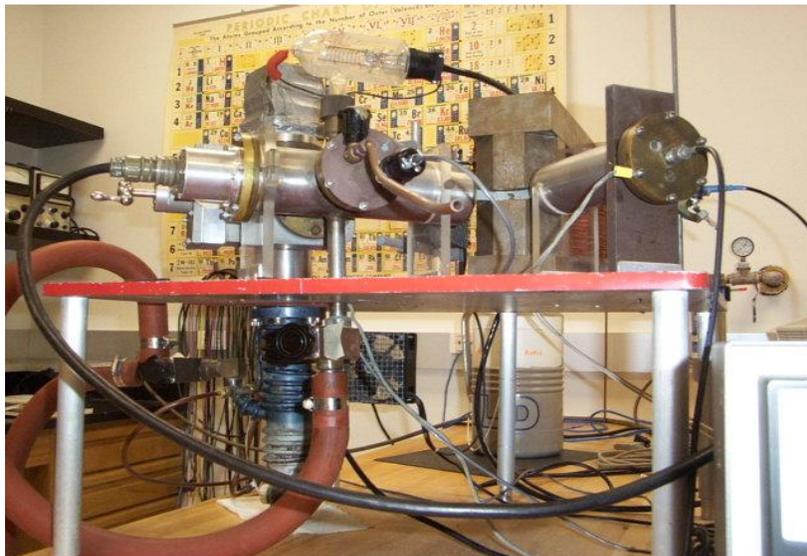## *MASS SPECTROMETER Rev. 2010*

Objectives:

1.) Resolve the mass separation of isotopes for the metal ions from any of a number of single isotope or multi-isotope salts such as Sodium Chloride or Potassium Chloride, respectively.

2.) Determine the resolving power (delta m/m) of the mass spectrometer.

3.) Measure the charge to mass ratio for the sample under test.

4.) Verify the relationship between magnetic field strength and accelerating voltage.

5.) Try to determine relative abundance of the isotopes.

Procedure:

The physics for this experiment, involving the Lorentz force, is very straight forward. The equipment however is somewhat complex, so here are some tips that should help insure that you will have good luck in obtaining good data.

Make sure you are familiar with, and understand the operation of all of the vacuum equipment (gauges, valves, pumps), and electronic equipment. The BPS manual in the lab is a good reference on vacuum components. Be able to locate every piece of equipment shown on the diagrams, and explain its function.

Inspect the ion gun and collector for alignment, and make sure they are very clean. Clean the apparatus with warm soapy water, and rinse first with distilled water and then with alcohol. Use the feeler gauge to measure the slit width and adjust the opening to <=.02 inches. Make sure the opening is centered, and that the filament is visible through the slit. The geometry of the ion gun optics largely determines the resolution, repeatability, and accuracy of the spectrometer. Resolving the potassium peaks is difficult, so spend a little time on alignment and you will be rewarded with improved resolution and better data. The geometry of the filament, lens, and slits affects the electrostatic focussing, and controls the collimation of your ion beam. ***Make sure that neither the filament nor the focussing slits are shorted to ground or to each other.***

Inspect the filament. Make sure it makes good electrical contact by taking a resistance measurement at the lead-in wires. BAD ELECTRICAL CONTACT TO THE FILAMENT IS THE SINGLE MOST COMMON PROBLEM IN LOW ION EMISSION. Study the wiring diagram.   There are two potentials applied to the filament.   The "Filament Supply" provides current to heat the filament. The high voltage supply provides an accelerating potential for the ions, and holds the filament at high voltage with respect to the slits (which are grounded). Ion emission is measured by placing an ammeter in series with the high voltage supply and the filament. Banana jacks are provided on the rack panel for this connection. The ammeter MUST be connected to these jacks in order to complete the circuit and provide high voltage to the filament.

The filament is made from a 2" x 3/8" piece of 1 mil tantalum foil, folded in half lengthwise to make a "V" boat. Scissors are usually all that is required to cut the foil. Tantalum becomes brittle after being heated, so don't try to bend it after it has been used. If the filament has any cracks it should be replaced. Consider that an ideal ion source would be infinitely small and that an extended source will tent to `blur' the focus of the ion beam. Students have reported good results by cutting a *SMALL* "v" notch near the center of the filament. This allows the filament to get the hottest right at the center. Please note the emphasis on the word '*SMALL*'. The inner surfaces of the filament holder assembly must be relatively free of oxidation to assure a good electrical connection.

The Sypris Gaussmeter comprises a hall probe and metering electronics.   Read the Sypris manual. The magnetic field is the single most important measurement you will make in this experiment. Therefore it is imperative that you verify the accuracy of this instrument and its computer interface. The hall probe is a delicate semiconductor device that does not respond well to abuse. The sensitive region is about 2 mm from the end of the probe. The plane of the probe should be oriented exactly perpendicular to the field (parallel to the pole faces). The probe is also responsive to field polarity. BEFORE BEGINNING the experiment you should CHECK CALIBRATION AND ZERO adjustments. Once the meter is calibrated and zeroed, run the experiment and compare the computer reading with the meter reading. If you discover a discrepancy, read the gaussmeter with a field applied, and apply a correction factor to your computer data. The meter should be set to either the 3 KGauss scale or the 30 KGauss scale depending on the range of your sweep. The voltage the meter outputs to the computer depends on the scale setting. The gaussmeter provides 3 volts for a full scale reading, and its output is connected differentially to the *National Instruments DAQ* inputs Ch2 and Ch10. Data from these inputs are displayed as the x-axis of the LabView virtual instrument program Mass'10.vi.

On the collector side of the mass spectrometer, a Faraday cup is used to collect the ions

which pass through the detector slit. The Keithley Electrometer measures the tiny currents produced by the ions collected by the Faraday cup. The electrometer produces a 0 to 3 volts output which is proprotional to the deflection of the meter on its front panel. The output from the electrometer is read differentially at DAQ inputs Ch3 and Ch11. This signal is displayed on the y-axis of the LabView virtual instrument program Masspec'10.vi.

The mass spec uses O-ring seals and just as did the Space Shuttle Challenger. Obviously these can leak. So be careful not to scratch the flanges or O-ring grooves, and do not force or bend the ceramic electrical feed-throughs. Keep the seals very clean, and lubricate the O-rings <u>sparingly</u> with vacuum grease. Keep the chamber clean and dry. Use alcohol to remove any residue from the chamber walls after each data run. Avoid dropping salts down into the roughing valve as this will damage the valve seat and seal. Unlike fire hydrant valves, it is not necessary to force vacuum valves closed. Vacuum valves seat against rubber seals that can be damaged by over-compression. Usually the pressure differential does a good job in holding the valves closed, so they need only be finger tight. Also: do not over tighten the flange screws as this will cause the flange to deform and not make a good seal, but make sure the o-ring is adequately compressed.

Other Precautions:

Some of the biggest mistakes you can make involve operating certain vacuum components at pressures above 100 microns. The diffusion pump and the ionization gauge for example, must never be operated in the presence of oxygen. At pressures above 100 microns hot diffusion pump oil and hot tungsten filaments will oxidize (burn). Always follow the vacuum procedure outlined below, and anticipate the consequences of each step you take. Make sure the diffusion pump has been evacuated before turning it on.

Chilled water must be supplied to the upper cooling coils of the diffusion pump. However do not overcool the heater. The heater must be allowed to get hot enough to vaporize the pump oil inside. Use the chilled water supply valve to adjust the flow of chilled water. The fans must also be on. Liquid nitrogen must be in the cold trap to prevent vaporized pump oil from backstreaming into the chamber thus depleting the pump of oil.
The chamber pressure must be below 100 microns before the HV valve can be opened to the diffusion pump. Use the ionization gauge only after the chamber pressure is below 1 micron.

Sample Preparation:

Make a saturated solution in distilled water by first grinding your salt sample into a fine powder with the mortar and pestle. Adjust the consistency so that it is thin enough to be picked up with an eye dropper. Use the eye dropper to deposit the sample into the filament/boat. Run 2-3 amps of current through the filament in order to warm it just enough to help dry the sample. The sample should be dry when you put it into the chamber, or the pump-down time will be extended considerably.

For your first run use NaCl as your sample. Sodium has only one isotope and therefore makes a good calibration source. Assume the accepted mass of sodium, and from your measurements, calculate the radius of curvature. Use this radius in all subsequent data runs. Next try a potassium or lithium salt. Always use water soluble salts!

Pump Down Procedure:

1) Make sure the vacuum system is sealed. Do not over tighten the allen screws securing the flanges, or the valves.   Make sure the vent valve and the HV (high vacuum) valve are closed.

2) Turn on the roughing pump and open the roughing valve and the foreline valve. Turn on the vacuum gauge controller.

3) When the foreline and chamber pressures have fallen below 60-80 microns;

a) Turn on the fans
b) Turn on the chilled water to the upper cooling coil of the diffusion pump - return line first, then supply line.
c) Turn on the diffusion pump.
d) WAIT about 20 minutes for the diffusion pump to warm up and outgas before continuing to the next step.
e) Pour liquid nitrogen into the cold trap
4) When the diffusion pump has begun operating, you will hear a slight pinging sound from inside the pump. Then;
a) Close the roughing valve.
b) Slowly open the HV valve.
c) Turn on the ionization gauge (read the manual for instructions on it's use).

5) Be prepared to allow an hour for the sample to outgas and chamber to pump down. If

the sample is wet or the chamber is dirty, the pump down time will increase. When the pressure reaches 5 x 10-5 or lower, you may begin taking data. The mean free path of the ions depends on the pressure. The higher the pressure, the fewer ions make it to the detector.

6) Next;

a) Set the accelerating voltage (250-425 volts).

b) Turn on the filament current (approx. 9 amps depending on the size and resistance of your filament). Adjust the filament current to produce a few microamps of ion emission current. There may also be a leakage current flowing from filament to ground through the ion gun. The amount of leakage current will vary with high voltage but not with filament current/temperature. You will see a slight rise in pressure when the sample begins to evaporate and fill the chamber (remember the thing about mean free path, above).

c) Turn on the magnet power supply and check the setting of the Magnet Supply switch on the rack panel. It is recommended that you first sweep manually so as to; locate the ion current peaks, optimize the lens voltage (approx. 1/2 accelerating potential), determine electrometer setting (approx. 1 x 10-9 amps), and filament current. Set the switch on the rack panel to manual in order to manually control the magnet using the controls on the magnet power supply.

NOTE: the magnetic field will affect the Ion Gauge measurement, so take pressure readings with the magnet turned off.

7) To TAKE DATA, set the Magnet Supply switch to Ext. Sweep, and run the LabView Mass'10.vi computer program. This program will control the magnet supply, measure magnetic field, measure ion current using the electrometer, and save data files in an Excel spreadsheet format.

To Bring The Chamber Up To Atmosphere:

1) Turn off the ionization gauge, high voltage supply, filament supply, and magnet supply.

2) Close the HV valve. You may leave the diffusion pump on if you intend to pump down again the same day. Always leave the foreline valve open when operating the diffusion pump. Make sure the roughing valve is closed.

3) Open the vent valve. Do this slowly to prevent the rapid expansion of gas and resulting condensation of water in the chamber.

To Resume Pumping After Changing The Sample:

1) Seal the chamber, close the vent valve.

2) Close the foreline valve and Open the roughing valve. Monitor the chamber pressure and WAIT until it is below 80 microns before proceeding. Do not leave the foreline valve closed for extended lengths of time while the chamber is roughing. During this time you should occasionally close the roughing valve, and open the foreline valve in order to "back up" the diffusion pump and prevent an overpressure from developing.

3) Close the roughing valve after the pressure levels off (below 50 to 100 microns). If the chamber pressure then rises rapidly, re-open the roughing valve and continue pumping until the outgassing rate stabilizes--and check for leaks.

4) Open the foreline valve. Make sure the foreline pressure is below 50-100 microns before proceeding.

5) Slowly open the HV valve. Turn on the ionization gauge and high voltage supply. Once pressure has returned to 1×10^{-5} Torr, you may resume experiment.

UCI MASS SPECTROMETER CONTROL PROGRAM

MASS'10.vi

This UCI *LabView* virtual instrument program performs magnetic field control, data acquisition and data recording functions by controlling the various inputs and outputs of the *National Instruments* data acquisition (DAQ) system connected to the computer. The digital to analog converter (DAC) in the DAQ module produces a 0-100 mv ramp signal required to drive the programming input of the magnet power supply. The programming input on the magnet power supply generates approximately 1 amp of output current for each 50 mV of input voltage (2 amps for 100 mV, etc.). To take advantage of the full resolution and 10 volt range of the digital to analog convertor we use a 100 to 1 voltage divider on the External Sweep input. e.g. 10 volts from the D to A convertor will produce 2 amps of power supply current. The 16 bit resolution equates to approximately 1 mV per LSB, so with 100 to 1 attenuation the minimum step size at the programming input of the power supply will be 10 uV.
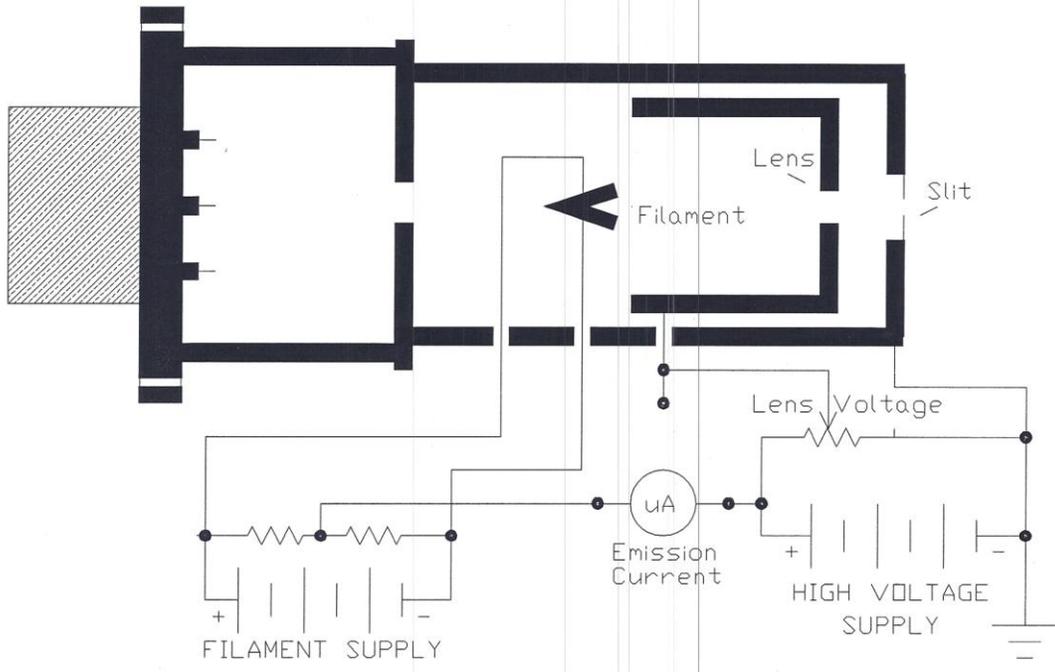
The *LabView* program allows you to set the minimum and maximum field sweep limits in Gauss. It is advisable to set the sweep over a relatively narrow range in order to improve resolution near the mass peak and minimize the sweep time and file size. The program also allows you to adjust the number of steps per sweep. Since each step is a unique DC voltage output by the DAC, you can affect control of the size of incremental change in magnetic field for a give sweep range by increasing or decreasing the number of steps per sweep. Increasing the number of steps for example makes each increment of change in field smaller, and improves field resolution.

Sweep times on the order of a few 10's of seconds, over a narrow range of magnetic fields usually provide the best results. Remember that the magnet has an enormous inductance and it is not possible to rapidly change the current through the magnet coil. In order to limit the rate at which we ask the current to change, adjustable time delays have been included in the program code. This delay also allows time for the magnetic field to settle before a measurement is made by the ADC's.
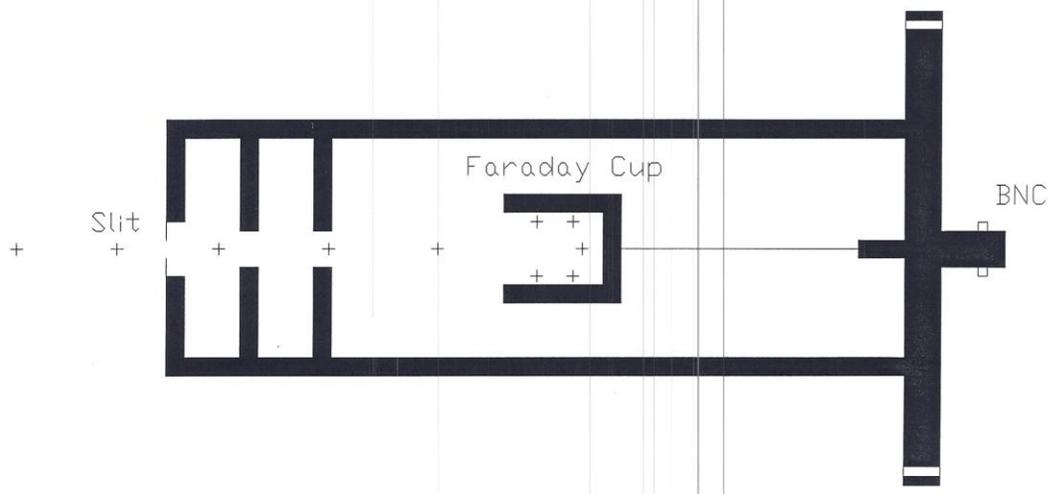
As the magnetic field is swept by the D/A converter, the analog to digital (A/D) converters acquire two channels of data. The channel 2 and 10 differential inputs, with 3 volt full scale sensitivity, record the output of the gaussmeter. The gaussmeter has a zero to 3 volt ratiometric output. This means the voltage the A/D sees is proportional to the percent of full scale of the gaussmeter reading. The program converts the measurement in volts to kilogauss depending on the gaussmeter range parameter you provide.

The channel 3 and 11differential inputs, also with 3 volt full-scale sensitivity, record the output of the electrometer. This voltage is also ratiometric with scale reading, however only the relative ion current is of importance. For best results, set the sensitivity to give as high a reading as possible without going beyond full scale.   Do not take data until you have 'tuned-up' the mass spectrometer by sweeping manually. The electrometer should be registering peaks on each sweep. In order to sweep the magnetic field manually, use the voltage controls on the magnet power supply and locate the setting that produces a peak on the electrometer. Adjust the electrometer to the appropriate range, and adjust the lens voltage (on the rack panel) to maximize the ion current reading. Take note of the field at which your peak appears, and set up your sweep so that it encompasses the peak.

ION GUN

Lens

Slit

Filament

Lens Voltage

uA

Emission
Current

HIGH VOLTAGE
SUPPLY

+

−

FILAMENT SUPPLY

+

−

COLLECTOR

Faraday Cup

BNC

Slit

+

+

+

+

+

+

+ +

+ +

+ +
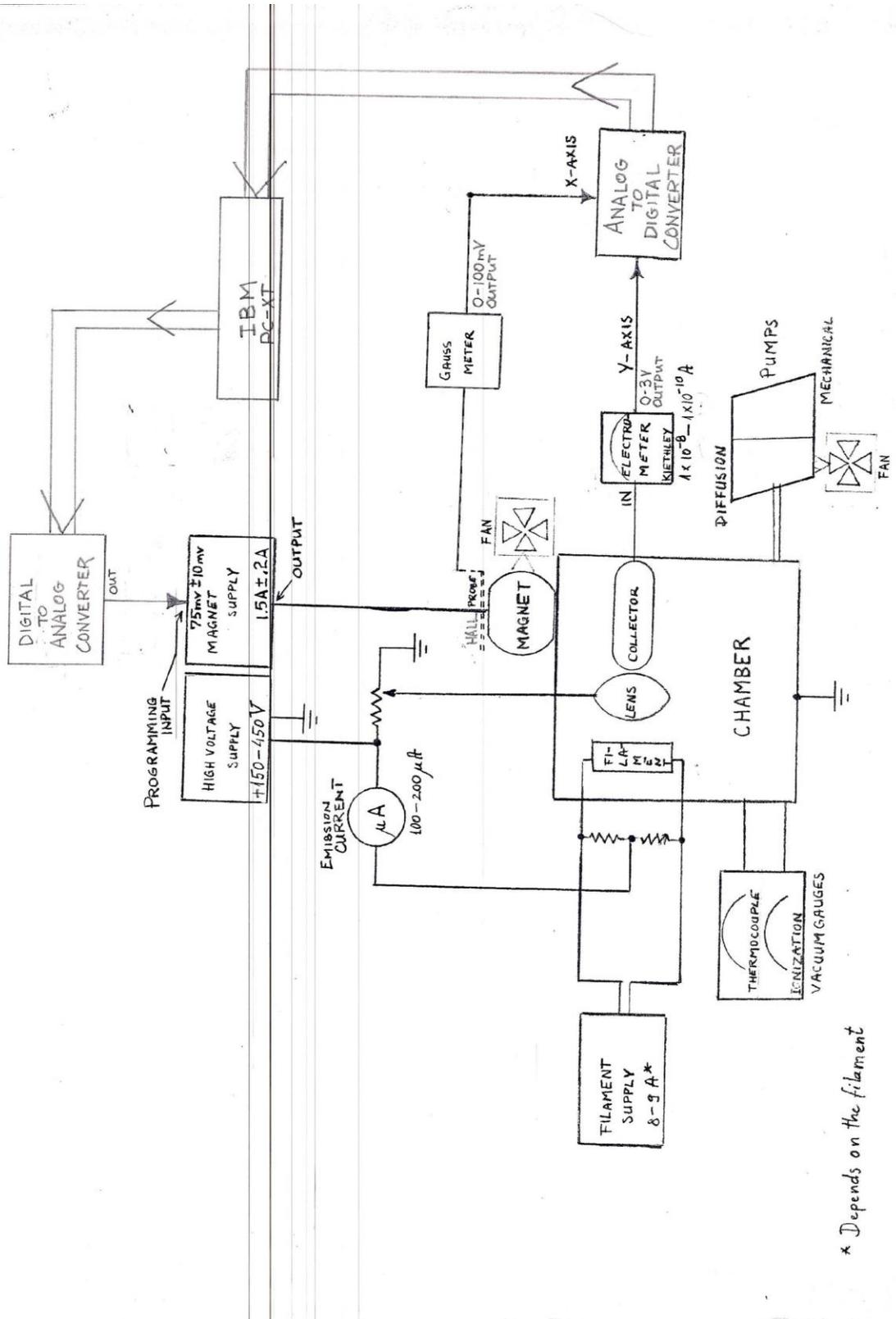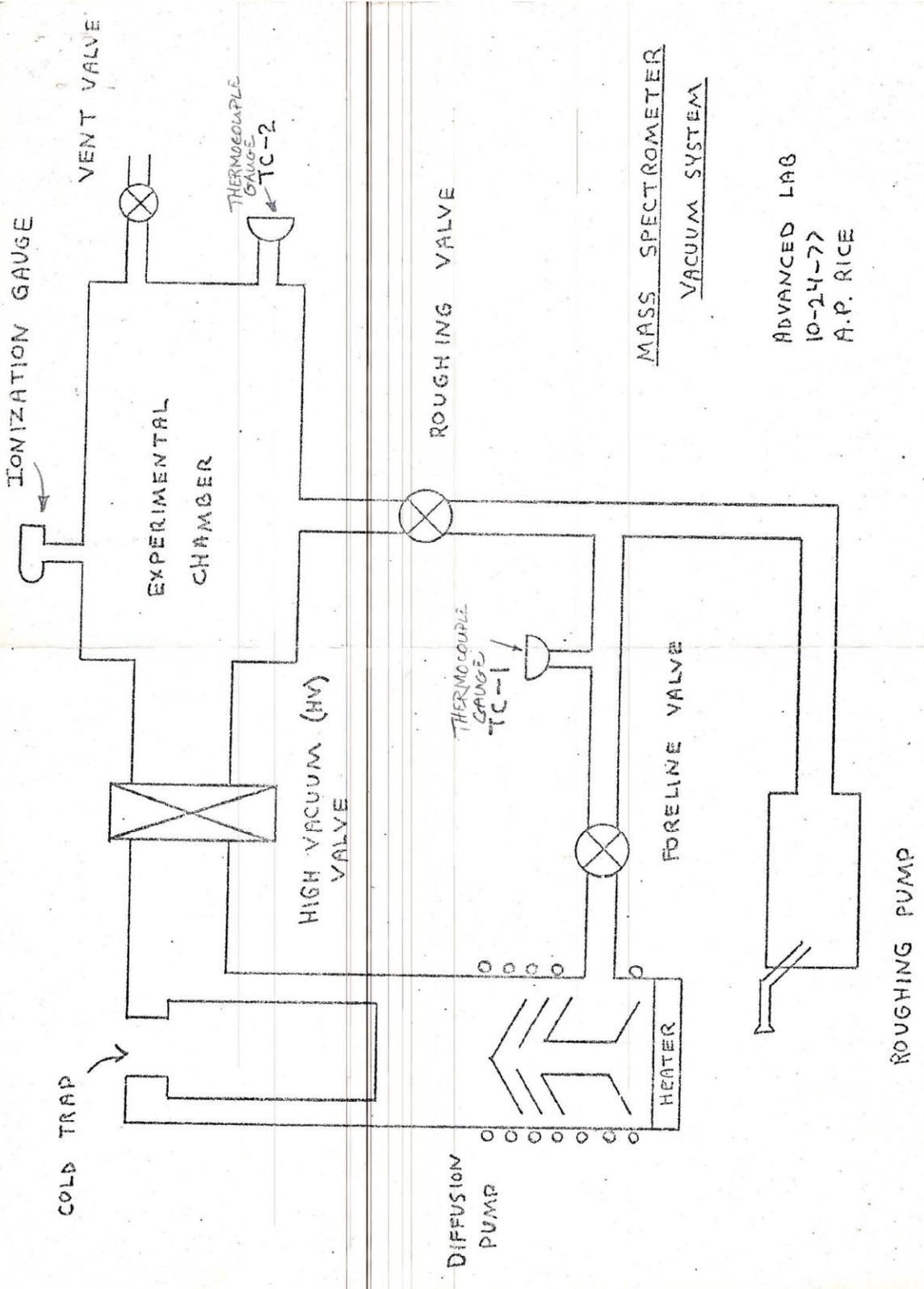
MASS SPECTROMETER BLOCK-DIAGRAM

* Depends on the filament

MASS SPECTROMETER
VACUUM SYSTEM

ADVANCED LAB
10-24-77
A.P. RICE