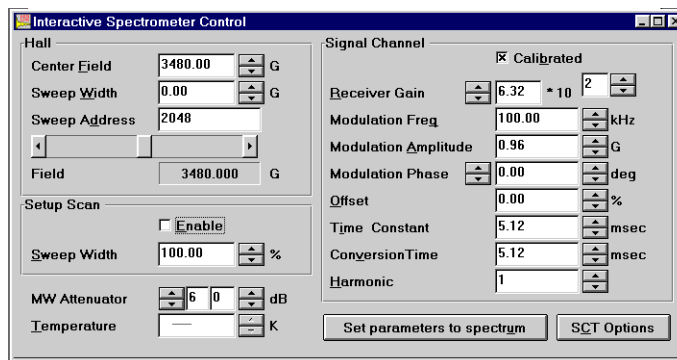
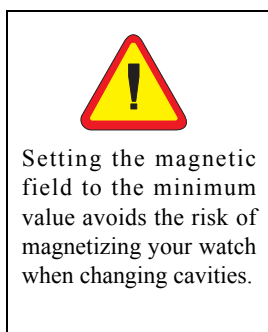


Figure 5-18 The Interactive Spectrometer Control dialog box.



2. **Set the modulation amplitude to zero.** Enter a value of 0.00 in the Modulation Amplitude box.
3. **Set the magnetic field to the minimal value.** Enter a value of 0.00 in the Sweep Width box and a value of 0.00 in the Center Field box.
4. **Close the Interactive Spectrometer Control dialog box.** Click the Interactive Spectrometer Control (the button labeled I) in the tool bar. The button toggles the dialog box on and off. The Interactive Spectrometer Control dialog box will then disappear. (See Figure 5-17 and Figure 5-18.)
5. **Open the Microwave Bridge Control dialog box.** If this window is not already open, click its button (See Figure 5-17.) in the tool bar. The button toggles the dialog box open and closed. The Microwave Bridge Control dialog box will then appear. (See Figure 5-19.)

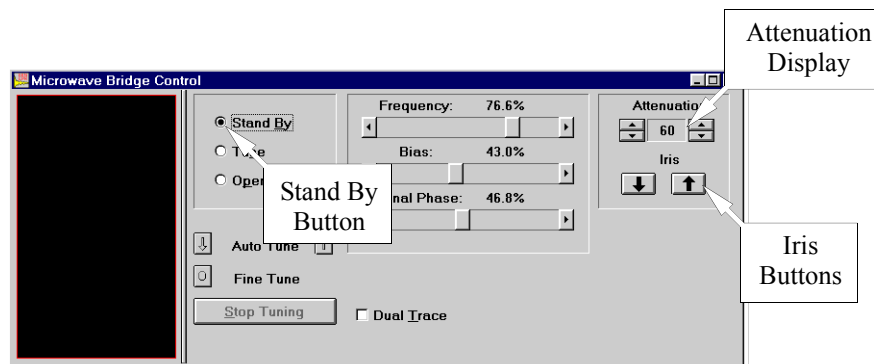


Figure 5-19 The Microwave Bridge Control dialog box.

6. **Switch the microwave bridge to Stand By mode.** Click the Stand By button in the dialog box to change to the Stand By mode. (See Figure 5-19.)

## Changing Cavities

### 5.6.3

1. **Disconnect any accessories.** If a variable temperature dewar assembly is installed, disconnect the coolant transfer line and the thermocouple connections from the cavity.
2. **Disconnect the modulation cable from the cavity.** This is the twin-ax cable labeled with a white connector and attached to the front of the standard cavity. To remove it, push the connector in, turn counter-clockwise and gently pull the cable away. (See Figure 5-20.)
3. **Disconnect the rapid scan cable from the cavity.** Disconnect the 50 Gauss rapid scan cable if it is connected to the cavity. This is the twin-ax cable labeled with a yellow connector and attached to the front left of the standard cavity.
4. **Disconnect the nitrogen purge line from the port on the waveguide.** The port is half way down the waveguide attached to the cavity. (See Figure 5-20.)



Not all cavities have a rapid scan cable.

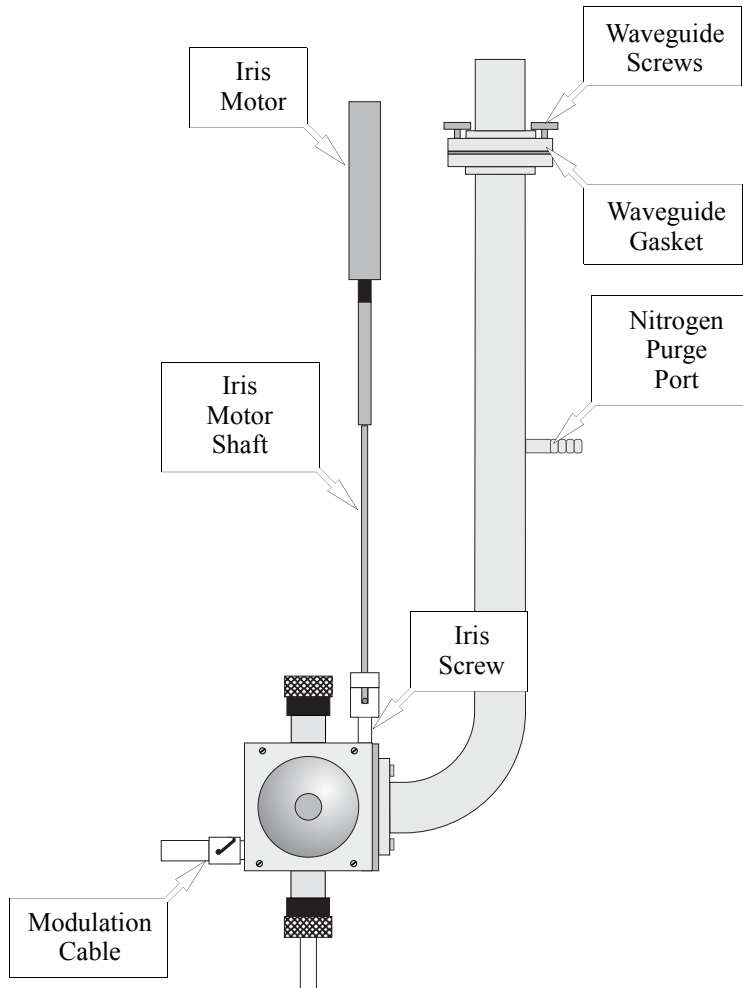
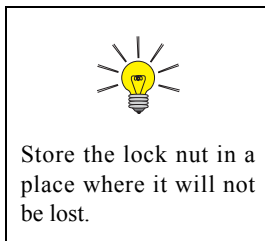


Figure 5-20 Connections on the ER 4122SHQ cavity.



5. **Disconnect the iris motor shaft from the iris screw.** First unscrew the lock nut from the iris screw. Lift the shaft upwards to disconnect. Move the iris motor to the side where it is out of the way. (See Figure 5-21.)

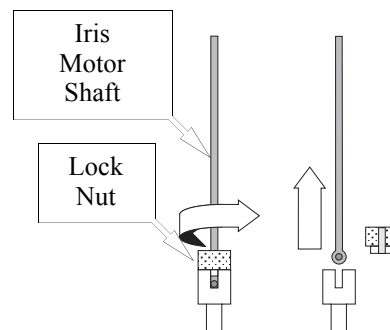


Figure 5-21 Disconnecting the iris motor shaft from the iris screw.

6. **Disconnect the cavity.** While grasping the waveguide attached to the cavity with one hand, unscrew the four waveguide screws joining the two sections of waveguide. (See Figure 5-20.) Loosen the waveguide stabilizers by rotating the screws and carefully remove the cavity from the air gap of the magnet. (See Figure 5-22.) Take care not to lose the gasket which was between the two wave guide flanges. Remove the waveguide stabilizers. (See Figure 5-23.) Seal the cavity with the solid collets and put the cavity in a safe clean place.

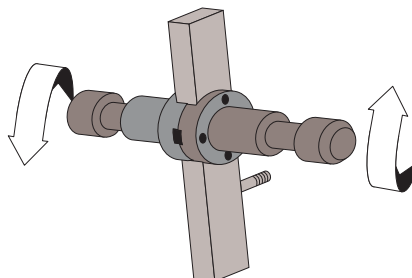


Figure 5-22 Loosening the waveguide stabilizers.

7. **Install the waveguide stabilizers on the new cavity.** (See Figure 5-23.) Visually position them just above the magnet pole caps.

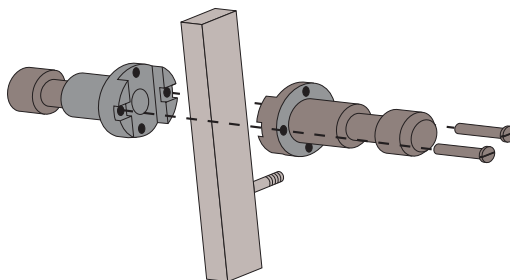


Figure 5-23 Removing/Installing the waveguide stabilizers.



Steps 9. and 15. are used to set the limit switches in the iris motor. The limit switches prevent you from screwing the iris in too far and thereby breaking the iris screw.



Make sure you connect the modulation cable to the MOD (modulation) connector and not the R.S. (Rapid Scan) connector. Not all cavities have rapid scan coils.

8. **Attach the appropriate size collet and pedestal on the cavity.**
9. **Screw in the iris.** Manually (By hand!) turn the iris screw almost all the way in. The iris screw will stop rotating. Back the screw out at least one turn after it hits the bottom. This will further decrease your chances of accidentally breaking the iris screw during the tune procedure.
10. **Connect the modulation cable to the cavity.** (See Figure 5-20.)
11. **Connect the 50 Gauss Rapid Scan cable to the cavity. (Elexsys only.)** If you plan to do rapid scan experiments and there are rapid scan coils on your cavity, connect the 50 G rapid scan cable (the yellow twinax cable) to the front left connector of the cavity.

12. **Reconnect the waveguide sections and tighten the stabilizers.** Do not forget to install the waveguide flange gasket between the two flanges; make sure it is oriented correctly. (See Figure 5-24.) Position the cavity in the center of the magnet air gap by moving the bridge on the table. Carefully tighten the stabilizers. Be careful not to stress the waveguide when expanding the stabilizers. Reconnect the nitrogen purge line and adjust the flow rate for a light flow.

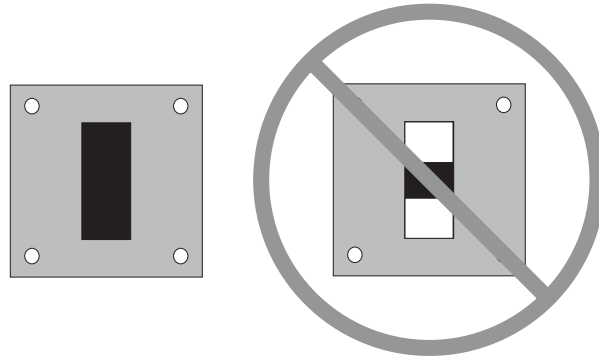


Figure 5-24 Installing the waveguide gasket properly.

13. **Reposition the iris motor.** Move the iris motor into a position such that the iris motor shaft hangs freely in the magnet. It should not be in contact with other objects.

## Finishing Up (Elexsys)

## 5.6.4

1. **Lower the iris motor.** Open the Microwave Bridge Tuning dialog box. Click the Options button.

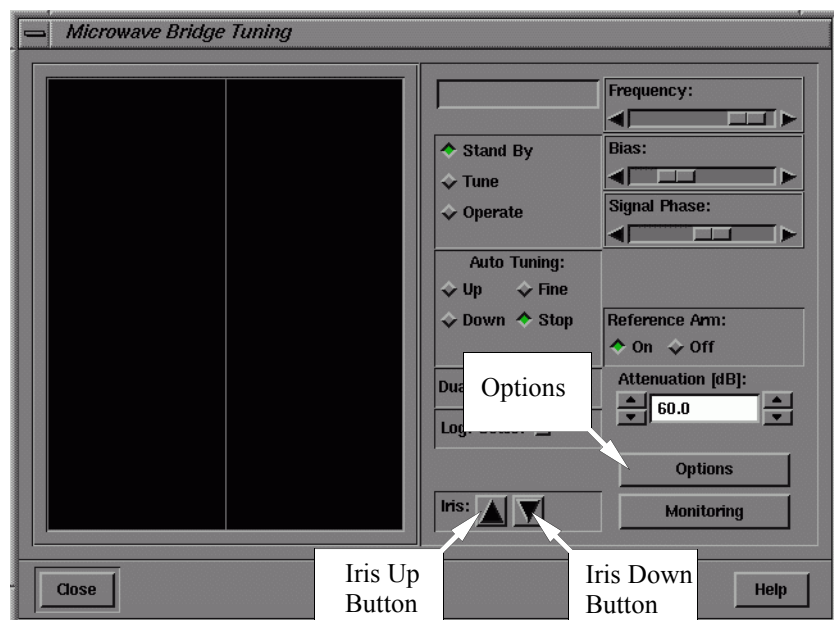


Figure 5-25 Iris controls in the Microwave Bridge Tuning dialog box.

A dialog box for lowering the iris screw appears. (See Figure 5-26.)



Figure 5-26 The Iris Control dialog box.

Click the button next to Iris Run Down. A message reminding you to disconnect the iris motor pops up. Heed the message and then click the **Yes** button to close the dialog box. (See Figure 5-27.) The iris motor will turn until it reaches the lower limit. Click **Close** on the Iris Control dialog box when the iris motor stops. You can also manually lower the iris motor. Click and hold the Iris Down button. In the Microwave Bridge Tuning dialog box (See Figure 5-25.), activate the Iris Down button until the iris motor stops; this is the lower limit of the motor.

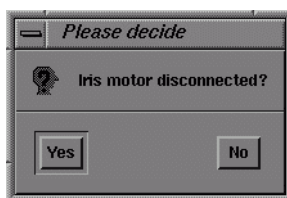


Figure 5-27 Make sure disconnecting the iris motor.



The end of the iris motor shaft should fit easily in the top of the iris screw. If it does not, rotate the iris screw until it fits easily.

2. **Reconnect the iris motor shaft to the iris screw.** The procedure here is like Step 5. performed in reverse. Reposition the iris screw motor. Screw the lock nut on the iris screw. Click and hold the up iris button in the Microwave Bridge Tuning dialog box (See Figure 5-16.) until the iris screw is approximately 0.5 to 1 cm. above the top surface of the cavity body.

3. **Read in the calibration file for the cavity.** Click Acquisition in the menu bar and then click Spectrometer Configuration. (See Figure 5-28.) The Spectrometer Configuration dialog box will then appear. (See Figure 5-29.)

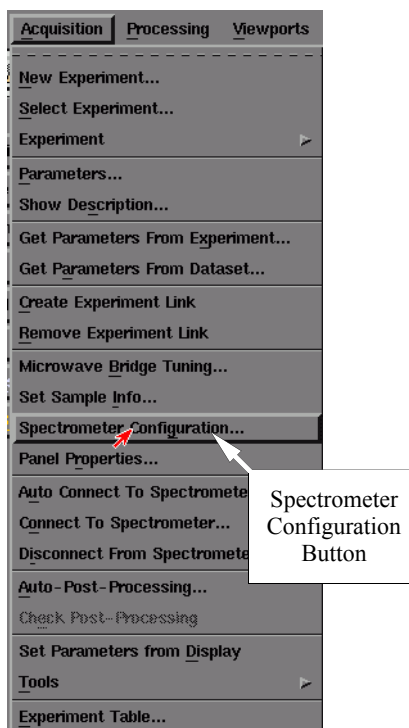


Figure 5-28 The Spectrometer Configuration button.

Click the Signal Channel tab. In the Standard Calibration folder there is a box called Calibration Data Set. Click the arrow to view the drop-down list. Select the appropriate calibration file for your cavity and click the Apply button at the bottom of the folder. This will automatically load the calibration data you have selected.

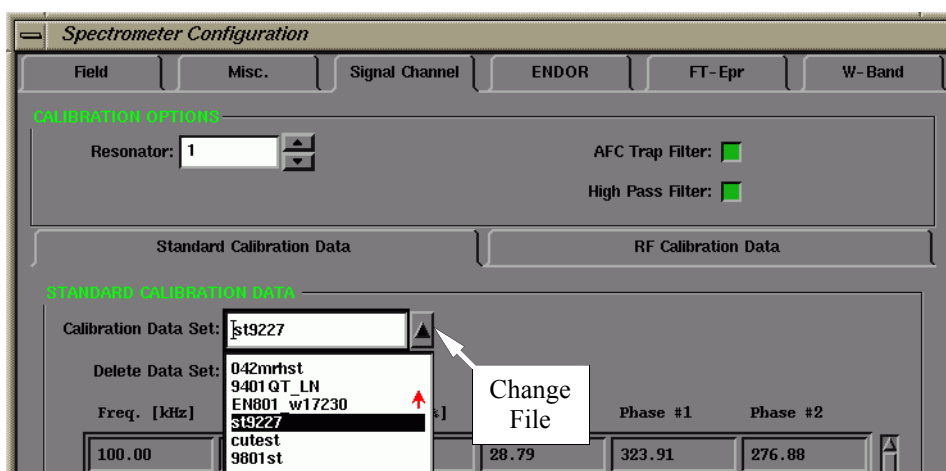


Figure 5-29 Selecting a calibration file.

Confirm that the calibration file is the correct one for the cavity. The calibration file name usually consists of two or three letters that identify the type of cavity (ST for ER 4102ST or TM for ER 4103TM) followed by the serial number of the cavity. This number is found on the back or front of the cavity. Click **Close** to exit. If you can not find the calibration file for the cavity you need to do the calibration first. Follow the instruction in Chapter 2 of the Elexsys E 500 User's Manual: Advanced Operations to create a calibration file.

## Finishing Up (EMX)

### 5.6.5



The end of the iris motor shaft should fit easily in the top of the iris screw. If it does not, rotate the iris screw until it fits easily.

1. **Click and hold the down Iris Button.** Activate this button (See Figure 5-19.) until the iris motor stops turning; this is the lower limit of the motor.
2. **Reconnect the iris motor shaft to the iris screw.** The procedure here is like Step 5. performed in reverse. Reposition the iris screw motor. Screw the lock nut on the iris screw. Click and hold the up iris button in the **Microwave Bridge Tuning** dialog box (See Figure 5-19.) until the iris screw is approximately 0.5 to 1 cm. above the top surface of the cavity body.
3. **Read in the calibration file for the cavity.** Open the **Experiment Options** dialog box in order to read in the calibration information. If this window is not already open, click its button (See Figure 5-30.) in the tool bar. The button toggles the dialog box open and closed. The **Experiment Options** dialog box will then appear. Click on the **Change File** button. A new dialog box, **Open Calibration File** will appear. Select the appropriate calibration file for your cavity and click **OK**. This will automatically load the calibration data you have selected. Confirm that the calibration file is the correct one for the cavity. The calibration file name usually consists of two or three letters that identify the type of cavity (ST for ER 4102ST or TM for ER 4103TM) followed by the serial number of the cavity. This number is located on either the front or back of the cavity. Clicking **Cancel** returns you to the **Experiment Options** dialog box.



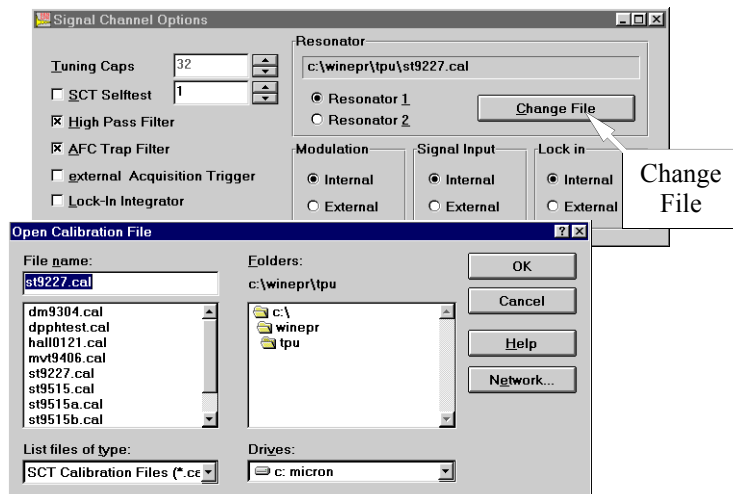


Figure 5-30 The Experiment Options and Open Calibration File dialog boxes.

Service engineers often save the calibration files in the c:\...\acquisit\tpu directory during the installation of the spectrometer.

## B<sub>1</sub> Conversion

## 5.7

The resonator converts microwave power into B<sub>1</sub> (microwave magnetic magnetic field) at the sample. Each type of resonator has a different conversion efficiency. The following table helps you to calculate an approximate B<sub>1</sub> value for different types of resonators.

The values are calculated by using the following equation:

$$B_1 [G] = C \cdot \sqrt{P[W]}, \quad [5-7]$$

where C is the constant in the table and P is the microwave power. To take into account different bandwidths the C' constant can be used in the following equation:

$$B_1 [G] = \frac{C' \cdot \sqrt{P[W]}}{\sqrt{\Delta\nu[\text{MHz}]}} \quad [5-8]$$

where  $\Delta\nu$  is the bandwidth of the resonator.

<b>B<sub>1</sub> Conversion</b>	<b>Mode</b>	<b>Q</b>	<b>C [G / √W]</b>	<b>C' [G√MHz / √W]</b>
ER 4102ST Standard Rectangular	TE <sub>102</sub>	2500 150	1.4 0.3 (overcoupled)	2.7 2.4
ER 4105DR Double Rectangular	TE <sub>104</sub>	3000	0.6	1.1
ER 4104OR Optical Transmission	TE <sub>103</sub>	3000	1.0	1.8
ER 4116DM Dual Mode Resonator	TE <sub>102</sub> ⊥	3000	1.3	2.3
ER 4103TM Cylindrical TM-Mode	TM <sub>110</sub>	3500	1.0	1.7
ER 4108TMH High Sensitivity TM-Mode	TM <sub>110</sub>	2500	1.2	2.4
ER 4106ZRC Cylindrical Concentric	TE <sub>011</sub>	7500	1.5	1.7
ER 4115ODC Optical Cylindrical Concentric	TE <sub>011</sub>	5000	1.3	1.8
ER 4122 SHQ Super High Q Resonator	N/A	7500	2.0	2.3
EN 801 ENDOR Resonator	TM <sub>110</sub>	1000	0.7	2.2
ER 4118 X-MD5 Dielectric Resonator	TE <sub>011</sub>	4000 150	4.2 1.0 (overcoupled)	6.5 9
ER 4118 X-MS5 Split Ring Resonator	N/A	500 150	2.0 1.2 (overcoupled)	8.8 9.6
EN 4118 X-MD5 Pulsed ENDOR Resonator	TE <sub>011</sub>	500 150	1.8 1.0 (overcoupled)	7.9 8

Table 5-1 Conversion factors for Bruker EPR resonators.