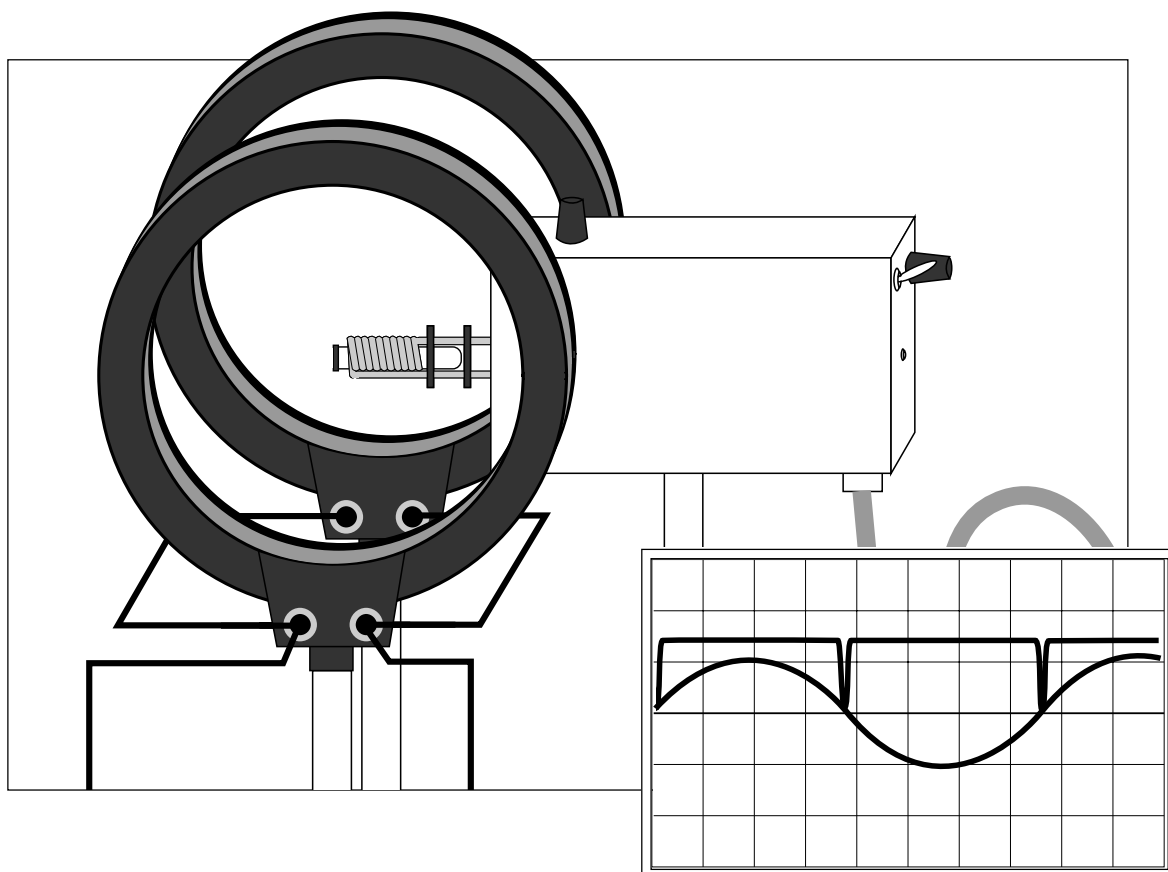


**Instruction Manual and
Experiment Guide for
the PASCO scientific
Model SE-9634, 9635, and 9636**

012-04273D
10/95

ESR APPARATUS



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The lightning flash with arrowhead, within an equilateral triangle, is intended to alert the user of the presence of uninsulated “dangerous voltage” within the product’s enclosure that may be of sufficient magnitude to constitute a risk of electric shock to persons.



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The exclamation point within an equilateral triangle is intended to alert the user of the presence of important operating and maintenance (servicing) instructions in the literature accompanying the appliance.

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Introduction

ESR in Theory

The basic setup for electron spin resonance is shown in Figure 1. A test sample is placed in a uniform magnetic field. The sample is also wrapped within a coil that is connected to an RF oscillator. The smaller magnetic field induced in the coil by the oscillations of the oscillator is at right angles to the uniform magnetic field.

Consider, for the moment, a single electron within the test sample. The electron has a magnetic dipole moment (μ_s) that is related to its intrinsic angular momentum, or spin, by the vector equation:

$$\mu_s = g_s \mu_B \frac{S}{\hbar}; \text{ (equation 1)}$$

where:

g_s = a constant characteristic of the electron, the g-factor

μ_B = the Bohr magneton = $\frac{eh}{2m_e} = 5.788 \times 10^{-9} \frac{eV}{G}$

s = the spin of the electron

\hbar = Planck's constant = 6.582×10^{-16} eV-sec.

The magnetic dipole moment of this electron interacts with the uniform magnetic field. Due to its quantum nature, the electron can orient itself in one of only two ways, with energies equal to $E_0 \pm g_s \mu_B / 2$; where E_0 is the energy of the electron before the magnetic field was applied. The energy difference between these two possible orientations is equal to $g_s \mu_B B$; where B is the magnitude of the magnetic field.

Resonance occurs when the RF oscillator is tuned to a frequency ν , such that the energy of the irradiated photons, $h\nu$, is equal to the difference between the two possible energy states of the electron. Electrons in the lower energy state can then absorb a photon and jump to the higher energy state. This absorption of energy effects the permeability of the test sample, which effects the inductance of the coil and thereby the oscillations of the RF oscillator. The result is an observable change in the current flowing through the oscillator.

The condition for resonance, therefore, is that the energy of the photons emitted by the oscillator match the energy difference between the spin states of the electrons in the test sample. Stated mathematically:

$$h\nu = g_s \mu_B B$$

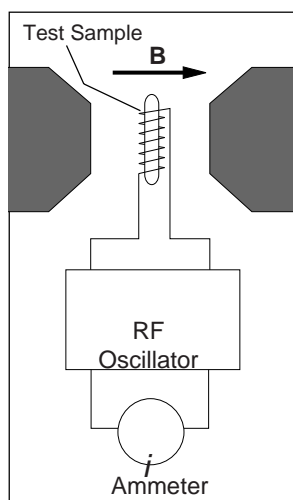


Figure 1 ESR Diagram

ESR in Practice

For an electron with only two energy states, in a magnetic field of a given magnitude, it would be necessary to set the RF frequency with considerable accuracy in order to observe resonance. In practice, this difficulty is solved by varying the magnitude of the magnetic field about some constant value. With the PASCO ESR Apparatus, this is done by supplying a small AC current, superimposed on a larger DC current, to a pair of Helmholtz coils. The result is a magnetic field that varies sinusoidally about a constant value.

If the RF frequency is such that equation 2 is satisfied at some point between the minimum and maximum values of the sinusoidally varying magnetic field, then resonance will occur twice during each cycle of the field. Resonance is normally observed using a dual trace oscilloscope. The oscilloscope traces, during resonance, appear as in Figure 2. The upper trace is a measure of the current going to the Helmholtz coils, which is proportional to the magnetic field. The lower trace shows the envelope of the voltage across the RF oscillator, which dips sharply each time the magnetic field passes through the resonance point.

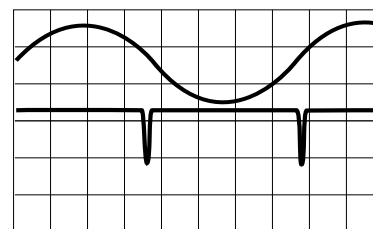


Figure 2 ESR on the Oscilloscope

ESR in Research

In research, ESR measurements are considerably more complicated than equation 2 would indicate. The electrons and protons in an atom or molecule form a complicated electromagnetic environment, which is affected by the externally applied magnetic field. The various energy splittings and shifts that show up in ESR measurements can therefore provide sensitive information about the internal structure of the atoms and molecules.

The test sample included with the PASCO ESR Apparatus, DPPH*, is a particularly simple substance for ESR measurements. It has an orbital angular momentum of zero, and only one unpaired electron. Therefore, for a given value of the external magnetic field, it has only a single resonant frequency. This makes it possible to investigate some of the basic principles of electron spin resonance, without (or before) getting into the more complex world of ESR analysis.

(* Diphenyl-Picryl-Hydrazyl)

The ESR Equipment

Included Equipment

The ESR Apparatus is available in three separate packages (see Figure 3):

- **The ESR Probe Unit (SE-9634)** includes:
 - The Probe Unit with base-
 - Three RF Probes and a DPPH sample in a vial
 - The Passive Resonant Circuit
 - The Current Measuring Lead for the Probe Unit
- **The ESR Basic System (SE-9635)** includes:
 - The ESR Probe Unit (SE-9634)
 - A pair of Helmholtz Coils with bases
 - The ESR Adapter (SE-9637)
- **The Complete ESR System (SE-9636)** includes:
 - The ESR Probe Unit (SE-9634)
 - A pair of Helmholtz Coils with bases
 - The Control Unit

Additional Equipment Needed:

If you're using the SE-9636 Complete ESR System, you'll need the following additional equipment:

- a DC ammeter capable of measuring up to 3 A (such as PASCO's SB-9599)
- a dual trace oscilloscope (such as PASCO's SE-9533)
- connecting wires with banana plug connectors

If you're using the SE-9635 Basic ESR System, you'll need the following additional equipment:

- **Frequency Meter** (to 130 kHz) (such as PASCO's SB-9599 Universal Multimeter)
- **SF-9584 Low Voltage AC/DC Power Supply** (or an equivalent supply providing a 10 volt, 3 amp DC output and a 0 - 4 volt, 1 amp AC output)
- **Power Supply** providing ± 12 VDC
- **DC Ammeter** (0 - 3 amp) (PASCO Model SB-9599)

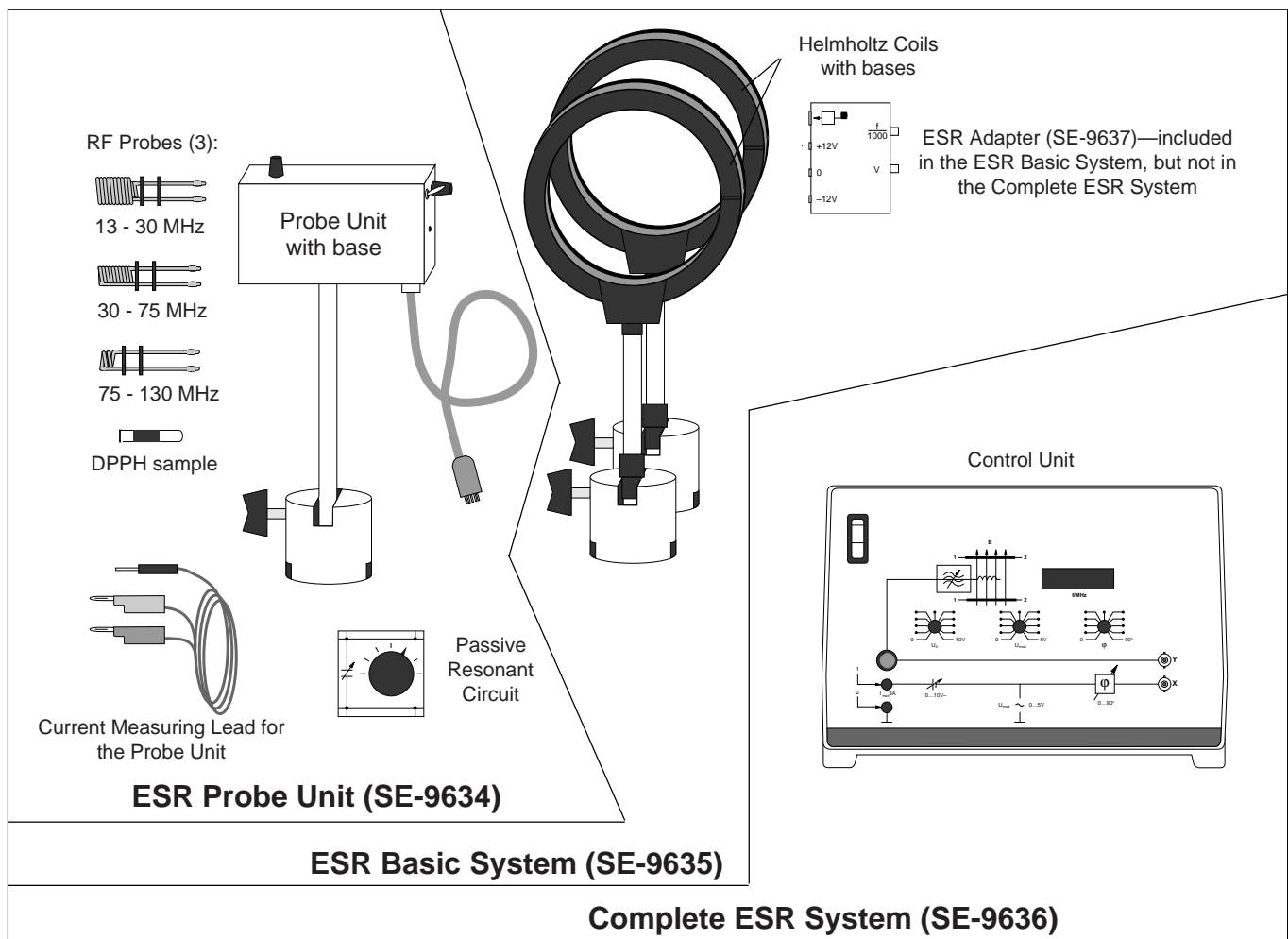


Figure 3 The ESR Apparatus

- **Oscilloscope** (dual trace) (PASCO Model SE-9533)
- **Circuit components:**
capacitors: 1000 μF , 25 V; and 0.1 μF
potentiometer (100 k Ω)
connecting wires

If you're using the SE-9634 ESR Probe Unit, you'll need the same equipment as is required with the Basic ESR System, except that you will also need:

- **SE-9637 ESR Adapter** (or a junction box of your own making—see Appendix)
- a pair of Helmholtz coils.

About the ESR Apparatus

The Probe Unit

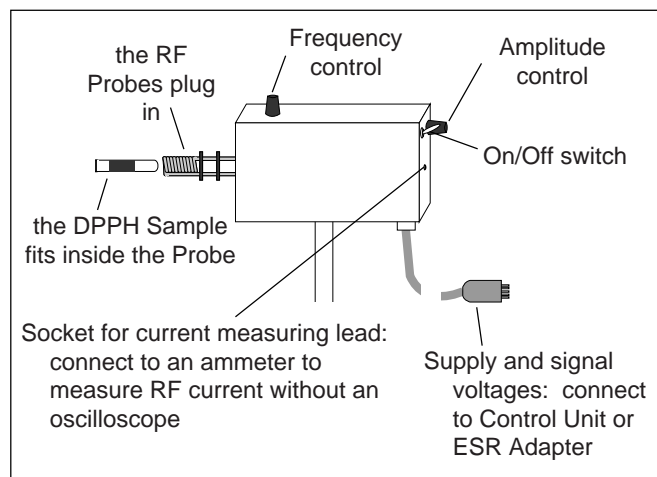


Figure 4 ESR Probe Unit

The ESR Probe Unit (Figure 4) is the heart of the ESR apparatus. It contains an RF oscillator with a built-in signal amplifier, and a 1000:1 frequency divider. The frequency divider allows the RF frequency, which is in the MHz range, to be measured using a standard kHz frequency meter.

The frequency and amplitude of the RF signal can be controlled using the knobs shown in the figure. The range of frequencies provided by the oscillator depends on which RF Probe is being used. This is because the inductance of the probe determines, in part, the inductance of the oscillator circuit. See Figure 3 for the frequency range associated with each probe.

Normally the Probe Unit is plugged into the Control Unit, which provides all necessary operating voltages. However, it can also be connected to separate laboratory instruments using the ESR Adapter, or a home made adapter. See *Setup with the Basic ESR System* for details.

Probe Unit Technical Data:

Power Requirements: ± 12 V, 175 mA

Frequency ranges: 13 - 130 MHz depending on RF Probe (see Figure 1)

Voltage across the RF Probe: 6 V peak-to-peak at 13 MHz and maximum amplitude

ESR signal: 1 - 6 V depending on frequency

Frequency divider: 1000:1

Frequency output for digital counter: TTL

DC current output: 100 μA approximately

Test substance: Diphenyl-Picryl-Hydrazyl (DPPH)

The Control Unit

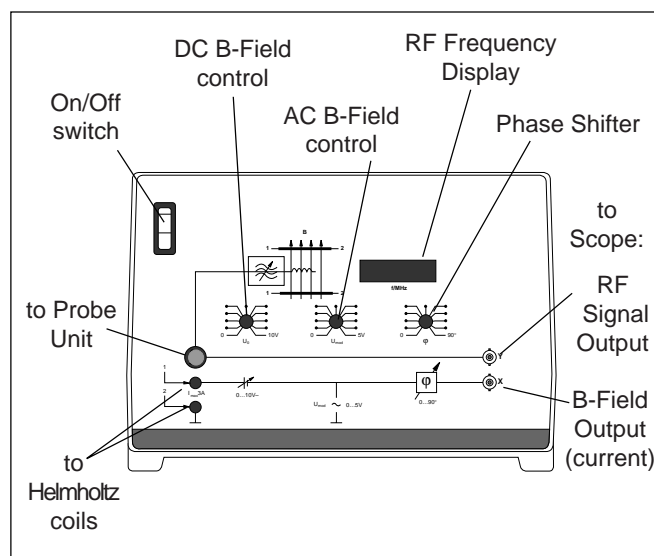


Figure 5 ESR Control Unit

The Control Unit provides most of the instrumentation needed to use the ESR Probe Unit. The front panel controls are explained in Figure 5. The Control Unit serves three functions:

- ① it supplies the voltages needed to drive the Probe Unit and the Helmholtz coils;
- ② it provides a digital readout of the RF oscillations produced by the Probe Unit;
- ③ it provides outputs for a dual trace oscilloscope. One output is proportional to the current in the RF oscillator, and is used to observe the resonance pulses. The other output is proportional to the current that is supplied to the Helmholtz coils, and indicates the magnitude of the magnetic field.

►NOTE: There is an inherent phase shift between the current in the Helmholtz coils and the voltage signal going to the oscilloscope. This shift is caused by the inductance of the coils. The Control Unit includes a phase shifter that lets the user compensate for this delay, so the magnetic field and the resonance pulses can be observed with their proper phase relationship.

The Control Unit operates on a line voltage of 110, 130, 220, or 240 VAC, 60 Hz. See the Appendix for information about adjusting the unit for the proper line voltage.

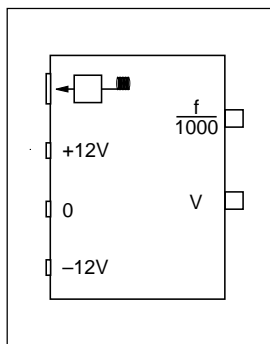


Figure 6 ESR Adapter

Control Unit Technical Data:

- Power Requirements: 120 or 240 VAC; 60 Hz
- Fuse: 1.6 A, 220 V (slo-blo) for 120 VAC
0.8 A, 220 V (slo-blo) for 240 VAC
- Magnetic Field Supply: 0 - 10 VDC; 0 - 5 VAC; maximum current 3 A (not overload protected)
- Phase Shifter: 0 - 90°
- Digital Frequency Display: 4 digits

ESR Adapter

If you are not using the Control Unit, the ESR Adapter can be used to connect the Probe Unit to the necessary power supply, frequency meter, and oscilloscope. See the section, *Setup with the Basic ESR System*, for details of the connections. See the Appendix if you would like details for building your own adapter.

Helmholtz Coils

The Helmholtz coils provide a highly uniform magnetic field in which to place the sample material for the ESR measurement. They should be connected in parallel and placed so that the separation between them is equal to the radius (see Figure 7). When this is the case, the magnetic field in the central area between the two coils is highly uniform, and is equal to the value shown in Figure 7.

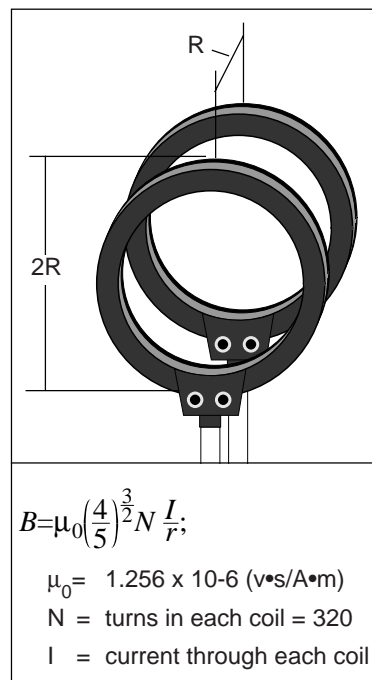


Figure 7 The Helmholtz Coils

►IMPORTANT: Current to each of the Helmholtz Coils should never exceed 3A. Therefore, if they are connected in parallel, the total current to them should never exceed 6 A.

The Passive Resonant Circuit

The Passive Resonant Circuit can be used to demonstrate resonant energy absorption in a non-quantum system. It is just an LC circuit with an adjustable capacitance. It replaces the test sample and the Helmholtz coils in the ESR experiment. See the Appendix for details on using the Passive Resonant Circuit.

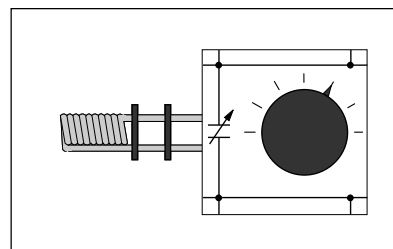


Figure 8 The Passive Resonant Circuit

Setup with the Complete ESR System

Required Equipment

To perform ESR experiments with the Complete ESR System, you'll need the following additional equipment:

- a DC ammeter capable of measuring up to 3 A
- a dual trace oscilloscope
- connecting wires with banana plug connectors.

Setup

- ① Connect the Helmholtz coils to the Control Unit, as shown in Figure 9. (The coils should be connected in parallel—terminal A to terminal A, and Z to Z.) Connect an ammeter in series, as shown, to monitor the current to the coils.

►IMPORTANT: the DC current supply in the Control Unit is *not* overload protected. Do *not* let the current to the Helmholtz coils exceed 3A.

- ② Position the Helmholtz coils so that they are parallel and facing in the same direction, and their separation is equal to approximately one half their diameter.

- ③ Connect the X output of the Control Unit to channel 1 of a dual trace oscilloscope. Set the oscilloscope controls as follows:

Sensitivity: 1 or 2 V/div
Sweep Rate: 2 or 5 ms/div
Coupling: DC

- ④ Set U_{mod} , the center knob on the Control Unit, to zero, then slowly vary U_0 , the left knob, from 0 to 10 V and observe the trace on the oscilloscope. It should be a clean, straight line, showing that the DC component of the current to the Helmholtz coils is constant. (U_0 controls the DC current going to the Helmholtz coils.)

►NOTE: If the oscilloscope trace is not straight, your Control Unit is probably not set for the proper line voltage. See the Appendix for help.

- ⑤ Set U_0 at approximately midscale, then turn U_{mod} clockwise, to increase the AC component of the current to the Helmholtz coils. The trace on the oscilloscope should now show a smooth sine wave, as in Figure 10, corresponding to an AC magnetic field that is superimposed upon a constant DC field.

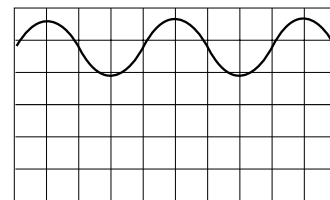


Figure 10
Oscilloscope—current to the Helmholtz coils

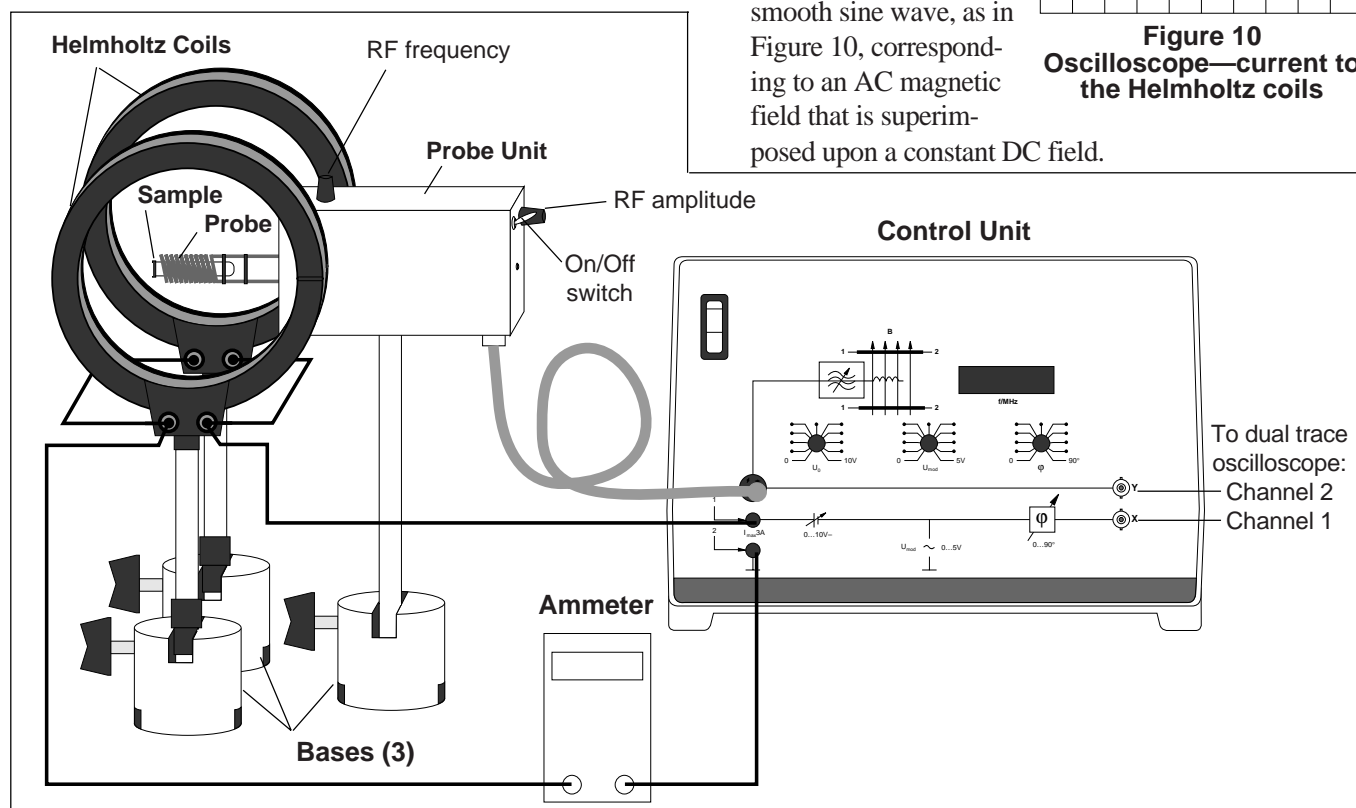


Figure 9 Setup using the Control Unit

- ⑥ Connect the Y output of the Control Unit to channel 2 of the oscilloscope. Set the oscilloscope controls for channel 2 as follows:
Sensitivity: 0.5 or 1 V/div
Coupling: DC
- ⑦ Connect the Probe Unit to the Control Unit, as shown in Figure 9.
- ⑧ Plug the medium sized RF Probe into the Probe Unit, and insert the sample of DPPH into the coil of the probe.
- ⑨ Turn on the Probe Unit by flipping the On/Off switch to the up (I) position. Then turn the Amplitude knob on the Probe Unit to a medium setting.
- ⑩ The frequency meter on the Control Unit should now indicate the frequency of the RF oscillations. Adjust the Frequency control knob on the Probe Unit to produce an output of approximately 50 MHz.
- ⑪ Set U_{mod} to about the 4th position above zero (at about the 11 o'clock position).

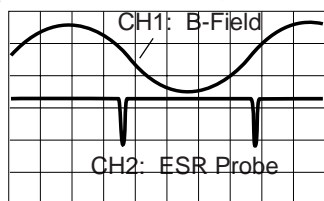


Figure 11 the Oscilloscope Display

- ⑪ Increase U_0 from zero to a medium value, so the Helmholtz coil current is about 1.0 A. The oscilloscope traces should now look as in Figure 11. The channel 1 trace shows the current to the Helmholtz coils, which is proportional to the magnetic field produced by the coils. The channel 2 trace shows the envelope of the voltage across the RF oscillator, with the pulses showing the points of resonance absorption.

If you see no resonance pulses, slowly vary U_{mod} or the RF frequency, until you do.

- ⑬ Your traces may not be symmetrical, as they are in Figure 11. This is because of the inductance of the Helmholtz coils, which causes the current through them, and therefore the magnetic field they produce, to lag the voltage that drives them. You can compensate for this delay by adjusting j , the Phase Shifter control knob, until the traces are symmetrical. When symmetrical, the traces properly reflect the relationship between the modulating magnetic field and the resonant pulses.

ESR in the X-Y Mode

- ⑭ ESR is often observed with the oscilloscope in the X-Y mode. For this mode of observation, connect the X and Y outputs of the Control Unit to the X and Y inputs of the oscilloscope, respectively. In this mode, the horizontal displacement of the trace indicates the magnitude of the magnetic field between the Helmholtz coils. The vertical displacement indicates the signal from the Probe Unit.

As before, two resonance pulses can be observed since the magnetic field passes through the correct value twice each cycle. By adjusting the Phase Shifter, the two peaks can be brought into coincidence. The resulting trace will appear as in Figure 12.

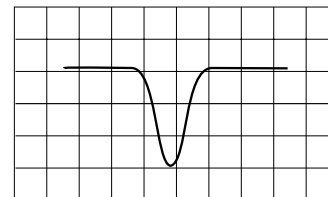


Figure 12 the Oscilloscope Display

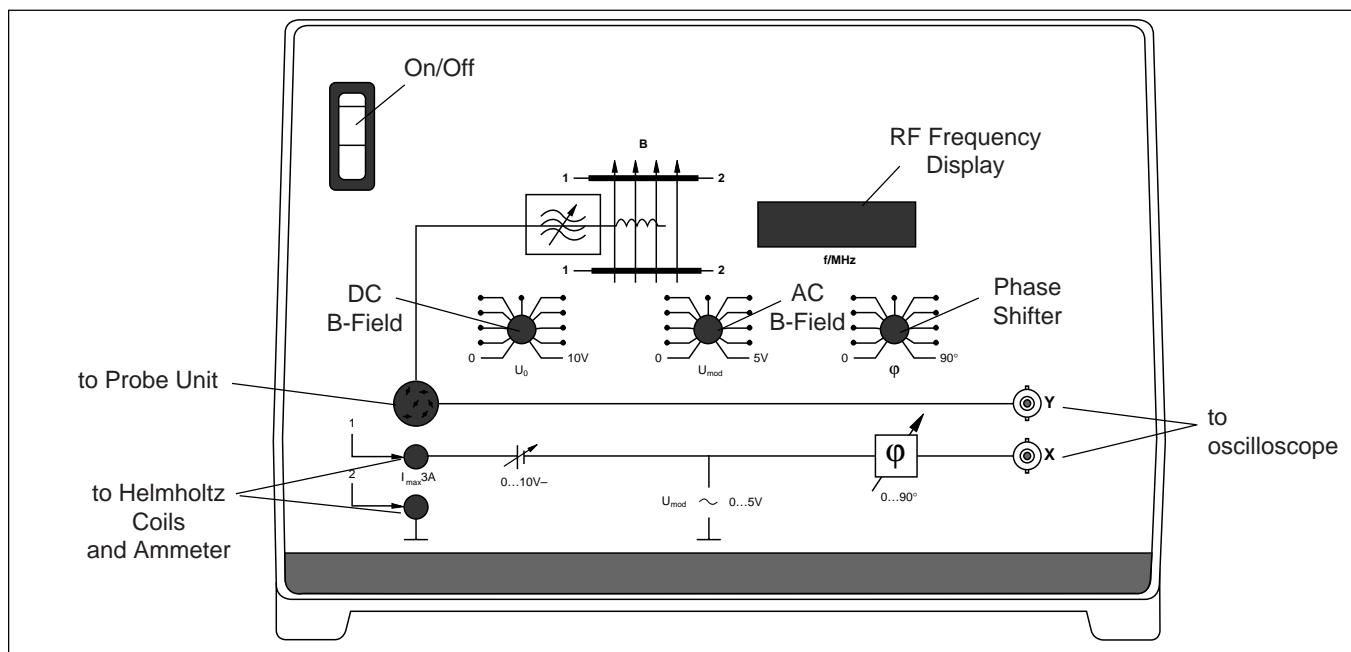


Figure 13 Using the Control Unit

Setup with the ESR Basic System

Required Equipment

To perform ESR experiments with the Basic ESR System, you'll need the following additional equipment:

- **Frequency Meter** (to 130 kHz) (such as the SB-9599)
- **SF-9584 Low Voltage AC/DC Power Supply**
(or equivalent supply providing a 10 V, 3 A DC output, and a 0 - 4 V, 1 A AC output)
- **Power Supply** providing ± 12 VDC
- **DC Ammeter** (0 - 3 amp) (such as the SB-9599)
- **Oscilloscope** (dual trace) (such as the SE-9533)
- **Circuit components:**
 - capacitors: 1000 μ F, 25 V; and 0.1 μ F
 - potentiometer (100 k Ω)
 - connecting wires

Setup

- ① Connect the Helmholtz coils in parallel, as shown in Figure 14. (Connect terminal A on one coil with terminal A on the other, and likewise connect Z to Z.)
- ② Position the Helmholtz coils so that they are parallel and facing the same direction. The separation between the coils should be approximately equal to their radius. This arrangement produces a highly uniform magnetic field around the center point between the two coils.
- ③ Connect the power supplies, ammeter, oscilloscope, and circuit components to the Helmholtz coils as shown in Figure 15.

Circuit Explanation: The Helmholtz coils require a small AC current superimposed on a larger DC current. This is supplied by the AC and DC power supplies connected in parallel. The 1000 μ F capacitor isolates the AC supply from the DC supply to prevent wave distortion.

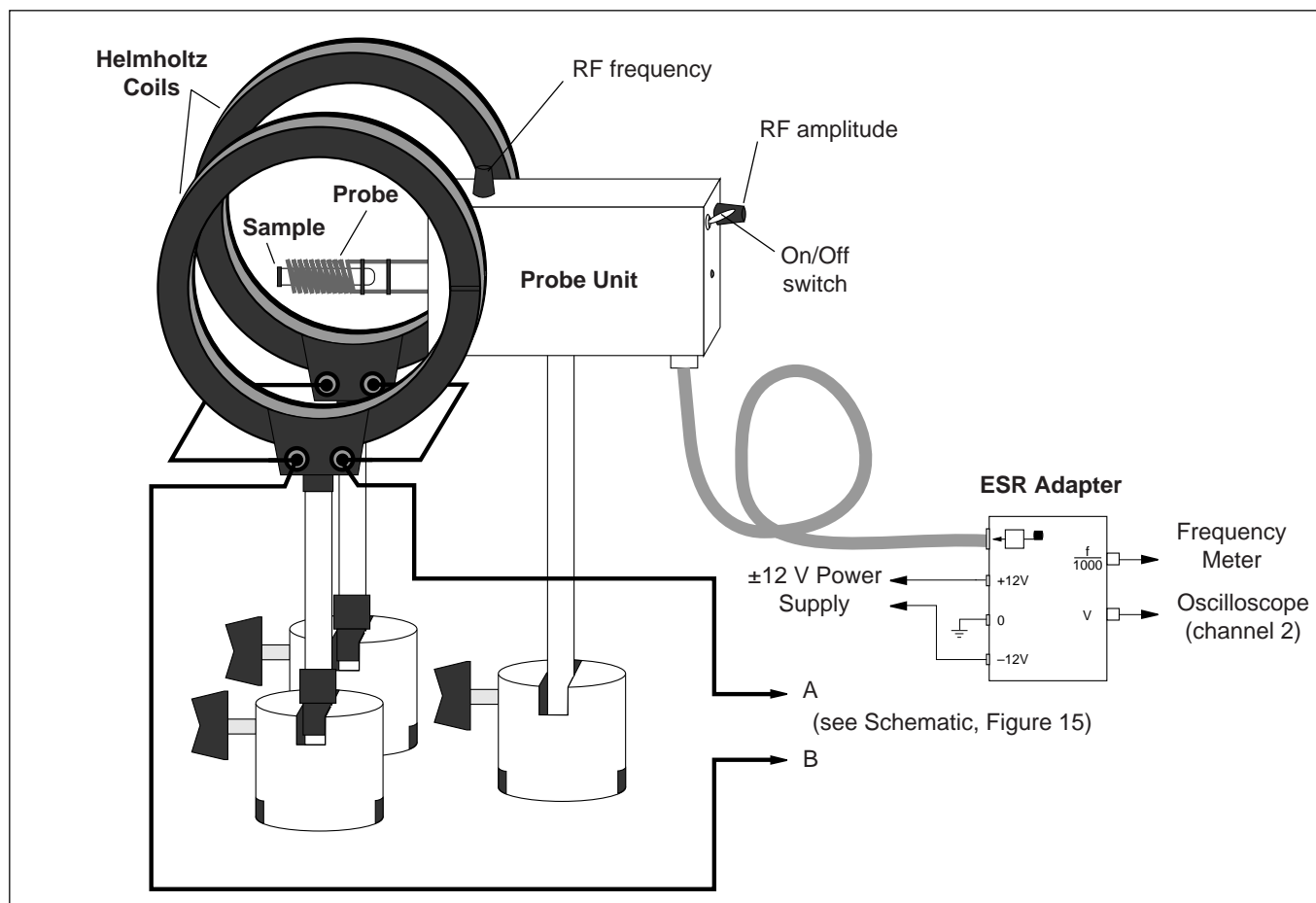


Figure 14 Setup Using the Basic ESR System

Because of the inductance of the Helmholtz coils, the current in the coils is out of phase with the voltage that is observed on the oscilloscope. To correct this, a 100 kΩ variable resistor and a 0.1 μF capacitor are used to shift the phase of the voltage that is displayed on the oscilloscope. This allows the experimenter to adjust the phase between the oscilloscope traces, so that the AC current to the Helmholtz coils and the ESR resonance pulses appear symmetrical, which in reality, they are.

- ④ Turn on the power supplies. Adjust the DC supply to provide approximately 1 amp to the Helmholtz coils. Set the output of the AC supply to about 2 volts.

With the oscilloscope controls set to the values shown in Figure 15, channel 1 of the oscilloscope will show the current to the Helmholtz coils, except for the phase

shift caused by the induction of the coils. The trace should be a simple sine wave. (If you switch channel 1 to DC coupling, it should show an AC voltage superimposed on a DC voltage.)

- ⑤ Connect the Probe Unit to the ESR Adapter, and connect the Adapter to the power supply, frequency meter, and oscilloscope, as indicated in Figure 14. (If you aren't using the ESR Adapter, the connections to the Probe Unit are as shown in Figure 15. If you wish to build your own adapter box, see the Appendix.)

►NOTE: The Probe Unit contains a frequency divider, so that the frequency of the signal sent to the frequency meter is actually a factor of 1000 less than the frequency of the RF oscillations. Because of this, your frequency meter need only measure accurately into the 130 kHz range.

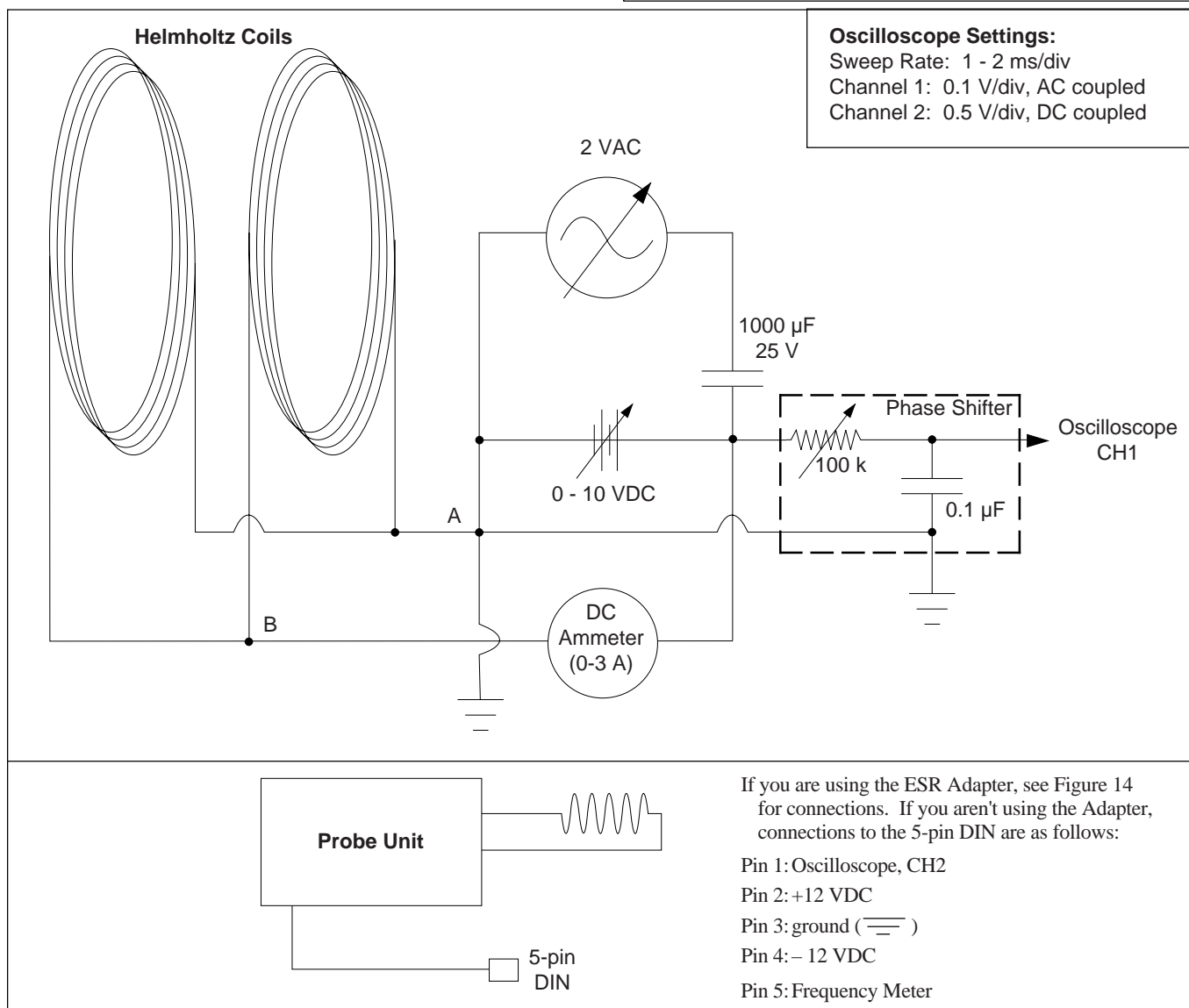


Figure 15 Schematic for Setup without Control Unit

- ⑥ Insert the medium-sized RF Probe into the connectors on the Probe Unit, and turn on the Probe Unit. Adjust the frequency to about 50 MHz (50 kHz on your frequency meter) and the amplitude to a midrange value. Then insert the test sample into the RF Probe and place the probe and sample in the center of the Helmholtz coils, with the axis of the Helmholtz coils perpendicular to the sample (see Figure 14). The oscilloscope traces should now appear approximately as in Figure 16.

- ⑦ If you don't see the resonance pulses, slowly vary the DC current to the Helmholtz coils, or vary the RF frequency, until you do.

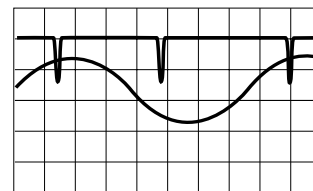


Figure 16 Scope Display

Making ESR Measurements

Whether you are using the Complete ESR System or the ESR Basic System, the same technique is used for making measurements.

- ① Setup the apparatus as described in the appropriate section.
- ② Adjust the RF frequency and the DC current to the Helmholtz coils until you locate the resonance pulses.
- ③ Adjust the phase shifter so that the resonance pulses are symmetric with respect to the oscilloscope trace that shows the current to the Helmholtz coils.
- ④ Refine the adjustment of the DC current until the resonance pulses occur when the AC component of the current to the Helmholtz coils is zero.

To do this:

- a. Make sure that channel 1 of the oscilloscope (the trace showing the current to the Helmholtz coils) is in the AC coupled mode.
- b. Using the oscilloscope controls, ground the input to channel 1, zero the trace, and then unground the input.
- c. Adjust the DC current. As you do, notice how the resonance pulses move closer together or farther apart. Adjust the DC current, and the phase shifter if necessary, until the pulses occur just when the AC current to the Helmholtz coils is zero. (This is most accurately accomplished if you adjust the vertical position of the channel 2 trace so that the bottom of the resonance pulses are just at the zero level of the channel 1 trace.)

With these adjustments, the oscilloscope traces should appear as in Figure 17. Everything is set for making ESR measurements. Since the current has

been adjusted so that the resonance pulses occur when the AC current to the coils is zero, the current to the Helmholtz coils at resonance is just the DC value indicated by the ammeter. The resonant frequency is equal to the value on the Control Unit display (or the value indicated by your frequency meter multiplied by 1,000).

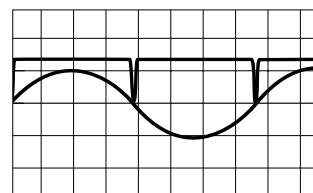


Figure 17 Scope Display

- ⑤ Measure the RF frequency and the DC current. Then vary the current and find the new resonance frequency. Do this for several values of the frequency.
- ⑥ The magnitude of the magnetic field between the Helmholtz coils is directly proportional to the current supplied to the coils. You can determine the magnitude of the field using the following equation (easily derived from the Biot-Savart Law):

$$B = \mu_0 \left(\frac{4}{5} \right)^{\frac{3}{2}} N \frac{I}{r};$$

where:

$$\mu_0 = 1.256 \times 10^{-6} \text{ Vs/Am}$$

N = number of turns in each coil

r = the radius of the Helmholtz coils (which is equal to their separation when they are properly arranged)

I = current passing through the coils.

- ⑦ If you are using the test sample of DPPH, you can now determine the g -factor for the electron using the equation $h\nu = g_S \mu_B B$ (see the *Introduction* for an explanation of the equation).

Appendix

Using the Passive Resonant Circuit

The Passive Resonant Circuit can be used to demonstrate the principles of ESR using a classical (non-quantum) electromagnetic system. Set up the Probe Unit as for a standard ESR measurement, using the medium inductance probe coil. Instead of using the Helmholtz coils and the test sample, position the Passive Resonant Circuit as in Figure A1. To observe resonance absorption, tune the Probe Unit and the Resonant Circuit to the same frequency. This simple demonstration shows how energy from the RF oscillator can be absorbed by a resonant system that is tuned to the same frequency.

►**NOTE:** For a slightly simpler setup than is used for standard ESR measurements, you can use the Current Measuring Lead to connect the Probe Unit to a Ammeter (see Figure A1). This eliminates the need for an oscilloscope. You will be able to see the drop in current in the ammeter when resonance is achieved.

You may also find it useful to show how the current drop in the RF oscillator corresponds to oscillations within the Passive Resonant Circuit. You can do this by connecting a voltmeter (1 volt scale) across the terminals of the Passive Resonant Circuit.

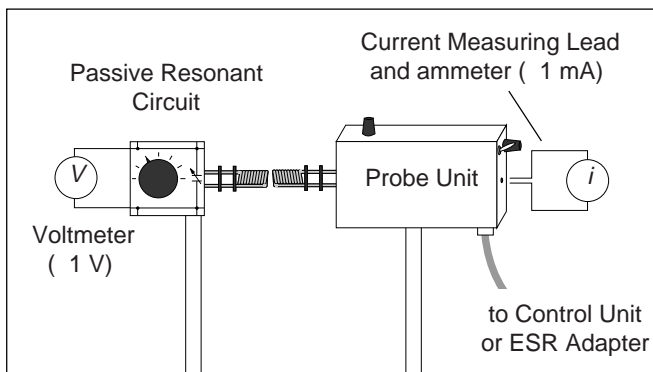


Figure A1 Setup for Using the Passive Resonant Circuit

The Control Unit

Setting the Line Voltage

The Control Unit can be used with a line voltage of 110 V, 130 V, 220 V, or 240 V. To set the Control Unit for the proper line voltage (see Figure A2):

- ① Turn the unit upside down and use a phillips head screwdriver to remove the four screws (deeply recessed) that hold the case together.
- ② Turn the unit right side up again, and remove the top of the case.

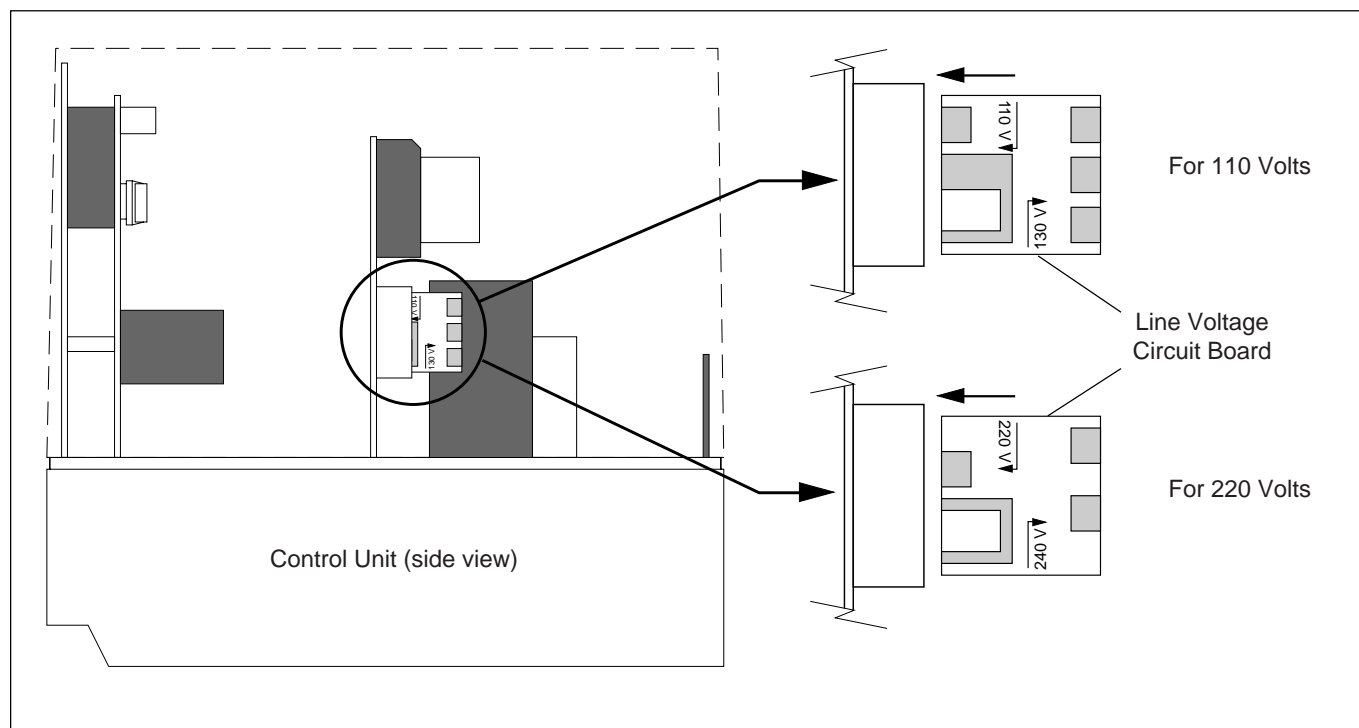


Figure A2 Setting the Line Voltage for the Control Unit

- ③ Pull the line voltage circuit board from its socket and replace it so that the arrow for the proper voltage faces out toward the side of the control unit and points in toward the socket. (Figure A2 shows the proper orientation of the circuit board for 110 V and 220 V.)

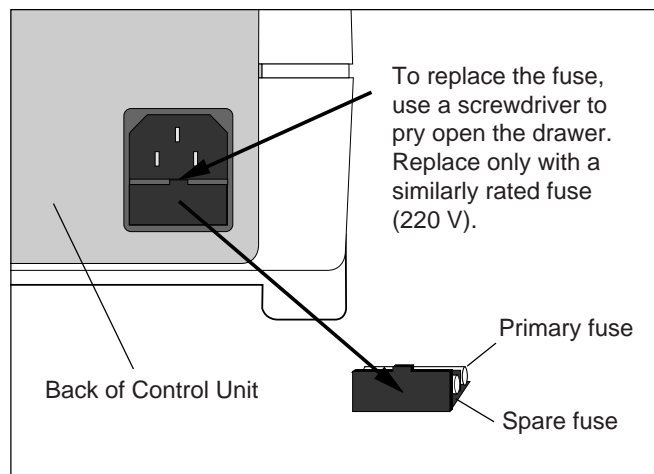
Replacing the Fuse

The fuse for the Control Unit is located in a pullout drawer just underneath the socket for the power cord (see Figure A3). Replace only with a similarly rated fuse:

- 1.6 A, 220 V (slo-blo) for 120 VAC
- 0.8 A, 220 V (slo-blo) for 240 VAC

Building Your Own ESR Adapter

You can purchase an ESR Adapter from PASCO scientific (Model number SE-9637). However, if you prefer to build your own, the connections are shown in Figure A4.



A3 Replacing the Fuse in the Control Unit

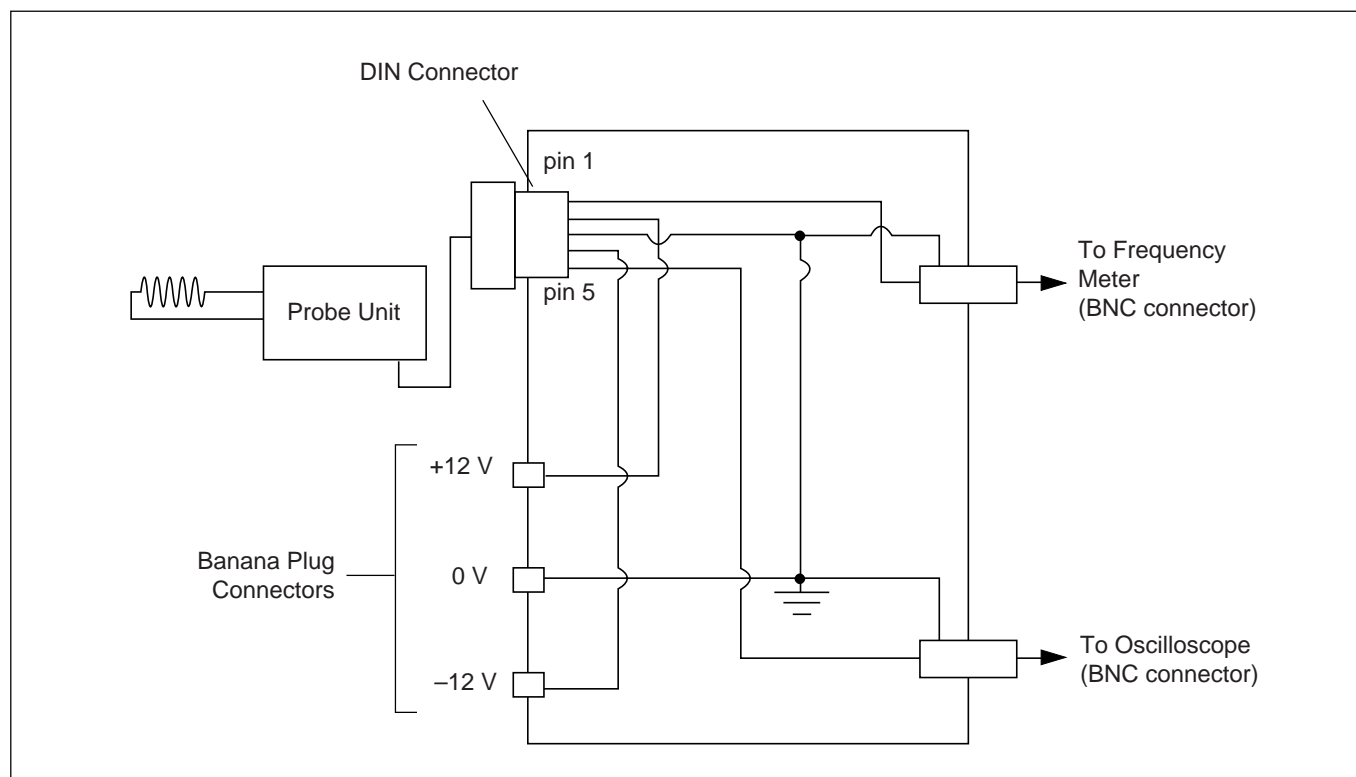


Figure A4 Schematic for the ESR Adapter

Notes:

Technical Support

Feed-Back

If you have any comments about this product or this manual please let us know. If you have any suggestions on alternate experiments or find a problem in the manual please tell us. PASCO appreciates any customer feedback. Your input helps us evaluate and improve our product.

To Reach PASCO

For Technical Support call us at 1-800-772-8700 (toll-free within the U.S.) or (916) 786-3800.

email: techsupp@PASCO.com

Tech support fax: (916) 786-3292

Contacting Technical Support

Before you call the PASCO Technical Support staff it would be helpful to prepare the following information:

- If your problem is with the PASCO apparatus, note:
Title and Model number (usually listed on the label).

Approximate age of apparatus.

A detailed description of the problem/sequence of events. (In case you can't call PASCO right away, you won't lose valuable data.)

If possible, have the apparatus within reach when calling. This makes descriptions of individual parts much easier.

- If your problem relates to the instruction manual, note:
Part number and Revision (listed by month and year on the front cover).

Have the manual at hand to discuss your questions.

