

MicroMag Vibrating Sample Magnetometer (μ VSM)

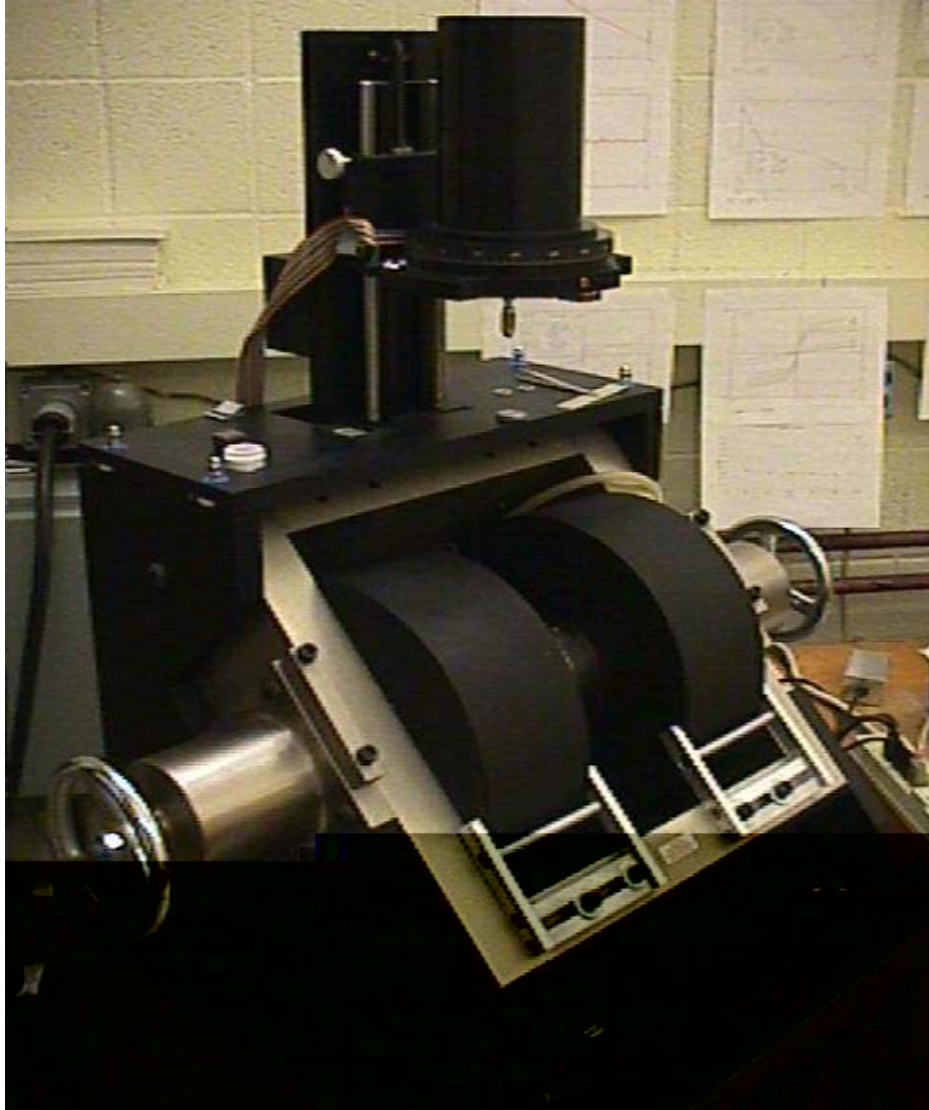
[Princeton Measurements Corporation] - Room 298 (μ VSM)

Sensitivity: 10^{-7} Am²

Maximum magnetic moment: 10^{-2} Am²

Temperature range: RT - 750 C

Maximum applied field: 1.8 T (depending on pole gap)



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I. SAMPLE PREPARATION

If the furnace is installed, you **MUST** follow instructions for high-temperature sample preparation, even if you only plan to measure at room temperature. The furnace is housed in a silvered, double-walled glass tube, which, when installed, occupies the space between the poles of the electromagnet (Fig. 1).

1) Furnace not installed.

a) **Sample size.** Samples must fit between the pole pieces of the magnet. The pole gap can be adjusted to accommodate a range of sample sizes (but **only by IRM staff**). Standard paleomagnetic cores will not fit, and must be trimmed or subsampled first. Trimming can be performed on the rock saws in the basement of Pillsbury Hall (Geology and Geophysics Department Building). Chips can be removed with a rock hammer. Samples can be crushed in the *IRM* sample-prep lab (room 292), using the pneumatic press on the central table and/or in the mortar and pestle on the shelves by the sink.

b) Sample holder.

- i) **Unconsolidated samples** should be packed in containers such as plastic P1 boxes or gelcaps.
- ii) **Small solid materials** (< ~5-6 mm) such as rock chips or thin films can be mounted directly on a cylindrical plastic sample holder, using a spot of Vaseline or vacuum grease; for this arrangement there should be at least one flat face on the specimen.
- iii) **For large or irregular solid** samples, consult an *IRM* staff member.

2) Furnace installed.

a) **Sample size.** Rock chip (no larger than about 5 x 5 x 2 mm) or powder.

b) **Sample mount.** When the furnace is installed, there is **only one permissible sample arrangement**, even if you are only planning to measure at room temperature. High-temperature samples **must** be cemented to a cylindrical ceramic sample mount, using the two-part Omega high-temperature epoxy. Plastic sample mounts, vacuum grease, gelcaps, etc are **not** allowed in the furnace, even when cold!

- i) Prepare a small amount of the high-T epoxy by mixing one drop of the liquid into a roughly equal volume of the powder. If your sample is in powder form, you may add it to the mix. Add more liquid or powder as needed to obtain a smooth, pasty consistency.
- ii) Apply the paste to the flat surface of the ceramic holder. If your sample is a solid chip, embed it into the paste.

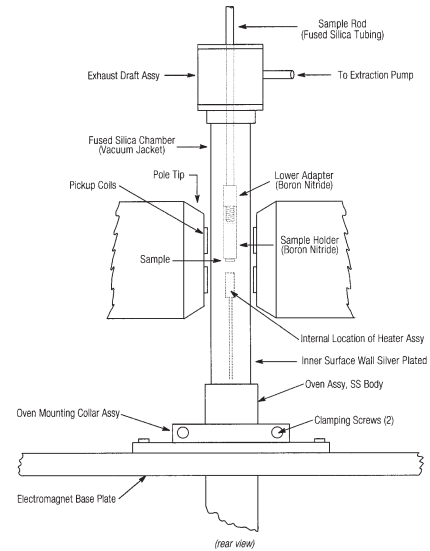


Figure 1. Diagram of VSM configuration with furnace installed.

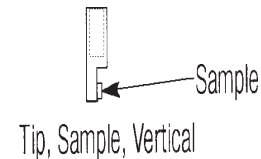


Figure 2. Example of sample mount.

iii) Wait for the epoxy to become fully hardened before running the sample in the μ VSM. At room temperature this requires 8-12 hours; at 100C in the Precision furnace in Rm. 292 the epoxy will be fully cured in about one hour.

II. SYSTEM START-UP

1) Furnace NOT installed.

- a) **Turn on the red switch on the power strip** on the left side of the desktop to power up the control electronics.
- b) The computer should already be on. If the screen is dark, move the mouse to wake it up.
- c) **Create user directory** for your data (if necessary).
 - i) Click on the VSM_HOT computer icon in the upper left corner of the computer desktop. This should bring up a window with the computer's file structure.
 - ii) Click on 'Local Disk D:', then 'USR', then 'GUEST'. (In other words, the path is D:\USR\GUEST).
 - iii) From the menu bar, select File -> New -> Folder. A new folder called 'Untitled' will be placed inside the GUEST folder. Change 'Untitled' to your name.
- d) **Start Program.** Click on the *μ Mag VSM Remanence* icon on the desktop. You will see a blue screen with the manufacturer's start-up instructions.
 - i) Steps 1 and 2, telling you to ensure the integrity of electrical grounds and connections are the responsibility of the IRM staff, not users (so don't worry about it).
 - ii) Steps 3 and 4 instruct you to turn on the power to the controller and cooling water to the electromagnet; these should be activated automatically when you turn on the power strip. Verify that the MicroMag Controller has power (the switch labelled 'Leave Me On' should always be in the On position and the red light should be lit). After a few minutes you should be able to feel the electromagnets begin to cool, as the chilled water circulates through them.
 - iii) Step 5 tells you turn on power to the Bipolar Magnet Power Supply. **This you DO need to do:** flip up the toggle switch marked 'Control Power', located in the lower left corner of the instrument panel.
 - iv) Click the *Continue* button to close the introductory screen.

2) Furnace installed (and you will be making high-T measurements). If you will be making room temperature measurements, follow procedure above in II.1.

- a) **Turn on the red switch on BOTH power strips** on the left side of the desktop to power up the control electronics. You should hear the vacuum pump begin to work.
- b) **Open the valve on the helium gas tank** in the corner and note the pressure. At the proper flow rate, the pressure will drop by approximately 200-300 PSI/hour (pounds per square inch per hour). Most high-temperature experiments require at least two hours, so in general you should not begin an experiment if the pressure is less than about 500 PSI. Find an IRM staff member if a new He tank is needed.
- c) **Observe the vacuum gauge**, which shows the pressure in the insulation space between the walls of the furnace tube. **When the pressure is less than about 50 millitorr, you can turn on the furnace current switch (heater).**
- d) The computer should already be on. If the screen is dark, move the mouse to wake it up.
- e) **Create user directory** for your data (if necessary). See II.1.c above.
- f) **Start Program.** Click on the *μ Mag VSM Remanence* icon on the desktop. See II.1.d above.

III. LOADING SAMPLES

1) Select a sample holder/rod from the black case sitting on the table across from the μ VSM.

a) For measurements in the furnace, use a glass rod; the ceramic tip is threaded to match the sample mount (Fig. 3).

b) For measurements without the furnace, find the appropriate holder for boxes or gelcaps, or use one of the brown phenolic sample rods with a plastic threaded tip to attach a cylindrical plastic mount. Affix your sample to the holder (e.g., use masking tape to attach the gelcap and straw to the sample holder).

2) If the furnace is installed, use the template to verify that the total length of your sample/holder/rod assemblage is not too long. This is very important! If it is too long, there is a chance that it will hit the fragile thermocouple inside the furnace, rendering the system unusable.

3) Insert the sample holder (with sample) into the chuck (collet; Fig. 4) on the bottom of the vibration unit. The sample holder rods are “keyed”, i.e., they have a flat section at the upper end (Fig. 3); press gently upward and twist the rod until the flat section snaps into place. Gently tighten the chuck.

4) Positioning of sample.

A) Without the furnace (If the furnace is installed, go to #4B below.):

a) Grasp the vibration head handle or the large vertical-position knob on the right side of the head.

b) Unlock the vibration head vertical-position lock (knob on the left).

c) Carefully lower the sample down to a position between the magnet poles. Be careful not to bump the Hall probe behind the poles or to touch the coils with the sample. When the sample is approximately centered vertically between the pick-up coils as shown, lock the vertical-position knob.

d) Use the X and Y adjustment screws to center the sample horizontally (approximate centering is adequate at this point; there is a software routine described below that can be used to “fine tune” the centering).

e) Turn on the vibration by clicking on the vibration toggle box in the program. A red LED on the vibration head will light up, indicating that the sample is vibrating.

B) With the furnace installed:

a) Grasp the vibration head handle or the large vertical-position knob on the right side of the head.

b) Unlock the vibration head vertical-position lock (knob on the left).

c) Carefully lower the sample down to a position *just above the top of the furnace tube*, and lock the vertical-position knob.

d) Use the X and Y adjustment screws to center the sample horizontally over the furnace tube.

e) Turn on the vibration by clicking on the vibration toggle box in the program. A red LED on the

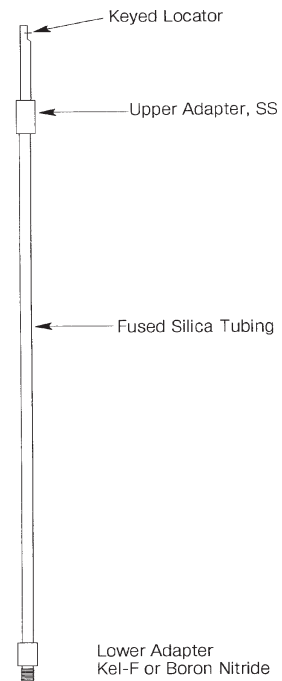


Figure 3. Sample rod. Note keyed end which can only be inserted into chuck/collet in one orientation.

vibration head will light up, indicating that the sample is vibrating.

f) Unlock the vibration head vertical-position lock on left while holding vertical-position knob on right side of head.

g) **Carefully lower the sample** down until the vibration head is resting on the adjustable stop. Along the way, **listen for a buzzing sound** that would indicate that the sample is in contact with the furnace tube; if you hear such a sound, stop the vibration, raise the sample back to the top of the furnace tube, and double-check the horizontal centering.

h) Make sure the **gold foil thermal shield** is in place between the pole pieces.

IV. CENTERING, SENSITIVITY, AND OTHER SETTINGS.

1) Measurement Units. The software has 3 possible units conventions. If you want to change this, select *Settings* from the program menu. Click on the 'Units of Measure - magnetism' box. Then select either Hybrid SI (usual), SI, or cgs. Click on the red box in the upper right corner to return to the main menu.

2) Set sensitivity. The software allows you to select from a set of predefined sensitivity levels (actually range settings). The ideal setting is a range somewhat larger than the maximum magnetic moment that will be measured during an experiment.

a) For hysteresis measurements, the maximum moment is usually attained in the maximum applied field. Click on the *Applied Field* box. Enter the field value that you will use as the maximum field for the hysteresis measurement (e.g., 1 T). The right-hand side of the display will show the actual applied field and the measured magnetic moment. If the sensitivity (range) is set too low, an overload indicator will usually show at the top of the status display. Click on the *Sensitivity* box on the menu (left-side display), and adjust the setting so it is just larger than the measured magnetic moment (right-side display). Press the Enter key to select that setting. [Note: for samples whose high-field behavior is dominantly diamagnetic, the magnetic moment will increase as the field decreases from its maximum value

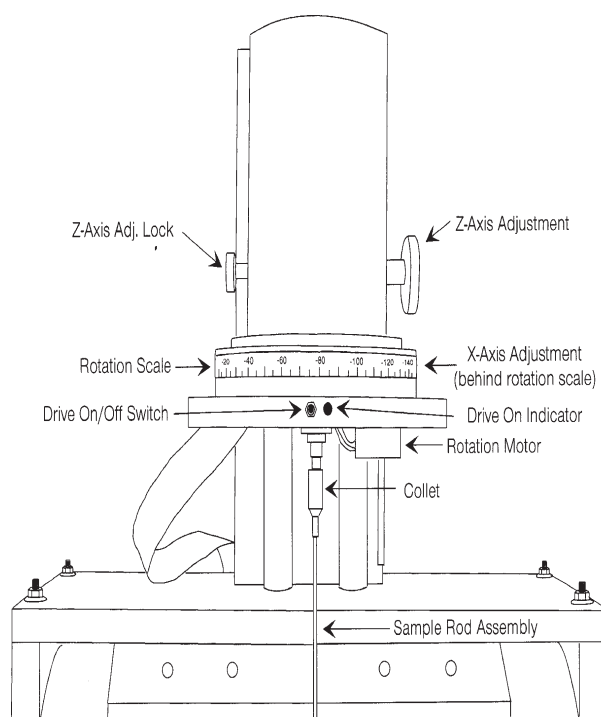


Figure 4. Vibration head showing locations of X-Y-Z adjustment knobs, Z-axis lock, and collet.

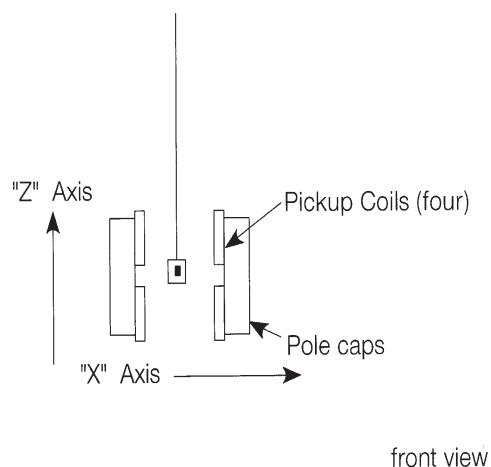


Figure 5. Positioning of sample between pick-up coils.

during the hysteresis measurement, and the moment may go off-scale (i.e., it may exceed the range setting). If this happens, try the next larger sensitivity (range) setting.]

b) In remanence experiments, such as determination of H_{cr} , the maximum moment is the saturation remanence. Apply a saturating field, and then set the field to 0 and set the sensitivity as above.

c) For thermomagnetic experiments, $M_s(T)$ typically has its greatest value at room temperature. Apply a field and set the sensitivity as described above for hysteresis experiments.

3) Centering. *If your sample is in the furnace, do not attempt to adjust the centering; go to step 2 below. If the furnace is not installed*, you can use the control software to help position the sample correctly. Select *Tools --> Optimize* from the menu. A screen will appear with a rising and falling green bar that corresponds to the signal strength. Three adjustment knobs located on the top and back of the instrument are used to displace the sample along corresponding axes (X, Y, or Z). Move the sample along one axis at a time until an extreme value (maximum or minimum; see below) is found for the signal strength. If the green bar goes off scale either change the scale or re-tare the signal.

<i>Axis:</i>	<i>Find:</i>
X (left-right; applied field direction)	minimum
Y (front-back)	maximum
Z (up-down)	maximum

4) Averaging Time: For weakly-magnetic samples (with maximum magnetic moments less than about 10^6 Am^2), it is generally necessary to use a relatively long *Averaging Time*. The minimum value allowed by the software is 100 ms, which gives good results for stronger samples. Longer averaging times give better signal/noise ratios, but increase the time required to complete an experiment. The upper limit enforced by the software is 1000 seconds, but in practice it is rarely useful to go beyond 1 or 2 seconds. *Note that averaging time set in the main menu will not affect the averaging time used in many of the measurement routines.* You must set averaging time within the sub-menu before starting the measurements.

5) Vibration Level: There are two circumstances in which it may be useful to decrease the *Vibration Amplitude* from its default (and maximum) relative value of 1. First, for powdered or granular samples packed in boxes or gelcaps, the vigorous shaking at the default setting may cause the particles in the container to move around, resulting in a noisy signal. Second, for very strongly magnetic samples, near the upper moment limit of the system (10^2 Am^2), you can sometimes measure an “off-scale” sample by reducing the vibration amplitude, which diminishes the induced voltage in the pick-up coils. In either case, to reduce the amplitude, choose *Vibration Amplitude* and select desired value.

V. ROOM-TEMPERATURE MEASUREMENTS

All of the following types of measurement are accessed from the main menu by selecting *Measurements...*

For all types of experiments, you may halt the measurement sequence at any point by pressing the Esc key. At this point you may save the data and/or change parameters and start over.

When the experiment is complete, you need to **save the data** by selecting *Save* from the *File* menu. If you do not do this, the data will be lost as soon as the next acquisition sequence is begun.

You can always return to the main menu by clicking the red button in the upper right corner of the sub-menu.

If you want to start in a demagnetized state, you can either do this from the main menu, or some of the measurement routines have a check box that will allow you to demagnetize the sample before proceeding.

1) Direct Moment vs. Field

a) Hysteresis Loop

Set desired values for maximum applied field, field increment, and averaging time.

b) First Order Reversal Curves (FORC)

Set the desired parameters. Consult an IRM staff member for assistance as needed.

c) It is also possible to measure **minor loops** or a user-defined set of **multiple loops** or **segments**. Ask an IRM staff member if you need assistance with these options.

2) Remanence Curves (for measurement of H_{cr} , IRM acquisition, DC demagnetization)

Select either *Single* or *Multiple* curve(s).

You will get a dialog box where you can choose what kind of curve(s) you would like to measure and set parameters. You must at least select one or both of (a) and (b).

a) **Isothermal Remanent Magnetization:** IRM acquisition curve. You probably want to start by demagnetizing the sample.

b) **DC Demagnetization:** program applies a user-defined saturating field, and then step-wise applies field in opposite direction to demagnetize. *This is the procedure to use for finding H_{cr} .* If this is all you want, you may terminate the routine by pressing Esc key shortly after the remanence curve crosses the zero axis.

c) **Include Direct Magnetization:** program will take in-field measurements in addition to remanence (zero-field) measurements.

3) Measurement vs. Orientation (Anisotropy Measurements)

Note: These measurements may **not** be made when the furnace is installed or when using a gelcap/straw sample holder.

This option allows you to make measurements as the sample is automatically rotated through a specified set of orientations.

a) **Moment vs. Orientation:** Sample moment is measured in or out of field at user-defined orientations.

b) Sequence vs. Orientation: A hysteresis loop and/or remanence curve can be measured at each orientation. Data for each orientation will be stored in a separate file, with sequential extensions; you will be asked for an initial file name.

4) Moment vs. Time (Viscosity Measurements)

This option allows you to monitor variations in moment after a change in the applied field (e.g., viscous acquisition or decay). Set desired values for initial and final applied fields, and timebase (duration of the experiment). Measurements will be made continuously, at a rate governed by the averaging time.

VI. HIGH-TEMPERATURE MEASUREMENTS

1) Thermomagnetic (M_s -T) Measurements

After the sample has been loaded, following the procedures of section III above, **apply the desired field** in the main menu. Set appropriate values for sensitivity and averaging time as described above in section IV. Then select *Measurements vs. Temperature* and press the Enter key. Set your desired values for minimum and maximum temperature, and for temperature increment. For $M_s(T)$ experiments, one degree is a suitable increment.

Pause after Temperature Increment allows the sample to equilibrate thermally after each temperature step. Allow about 6 seconds for each degree (e.g., for a 2-degree temperature increment, set the pause to 12 seconds). If *Retrace Initial-Final* is set to 'yes', measurements will be made on cooling from the maximum temperature, as well as on warming. This is very useful in M_s -T experiments, to assess the extent of alteration due to heating. Select the execute *Dipole Moment vs T* command to begin the experiment.

2) High-T Hysteresis Measurements

After the sample has been loaded, following the procedures of section III above, set appropriate values for sensitivity and averaging time as described above in section IV. Run a room-temperature hysteresis loop (see section V); the values you set there for maximum field and step size will be used for the high-T loops.

Select *Measurements versus Temperature* and press the Enter key. Set your desired values for minimum and maximum temperature, and for temperature increment. For hysteresis experiments, suitable increments range from about 25 to 100 degrees. (You can vary the increment by doing multiple experiments on the same sample, e.g., from 100 to 500C in 50-degree steps, and then from 510 to 600C in 10-degree steps).

Pause after Temperature Increment allows the sample to equilibrate thermally after each temperature step. Allow about 6 seconds for each degree (e.g., for a 100-degree temperature increment, set the pause to 600 seconds (10 minutes)). If *Retrace Initial-Final* is set to 'yes', measurements will be made on cooling from the maximum temperature, as well as on warming. This is very useful in M_s -T experiments, to assess the extent of alteration due to heating, but for hysteresis measurements it may not be worth the additional measurement time required (you can instead just cool directly to room-T and then run a loop).

Select the execute *Hysteresis Loops vs T* command to begin the experiment. The data for each temperature will be stored in a separate file, with sequential extensions; you will be asked for an initial file name.

VII. REMOVING SAMPLES

- 1) If the sample is at elevated temperature, return to the main menu (by pressing Esc one or more times) and set T to 25C (or 300K); **wait until the sample has cooled** before proceeding.
- 2) Turn off the vibration by clicking on *Toggle Vibration* in the main menu. The red light on the head should go out.
- 3) Grasp the large Z-adjust knob (on the right side of the vibration head) and unlock the head (using the smaller knob on the left).
- 4) Raise the vibration head all the way up, and lock it in place.
- 5) While holding the sample rod, loosen the sample chuck one full turn.
- 6) Remove the sample rod. Sometimes it sticks a bit; twist and wiggle it (loosening the chuck further will not help).

VIII. SYSTEM SHUTDOWN

1) High-Temperature Mode Shutdown

- a) Make sure that the applied field is zero.
- b) Make sure system is at room temperature (see VII.1. above).
- c) Turn off power to magnet.
- d) Exit software.
- e) Turn off both power strips.
- f) Turn off heater switch.
- c) Close valve on He gas tank.

2) Room-Temperature Mode Shutdown

- a) Make sure applied field is zero.
- b) Turn off power to magnet.
- c) Exit software.
- d) Turn off power strip.

APPENDIX A. Positioning of sample with shiny, new, transparent furnace tube.

- a) Gently **remove the exhaust assembly** from the top of the furnace tube by sliding it upward, and set it aside on one of the electromagnet pole pieces.
- b) **Grasp the vibration head** handle or the large vertical-position knob on the right side of the head.
- c) **Unlock the vibration head** vertical-position lock (knob on the left).
- d) **Carefully lower the sample** down to a position *just above the top of the furnace tube*, and lock the vertical-position knob.
- e) **Use the X and Y adjustment screws** to center the sample horizontally over the furnace tube.
- f) **Lower the gold foil thermal shield**, which is taped to one of the pole pieces; this will expose the transparent upper portion of the furnace tube and enable you to see the sample as you lower it into the tube.
- g) **Turn on the vibration** by clicking on the vibration toggle box in the program. A red LED on the vibration head will light up, indicating that the sample is vibrating.
- h) Unlock the vibration head vertical-position lock on left while holding vertical-position knob on right side of head.
- i) **Carefully lower the sample** down to a position at the top of the silvered section of furnace tube, where it should also be centered with respect to the pick-up coils. At this point the vibration head should be resting on the adjustable stop; if it is not, **do not** lower the sample beyond this point, because damage to the thermocouple may result.
- j) **Listen for a buzzing sound** that would indicate that the sample is in contact with the furnace tube; if you hear such a sound, stop the vibration, raise the sample back to the top of the furnace tube, and double-check the horizontal centering.
- k) Once the sample is centered, stop the vibration, unlock the head, raise it all the way up, and lock it into place. Do not change the orientation of the vibration head, because that would move the sample slightly off its properly centered position.
- l) **Reinstall the exhaust assembly** on the top of the furnace tube by gently sliding it downward. Make sure there is no tension from the plastic exhaust tube, which would cause displacement of the furnace tube.
- m) **Start vibration.**
- n) **Carefully lower the sample** down to position at the top of the silvered section of furnace tube, where it should also be centered with respect to the pick-up coils, and where the vibration head should be resting on the adjustable stop.
- o) **Raise the gold foil thermal shield** and attach it to the pole piece.