D-2 Electron Spin Resonance

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first experiment: II

Contents

LEARNING GOALS

- Determine the g-factor for the free electron.

- Calculate the energy levels for chromium ions in ruby by solving a 4x4 Hamiltonian.

- Measure the energy levels for chromium ions in ruby.

- Learn to use a lockin amplifier and understand its principle of operation

- Understand microwave waveguides and microwave cavity modes
- Work safely with high magnetic fields

INTRODUCTION

The subject of group theory deals with methods for exploiting symmetry to simplify solutions to physical problems. Techniques that make use of group theory are ubiquitous to most of our understanding of the physical world. For example the fact that the stationary states of all atoms can be characterized by angular momentum, regardless of atomic number and regardless of the complicated many body electron-electron interactions, can be attributed to the isotropic spherical symmetry of the world surrounding the atom. One of the purposes of this experiment is to illustrate the relation between symmetry and angular momentum by studying the effect that breaking the isotropic symmetry has on the ground state of the Cr^{+3} ion. The system chosen for this example is a single crystal of ruby. The ruby crystal is formed from a clear trigonal Al_2O_3 crystal in which Cr^{+3} has replaced a small fraction of the Al^{+3} ions. The red color of ruby is due to the Cr^{+3} ions: the more Cr^{+3}, the darker the ruby.

The electron configuration of the ground state of the isolated Cr^{+3} ion is $1s^2 2s^2 2p^6 3s^2 3p^6 3d^3$ ($^4F_{3/2}$) with only three 3-d electrons outside of closed shells. From Hund's rule the ground state has an orbital angular momentum L=2+1+0=3, or an F state and a spin angular momentum of S=3/2. These couple through the spin-orbit interaction $(\lambda \vec{L} \cdot \vec{S})$ to form different multiplets with total angular momentum that vary from J=3-3/2=3/2 to J=3+3/2=9/2. The multiplet with the lowest energy that forms the ground state of the Cr^{+3} ion is the 4-fold degenerate J=3/2 multiplet with eigenstates that we label |m> with m = -3/2, -1/2, +1/2 and +3/2. This ground state mutiplet is separated from the next higher multiplet ($^4F_{5/2}$) by 244 cm^{-1}.[1] A powerful theorem of group theory gives us a simple relation between the different quantum mechanical matrix elements within any single mutiplet.

The example that illustrates this theorem is actually the one that is needed for this experiment.[2] Suppose the Cr^{+3} ion is placed in an external potential that can be expanded as a power series in distance from the ion

$$V = V_0 \left[2z^2 - x^2 - y^2\right].$$

The theorem says that for all m within the multiplet there is a constant such that

$$\langle m|V|m'\rangle = const V_0 \langle m|2S_z^2 - S_x^2 - S_y^2|m'\rangle$$

where $\vec{S}$ is the angular momentum operator for the multiplet. In view of the fact that the m states are linear combinations of products of spin and orbital momentum wave functions $\vec{S}$ is referred to as an *effective spin*. The power of the theorem is that the matrix elements of the Zeeman interaction can also be written as

$$\langle m| \left[\vec{\mu} \cdot \vec{H}\right] |m'\rangle = const' V_0 \langle m|\vec{S}|m'\rangle \cdot \vec{H}$$

where $const'$ is a second constant.

It is therefore convenient to describe the manner in which the degeneracy of the multiplet is lifted due to these two perturbations in terms of an effective Hamiltonian

$$H_{eff} = -g\mu_B \vec{S} \cdot \vec{H} + \frac{D}{3}\left[2S_z^2 - S_x^2 - S_y^2\right]$$
$$= -g\mu_B \vec{S} \cdot \vec{H} + D\left[S_z^2 - \frac{S(S+1)}{3}\right]$$

where μ_B is the Bohr magneton, and D and g are constants that depend on the details of the electronic wavefunctions.

The g tensor is defined by the magnetic moment

$$\vec{\mu} = \mu_B(\vec{L} + 2\vec{S}) \equiv g\mu_B S_{eff}.$$

Here S_{eff} is the effective spin with multiplicity $2S_{eff} + 1$ such that the low-lying manifold of angular momentum states is spanned. The g tensor is found by evaluating matrix elements of $\vec{\mu}$, using the eigenstates in the crystal field and spin-orbit Hamiltonian.

BACKGROUND

In preparation for the experiment you should solve the model problem illustrated in the following figure. It is designed to mimic the effects that you will observe with the electron spin resonance spectrometer.

A voltage source $E_0 \exp(i\omega t)$ is attached to a simple two-wire transmission line with internal impedance Z_0. It generates a voltage that propagates to the right. At the position of MM` there is a device which has the special property that the wave coming from the left is transmitted to the right without any change. This wave will strike some complex impedance $Z(\omega)$ at the end of the line and a wave will be reflected back towards MM`. Assume that waves coming from the right are reflected downward as shown. Don’t worry about the different position of MM` on the two wires.

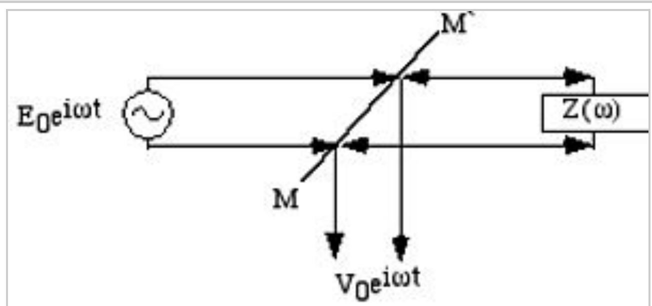

Figure 1. Transmission line model.

Calculate and plot the ratio of $|\frac{V_0}{E_0}|^2$ as a function of frequency if $Z(\omega)$ is a parallel combination of R, L and C and think about how different values of R,L,C and Z_0 affect the results. Consider cases when $R = Z_0$ and when $R \neq Z_0$. This resonant circuit models the microwave cavity that you will use in the laboratory.

The effects of the electron spin resonance can be modeled by assuming that $R \rightarrow Z_0 + r$ where $r \ll Z_0$. When ω is tuned to resonance r is positive, otherwise it is zero. The signal you will measure will be proportional to $Signal(r) = |\frac{V_0(r) - V_0(r=0)}{E_0}|^2$. Plot $Signal(r) vs. \omega$ when $R = Z_0$ and when $R \neq Z_0$ for $Z_0 \gg |Z_0 - R| \gg r$.

APPARATUS

Equipment List

Microwave and magnetic field apparatus

- Gunn diode oscillator (Epsilon Lambda ELM X6/M)
- Gunn Diode power supply (ELD101)
- Sawtooth modulation box
- X-band microwave components
- Rectangular TE_{102} cavity
- Frequency absorption meter (Hewlett Packard X532B)
- Electromagnet (Magnion L-75B)
- Magnet power supply (Magnion HSR-1050B)
- Modulation coils with power supply
- Gaussmeter (Bell 615)
- Preamplifier
- Oscilloscopes (Tektronix 2245A and TDS3012B)

Lockin amplifier and field sweep apparatus

- Lockin amplifier(Stanford Research Systems SR510)
- Function generator with low impedance output (Pasco PI-8127)
- Dual filter (Ithaco 4302)
- Function generator (Wavetek 164)
- Digital multimeter (Keithley 169)
- Computer (Dell Optiplex 980)
- Multifunction data acquisition board (National Instruments PCI-6023e)
- Breakout box for PCI card (National Instruments NI-BNC 2110)

Microwave Spectrometer

Figure 2 shows a schematic illustration of a spectrometer that closely resembles the one in the laboratory.

- **M** The microwave source is a Gunn diode, a solid-state device operated in its negative impedance regime. The frequency of the oscillator may be controlled by mechanically tuning the size of the cavity and by varying the diode bias voltage. These are used respectively as a coarse frequency adjustment and as a way to modulate the oscillator frequency. When the bias voltage is modulated, the detected power can be viewed on an oscilloscope with the time base corresponding to microwave frequency. The microwave frequency is tunable from 8.5 to 9.5 GHz.

- **I** Isolator. Power is only transmitted in the direction of the arrow. Waves reflected back towards the Gunn diode are attenuated.

- **F** The HP frequency meter is a cavity, which absorbs microwaves in a narrow band. If the band absorbed by the meter is within the frequency range generated by the Gunn oscillator, a dip will appear in the oscilloscope display of microwave power vs. frequency. Changing its dimensions changes the resonant frequency of the cavity; this is accomplished by rotating the black cap of the meter. The intersection of the scale above the red line with the thin vertical black line gives the absorption frequency in GHz.

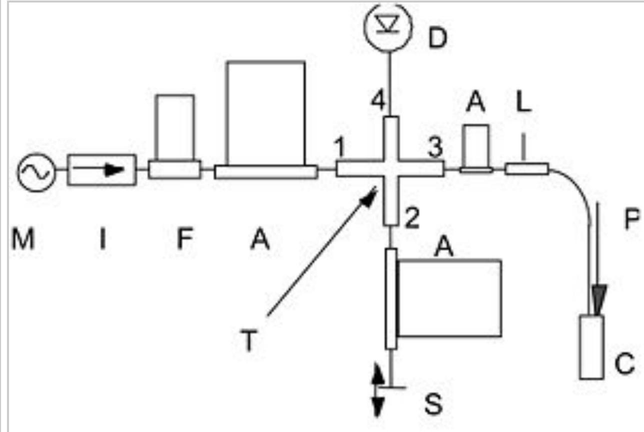

Figure 2. Microwave spectrometer schematic.

- **A** An Attenuator allows control of the power transmitted. Two of the attenuators have calibrated dials.

- **T** The magic tee transmits power according to the table:

Input Arm	Power Out of Arm	No Power Transmitted
1	2 & 3	4
2	1 & 4	3
3	1 & 4	2
4	2 & 3	1

You should look into the interior of the magic tee. You can use symmetry considerations to understand why it behaves the way it does.

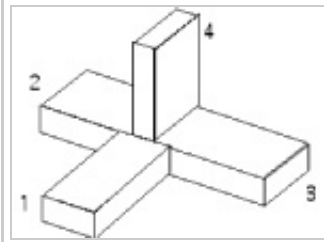

Figure 3. Magic Tee.

- **D** Crystal detector (diode 1N23E) rectifies a microwave signal. Output voltage is proportional to microwave power. The detector crystals are fragile, rare and expensive. When installing a new crystal, avoid electrostatic discharge through it.

- **L** Slotted Line. You will probably not use this. It allows measurement of the average electric field at different points along the length of the waveguide.

- **C** Rectangular sample cavity.

- **P** Adjusts coupling between the cavity (C) and the waveguide. Its effect is similar to varying the value of R in the model calculation.

- **S** Position of the short circuit termination of waveguide can be varied

Homodyne *vs* Heterodyne

Power incident on port 1 of the magic-T is divided between port 3 (to the sample) and port 2 (we call this the reference arm). The electric field incident on the cavity is denoted as E_0. For the simplest usage the attenuator A in the reference arm is adjusted to completely absorb all the power in this arm. In this case the reference arm is not used. Power that is returned to the magic-T after being reflected from the sample (port 3) is divided between port 1 (the Gunn diode) and port 4 (the crystal detector). The combination of the attenuator (A) and isolator (I) in the arm connected to port 1 should be sufficient to prevent any of the power coming back from the sample reaching the Gunn diode. The measured signal arises from the power that is deflected to port 4.

In general the detector rectifies the microwave electric field E to produce a dc voltage $V \sim |E|^2$. On the other hand since the reflected microwave field is modulated at some audio frequency (60 Hz or other) the reflected field can be expressed as

$$E = \chi_c E_0 + E_1(t)$$

where χ_c is the reflection coefficient from the cavity. For critical coupling $\chi_c = 0$; however, in normal usage there is a small non-zero value for χ_c. If the frequency of the Gunn diode is not modulated E_0 is constant and a small slowly varying modulated reflection can be expressed as

$$|E_1(t) \approx [\chi'(t) + i\chi''(t)]E_0|$$

where $\chi'(t) + i\chi''(t)$ is the time dependent variation in the cavity reflection coefficient due to the modulation of the sample. Note that E_0 is a complex number (i.e. $E_0 \sim exp[(i\omega t + \phi)]$) where ω is the microwave frequency.

If we assume that $|E_1(t)| \ll |E_0|$ is small and that χ_c is real then $V \sim \chi_c^2|E_0|^2 + 2\chi_c\chi'(t)|E_0|^2$. The amplifier that follows the detector can discriminate against the time average voltage such that the measured signal is just the time dependent portion $v(t) \sim 2\chi_c\chi'(t)|E_0|^2$. In this method, which is known as *homodyne detection* one only observes one component of the complex reflection coefficient.

The *heterodyne detection* method, which uses port 2 of the magic T can be used to select either $\chi'(t)$ or $\chi''(t)$. In this method the cavity coupling is set to critical coupling and $\chi_c = 0$. On the other hand, the attenuator in the reference arm (port 2) is now relaxed such that some fraction of the power that was originally diverted to port 2 is now reflected back from the reflecting termination of this arm. A fraction of this power from port 2 is directed towards the detector such the total field at the detector can be written as

$$E(t) = \chi_r E_0 + (\chi'(t) + i\chi''(t))E_0$$

where the first term comes from port 2 and the second comes from port 3. In general one can write $\chi_r = |\chi_r|exp(i\phi_r)$ where $|\chi_r|$ is determined by the attenuator and ϕ_r is determined by the position of the movable termination S at the end of the reference arm. Assuming that $|\chi_r| \gg |\chi'(t) + i\chi''(t)|$ case the time dependent measured signal can be written as

$$v(t) \sim 2Re[(\chi'(t) + i\chi''(t))|\chi_r|exp(-i\phi_r)].$$

In this *heterodyne method* one can chose ϕ_r such that $v(t)$ is proportional to either $\chi'(t)$ or $\chi''(t)$ or any combination of the two.

Electronics

Gunn diode power supply

The Gunn diode power supply (ELD101) provides 470 mA forward bias current at 10.0 V to the Gunn diode. These parameters are specified by the plug-in module on the front of the supply. Do not unplug this while the unit is on; doing so would destroy the diode. The ELD101 has an external modulation input which adds a small modulation to this dc level. The modulated current produces a frequency modulation of the microwaves. This is useful while searching for the cavity resonance.

Sawtooth frequency modulation

The sawtooth modulation signal is generated in a home made circuit by a current source charging a capacitor. A 555 oscillator resets the capacitor voltage to zero when it reaches a threshold. A schematic diagram is available in the Bench Notes. The sawtooth output (Figure 4-1) amplitude is adjustable (Figure 4-2) from approximately 0 to 8 V. Frequency is fixed at 190 Hz. A dc level (Figure 4-3) varying from 0 to 3 V is added to the sawtooth. DC ONLY (Figure 4-4) eliminates the sawtooth. A trigger pulse (Figure 4-5) (compliment of TTL) has the same frequency as the sawtooth. The power input requires +15 V relative to common, and a ground connection for shielding.

Figure 4. Sawtooth modulation box panel.


Figure 5. Sawtooth modulation box circuit.

Microwave Detection

The 1N23E detector diode is mounted in an arm of the Magic Tee as shown in Figure 2. It produces a current proportional to microwave power. The simplest detection method is to connect the diode directly to the oscilloscope. The scope input impedance of 1 MΩ converts the current into a voltage on the order of tens or hundreds of millivolts. Optionally, a PAR 113 amplifier can be used to amplify and filter the signal. Use a 1 MΩ load on the PAR 113 input. See Figure 5a.

While searching for the cavity resonance, either use the oscilloscope in time base mode, triggering from the sawtooth box, or XY mode, driving the horizontal axis with the sawtooth signal itself. Should the vertical axis be dc or ac coupled?

For a certain position of the teflon coupler (P for "plunger" in Figure 2), the power reflected from the cavity can be made close to zero. After finding this signature of the cavity resonance, gradually reduce the microwave modulation amplitude while staying centered on the cavity resonance. Switch to DC ONLY mode and adjust the dc level carefully to drive the cavity at resonance. Adjust the teflon coupler for slightly non-critical coupling.

Next set up for field modulation. The small coils inside the magnet pole pieces connect to a 60 Hz modulation box. The box has a variac output for the coils and a separate section for driving the horizontal axis of the oscilloscope. This output has variable amplitude and phase.

For the ruby sample, some angle/field combinations have weak absorption. A phase sensitive detector can solve the problem of locating these signals. The lockin amplifier is sensitive enough to find signals buried in noise which is orders of magnitude larger. See Appendix 9. While using the lockin one prefers to choose a modulation frequency other than 60 Hz since line noise is ubiquitous. The Pasco PI-8127 function generator has variable frequency. Choose a prime number for your modulation frequency, or at least avoid multiples and submultiples of 60 Hz. The Pasco output has a low impedance, so it can drive the modulation coils directly. Currents on the order of 100 mA are adequate to see a signal. Choose the modulation amplitude carefully because modulation which is too large causes decreased resolution. Figure 5b shows the lockin setup.

As a strategy for collecting data, one might choose to follow a transition after finding it at 0 degrees by making small changes in angle and corresponding small changes in field. This works for oscilloscope and for lockin detection.

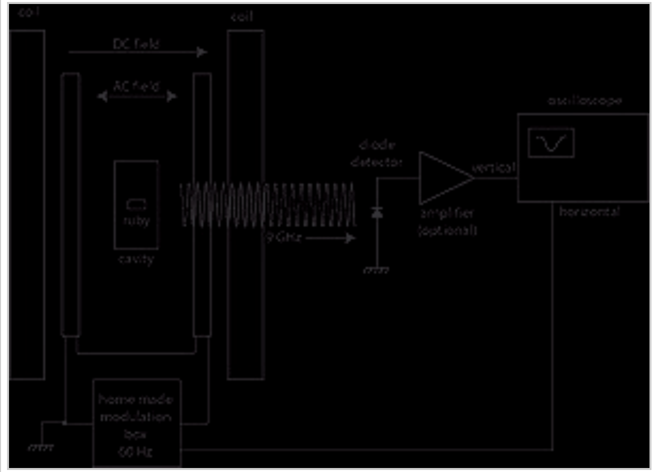

Figure 5a. Apparatus schematic -- oscilloscope.

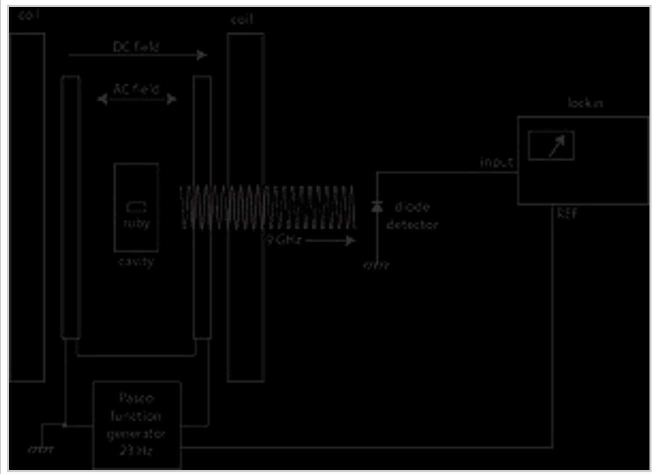

Figure 5b. Apparatus schematic -- lockin.

Magnet Power Supply and Gaussmeter

The Magnion HSR-1050B power supply is capable of delivering 50 Amps at 8 Volts to the magnet for a maximum field of approximately 8 kGauss. Water cooling removes heat from the coils and an interlock prevents the power supply from operating unless the water is flowing.

A bootstrap procedure is needed to energize the magnet. Begin by turning the Mode Selector to Current Control. Check the three dials also labelled Current Control on the right side of the upper panel. The last person to use the supply should have left them all in the fully CCW position. Next hold the inconspicuous red button on the lower panel labelled Water Supply. Then push REV while continuing to hold Water Supply. After a few seconds, look for exhaust water at the sink on the west wall near the fume hood and release Water Supply when a steady stream of water is visible.

The red button temporarily shorts out the water interlock. This is the bootstrap. Otherwise, the interlock, which has a time constant of a few seconds, would shut down the power supply immediately. The power supply and the solenoid water valve both get power when the REV button is pressed.

Now set the three Current Control dials (coarse, medium and fine) for the desired field value as measured by the Bell 615 Gaussmeter. Make sure that the Gaussmeter probe is properly mounted and perpendicular to the field lines.

To shut down, turn the three Current Control dials fully CCW, press the red OFF button next to REV, and return the Mode Selector to OFF.

Automated data acquisition

Alternately, one may sweep the DC magnet current automatically and record lockin output as a function of magnetic field with a computer.

Magnet Sweep

The Magnion HSR-1050B power supply is equipped with an Ext. Sweep Input. The input impedance is 20 kΩ and a fully floating input voltage must be used. The sensitivity is 20 A/V, so a sweep from zero to 2.5 V to zero sweeps the entire 50 Amp range of the supply. The Wavetek 164 function generator can satisfy these requirements. It also has variable phase and manual trigger, so single sweeps can be set up to start and finish at zero. We wish to change the magnet current **slowly** to avoid dangerous back EMF. Do not use a sawtooth function. Triangle is a good choice. **Slowly** means a minimum of 100 seconds sweep time.

Magnetic Field detection

The Bell 615 Gaussmeter has an analog output voltage which is proportional to magnetic field. Filter out the AC field component and amplify as needed with the Ithaco 4302. Calibrate this voltage against the Gaussmeter's digital readout, and record it with the NI card as described below.

National Instruments PCI card and LabVIEW program

We wish to record the lockin output as a function of magnetic field. To this end, the Dell computer houses a National Instruments PCI-6023 analog to digital converter card. A connector block, NI-BNC-2110 adapts BNC connectors to the card. A LabVIEW program, xyt_mx_jul10.vi talks to the card and records analog inputs number zero and one (AI0 and AI1) and plots them. More information about this "virtual instrument" is available at the superconductivity wiki.

EXPERIMENTAL PROCEDURE

Begin by optimizing the operation of the spectrometer (bullets 1 - 4).

- 1) Find the cavity resonance and experimentally determine the optimum bridge conditions (balanced or unbalanced, dispersion or absorption mode).
- 2) Find the DPPH signal.
- 3) Determine in what microwave power range the spectrometer must be operated in order to avoid saturation.
- 4) Identify the major limitations on the sensitivity of the spectrometer.
- 5) Observe all possible resonances for a ruby crystal oriented with its C-axis parallel to the external field; measure the field at which each resonance occurs. From this data and the ruby Hamiltonian, identify the transitions observed, calculate g and D and compare the results with published values. Study the angular dependence of the ruby resonance.

NOTES

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BENCH NOTES

- Appendix 12: Magnetic Precession in Static and Oscillating Magnetic Fields (http://www.fas.harvard.edu/~phys191r/pdf/ap12.pdf)

Microwave and magnetic field apparatus

- HP X532B Microwave Frequency Meter (http://www.fas.harvard.edu/~phys191r/Bench_Notes/D2/Frequencymeter.pdf)
- Bell 615 Gaussmeter (http://www.fas.harvard.edu/~phys191r/Bench_Notes/bell615.pdf)
- Epsilon Lambda ELMGX6 Gunn Oscillator (http://www.fas.harvard.edu/~phys191r/Bench_Notes/D2/Gunn.pdf)
- Model L-75B Magnet (http://www.fas.harvard.edu/~phys191r/Bench_Notes/D2/Magnet.pdf)
- PAR 113 Preamplifier (http://www.fas.harvard.edu/~phys191r/Bench_Notes/par113.pdf)
- Sawtooth Box (http://www.fas.harvard.edu/~phys191r/Bench_Notes/D2/Sawtooth.pdf)
- Power Supply for Sawtooth Box (http://www.fas.harvard.edu/~phys191r/Bench_Notes/D2/ESR_15V.pdf)

Lockin and field sweep apparatus

- Stanford Research Systems SR510 Lockin Amplifier (http://www.fas.harvard.edu/~phys191r/Bench_Notes/SR510.pdf)
- Pasco PI8127 Function Generator (http://www.fas.harvard.edu/~phys191r/Bench_Notes/D2/pasco_pi8127.pdf)
- National Instruments BNC2110 Breakout Box (http://www.fas.harvard.edu/~phys191r/Bench_Notes/D2/ni_bnc2110.pdf)
- National Instruments PCI-6023e Multifuction DAQ card (http://www.fas.harvard.edu/~phys191r/Bench_Notes/A10/ni6023e.pdf)
- Ithaco 4302 Dual Filter (http://www.fas.harvard.edu/~phys191r/Bench_Notes/A7/ithaco4302.pdf)
- Keithley 169 Digital Multimeter (http://www.fas.harvard.edu/~phys191r/Bench_Notes/B5/Keithley169.pdf)
- Wavetek 164 Function Generator (http://www.fas.harvard.edu/~phys191r/Bench_Notes/D2/Wavetek164.pdf)