

I R M
Institute for Rock Magnetism

IRM Users' Manual

Written by the Staff, Students, and Users
of the *Institute for Rock Magnetism*

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ACKNOWLEDGMENT GUIDE

The Visitor's Guide to *IRM* Acknowledgments:

If any work you do at the *IRM* contributes to or results in a publication, we would appreciate seeing some variation on the following in the acknowledgment section:

I/we wish to thank the *Institute for Rock Magnetism* in Minneapolis for the use of its facilities. The *IRM* is funded by the Keck Foundation, the National Science Foundation, and the University of Minnesota.

And don't forget to send us a copy of your paper.

BROCHURE

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Future additions????

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INTRODUCTION

The *Institute for Rock Magnetism (IRM)* is dedicated to providing state-of-the-art facilities and technical expertise free of charge to any qualified researcher. This manual is intended for use by experimenters at the *IRM* after they have received initial training on the equipment.

The reader is assumed to have some DOS and Mac literacy. Samples are assumed to be either 1" paleomag cores, rock chips, or powders/sediments packed in appropriate containers such as "P1" plastic boxes or pharmaceutical "gel-caps". No further special preparation is necessary, except where noted. All computer menu items, directories, folders, etc. will be written in *italics*. All items that must be entered on a keyboard will be noted in **bold**. In most cases, such entries will be followed by pressing **return** or **enter**, which will not be noted. The **escape** key usually will get you out of something you don't want to do. All datafiles produced by *IRM* instrument control programs are IBM-compatible ASCII text files, unless otherwise noted.

INSTRUMENTS

Each entry will contain a summary header for each instrument along the following lines:

Instrument name (nickname)

[Manufacturer]—Location (*computer network name*)

specifications [time needed for a typical measurement (typical sample size)]

Reference to *IRM Quarterly* article for more information

Then comes a schematic diagram and brief description of the physical parts of the instrument. The main body of text for each instrument will consist of a detailed step-by-step set of instructions for using the machine. At the end will be any other pertinent information, such as calibrations, units, or special procedures.

Susceptibility Bridge (Bartington)

[Bartington Instruments]—Room 288 (*no computer*)

sensitivity 1.2×10^{-5} SI; frequency 470 Hz [~ 10 sec/sample (8 cm³)]

A computer-controlled dual-frequency (470 Hz and 4700 Hz) Bartington bridge is available in the Limnological Research Center's Core Lab in Civil and Mineral Engineering (CME) Room 668

See *IRM Quarterly*, Summer 1993, Vol. 3, No. 2

See *IRM Quarterly*, Spring 1994, Vol. 4, No. 1 (magnetic quantities and units)

The Susceptibility bridge consists of a meter, a cable, a sensor, and assorted plastic or cardboard sample supports. The meter should always be left on (i.e. the left-hand knob on the meter should be set to SI or cgs). The sensitivity is controlled by the right-hand knob (and is usually set to 0.1 for most geological samples).

I. MEASURING SUSCEPTIBILITY

- 1) Place any necessary sample support in the coil, and zero the meter: Momentarily move the toggle switch on the left side of the meter to the "Zero" (up) position, and then return it to the middle position. In about 15 seconds, the meter will beep, and "000.0" will appear on the readout.
- 2) Insert your sample into the coil (use the sample support to insure uniformity of measurements), and measure: Momentarily move the toggle switch on the left side of the meter to the "Measure" (down) position, and then return it to the middle position. In about 15 seconds, the meter will beep, and a reading will appear on the readout.
- 3) If you are worried about drift, or have particularly weak (< 5 Bartington units) samples, measure again without the sample in the coil to get a background reading. The corrected raw

value is then (reading) - .

- 4) Record the data by hand; there is no computer connected to our Bartington.
- 5) If you are using plastic boxes, be sure to measure an empty box to further correct your data.
- 6) Convert your data to real units according to the scheme below.

II. USING THE CALIBRATION CONSTANTS

Magnetic low-field susceptibility can be determined from the following formula:

$$\text{Susceptibility} = (\text{Sample reading} - \text{Empty-box reading}) \cdot K$$

where K is a conversion constant from the table below.

For SI volume susceptibility units [k (dimensionless SI)]

- P1 boxes $K = 1.015 \times 10^{-4} / (\text{sample volume in cm}^3)$
- 1" cylinders, measured along z axis $K = 1.00 \times 10^{-4} / (\text{sample volume in cm}^3)$
- 7.1mm D, 7.5mm L cylinders $K = 1.01 \times 10^{-4} / (\text{sample volume in cm}^3)$

For SI mass susceptibility units [χ ($\text{m}^3 \text{ kg}^{-1}$)]

- P1 boxes $K = 1.015 \times 10^{-7} / (\text{sample mass in g})$
- 1" cylinders, measured along z axis $K = 1.00 \times 10^{-7} / (\text{sample mass in g})$
- 7.1mm D, 7.5mm L cylinders $K = 1.01 \times 10^{-7} / (\text{sample mass in g})$

For cgs volume susceptibility units [k (dimensionless cgs)]

- P1 boxes $K = 1.015 \times 10^{-5} / (\text{sample volume in cm}^3)$
- 1" cylinders, measured along z axis $K = 1.00 \times 10^{-5} / (\text{sample volume in cm}^3)$
- 7.1mm D, 7.5mm L cylinders $K = 1.01 \times 10^{-5} / (\text{sample volume in cm}^3)$

For cgs mass susceptibility units [χ ($\text{emu Oe}^{-1} \text{ g}^{-1}$)]

- P1 boxes $K = 1.015 \times 10^{-5} / (\text{sample mass in g})$
- 1" cylinders, measured along z axis $K = 1.00 \times 10^{-5} / (\text{sample mass in g})$
- 7.1mm D, 7.5mm L cylinders $K = 1.01 \times 10^{-5} / (\text{sample mass in g})$

For example, let's assume that 8.13 g of a typical lake sediment has been repacked into a P1 box with a volume of 5.28 cm^3 . Suppose that, using the 0.1 SI scale, an empty P1 box measures -0.5 Bartington units (plastic is diamagnetic), and the box with the sample in it measures 29.7 Bartington units. These numbers would yield the following susceptibilities:

volume susceptibility: $k = 5.81 \times 10^{-4}$ SI

mass susceptibility: $\chi = 3.77 \times 10^{-7} \text{ m}^3 \text{ kg}^{-1}$

If the same sample had been measured using the 0.1 cgs scale, the reading would have been 23.6 Bartington units, yielding susceptibilities of $k = 4.62 \times 10^{-5} \text{ cgs}$ and $\chi = 3.00 \times 10^{-5} \text{ emu Oe}^{-1} \text{ g}^{-1}$, respectively.

Without volume or mass normalization, the Bartington units are meaningless!

Susceptibility Anisotropy Bridge (Roly-Poly)

[in-house]—Room 262 (*Aniso*)

sensitivity 1.2×10^{-6} SI; frequency 680 Hz [~2 min/sample (8 cm³)]

See *IRM Quarterly*, Fall 1993, Vol. 3, No. 3

See *IRM Quarterly*, Spring 1994, Vol. 4, No. 1 (magnetic quantities and units)

The Roly-Poly consists of a sample handler/susceptibility bridge, a computer, sample holders, a ThinkJet printer, and supplies

I. SAMPLE PREPARATION

- 1) Pick a plastic sample holder from the cardboard box on the Bartington table in Room 288. It should hold your sample snugly and fit into the chuck on the Roly-Poly. Carefully align the orientation mark on your sample with the orientation mark on the sample holder (or draw a new one). See the diagram on the wall, or in Appendix 2, for the *IRM*'s orientation scheme.

II. COMPUTER SET-UP

- 1) Turn on the computer with the red switch on the power strip.

- 2) Set the toggle switch on the Roly-Poly to the “Up” position.
- 3) After the computer boots, get into your data directory:
type **guest** to get to the guest directory (C:\usr\guest)
or type **cd\usr\xxxx** where xxxx is the name of any other user directory.

III. RUNNING THE CONTROL PROGRAM

- 1) Start the control program by typing **measure**. (You can get out of the program any time by pressing **control-break** (text mode) or **alt-s** (graphics mode or when motors are running).)
- 2) Make appropriate choices given by the program. Choices are generally typed in, and then registered with the **enter** key.
- 3) Type **o** to read about the orientation scheme for the program. Compare with the *IRM* scheme on the wall and in Appendix 2. The program calculates all values in three coordinate systems: sample, in-situ (Earth), and tilt-corrected (for bedding). This means that you must tell it the sample orientation in space (the azimuth of the x-axis [= $\alpha - 90^\circ$ in the *IRM* scheme] and the plunge of the z-axis [= β in the *IRM* scheme]), and the orientation of any bedding plane (the strike and dip).
- 4) Type **a** to start the anisotropy program.
- 5) Enter the information requested
 - a) Enter the sample shape (**P1**, **ODP**, **lund**, **cube**, **cyl** [cylinder], or **arb** [arbitrary]). This sets a volume and a shape correction, unless you choose “arb.” Examples of sample shapes are displayed on the side of the Superconducting Rock Magnetometer in Room 288, and are depicted in Appendix 3.
 - b) If you chose P1, ODP, or lund for a sample shape, enter the short axis of the box (usually the x-axis).
 - c) Press **enter** or **s** to get SI units, or **c** to get cgs units.
 - d) Enter the sample name.
 - e) Enter a filename (8 letters and/or numbers, no extensions). The data files are placed in the C:\usr directory with a .an extension. Move them to your directory when finished.
 - f) Enter as integers (separate by spaces) the bearing, plunge, strike, and dip appropriate for the sample. For an unoriented sample, use the defaults which make no corrections: **0 90 0 0**.
 - g) If you chose arb for a sample shape, enter the volume in cm³.
 - h) Press **enter** to use the default measurement parameters for your first attempt.
- 6) At the prompt in the upper right box on the screen, fit your sample in the chuck in the appropriate orientation according the diagram on the computer screen. Tape it in place with masking tape to keep it snug.
- 8) Follow the prompts, and let the machine move your sample.

- 9) When the sinusoidal data (χ vs. rotation angle) is displayed, you will be asked if the sinusoid is OK.
 - a) Type **y** to go on
 - b) Type **n** to change the measurement parameters (number of repetitions or averages). Then repeat the step following the prompts.
- 10) When the program finishes measuring, remove your sample from the chuck, and repeat steps 7 through 10 for each orientation until finished.
- 11) After the program fits an ellipsoid to your data, it will tell how good a fit it obtained ([RMS residual]/[RMS k]). Smaller is better. Press **enter** to continue.
- 12) After the program displays the susceptibility ellipsoid data, and other calculations, follow the prompts for printing and filing the results. Susceptibilities are in volume SI units.
- 13) Repeat steps 5d through 13 until finished by pressing **enter**. Put several samples into the same datafile when you would like to compare them in future calculations (mean directions, stereonet plots, Flinn plots, etc.).

IV. SHUTTING DOWN

- 1) Turn off the Roly-Poly toggle switch
- 2) Exit the control program by typing **e** from the main menu
- 3) Turn off the computer and printer with the red switch on the power strip.
- 4) Return the sample holder to Room 288.

V. PLOTTING AND MANIPULATING DATA

- 1) From the *Main* computer in 288, logon to the *Ani* computer over the network as drive S:, e.g. [See "Using the IBM computer network" in the Data Manipulation chapter.]
- 2) Type **aniso2** to start the anisotropy data manipulation program.
- 3) Type **o** to open your files. Enter the path as, e.g., **s:\usr\guest**.
- 4) Select the appropriate file with the arrow keys. The program will then count how many samples are in the file.
- 5) Type **c** to compute the mean directions and confidence ellipses for your samples. Enter the appropriate coordinate system at the prompt.
- 6) Type other letters to display and print or plot stereoplots (**s**), Flinn diagrams (**f**), and other plots. Follow the prompts for printing and plotting.

7) When finished, type `e` to exit the program, and logout of the *Ani* computer.

High-Temperature Susceptometer (Kappabridge)

[Kappa]—Room 262 (*Kappa*)

sensitivity 4×10^{-8} SI; frequency 920 Hz; temperatures from ambient to 1000 K [several hours/sample (= 200 mg chip or powder)]

See *IRM Quarterly*, Spring 1994, Vol. 4, No. 1 (magnetic quantities and units)

See *IRM Quarterly*, Summer 1994, Vol. 4, No. 2

The Kappabridge consists of an measurement station, an electronics box, numerous sample holders, a computer, a furnace control box, cooling water and pumps under the table, various small tools, and supplies.

PART 1. MANUAL ROOM-TEMPERATURE SUSCEPTIBILITY MEASUREMENT (WITHOUT COMPUTER)

I. SAMPLE PREPARATION

- 1) Your sample should fit into one of the plastic sample handlers for the Kappabridge. If necessary, pick a plastic sample holder from the cardboard box on the Bartington table in Room 288.

II. INSTRUMENT SET-UP

- 1) Make sure the cylindrical plastic spacer is in place in the measuring space.
- 2) Turn on the Kappabridge by setting its power switch to the on position (toggle switch at lower right corner of electronics box).
- 3) Adjust the meter: With the range set to any scale (left side of the electronics box) and no sample in the measurement space, adjust the large knob on the measurement box until the needle on the “Re” (“Real”) meter on the electronics box reads in the middle.
- 4) Adjust the phase: Turn the small knob just to the right of the meters until the “Im” (“Imaginary”) meter reads half scale (positive or negative). If necessary, readjust the large knob on the measurement box until the needle on the “Re” meter on the electronics box reads less than one division. When satisfied, turn the small knob again to return the “Im” needle to the middle.

III. MEASUREMENTS

- 1) When the “ready” light comes on, press the start/reset button (middle of the electronics box).
- 2) Insert the sample quickly into the measurement space.
- 3) When the machine beeps, quickly remove the sample.
- 4) Adjust the range switch if necessary to get the largest possible value on the LED display (full-scale is ± 1999). Repeat steps 1 through 4 if you change the range.
- 5) Calculate (by hand!) the value of “raw” susceptibility by multiplying the number on the LED display with the appropriate factor noted on the range setting.
- 6) Normalize this value by the measured volume or mass of your sample. The Kappabridge assumes that your sample has a volume of 10 cm^3 . See below and see the Bartington manual for more information about normalization and conversion of susceptibility values.
- 7) If you are using plastic boxes, be sure to measure an empty box to further correct your data.

For example, let's assume that 8.13 g of a typical lake sediment has been repacked into a P1 box with a volume of 5.28 cm^3 . Suppose that, using range 3 (and therefore multiplier 0.2), an empty P1 box measures -26 on the LED readout (plastic is diamagnetic), and the box with the sample in it measures 1508. These numbers would yield the following susceptibilities:

volume susceptibility: $k = 5.81 \times 10^{-4} \text{ SI}$

mass susceptibility: $\chi = 3.77 \times 10^{-7} \text{ m}^3 \text{ kg}^{-1}$

~~Without volume or mass normalization, the Kappabridge units are meaningless!~~

IV. SHUTTING DOWN

- 1) Turn off the Kappabridge by setting its power switch to the off position (toggle switch at lower right corner of electronics box).
- 2) Remove the cylindrical plastic spacer from the bottom of the measuring space using the suction-cup toy.

PART 2. ANISOTROPY OF MAGNETIC SUSCEPTIBILITY

I. SAMPLE PREPARATION

- 1) Your sample should fit into one of the plastic sample handlers for the Kappabridge. If necessary, pick a plastic sample holder from the cardboard box on the Bartington table in Room 288. Draw a single arrow on the top of your sample that corresponds to the x-axis. See the diagram on the wall, or in Appendix 2, for the *IRM*'s orientation scheme.

II. COMPUTER SET-UP

- 1) Turn on the computer with its switch and wait for it to finish "booting."
- 2) After the computer boots, get into your data directory:
type **guest** to get to the guest directory (C:\usr\guest)
or type **cd\usr\xxxx** where xxxx is the name of any other user directory.

III. INSTRUMENT SET-UP

- 1) Put the cylindrical plastic spacer into bottom of the measuring space.
- 2) Turn on the Kappabridge by setting its power switch to the on position (toggle switch at lower right corner of electronics box).
- 3) Adjust the meter: With the range set to any scale (left side of the electronics box) and no sample in the measurement space, adjust the large knob on the measurement box until the needle on the "Re" ("Real") meter on the electronics box reads in the middle.
- 4) Adjust the phase: Turn the small knob just to the right of the meters until the "Im" ("Imaginary") meter reads half scale (positive or negative). If necessary, readjust the large knob on the measurement box until the needle on the "Re" meter on the electronics box reads less than one division. When satisfied, turn the small knob again to return the "Im" needle to the middle.

IV. USING THE CONTROL PROGRAM

- 1) Start the control program by typing **ani**. (You can get out of the program any time by pressing **control-break**.)
- 1) At the *Measurement Options* menu, the defaults that will work are L, S, 1, Y, Y, N, Y, and M.
- 2) At the *Orientation Parameters* menu, the *IRM*'s orientation scheme will be used if the coordi-

nate system parameters are $P1 = 12$, $P2 = 90$, $P3 = 3$, and $P4 = 90$.

- 3) At the *Anisotropy Factors* menu, the defaults that will work are [9], [13], [4], [2], [31], and [24].
- 4) At the prompt, enter a **filename** (8 characters, letters and numbers only).
- 5) At the prompt, enter a path for the file, e.g., **c:\usr\guest**.
- 6) At the prompt, enter your initials as the operator, if desired.
- 7) At the prompt, enter a specimen name (anything you want).
- 8) At the prompt, enter a specimen volume (in cm^3).
- 9) At the prompt, if you have weak samples, enter the susceptibility of the empty sample holder:
 - a) Set the range to 1, and zero the bridge, if necessary.
 - b) When the “ready” light comes on, press the start/reset button (middle of the electronics box).
 - c) Insert the empty sample holder quickly into the measurement space.
 - d) When the machine beeps, quickly remove the sample holder.
 - e) Press the start/reset button again, so that the “halt” light goes out, and “ready” light goes on.
 - f) The number on the LED display should be entered as the susceptibility (without multipliers), e.g., **-106**.
- 10) At the prompt, enter the orientation of the sample, according to the *IRM*'s orientation scheme. For unoriented samples, enter **90,90**.
- 11) At the prompt, enter the code for foliations and lineations [see Kappa manual for a list of codes???]. Repeat for other foliations and lineations.
- 12) Find the measurement range by hand:
 - a) Insert your sample into the sample holder.
 - b) Select any range, and zero the bridge, if necessary.
 - c) When the “ready” light comes on, press the start/reset button (middle of the electronics box).
 - d) Insert the sample quickly into the measurement space.
 - e) When the machine beeps, quickly remove the sample.
 - f) Press the start/reset button again, so that the “halt” light goes out, and “ready” light goes on.
 - g) Adjust the range switch if necessary to get the largest possible value on the LED display (full-scale is ± 1999). Repeat steps b through f if you change the range.
- 13) Place your sample in position 1 (see appropriate diagram on the wall), and measure:
 - a) Press any key when ready to begin
 - b) When the computer beeps loudly, insert the sample quickly into the measurement space.
 - e) ~~When the machine beeps softly, quickly remove the sample.~~

- 14) Repeat step 13 for a total of 15 positions.
- 15) When finished, the control program will calculate various anisotropy parameters, and display them on the screen. Select your choice at the bottom of the screen by entering the appropriate number (2 allows you to redo a given measurement step).
- 16) Select your choice at the bottom of the new screen by entering the appropriate number (1 adds another specimen to the same file; 3 returns to DOS).

IV. SHUTTING DOWN

- 1) Exit the control program by typing 3 from the last screen.
- 2) Turn off the computer with its power switch (lower right push button).
- 3) Turn off the Kappabridge by setting its power switch to the off position (toggle switch at lower right corner of electronics box).
- 4) Remove the cylindrical plastic spacer from the bottom of the measuring space using the suction-cup toy.

PART 3. TEMPERATURE DEPENDENCE OF SUSCEPTIBILITY

I. SAMPLE PREPARATION

- 1) Your sample should fit into the small test tubes that go inside the furnace. Small chips or powders (=100 mg or so) should be weighed beforehand. You may want to take a difference weight between the empty sample tube and the full one.

II. COMPUTER SET-UP

- 1) Turn on the computer with its switch and wait for it to finish “booting.”
- 2) After the computer boots, get into your data directory:
type **guest** to get to the guest directory (C:\usr\guest)
or type **cd\usr\xxxx** where xxxx is the name of any other user directory.

III. INSTRUMENT SET-UP

- 1) Make sure the cylindrical plastic spacer is not in the measuring space. If it is, remove it with the toy plunger.
- 2) Turn on the Kappabridge by setting its power switch to the on position (toggle switch at lower

right corner of electronics box).

- 3) Adjust the meter: With the range set to any scale (left side of the electronics box) and no sample in the measurement space, adjust the large knob on the measurement box until the needle on the “Re” (“Real”) meter on the electronics box reads in the middle.
- 4) Adjust the phase: Turn the small knob just to the right of the meters until the “Im” (“Imaginary”) meter reads half scale (positive or negative). If necessary, readjust the large knob on the measurement box until the needle on the “Re” meter on the electronics box reads less than one division. When satisfied, turn the small knob again to return the “Im” needle to the middle.
- 5) Remove the furnace from its storage holder, and place it in the transport ring above the measurement space, being careful of the hoses and wires. The keyway on the furnace fits into the nub on the left side of the ring.
- 6) Insert your sample into the test tube (unless this is your first run with this test tube and/or temperature sensor, in which case run the test tube with only the temperature sensor for use as a furnace correction data file). You can fill the test tube to a height of about 1 cm or less. Rock chips are generally more stable than powders.
- 7) Slide the test tube into the black plastic collar.
- 8) Replace the assembly into the furnace.
- 9) Insert the thermocouple until it touches the sample. Loosely insert the argon tube.
- 10) Open the valve on the argon bottle in the corner. Make sure that the pressure regulator is set at the marked value. Check the flow gauge; adjust the flow with the black knob to approximately 100 cc/minute. This will help prevent oxidation of your sample at high temperature.
- 11) Turn on the furnace control box with its power switch (upper right).

IV. USING THE CONTROL PROGRAM

- 1) Start the control program by typing **measure**. (You can get out of the program any time by pressing **control-break**.)
- 2) Use the function (F) keys to change parameters for your run. The maximum temperature that the Kappabridge can reach is 700°C. Leave the susceptibility values at 0; the bridge will autorange. Leave F6 (furnace correction) set to “no”; you will correct for the furnace afterwards in your datafile. Press **F5** when ready.
- 3) At the prompt, enter a filename.
- 4) ~~When a blank plot appears on the screen, register your choices with the function (F) keys:~~

- a) Press **F3** to test the range. After the measurement is made, adjust the range switch if necessary to get a value on the LED display between ± 1000 and ± 1500 (full-scale is ± 1999 ; initial values between ± 1000 and ± 1500 allow for a moderate increase without going off-scale). Repeat this step as many times as necessary.
 - b) Press **F5** to start the heating run.
- 5) The program will plot the data in real time. Use the function (F) keys to change run parameters at any time.
 - a) Press **F1** to reset temperature range.
 - b) Press **F5** to stop the run at any time.
 - 6) If necessary, change the range setting and/or re-zero the bridge while the “wait” light is on between measurements.
 - 7) When the run is finished, you will be prompted to press **enter** to return to DOS.
 - 8) Susceptibility values in the datafile are in quasi-volume-normalized SI units, assuming a sample volume of $10 \text{ cm}^3 = 10^{-6} \text{ m}^3$. Un-normalize by this volume and then re-normalize by the correct volume or mass to get true susceptibility values.

V. REMOVING SAMPLES

- 1) When the sample is cool enough ($< 50^\circ\text{C}$), remove the sample holder test tube by lifting the black plastic collar at the top of the furnace.
- 2) Carefully remove the argon tube and return it to its holster. Close the valve on the argon tank.
- 3) Remove the thermocouple and return it to its holster.
- 4) Remove your sample from the test tube. Then, carefully and thoroughly clean the test tube with water, alcohol, and acetone, using the test tube brush and the Q-tips to get it clean and dry. Mop off the temperature sensor with alcohol. Citrate-bicarbonate-dithionite (CBD) solution may also be used for occasional cleaning of the test tubes and thermocouples.

VI. DATA MANIPULATION

- 1) Start the data manipulation software by typing **plot**.
- 2) Choose your data file by selecting the appropriate directory and filename.
- 3) At the *Main Menu*, select your choices with the **arrow** keys, and press **enter**.
 - a) Select 2 to remove bad data points from your file.
 - b) **Select 3 to smooth the empty furnace data for use in correcting future data files.**
 - c) Select 4 to correct for the (smoothed) susceptibility of the empty furnace, and follow the prompts.
 - d) **Select 5 to go on when ready.**

- 4) At the *Plotting Options* menu, select the type of graph you want, and then enter the appropriate minimum and maximum susceptibility values for that graph, e.g., 0,1 for a normalized graph.
- 5) After viewing the graph, press PrintScreen to dump the graph to the ThinkJet printer, or press any other key to return to the *Plotting Options* menu to make any changes.
- 6) From the *Plotting Options* menu, choose “end of plotting” to return to the *Main Menu*. The corrected data which had been plotted is now saved in your original data file in the fourth column. You can import the data file into a spreadsheet/plotting program for additional processing and plotting.
- 7) From the *Main Menu*, choose “end of work” to return to DOS.

VII. SHUTTING DOWN

- 1) Remove the furnace from the transport ring, and replace the furnace into its storage holder, being careful of the hoses and wires.
- 2) Turn off the furnace control box with its power switch (upper right).
- 3) Turn off the computer with its power switch (lower right push button).
- 4) Turn off the Kappabridge by setting its power switch to the off position (toggle switch at lower right corner of electronics box).

PART 4. CALIBRATION

Periodic calibrations are done by *IRM* staff; visitors generally do not need to do this.

- 1) Set range to 5.
- 2) When the “ready” light comes on, press the start/reset button.
- 3) Insert the standard [“ETALON No. 157, R=5, Xn=167”] quickly into the measurement space.
- 4) At the beep, quickly remove the standard.
- 5) If the LED display does not read 1167 (within 1%, i.e., between 1155 and 1179), adjust the calibration screw (lower left corner) with the small screwdriver and repeat steps 2-5 as necessary.
- 6) Zero the meter and adjust the phase.

Low-Temperature AC Susceptometer (LakeShore)

[LakeShore Cryotronics]—Room 262 (*Lake*)

sensitivity 2×10^{-8} SI; frequencies 1 Hz to 10,000 Hz, real and imaginary components plus harmonics; temperatures 15 K to 325 K [several hours/sample
(= 200 mg chip or powder)]

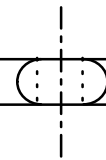
See *IRM Quarterly*, Spring 1994, Vol. 4, No. 1 (also for magnetic quantities and units)

The LakeShore consists of an instrument station, a sample holder rod, a computer, a DeskJet printer, various small tools, and supplies.

PART 1. ROOM-TEMPERATURE FREQUENCY SWEEPS (10 HZ TO 10,000 HZ)

I. SAMPLE PREPARATION

- 1) Cut a drinking straw to the proper length, shown below and by the marks on the table.
- 2) Prepare the sample for the drinking straw holder:
 - a) Gel-cap method: First, put in some fiberfrax or bits of kimwipe in the ends of a gel-cap (to keep the sample snug). After placing the sample in the gel-cap and closing it until it snaps shut, seal the side of the gel-cap with enough Capton tape to make a snug fit in the drinking straw.
 - b) Second straw method: First, place the sample chip in a short piece of drinking straw. Then, insert this sideways into the main straw. Recommended only for single chips.
- 3) Position the sample in the straw so that it is somewhat less than half-way up from the bottom. Measure against the diagram below or against the mark on the table. This aids in the centering procedure later.



- 4) Prevent the sample from falling out of the straw by placing a small piece of Capton tape over the bottom of the straw. Then, make a hole in the tape with a paper clip to allow air to pass. Make small holes in the side of the drinking straw above the sample to allow air to pass.
- 5) Select the large diameter sample rod for room-temperature measurements.
- 6) Slide the drinking straw onto the sample rod until it is correctly positioned on the template. Tape the straw securely to the rod with a fresh piece of Capton tape.

II. SAMPLE INSTALLATION

- 1) Loosen the hold-down screws and remove the plug. Be sure that the lower O-ring does not come out with the plug.
- 2) If it is not already open, open the Load Lock valve by turning the handle counter-clockwise until vertical.
- 3) Insert the sample rod assembly until it rests on the lower O-ring, and gently tighten the hold-down screws.
- 4) Slide the sample rod down until the top threaded end is near the movement extension, if it is not already there.

- 5) Swing the movement extension so the threaded end of the sample rod and the threaded end of the movement extension abut. Then screw the knurled finger nut up to connect the two pieces, and lock the assembly with the clamping lever by rotating it to the left.

III. USING THE CONTROL PROGRAM

- 1) Turn on the power with the master toggle switch (left side of the bottom panel in the electronics rack).
- 2) After the computer boots, get into your data directory
type **guest** for C:\usr\guest
or type **cd \usr\xxxx** where xxxx is another user directory.
- 3) Start the control program by typing **measure**.
- 4) Get into the *Collection* menu.
- 5) Get into the *Position* menu.
 - a) Select *Autoposition* to center the sample automatically. The “sample position” box on the screen should read “bottom coil” after about a minute, and the black marks on the head and sample transport mechanism should align approximately.
 - b) If this fails, select *Manual* to center it manually. The idea is to find the maximum (negative) voltage on the output meter (right side of bottom panel) when the sample is near the bottom

coil position. Move the sample up and down with the number pad keys indicated in the menu, using the black marks on the head and sample transport mechanism as a guide. When satisfactory, select *Bottom Coil* to register the position of the sample as centered in the bottom coil.

- c) If manual centering also fails, exit to DOS, turn the lock-in amplifier off for a moment and then turn it back on, and restart the program.
- 6) Press **escape** once to get back from the *Position* menu to the *Collection* menu, and select *Recall* to call up the sequence of steps used in a previous experiment. When asked for a path\file, enter \seq\ and a list of files will be displayed, from which you can select. The file 32FREQ_1S, for example, will measure susceptibility at thirty-two frequencies between ten and 10000 Hz, using a 1-second time constant. You will be asked to provide a new data file name (8 characters, letters and numbers only, no extension), and to enter information about the sample.
- 7) Get into the *Auto* menu and select *Begin* to start the experiment.
- 8) Then select *Realtime* followed by *Graphical* to watch the progress of your experiment plotted on the screen. It is not possible to obtain a hard copy of the plot through the control software, but you can use the file output later to generate graphs on paper (see Part 3. Data Files and Processing/Plotting Results).
- 9) When the experiment is complete, press **escape** twice to return to the *Collection* menu.

IV. REMOVING SAMPLES

- 1) Unscrew the knurled finger nut, unlock the movement extension with the clamping lever, and swing it to one side.
- 2) Loosen the two hold-down screws, and pull the sample rod assembly up and out of the machine. Be sure that the lower O-ring does not come out with the sample rod.
- 3) Replace the plug to keep dust out of the machine.
- 4) Repeat sections I through IV for other samples until finished.

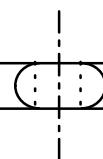
V. SHUTTING DOWN

- 1) Make sure that the plug is in, and that all of the valves on the instrument are closed (Load Lock Vent, Load Lock, Sample Space, Vacuum Space, He Exchange Gas, and Vacuum Pump valves).
- 2) Turn off the power with the master toggle switch (left side of the bottom panel in the electronics rack).

PART 2. DUAL-FREQUENCY TEMPERATURE SWEEPS (15 K TO 300 K AT 1000 Hz)

I. SAMPLE PREPARATION

- 1) Cut a drinking straw to the proper length, shown below and by the marks on the table.
- 2) Prepare the sample for the drinking straw holder:
 - a) Gel-cap method: First, put in some fiberfrax or bits of kimwipe in the ends of a gel-cap (to keep the sample snug). After placing the sample in the gel-cap and closing it until it snaps shut, seal the side of the gel-cap with enough Capton tape to make a snug fit in the drinking straw.
 - b) Second straw method: First, place the sample chip in a short piece of drinking straw. Then, insert this sideways into the main straw. Recommended only for single chips.
- 3) Position the sample in the straw so that it is somewhat less than half-way up from the bottom. Measure against the diagram below or against the mark on the table. This aids in the centering procedure later.



- 4) Prevent the sample from falling out of the straw by placing a small piece of Capton tape over the bottom of the straw. Then, make a hole in the tape with a paper clip to allow air to pass. Make small holes in the side of the drinking straw above the sample to allow air to pass.
- 5) Slide the drinking straw onto the sample rod until it stops. Tape the straw to the rod with another piece of Capton tape.

II. INSTRUMENT SET-UP

The idea is to keep only helium in the machine when the instrument is at low temperature to avoid freeze-up. Initial cooling takes overnight.

- 1) Make sure that the plug is in, and that all of the valves on the instrument are closed (Load Lock Vent, Load Lock, Sample Space, Vacuum Space, He Exchange Gas, and Vacuum Pump valves).
- 2) Open the Helium Tank valve on the helium gas tank by the wall. The regulator valve should be set to about 1 psi, and the balls in the gas flow meter on top of the instrument should be in the middle of the scale.
- 3) Turn on the power with the master toggle switch (left side of the bottom panel in the electronics rack).

- 4) Turn on the vacuum pump with its toggle switch (right side of the middle panel in the electronics rack). In a few seconds, you will hear a whirring whose pitch will increase as the pump spins up to speed. The green light on the pump (in the corner) will come on when it is ready. (Do not start the pump on a hard vacuum.)
- 5) Open the Vacuum Pump, Vacuum Space, Sample Space, and Load Lock valves, and allow the pump to evacuate these areas for about 10 minutes (until the pressure reads below 5 μm on the meter near the valves).
- 6) Open the Load Lock Vent for a few seconds to bleed helium gas into the machine, and then close it again.
- 7) Allow the pump to evacuate the machine again.
- 8) Repeat steps 6 and 7 at least one more time for a total of at least three evacuations.
- 9) Close the Load Lock, Sample Space, Vacuum Space, and Vacuum Pump valves again (All instrument valves should be closed, but the Helium Tank valve should stay open).
- 10) Turn on the cryo-pump with its toggle switch (left side of the middle panel in the electronics rack). You will hear a heart-beat and a murmuring sound from the pump out in the core behind the wall. Allow the cryo-pump to work overnight to bring the system down to about 13 K. The vacuum pump can stay on overnight.

III. SAMPLE INSTALLATION

- 1) Make sure that all of the valves on the instrument are closed (Load Lock Vent, Load Lock [handle horizontal], Sample Space, Vacuum Space, He Exchange Gas, and Vacuum Pump valves), and that the Helium Tank valve is open and helium is flowing.
- 2) Open the Load Lock Vent to bleed helium into the air lock.
- 3) Loosen the hold-down screws and remove the plug when the internal pressure is high enough to enable you to do so. Be sure that the lower O-ring does not come out with the plug.
- 4) With the top of the sample probe seal assembly positioned at the black mark on the sample rod, insert the sample rod assembly until it rests on the lower O-ring, and gently tighten the hold-down screws. Pressing and rotating the assembly aids in making a better vacuum seal.
- 5) Close the Load Lock Vent, and open the Vacuum Pump and Sample Space valves. Allow the pump to evacuate the air lock for about 10 minutes (until the pressure reads below 5 μm on the meter near the valves).
- 6) Open the Load Lock Vent for a few seconds to bleed helium gas into the machine, and then close it again. (The Sample Space valve can stay open during this operation.)
- 7) Allow the pump to evacuate the machine again.
- 8) Repeat steps 6 and 7 at least one more time for a total of at least three evacuations.
- 9) Close the Sample space valve.
- 10) Open the Load Lock Vent again to bleed helium into the air lock.
- 11) Open the Load Lock valve by turning the handle counter-clockwise until vertical, and very slowly slide the sample rod down until the top threaded end is near the movement extension. This should take a couple of minutes—the slower you go, the less likely you are to get air and moisture into the sample space.
- 12) Swing the movement extension so the threaded end of the sample rod and the threaded end of the movement extension abut. Then screw the knurled finger nut up to connect the two pieces, and lock the assembly with the clamping lever by rotating it to the left.
- 13) Close the Load Lock Vent, and open the Sample Space valve. Allow the pump to evacuate the sample space for about 10 minutes (until the pressure reads below 5 μm on the meter near the valves).
- 14) Open the Load Lock Vent for a few seconds to bleed helium gas into the machine, and then close it again.
- 15) Allow the pump to evacuate the machine again.

16) Repeat steps 14 and 15 at least one more time for a total of at least three evacuations.

17) After the final evacuation, close the Vacuum Pump valve, and then open and close the He Exchange Gas valve twice so that 500–1000 μm of helium is in the sample space. This aids in thermal conductivity between the sample and the heater.

18) Close the Sample Space valve. Open the Vacuum Pump and Vacuum Space valves.

During the experiment:

- the Load Lock Vent, Sample Space, and He Exchange Gas valves should be closed;
- the sample space should have about 500–1000 μm of helium in it;
- the vacuum space should be under a vacuum of $<1 \mu\text{m}$;
- the Load Lock, Vacuum Space, Vacuum Pump, and Helium Tank valves should be open;
- the vacuum pump and the cryo-pump should be on.

Note: The vacuum pump occasionally causes the lock-in amplifier (top of the electronics rack) to get kicked out of “remote.” Simply pressing the remote button is almost never sufficient. To be sure that everything is re-initialized properly, restart the control program from DOS.

IV. USING THE CONTROL PROGRAM

- 1) After the computer boots, get into your data directory
type **guest** for C:\usr\guest
or type **cd \usr\xxxx** where xxxx is another user directory.
- 2) Start the control program by typing **measure**.
- 3) Get into the *Collection* menu.
- 4) Get into the *Position* menu.
 - a) Select *Autoposition* to center the sample automatically. The “sample position” box on the screen should read “bottom coil” after about a minute, and the black marks on the head and sample transport mechanism should align approximately.
 - b) If this fails, select *Manual* to center it manually. The idea is to find the maximum (negative) voltage on the output meter (right side of bottom panel) when the sample is near the bottom coil position. Move the sample up and down with the number pad keys indicated in the menu, using the black marks on the head and sample transport mechanism as a guide. When satisfactory, select *Bottom Coil* to register the position of the sample as centered in the bottom coil.
 - c) If manual centering also fails, exit to DOS, turn the lock-in amplifier off for a moment and then turn it back on, and restart the program.
- 5) Select *Recall* to call up the sequence of steps used in a previous experiment. When asked for a path\file, enter \seq\ and a list of files will be displayed, from which you can select. The file 2FVSTEMP, for example, will measure susceptibility at two frequencies (400 and 4000 Hz) between 35 K and room temperature. You will be asked to provide a new data file name (8 characters, letters and numbers only, no extension), and to enter information about the sample.
- 6) Get into the *Auto* menu and select *Begin* to start the experiment.
- 7) Then select *Realtime* followed by *Graphical* to watch the progress of your experiment plotted on the screen.
- 8) When the experiment is complete, press **escape** twice to return to the *Collection* menu.

FREEZE UP!

If the machine freezes up so that the sample rod no longer moves (you'll feel it if you are trying to move it, and you'll hear it if the motor is trying to move it), press **escape** to stop automatic data collection and temperature control, turn off the cryo pump with its toggle switch (left side of middle panel in the electronics rack), allow the system to warm up (probably overnight), and then start over, being sure to pump out all areas thoroughly.

V. REMOVING SAMPLES

- 1) ~~Unscrew the knurled finger nut, unlock the movement extension with the clamping lever, and~~

swing it to one side.

- 2) Pull the sample rod up slowly until you see the black mark on the rod. The rod may be cold, especially near the bottom—allow it to warm as you pull it up. This should take a few minutes.
- 3) Close the Load Lock valve.
- 4) Make sure that all of the valves on the instrument are closed (Load Lock Vent, Load Lock, Sample Space, Vacuum Space, He Exchange Gas, and Vacuum Pump valves), and that the Helium Tank valve is open and helium is flowing.
- 5) Open the Load Lock Vent to bleed helium into the air lock.
- 6) Loosen the hold-down screws and remove the plug when the internal pressure is high enough to enable you to do so. Be sure that the lower O-ring does not come out with the plug.
- 7) Loosen the two hold-down screws, and pull the sample rod assembly up and out of the machine. Be sure that the lower O-ring does not come out with the sample rod.
- 8) Replace the plug to keep dust out of the machine.

VI. SHUTTING DOWN

- 1) Make sure that the plug is in, and that all of the valves on the instrument are closed (Load Lock Vent, Load Lock, Sample Space, Vacuum Space, He Exchange Gas, and Vacuum Pump valves).
- 2) Turn off the vacuum pump and the cryo pump with their toggle switches (left and right sides of the middle panel in the electronics rack).
- 3) Turn off the power with the master toggle switch (left side of the bottom panel in the electronics rack).

PART 3. DATA FILES AND PROCESSING/PLOTTING RESULTS

For each set of measurements, the control software produces three output files, with respective extensions .DAT, .CFG, AND .PRN. Only the last of these, the .PRN file, is necessary or useful for processing and/or plotting the results. These plain text files contain a few header lines, followed by the comma-delimited data. In the partial sample data file below, measurements were made for two frequencies over a range of temperatures. Each data line represents a single susceptibility measurement, with values given for temperature [K], rms AC field [A/m], DC field [A/m], frequency [Hz], in-phase susceptibility [SI], out-of-phase response, and a frequency-dependent correction/calibration factor that has been applied.

Comma-delimited text files can be imported by virtually any spreadsheet/plotting program, so you can use whatever software you prefer for processing and plotting.

Sample Data File

"TLC1761", " ", "11-07-1995", " ", "1-4" " SI vol norm units ", "v is ".0000001," m^3" " The U of MN amplitude correction is enabled: X'table = X'raw * X'corr "

" Temp ", " ACfld ", " DCfld", " Freq ", " X' ", " X" ", " X'corr"

29.6575,200,0,400,3.208465E-03,-1.048957E-05,.9936
29.665,200,0,4000,3.061031E-03,4.796873E-05,1.0068
39.715,200,0,400,3.271742E-03,-3.26061E-05,.9936
39.545,200,0,4000,3.215781E-03,-7.082183E-05,1.0068
49.1925,200,0,400,3.333918E-03,-3.080906E-05,.9936
48.9675,200,0,4000,.003293,-9.331932E-05,1.0068
57.98,200,0,400,3.376587E-03,-3.233923E-05,.9936
57.745,200,0,4000,3.33488E-03,-9.118684E-05,1.0068
68.71,200,0,400,3.392688E-03,-2.620257E-05,.9936
67.9,200,0,4000,3.352633E-03,-2.356551E-06,1.0068
78.37,200,0,400,3.415759E-03,-2.908538E-05,.9936
77.6425,200,0,4000,3.383731E-03,-3.307553E-05,1.0068
88.09,200,0,400,3.451857E-03,-3.026198E-05,.9936
87.43999,200,0,4000,3.395301E-03,-2.964692E-05,1.0068
95.8775,200,0,400,3.549342E-03,-3.616418E-05,.9936
95.515,200,0,4000,3.464885E-03,-5.453895E-05,1.0068
106.8625,200,0,400,3.748249E-03,-3.866125E-05,.9936
106.495,200,0,4000,3.662109E-03,-3.995508E-05,1.0068

Spinner Magnetometer (Spinner)

[Schonstedt]—Room 288 (*SSM*)

sensitivity 1×10^{-10} A m² [~ 3 min/sample (8 cm³)]

See *IRM Quarterly*, Fall 1993, Vol. 3, No. 3

See *IRM Quarterly*, Spring 1994, Vol. 4, No. 1 (magnetic quantities and units)

The SSM consists of a magnetometer inside magnetic shields, an electronics box, a computer, a dot-matrix printer, sample holders, and small tools

I. SETTING UP

- 1) Pick a plastic sample holder from the cardboard box on the Bartington table to the left. It should hold your sample snugly and fit into the chuck of the Spinner. Carefully align the orientation mark on your sample with the orientation mark on the sample holder (or draw a new one). See the diagram on the wall to the right, or in Appendix 2, for the *IRM's* orientation scheme.
- 2) Turn on the computer, monitor, and printer with the switch on the power strip at the right rear corner of the desk, behind the computer near the wall. Turn on the LED digital output device with the toggle switch at its lower right front corner. The main control box should always be left on (to avoid drift during the lengthy warm-up period).

II. USING THE CONTROL PROGRAM

- 1) After the computer boots, get into your data directory:
type **guest** to get to the guest directory (C:\usr\guest)

or type `cd\usr\xxxx` where `xxxx` is the name of any other user directory.

- 2) Type `ssm` to start the control program.
- 3) Make appropriate choices given by the program. Choices are selected with the **arrow** keys, and registered with the **enter** key. Mistakes can be fixed by cycling through the fields with the **enter** key.
 - a) Select the program mode: “ARM 1pos” (measures once on only one axis), “ARM 2pos” (measures twice on only one axis), or “NRM” (measures four times on all three axes using six sample positions). The “ARM 1pos” version is a quick and dirty way to measure laboratory remanences. The “ARM 2pos” version is used to measure laboratory-induced remanences (ARM, IRM, etc.) and demagnetizations. The “NRM” version is used for straightforward three-axis NRM measurements and demagnetizations.
 - b) Select Units: “SI” or “cgs.”
 - c) Choose the Data Organization: “Demagnetization” or “Strat.” The “Demagnetization” version (used for demagnetizing many samples, e.g.) assigns one file to each sample, so that levels correspond to demagnetization steps or acquisition steps. The “Strat” version (used for logging many NRMs, e.g.) makes just one file, so that levels corresponds to depths or sample numbers.
 - d) Input Number of Samples or Sites: Enter the number of samples you will be measuring at once. If you enter 2 for instance, you will be asked to measure sample 1, level 1; sample 2 level 1; sample 1, level 2; sample 2, level 2; etc.
 - e) Select Sample Type: “P1,” “CYL” (cylinder), “CUBE,” or “ARBITRARY.” Examples of sample shapes are displayed on the side of the Superconducting Rock Magnetometer to the left, and are depicted in Appendix 3.
 - f) Select Type of Demagnetization: “AF,” “THERMAL,” or IRMAQ” (IRM acquisition).
 - g) When all entries are satisfactory, press **F10**.
- 4) Enter the appropriate information about your sample:
 - a) Enter sample name (all characters are OK).
 - b) Enter filename (8 letters and numbers only, no extension). The program will put a `.tdb` extension on your filename.
 - c) When using the “NRM” version, enter the orientation angles, α and β , which are defined on the sheet on the wall to the right, and in Appendix 2. The default values for unoriented samples are $\alpha = 90^\circ$ and $\beta = 90^\circ$.
 - d) When all entries are satisfactory, press **F10**.

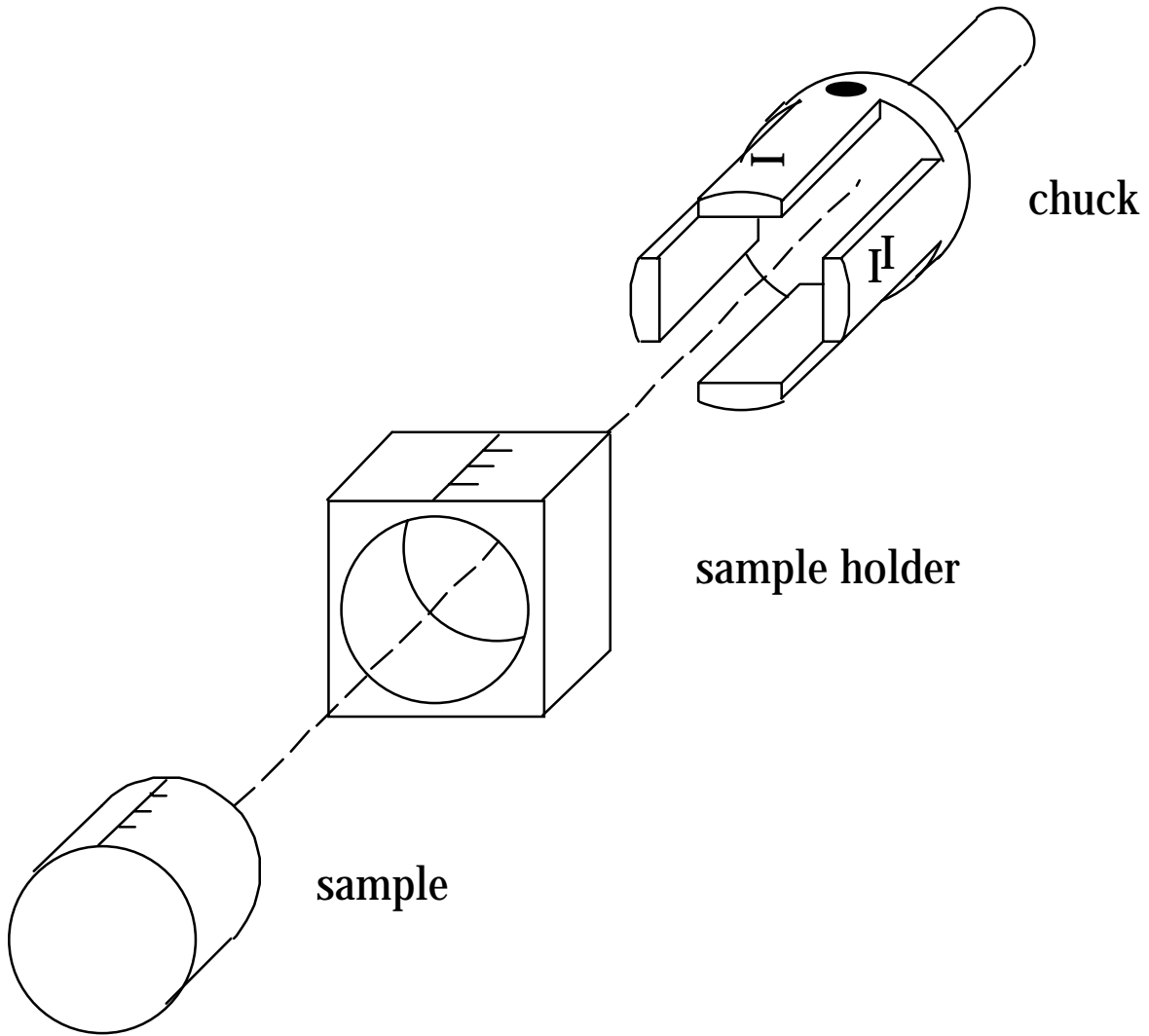
III. MEASUREMENTS

- 1) At the prompt, fit your sample in the chuck in the appropriate orientation for position 1 according the diagrams on the following pages.
- 2) Start the sample spinning by setting the toggle switch near the right end of the sample handler to the left position. Run the sample handler into the shield until it stops.
- 3) ~~Adjust the sensitivity with the multiplier and the decade multiplier switches (in the middle of~~

the SSM control box) until both meters read as high as possible without being off scale. Adjust the time constant (switch near lower left corner of the SSM control box) if the sample is too noisy. Further decades of sensitivity can be adjusted with the knurled knob at the right end of the sample handler—this changes the stop position of the handler (but avoid the closest stop to minimize geometrical problems with the signal pick-up).

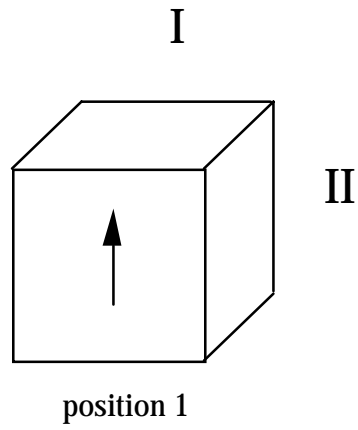
- 4) When satisfied, press the red push-button to take the measurement.

SPINNER ORIENTATION

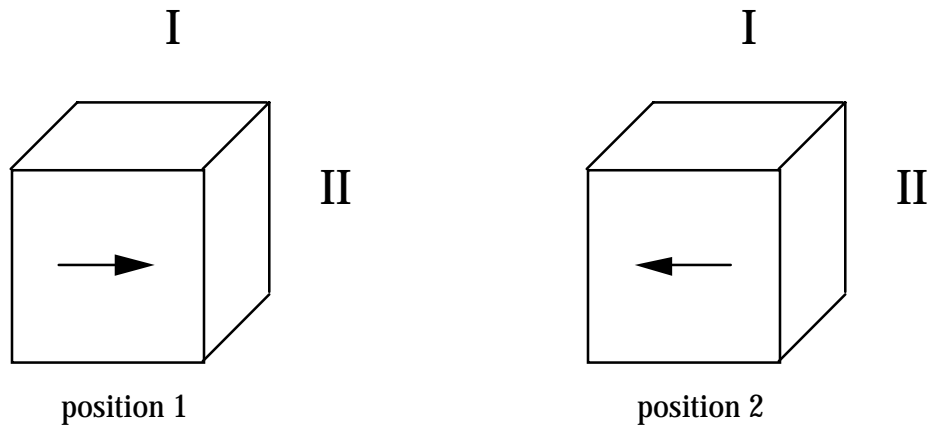


Position	Axis directed into shaft	Axis towards I direction	Axis towards II direction
1	+z	+x	+y
2	-z	+x	-y
3	+x	+y	+z
4	-x	+y	-z
5	+y	+z	+x
6	-y	+z	-x

SPINNER POSITIONS (ARMpos1)

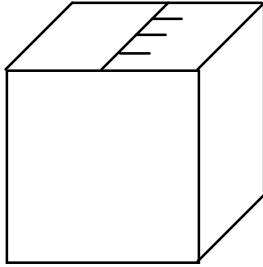


SPINNER POSITIONS (ARMpos2)



SPINNER POSITIONS (NRM)

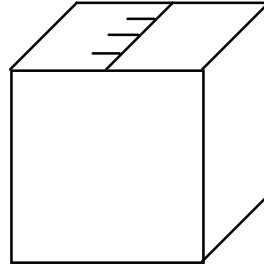
I



position 1

II

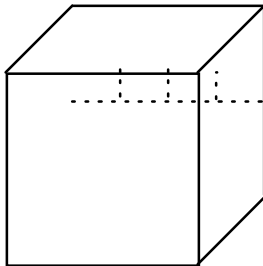
I



position 2

II

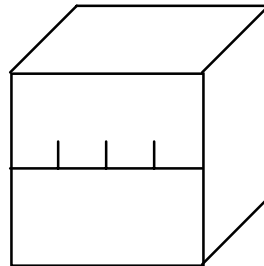
I



position 3

II

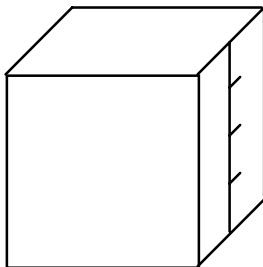
I



position 4

II

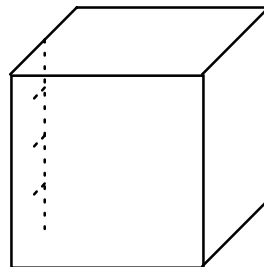
I



position 5

II

I



position 6

II

- 5) Pull the sample handler out and stop the spinning by setting the toggle switch to the right position.
- 6) Change the sample position, and repeat steps 9 through 11 as necessary until finished.
- 7) When a level is complete, the program will calculate the moment, and plot the value on the graphs and the top. Check the J_a/J_r (consistency and noise-to-signal) term at the lower left of the screen to make sure it is less than about 0.100. If the measurement was satisfactory, press **enter** to proceed to the next level. If the measurement was not satisfactory (wrong orientation, improper settings, high J_a/J_r , etc.), press **n** to repeat the measurement.
- 8) Repeat steps 1 through 7 for other demagnetization or acquisition levels, or for other samples as appropriate.
- 9) When finished, press **t** for a final table printout on the printer.

IV. SHUTTING DOWN

- 1) Put the sample holder back in the cardboard box.
- 2) Turn off the LED output device with its toggle switch.
- 3) Exit the SSM control program by typing **alt-q**, and type **bye** to park the heads of the computer.
- 4) Turn off the red switch on the power strip.

Superconducting Rock Magnetometer (SRM)

[2G Corporation]—Room 288 (*SRM*)

sensitivity 2×10^{-11} A m² [~1 min/sample (8 cm³)]

See *IRM Quarterly*, Fall 1993, Vol. 3, No. 3

See *IRM Quarterly*, Winter 1993–94, Vol. 3, No. 4

See *IRM Quarterly*, Spring 1994, Vol. 4, No. 1 (magnetic quantities and units)

The SRM consists of a magnetometer inside magnetic shields, sample handlers, electronics boxes, a computer, a pen plotter, a ThinkJet printer, sample holders, and small tools

I. SETTING UP

- 1) Pick a plastic sample holder from the cardboard box on the Bartington table to the right. It should hold your sample snugly. Carefully align the orientation mark on your sample with the orientation mark on the sample holder (or draw a new one). See the diagram on the wall near the corner above the freezer, or in Appendix 2, for the *IRM*'s orientation scheme.
- 2) Pick a sample chuck from the selection inside the SRM shields. Replace the chuck if necessary; it is held with the two small screws at the left end of the sample handler.
- 3) Turn on the following items: computer (push-button at upper right), electronics master power (toggle switch at lower left of the stack of four boxes), and printer (toggle switch at right

rear). The electronics will drift a little for the first 10 or 15 minutes.

- 4) Adjust the DC Offset dial at the middle of each electronics box to obtain a minimum value on the readout when nothing is inside the machine (one for each mutually perpendicular SQUID detector). If the readout is not steady in the first two decimal places, the SQUID is not tuned—see section V below.

II. USING THE CONTROL PROGRAM

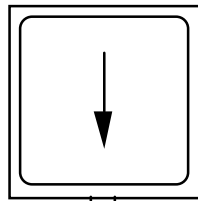
- 1) After the computer boots, get into your data directory:
type **guest** to get to the guest directory (C:\usr\guest)
or type **cd\usr\xxxx** where xxxx is the name of any other user directory.
- 2) Type **srm** to start the control program.
- 3) Make appropriate choices given by the program. Choices are selected with the **arrow** keys, and registered with the **enter** key. Mistakes can be fixed by cycling through the fields with the **enter** key.
 - a) Select the program mode: “ARM” (measures twice on only one axis), “NRM” (measures twice on all three axes using two sample positions), or “NRMWEAK” (measures four times on all three axes using four sample positions). The “ARM” version is used to measure laboratory-induced remanences (ARM, IRM, etc.) and demagnetizations. The “NRM” version is used for straightforward three-axis NRM measurements and demagnetizations. The “NRMWEAK” version is used for particularly weak NRM measurements and demagnetization.
 - b) Select Units: “SI” or “cgs.”
 - c) Choose the Data Organization: “Demagnetization” or “Strat.” The “Demagnetization” version (used for demagnetizing many samples, e.g.) assigns one file to each sample, so that levels correspond to demagnetization steps or acquisition steps. The “Strat” version (used for logging many NRMs, e.g.) makes just one file, so that levels corresponds to depths or sample numbers.
 - d) Input Number of Samples or Sites: Enter the number of samples you will be measuring at once. If you enter 2 for instance, you will be asked to measure sample 1, level 1; sample 2 level 1; sample 1, level 2; sample 2, level 2; etc.
 - e) Select Sample Type: “P1,” “CYL” (cylinder), “CUBE,” or “ARBITRARY.” Examples of sample shapes are displayed on the side of the Superconducting Rock Magnetometer to the right, and are depicted in Appendix 3.
 - f) Select Type of Demagnetization: “AF,” “THERMAL,” or IRMAQ” (IRM acquisition).
 - g) When all entries are satisfactory, press **F10**.
- 4) Enter the appropriate information about your sample:
 - a) Enter sample name (all characters are OK).
 - b) Enter filename (8 letters and numbers only, no extension). The program will put a .tdb extension on your filename.
 - c) When using the “NRM” version, enter the orientation angles, α and β , which are defined on the sheet on the wall to the right, and in Appendix 2. The default values for unoriented samples are $\alpha = 90^\circ$ and $\beta = 90^\circ$.

d) When all entries are satisfactory, press **F10**.

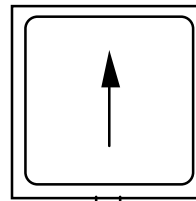
III. MEASUREMENTS

- 1) At the prompt, fit your sample in the chuck in the appropriate orientation for position 1 according to the diagram on the following page.

ARM/IRM POSITIONS (TOPVIEWS)



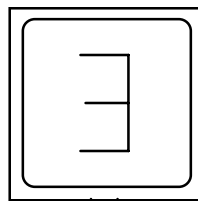
position 1



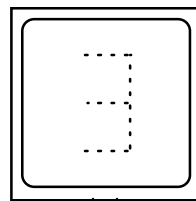
position 2

PROCEDURE: Measure baseline (shaft out), z (shaft in), baseline (shaft out). Rotation angle does not matter. Arrows show the dipole direction measured in each position.

NRM POSITIONS (TOPVIEWS)



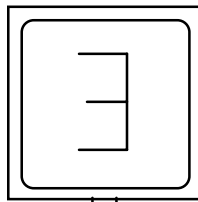
position 1



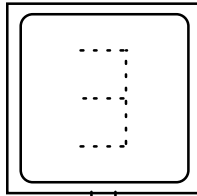
position 2

PROCEDURE: Measure baseline (shaft out), A (shaft in, fully CW), B (shaft in, fully CCW), baseline (shaft out).

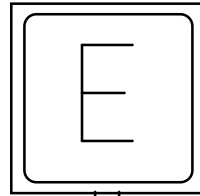
NRMWEAK POSITIONS (TOPVIEWS)



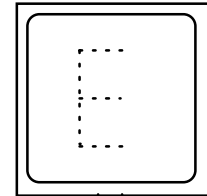
position 1



position 2



position 3



position 4

PROCEDURE: Measure baseline (shaft out), A (shaft in, fully CW), B (shaft in, fully CCW), baseline (shaft out).

- 2) With the sample handler out of the machine and in the fully clockwise position, press the red button to take a background measurement.
- 3) Slide the sample handler slowly into the machine until it stops. Press the red button again to take a reading.
- 4) [For “nrm” and “nrmweak” versions only:] Rotate the sample handler to the fully counter-clockwise position, and press the red button a third time to take another reading.
- 5) Pull the sample handler slowly out of the machine, and press the red button a last time to take a second background measurement.
- 6) Change the sample position, and repeat steps 2 through 5 as necessary until finished.
- 7) When a level is complete, the program will calculate the moment, and plot the value on the graphs and the top. Check the J_a/J_r (consistency and noise-to-signal) term at the lower left of the screen to make sure it is less than about 0.100. Also check the lower left of the screen to be sure there were no fluxjumps. If the measurement was satisfactory, press **enter** to proceed to the next level. If the measurement was not satisfactory (wrong orientation, improper settings, high J_a/J_r , fluxjump occurred, etc.), press **n** to repeat the measurement. If the sample is too strong ($> 5 \text{ A m}^2$, you'll have to use the Spinner.
- 8) Repeat steps 1 through 7 for other demagnetization or acquisition levels, or for other samples as appropriate.
- 9) When finished, press **t** for a final table printout on the printer.

IV. SHUTTING DOWN

- 1) Put the sample holder back in the cardboard box.

- 2) Turn off the electronics master power switch.
- 3) Exit the SRM control program by typing **enter**, type **bye** to park the heads of the computer.
- 4) Turn off the computer with its push-button.

V. TUNING THE SQUIDS

- 1) Turn on the electronics master power with its toggle switch at the lower left of the stack of four boxes.
- 2) Turn on the oscilloscope behind the electronics boxes with its toggle switch at the bottom.
- 3) Connect a BNC/co-axial cable between the “sync” output at the back of the z-axis SQUID electronics box and the “trigger input” of the scope, and between the “r.f. det. output” at the back of the z-axis box and the “input” of the scope.
- 4) At the front of z-axis box, turn the “rf level” knob clockwise (starting from 0 [fully counter-clockwise]) until the first sine wave (a node) appears on the scope screen. Adjust for the maximum amplitude sine wave.
- 5) Repeat steps 3 and 4 for the other two SQUID electronics boxes after reconnecting the cables to the appropriate corresponding points. (This is not necessary for ARM measurements, which read only the z-axis (top) SQUID box.)
- 6) Turn of the oscilloscope with its toggle switch when finished. The cables can stay connected.

VI. HELIUM TRANSFER

ØThis is a two-person job♦

ØGloves and safety goggles required♦

Only the people directly involved in the transfer allowed in the room. All food and beverages removed for duration.

Read all the way through these directions before starting, because things happen quickly once you begin!

- 1) Call Ron Fremstad (x6-2263) in Physics room 77 to reserve at least one dewar of liquid helium (about 60 liters).
- 2) Collect the following near the SRM:
 - liquid helium dewar(s) (from Physics)
 - helium gas tank (in the corner behind the door in room 242)
 - dewar exit valve adapter (in the top left drawer behind the door in room 242)

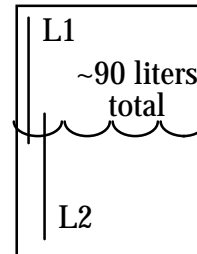
helium transfer tube adapter (from the MPMS transfer tube on the back wall in room 242)
two pair of gloves (in the cabinets in room 242)
heat gun (in the cabinets in room 242)
SRM helium transfer tube (on the back wall above the coffee machine in room 290)
SRM helium transfer accessories [adapter hoses with valve, pair of black “00” corks, and a smaller orange cork] (in the drawer in room 290)
crescent wrench and large slot screwdriver (on the tool board in room 292)
pen and paper

- 3) Attach the electronics adapter at the end of the wire under the SRM to the adapter at the upper left part of the SRM inside the shields. This allows you to monitor the helium levels as you fill. Turn on the electronics master power (toggle switch at lower left of the stack of four boxes).
- 4) Attach the dewar exit valve adapter to the dewar, and the transfer tube adapter to the long end of the transfer tube. Purge both the hose from the helium gas tank and the connector to the dewar with helium gas, then connect the hose to the dewar. Set the regulator for about 1 psi, but leave the inlet valve on the dewar closed.
- 5) Remove the brass caps from the vent valve and from the fill port on the left front of the SRM using the crescent wrench.
- 6) Make sure there is a “00” cork in the smaller end of the transfer adapter hoses, that the valve is closed, and that the hose clamp on the large end is loose.
- 7) With at least one glove on, quickly remove the exposed “00” cork from the fill port, and push the large end of the adapter hoses over the fill port. Some cold gas will escape from the fill port when you do this. Secure the adapter hoses by tightening the hose clamp.
- 8) Bleed the 0.5 psi relief valve on the dewar, then open the valve at the top of the dewar. Insert the long end of the transfer tube, making sure to seat the outer sleeve of the tube in the dewar connector. Push in slowly.
- 9) Close the 0.5 psi relief valve on the dewar.
- 10) When helium gas starts to come out of the short end of the transfer tube, open the SRM vent valve and the adapter hose valve. Then quickly remove the “00” cork from the adapter hoses, and insert the short end of the transfer tube a few inches into the adapter hoses (but not far enough to block the valve). Again, some cold gas will escape when you do this.
- 11) Open the inlet valve to the dewar to start gas flow into dewar.
- 12) When liquid helium comes out of the adapter hose valve, push the transfer tube all the way into the fill port. There should be less than an inch of narrow transfer tube exposed when it is all the way in—try to get past the fill port opening. Close the adapter hose valve.
- 13) Monitor the pressure in the dewar and that in the SRM (meter on left, outside the shields).
~~Adjust the regulator on the gas tank to maintain a pressure difference of about 1–2 psi be~~

tween the dewar and the SRM (but don't exceed 10 psi total pressure).

- 14) Monitor the helium level as you go by holding the L2 meter toggle switch (middle of bottom electronics box) up until the meter settles. Take readings every couple of minutes to estimate the transfer rate. Because the L1 meter does not work when transferring, you will have to "fly blind" at the end, and estimate when the dewar is empty or the SRM is full based on earlier transfer rates. (Past experience shows that, once the helium level starts to rise, the dewar will be nearly empty in approximately 25 minutes.) [100% on the meters equals 50 liters, and L2 = 0.96 (max) corresponds to

L1 = 0.19]



- 15) Periodically remove the ice from the SRM vent to avoid plugs.
- 16) Before the transfer is complete, make sure that the frost on the adapter hoses has melted (use the heat gun if necessary).
- 17) When you estimate that the transfer is complete, close the dewar inlet valve.
- 18) Then, using at least one glove, quickly pull the transfer tube all the way out of the adapter hoses, and plug the opening with the "00" cork. Plenty of cold helium gas will escape when you do this, so be careful!
- 19) Pull the transfer tube out of the dewar. CAUTION! The tube is dangerously cold! USE GLOVES!
- 20) Close the dewar exit valve at the top of the dewar, and open the 0.5 psi relief valve. Close the helium gas tank valve, disconnect the gas tank hose from the dewar, and remove the adapter. (Reopen the dewar exit valve to measure the liquid helium left in the dewar if possible, then close the dewar exit valve.)
- 21) Use the heat gun to thaw the SRM vent valve. Use a glove to hold the orange cork in the valve opening when possible. When the valve thaws enough to turn, close it.
- 22) When completely thawed, remove the orange cork, and replace the cap on the SRM vent. Also replace the cap on the fill port, leaving the "00" cork in place.
- 23) The gas pressure in the SRM should drop back to <1 psi in a few hours. The nodes for the SQUIDs should return by the next day.
- 25) For several days after a transfer, check that the gas flow (meter on top of the SRM shields) stays

non-zero. If the flow is zero, there is an ice plug: Turn off the power switch on the cryopump in the core (between room 288 and 242) for about 4 hours to melt the ice plug. When the gas flow returns, restart the cryopump.

24) Put everything away, and return the dewar to Physics room 77.

Alternating-Field (AF) Demagnetizers

[Schonstedt]—Room 288 (*no computer*)

peak applied fields from 0 to 100 mT; biasing fields from 0 mT to 0.2 mT; ARM and partial ARM capability [~ 1 min/demagnetization step or ARM (8 cm³)]

See *IRM Quarterly*, Fall 1993, Vol. 3, No. 3

See *IRM Quarterly*, Winter 1993–94, Vol. 3, No. 4

See *IRM Quarterly*, Spring 1994, Vol. 4, No. 1 (magnetic quantities and units)

Each AF Demagnetizer consists of a coil inside magnetic shields, a power supply, an electronics box, the ARM control box, and sample holders. Except for the details of the home-made ARM boxes, the machines are identical and function identically.

PART 1. STEPWISE AF DEMAGNETIZATION

- 1) Pick a plastic sample holder from the cardboard box on the Bartington table behind you. It should hold your sample snugly, and fit into the chucks for the AF Demagnetizers. Use masking tape as necessary to insure a good fit. Carefully align the orientation mark on your sample with the orientation mark on the sample holder (or draw a new one). See the diagram on the wall to the left, or in Appendix 2, for the *IRM's* orientation scheme.
- 2) Turn on the AF Demagnetizer with the white “Power” push-button at the bottom left of the control box. [The “Power” throw switch in the lower left corner of the power supply box below should always be set to “On.”]
- 3) Set the “Decay Rate” switch to 25.
- 4) Set the “Peak AC Field” wheels to the desired value for a given demagnetization level. With an unknown sample, try demagnetization levels of 0, 5, 10, 20, 40, 70, and 100 mT, and then adjust with the next sample accordingly so that less than 50% of the initial magnetization is

lost per step.

- 5) Place the sample into the chuck, and slide the chuck into the solenoid inside the shields until it stops.
- 6) Press the red "Start Cycle" push-button to demagnetize along the axis of the solenoid. When it is finished, the machine will (sometimes!) beep, and the red "Start Cycle" light will go out.
- 7) Pull out the chuck and remove the sample holder.
- 8) [For three-axis demagnetization only, e.g., NRM's.] Repeat steps 5 through 7 for each of the other two axes to completely demagnetize a sample which has an unknown magnetization at a given level.
- 9) Measure the sample on the SRM or the Spinner [See above].
- 10) Repeat steps 4 through 9 until finished.
- 11) Turn off the AF Demagnetizer with the white "Power" push-button.

PART 2. ARM AND PARTIAL ARM ACQUISITION

- 1) Pick a plastic sample holder from the cardboard box on the Bartington table behind you. It should hold your sample snugly, and fit into the chucks for the AF Demagnetizers. Use masking tape as necessary to insure a good fit.
- 2) Turn on the AF Demagnetizer with the white “Power” push-button at the bottom left of the control box. [The “Power” throw switch in the lower left corner of the power supply box below should always be set to “On.”]
- 3) Set the “Decay Rate” switch to 25.
- 4) Set the “Peak AC Field” wheels to 99.0 mT. [This allows for the possibility of removing the ARM later with a 100-mT AF demagnetization.]
- 5) Turn on the ARM power with its toggle switch, and set the desired DC Field with the knob (usually 50 μ T or 100 μ T). [Left-side instrument only: Make sure the upper toggle switch is set to “arm.”]
- 6) [For Partial ARM (pARM) only:] Set the window during which the DC field will be applied for pARM using the push-buttons (left-side instrument) or the knobs (right-side instrument). [The right-side instrument requires minor equipment modification to do pARM: If you need both set-ups, see an *IRM* Staff member for help.]
- 7) Place the sample into the chuck, and slide the chuck into the solenoid inside the shields until it stops.
- 8) Press the red “Start Cycle” push-button to impart an ARM (or pARM) along the axis of the solenoid. When it is finished, the machine will (sometimes!) beep, and the red “Start Cycle” light will go out.
- 9) Pull out the chuck and remove the sample holder.
- 10) Measure the sample on the SRM or the Spinner [See above].
- 11) [For pARM only:] Repeat steps 6 through 10, changing the pARM window each time until finished.
- 12) Turn off the ARM power with its toggle switch, and turn off AF Demagnetizer with the white “Power” push-button.

Thermal Demagnetizer

[Schonstedt]—Room 290 (*no computer*)

temperatures from ambient to 1100 K; optional applied fields from 0 mT to 0.2 mT [~1 hour/demagnetization step or TRM (8 cm³, 8 samples per batch)]

See *IRM Quarterly*, Winter 1993–94, Vol. 3, No. 4

See *IRM Quarterly*, Spring 1994, Vol. 4, No. 1 (magnetic quantities and units)

The Thermal Demagnetizer consists of a field-controlled furnace, an electronics box, quartz sample boats, supports for the boats, a timer, and push rods.

- 1) Select only competent samples for thermal demagnetization (paleomag rock cores, or sediments that have been impregnated with Sauereisen cement from the shelf by the door in Room 292). No plastic or combustibles!
- 2) Turn on the power by pressing the white “Power” push-button on the temperature controller.
- 3) Place samples in the quartz sample boat, and set the boat on the support rods.
- 4) Insert this sample assembly into the furnace and close the door.
- 5) Set the desired temperature with the thumb wheels on the temperature controller.
- 6) Press the red “Heat” push-button to start the furnace. It takes up to an hour to reach thermal equilibrium, depending on the temperature. Set the timer and go do something else for awhile.
- 7) After samples have sat a thermal equilibrium for about 10 minutes, press the red “Heat” push-button again to turn it off.
- 8) Open the door, and use the long glass rod to push the sample assembly into the cooling chamber. A mark on the rod shows the correct distance to clear the rear door.

- 9) Turn on the fan by pressing the blue “Cool” push-button. Closing the door again will help speed the cooling process.
- 10) When air coming out of the back of the machine is cool (at least 15 minutes), open the door and push the sample assembly out of the back of the machine from the front with the glass rod. Be careful not to push everything onto the floor.
- 11) Measure the samples on the SRM or the Spinner [See above].
- 12) Repeat steps 3 through 11 until finished.

Vibrating Sample Magnetometer (VSM)

[Princeton Applied Research]—Room 294 (*VSM*)

sensitivity 2×10^{-8} A m²; applied fields from 0 T to 1.7 T; temperatures from 77 K to 1200 K [\sim 30 min/sample for hysteresis loops (8 cm³) and \sim 2 hours/sample for Curie points (= 200 mg chip or powder)]

See *IRM Quarterly*, Fall 1993, Vol. 3, No. 3

See *IRM Quarterly*, Spring 1994, Vol. 4, No. 1 (magnetic quantities and units)

The VSM consists of an electromagnet assembly, a magnet power supply rack, a control electronics rack, a computer, a pen plotter, sample holders, a furnace, a cryogenic chamber, a vacuum pump system, a high-pressure helium gas tank (see Chemical Hygiene Plan for proper use of chemicals), various small tools, and supplies.

Contents:

PART 1. ROOM-TEMPERATURE HYSTERESIS LOOPS

PART 2. CURIE POINT DETERMINATIONS

PART 3. LOW-TEMPERATURE HYSTERESIS LOOPS

PART 4. HIGH-TEMPERATURE HYSTERESIS LOOPS

PART 1. HYSTERESIS LOOPS

I. SAMPLE PREPARATION

- 1) Samples must fit between the pole pieces of the magnet. Standard paleomag cores will not fit, and must be trimmed or subsampled first. Trimming can be done 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 pneumatic press on the central table and/or in the mortar and pestle on the shelf by the sink. Unconsolidated samples should be repacked in plastic P1 boxes (plastic cubes are too big). Be sure you know the mass of the sample before you continue [a small electronic scale should be on the counter in room 290].
- 2) Select a sample holder from the right-side table drawer that best suits your sample. Affix your sample to the rod (e.g., with masking tape, which is non-magnetic).
- 3) Fit the sample between the pole pieces of the magnet, and slide the sample holder onto the vertical shaft. Tape it in place for insurance.

II. COMPUTER SETUP

- 1) Turn on the red switch on the power strip on the left end of the table to power up the computer and the control electronics
- 2) After the computer boots, get into your data directory:
type **guest** to get to the guest directory (C:\usr\guest)
or type **cd\usr\xxxx** where xxxx is the name of any other user directory.
- 3) Type **measure** to start the control program. Follow the instructions on the screen for setting up the machine, which are also given below:

III. INSTRUMENT SETUP

- 1) Turn on the Magnet:
 - a) Turn on the magnet main power switch (at the bottom left of the magnet power supply = tall rack in the corner to the right). [The orange “ready” light should come on (left side of the tan Bipolar Power Supply panel in the smaller rack to the right of the table).]
 - b) Make sure the Mode Control knob is set to Off (middle of the Bipolar Power Supply panel).
 - c) Press the On button (left side of the bipolar power supply panel). [The “on” light should come on (left side of the Bipolar Power Supply panel) after two loud clicks from the magnet power supply.]
 - d) Set the Mode Control knob to Remote. [This allows the computer to control the magnetic field.]
 - e) ~~The other switches in the Bipolar Power Supply panel are not used.~~

Note: Never turn off the magnet when current is flowing (as read on the meter in the Bipolar Power Supply panel)—this blows fuses, blasts amplifiers, and otherwise causes headaches for the *IRM* staff!

- 2) Turn on the Teslameter:
 - a) Set the toggle switch to On (right side of the Teslameter panel in the electronics rack). [This allows the computer to measure the field at the Hall probe between the pole pieces of the magnet.]
 - b) Check that the left-hand knob in the panel is set to “measure,” and the right-hand knob is set to “computer control”.
- 3) Turn on the Vibration Control:
 - a) Set the toggle switch to On (right side of the Vibration Control panel in the electronics rack).
 - b) When the sample is in place, set the Vibration toggle switch to On (middle of the Vibration Control panel).
- 4) Turn on the Lock-In Amplifier:
 - a) Set the toggle switch to On (right side of the Lock-In Amplifier panel in the electronics rack).
 - b) Press the Remote Enable button so that its red light comes on (middle of the Lock-In Amplifier panel). [This allows the computer to control signal detection.]
 - c) The other buttons in the Lock-In Amplifier panel are not used.
- 5) The lower panels in the electronics rack are not used for room-temperature hysteresis loops.

IV. RUNNING THE CONTROL PROGRAM

- 1) Back at the computer, press any key to bring up the *Main Menu*. Type **loop** to run a hysteresis loop. The program first tests the magnet and communication with the Lock-In Amplifier.
- 2) At the prompts, enter the appropriate information about your sample:
 - a) Enter your sample name (all characters are OK).
 - b) Enter sample mass in mg.
 - c) Enter sample description (any 30 characters are OK).
 - d) Enter filename for data storage (8 letters and numbers, no extension). The program will not add an extension to files with the raw data, but it will add a .par or .dia extension to files with corrected data later.
 - e) Enter h to H_{cr} measured. Entering **h** will make the VSM do a step-wise DC demagnetization of a saturation IRM after it finishes the loop. This procedure usually takes about 15 extra minutes. [*Recommended.*]
 - f) Enter i for initial suscept curve. Entering **i** will make the VSM start its loop at zero field, but requires that you demagnetize your sample after centering it below. [*Not recommended.*]
 - g) Enter the maximum field (in mTesla) for the loop.

- 3) The program will then set up the Lock-In Amplifier (choosing sensitivity, phase, time constant, etc.). The phase (lower right meter in the Lock-In Amplifier panel) should read in the low thirties. If it does not, check to make sure that the sample is securely mounted to the rod, and that the sample is not touching the magnet or the sensors.
- 4) When the program prompts you, center the sample with the three translation screws near the head of the VSM so that you get a minimum reading on the Output meter (right side of the Lock-In Amplifier panel in the electronics rack) when adjusting the y direction (side-to-side: use the screw on the right), and maximum readings when adjusting the x direction (front-to-back: use the screw at the front) and the z direction (up-and-down: use the screw underneath the head). Press the **n** key to normalize the sound if you wish to use audio cues to help center the sample (the computer will issue tones whose pitch [frequency] increases with the Output reading). Press any key when satisfied.
- 5) The program will double-check the Lock-In Amplifier settings, prompt you to press any key one last time, and then run the hysteresis loop, plotting the data in real time as it goes.
- 6) If your sample falls off, or the loop is otherwise unsatisfactory, press **alt-s** to stop the loop.
- 7) When finished, the program will compute the high-field slope, which corresponds to the para/diamagnetic susceptibility. If you are interested in this number write it down, because it will not appear on the hardcopy plot. The program will ask if you want to make a paramagnetic or a diamagnetic correction to the loop (i.e., subtract the high-field slope). Entering **p** or **d** as appropriate will create a new datafile which contains the corrected data. It will be denoted with a .par or .dia extension, as appropriate. Pressing any other key will save only the uncorrected data. The program will then return you to the *Main Menu*.

V. PLOTTING YOUR DATA

- 1) Turn on the pen plotter on the table by pressing its power switch (rear-left button in the bank of buttons). Align a piece of paper with the white line and press the paper-grabber button (left-middle) to load the paper.
- 2) From the *Main Menu*, type **plot** to get to the *Plot Menu*. Then type **loop** to call up the plotting program.
- 3) When prompted for a filename, press **enter** for choices, or type in the filename you want. The plotter should start plotting immediately. You can plot a second loop (the corrected data, for instance) on the same piece of paper by following the prompts.
- 4) Type **exit** to return to the *Main Menu*.
- 5) When the plotter finishes, remove the plot by pressing the paper-grabber button again.

VI. CHANGING SAMPLES

- 1) Set the Vibration toggle switch to Off (middle of the Vibration Control panel).
- 2) Remove the sample rod from the vertical shaft and change your sample.
- 3) Replace the sample rod on the vertical shaft, again using masking tape for insurance.
- 4) Set the Vibration toggle switch to On (middle of the Vibration Control panel).
- 5) Go to section IV above to measure another loop.

VII. INSTRUMENT SHUT DOWN

- 1) Set the Vibration toggle switch to Off (middle of the Vibration Control panel).
- 2) Remove the sample holder from the vertical shaft, remove your sample, and put the sample holder back in the drawer.
- 3) From the *Main Menu*, type **exit** to quit the program.
- 4) Turn off the Magnet:
 - a) Check that the magnet current is zero (meter in the middle of the Bipolar Power Supply panel).

Note: Never turn off the magnet when current is flowing (as read on the meter in the Bipolar Power Supply panel)—this blows fuses, blasts amplifiers, and otherwise causes headaches for the *IRM* staff!

- b) Set the Mode Control knob to Off (middle of the Bipolar Power Supply panel).
 - c) Press the Off button (left side of the Bipolar Power Supply panel). [The “on” light should go off (left side of the Bipolar Power Supply panel) after one loud click from the magnet power supply.]
 - d) Turn off the magnet main power switch (at the bottom left of the magnet power supply = tall rack in the corner to the right).
- 5) Turn off the Teslameter:
 - a) Set the toggle switch to Off (right side of the Teslameter panel in the electronics rack).
- 6) Turn off the Vibration Control:
 - a) Set the toggle switch to On (right side of the Vibration Control panel in the electronics rack).
 - b) When the sample is in place, set the Vibration toggle switch to On (middle of the Vibration Control panel).
- 7) Turn off the Lock-In Amplifier:
 - a) Set the toggle switch to Off (right side of the Lock-In Amplifier panel in the electronics rack).

8) Turn off the computer: Type **bye** to park the heads, and then turn off the red switch on the power strip on the end of the table.

PART 2. CURIE TEMPERATURE DETERMINATIONS

I. INSTRUMENT MODIFICATION

- 1) Remove the VSM head dust cap from the top of the VSM head.
- 2) Remove the normal sample rod by unscrewing its threaded end at the top of the VSM head, and pulling it all the way up and out. Rotating it will help to get past the two keyways. Hang it up on the wall to the right next to the magnetic power supply.
- 3) Remove the sample-rod guide by unscrewing the threaded cap under the VSM head near the z-direction translation screw, and pulling the guide gently straight down until its O-ring seal releases. Hang it up on the wall also.
- 4) Attach the furnace by guiding it between the pole pieces of the magnet with the copper tubing facing towards the back, and then pushing it up until its O-ring seals. Then screw the threaded cap on until firm. Be careful not to hold the bottom part of the furnace for leverage, because it will twist and snap the delicate furnace leads inside.
- 5) Attach the white plastic tubing that comes from the vacuum system to the mating piece of copper tubing at the back of the furnace. After they are made finger tight, the nuts should be given another quarter turn with the crescent wrenches (on the narrow shelf behind the VSM) to insure a good vacuum seal.
- 6) Attach the furnace power supply and thermocouple lead wires to the mating connector at the front of the furnace.
- 7) Connect the hose from the regulator of the high-pressure helium tank to the hose dangling from the vacuum system Bleed Valve.

II. SAMPLE PREPARATION

- 1) Samples must fit into the quartz sample rod for the furnace which lives on the wall near the magnet power supply (< 4 mm in diameter, 1–2 cm long; usually about 100 mg).
 - a) Rock chips should be weighed on the balance which is on the counter near the door. They should then be wrapped in silver foil to aid thermal contact and to prevent contamination of the sample holder. Silver foil can be found near the balance.
 - b) Unconsolidated samples should be packed into silver foil pellets. Make a small cylindrical pellet wrapper out of the silver foil by wrapping the foil around the end of one of the tools in the plastic container near the balance. Fold the edges over to seal one end, remove the shell, and weigh it on the balance. Fill the shell with your sample, tamping often to get as tight a packing as possible. Seal the other end and weigh the full pellet. Finally subtract the mass of the empty shell from that of the full pellet to get your sample mass.

- 2) Pack your sample into the end of the quartz sample rod with the fiberfrax (spun alumina) from the right-side table drawer. The end of the thermocouple should touch or be very near your sample. Take time with this step to make sure your sample is very snug and secure, because thermal cycling and vibration try hard to loosen your sample and degrade your signal.
- 3) Load the sample rod into the VSM from the top, making sure both keyways pass on the way down. The threaded end of the rod should screw in many turns at the end, after the final keyway prevents the rod from rotating.
- 4) Attach the connector for the sample thermocouple in the VSM cap to the pins protruding from the sample rod, making sure the black and red marks on each piece match each other.
- 5) Fit the cap onto the top of the VSM head and seat it, making sure the O-ring seals well.

III. COMPUTER SETUP

- 1) Turn on the red switch on the power strip on the left end of the table to power up the computer and the control electronics
- 2) After the computer boots, get into your data directory:
type **guest** to get to the guest directory (C:\usr\guest)
or type **cd\usr\xxxx** where xxxx is the name of any other user directory.
- 3) Type **magsi** to start the control program. Follow the instructions on the screen for setting up the machine, which are also given below:

IV. INSTRUMENT SETUP

- 1) Turn on the Magnet:
 - a) Open the cooling water valve (just to the right of the table on the wall).
 - b) Turn on the magnet main power switch (at the bottom left of the magnet power supply = tall rack in the corner to the right). [The orange “ready” light should come on (left side of the tan Bipolar Power Supply panel in the smaller rack to the right of the table).]
 - c) Make sure the Mode Control knob is set to Off (middle of the Bipolar Power Supply panel).
 - d) Press the On button (left side of the bipolar power supply panel). [The “on” light should come on (left side of the Bipolar Power Supply panel) after two loud clicks from the magnet power supply.]
 - e) Set the Mode Control knob to Remote. [This allows the computer to control the magnetic field.]
 - f) The other switches in the Bipolar Power Supply panel are not used.

Note: Never turn off the magnet when current is flowing (as read on the meter in the Bipolar Power Supply panel)—this blows fuses, blasts amplifiers, and otherwise causes headaches for the IRM staff!

- 2) Turn on the Teslameter:
 - a) Set the toggle switch to On (right side of the Teslameter panel in the electronics rack). [This allows the computer to measure the field at the Hall probe between the pole pieces of the magnet.]
 - b) Check that the left-hand knob in the panel is set to “measure,” and the right-hand knob is set to “computer control”.
- 3) Turn on the Vibration Control:
 - a) Set the toggle switch to On (right side of the Vibration Control panel in the electronics rack).
 - b) When the sample is in place, set the Vibration toggle switch to On (middle of the Vibration Control panel).
- 4) Turn on the Lock-In Amplifier:
 - a) Set the toggle switch to On (right side of the Lock-In Amplifier panel in the electronics rack).
 - b) Press the Remote Enable button so that its red light comes on (middle of the Lock-In Amplifier panel). [This allows the computer to control signal detection.]
 - c) The other buttons in the Lock-In Amplifier panel are not used.
- 5) Turn on the Thermocouple Sensors:
 - a) Set the toggle switch to On (right side of Thermocouple panel in the electronics rack). [Both thermocouples should read about 20°C after a few seconds. If either is flashing or shows a temperature of “---,” check the connections at the furnace or at the top of the sample rod, as appropriate.]
- 6) Turn on the Temperature Controller:
 - a) Set the toggle switch to On (right side of Temperature Controller panel in the electronics rack). [The temperature lock-in meter near the switch should move to “over temp.” The furnace voltage, as read from the left-hand meter in the lowest panel in the electronics rack, should be about 30 V; the furnace current, as read from the right-hand meter, should be 0 A.]
 - b) Check that the left-hand toggle switch is set to Computer Control.
- 7) Open the air valve on the right wall near the magnet power supply by turning it clockwise until you can hear and feel some air flowing from the tube near the sensors between the pole pieces of the magnet. [This keeps the sensors from getting too hot when the furnace is at high temperature.]

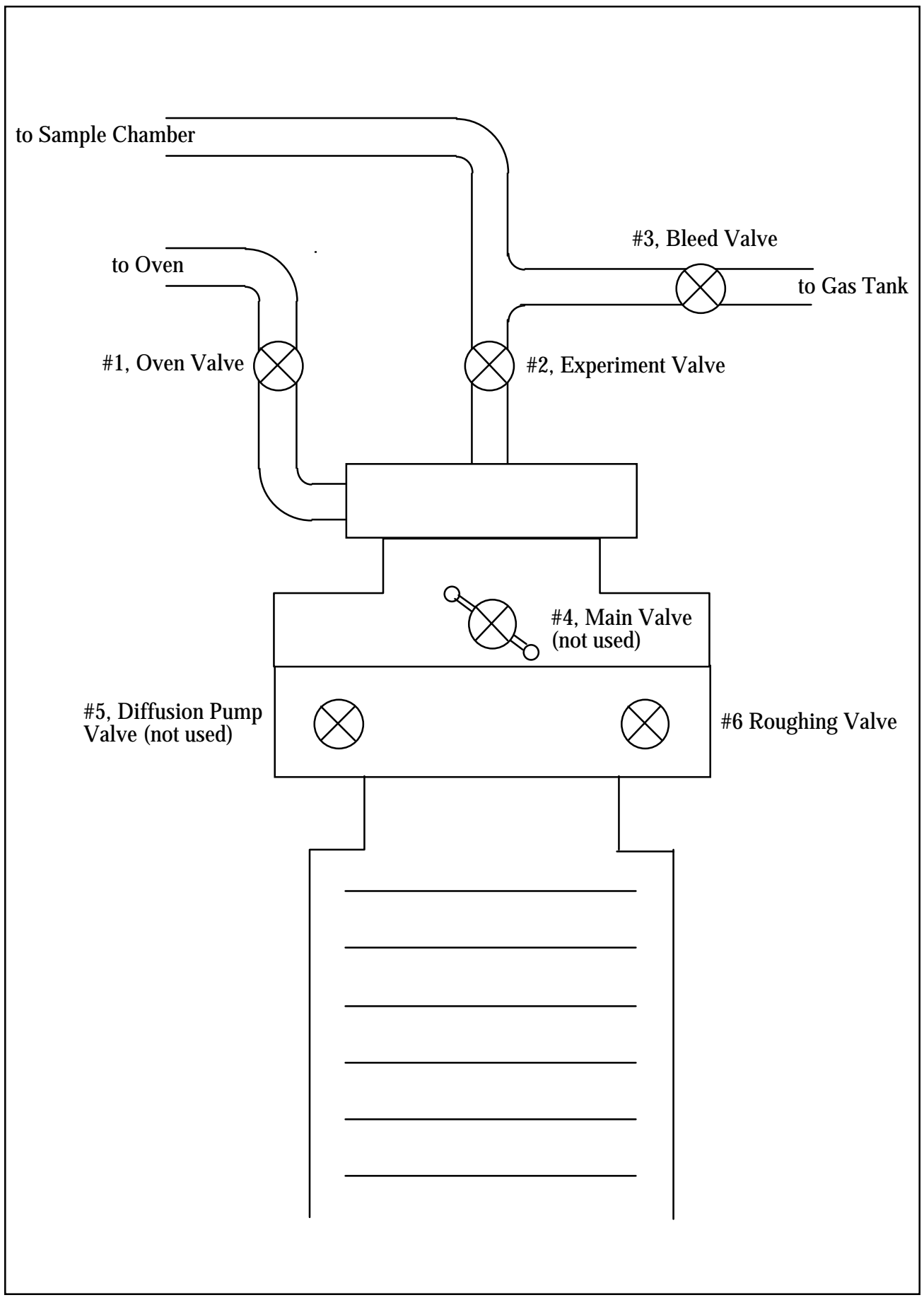
V. USING THE VACUUM SYSTEM

The goal is to get only inert gas into the sample chamber, and keep the furnace jacket under vacuum during the entire heating experiment. Refer to the schematic diagram on the next page and on the wall behind the vacuum system.

- ~~1) Close the Bleed Valve (#3) and make sure the other five vacuum system valves are also closed~~

[see schematic on the wall and below].

- 2) Turn on the mechanical vacuum pump:
 - a) Turn the Main Power switch to On (left side of the blue power box on the bottom of the pump cart).
 - b) Turn the Mech. Pump switch to On (middle of the blue power box on the bottom of the pump cart).
 - c) Wait for about 10–15 seconds until the pump settles down and stops gurgling, then open the Roughing Valve (#6).
- 3) Open the Experiment Valve (#2) to purge the sample chamber of gas. Again wait for 10–15 seconds until the pump settles down and stops gurgling.
- 4) Close the Experiment Valve (#2) and open the Bleed Valve (#3).



- 5) With the gas tank regulator valve closed (left side of regulator), crack the gas tank main valve to pressurize the regulator, and then close it again.
- 6) Open the gas tank regulator valve to bleed the gas into the sample space, and then close it again.
- 7) Repeat steps 5 and 6 twice for a total transfer of three regulators-full of gas.
- 8) Close the Bleed Valve (#3)
- 9) Repeat steps 3 through 8 twice for a total of three purges and gas fills. The final slug of gas stays in the sample chamber during the experiment to aid in heat transfer and prevent oxidation.
- 10) Open the Oven Valve (#1) to pump on the furnace jacket during the experiment.

VI. RUNNING THE CONTROL PROGRAM

- 1) Back at the computer, press any key to bring up the *Main Menu*. Type **curie** to obtain a thermomagnetic curve. The program first tests the magnet and communication with the Lock-In Amplifier.
- 2) At the prompts, enter the appropriate information about your sample:
 - a) Enter your sample name (all characters are OK).
 - b) Enter sample mass in mg.
 - c) Enter sample description (any 30 characters are OK).
 - d) Enter filename for data storage (8 letters and numbers, no extension). The program will not add an extension to files with the raw data, but it will add a .par or .dia extension to files with corrected data later.
 - e) Enter (separate by a space) maximum-temperature and field (mT). A good rule of thumb is to choose a temperature a bit higher than the highest Curie temperature you expect, and to choose the minimum field that will saturate the sample, e.g., **620<space>500<return>** for magnetite.
- 3) The program will then set up the Lock-In Amplifier (choosing sensitivity, phase, time constant, etc.). The phase (lower right meter in the Lock-In Amplifier panel) should read in the low thirties. If it does not, follow the steps for changing samples below, and check to make sure that the sample is securely mounted in the holder.
- 4) When the program prompts you, center the sample with the three translation screws near the head of the VSM so that you get a minimum reading on the Output meter (right side of the Lock-In Amplifier panel in the electronics rack) when adjusting the y direction (side-to-side: use the screw on the right), and maximum readings when adjusting the x direction (front-to-back: use the screw at the front) and the z direction (up-and-down: use the screw underneath the head). Press and key when satisfied.
- 5) Be sure that the soon-to-be-hot furnace does not touch the plastic-coated sensors in the pole pieces of the magnet. The furnace usually wants to be right up against the right hand sensors.

If necessary, you must sacrifice your centering to be sure you can see light between the furnace and the sensors.

- 6) The program will double-check the Lock-In Amplifier settings, prompt you to press any key to continue, ask you to "re-enter" the time for the Curie run (enter **30** minutes), and prompt you to press any key one last time to acquire data. Then it will run the heating experiment, plotting the data in real time as it goes.
- 7) If your sample falls off, or the loop is otherwise unsatisfactory, press **alt-s** to stop the program (and turn off the furnace).
- 8) If your sample has reached its Curie temperature (zero magnetization) before the furnace has reached its maximum temperature, you can turn the temperature control around in a controlled way by pressing **alt-r** to reverse it.
- 9) When the temperature returns to 30°C, the program will prompt you to press any key to return you to the *Main Menu*.

VII. PLOTTING YOUR DATA

- 1) Turn on the pen plotter on the table by pressing its power switch (rear-left button in bank of buttons). Align a piece of paper with the white line and press the paper-grabber button (front-left) to load the paper.
- 2) From the *Main Menu*, type **plot** to get to the *Plot Menu*. Then type **curie** to call up the plotting program.
- 3) When prompted for a filename, press **enter** for choices, or type in the filename you want. Your data should reappear plotted on the screen.
- 4) If you choose to fit lines to your data in order to calculate Curie temperatures, you will be asked a series of questions. Reversible loops will be fit with up to three lines (one at the heating and cooling inflection points just above the Curie temperature, and one to the paramagnetic tail above the Curie temperature). Irreversible curves can be fit for two Curie temperatures in the same way.
- 5) Following the prompts should yield a plot on the plotter.
- 6) Type **exit** to return to the *Main Menu*.
- 7) When the plotter finishes, remove the plot by pressing the paper-grabber button again.

VI. CHANGING SAMPLES

- 1) Disconnect the hoses between the gas tank and the Bleed Valve (#3).

- 2) Close the Oven Valve (#1) and Open the Bleed Valve to let air into the sample space.
- 4) Set the Vibration toggle switch to Off (middle of the Vibration Control panel).
- 5) Remove the VSM cap and disconnect the sample thermocouple leads.
- 6) Remove the sample rod by unscrewing the threaded end and pulling the sample rod straight up, again making sure all keyways pass on the way up.
- 7) Change your sample
- 8) Reload the sample rod, reattach the sample thermocouple leads, and replace the VSM cap as before.
- 9) Set the Vibration toggle switch to On (middle of the Vibration Control panel).
- 10) Go to section V above to measure another Curie temperature.

VII. INSTRUMENT SHUT DOWN

- 1) Disconnect the hoses between the gas tank and the Bleed Valve (#3).
- 2) Close the Oven Valve (#1) and Open the Bleed Valve to let air into the sample space.
- 3) Set the Vibration toggle switch to Off (middle of the Vibration Control panel).
- 4) Remove the VSM cap and disconnect the sample thermocouple leads.
- 5) Remove the sample rod by unscrewing the threaded end and pulling the sample rod straight up, again making sure all keyways pass on the way up. Remove your sample and hang the sample rod back up on the wall.
- 6) From the *Main Menu*, type **exit** to quit the program.
- 7) Turn off the Magnet:
 - a) Check that the magnet current is zero (meter in the middle of the Bipolar Power Supply panel).

Note: Never turn off the magnet when current is flowing (as read on the meter in the Bipolar Power Supply panel)—this blows fuses, blasts amplifiers, and otherwise causes headaches for the IRM staff!

- b) Set the Mode Control knob to Off (middle of the Bipolar Power Supply panel).
- c) Press the Off button (left side of the Bipolar Power Supply panel). [The “on” light should go off (left side of the Bipolar Power Supply panel) after one loud click from the magnet power supply.]
- d) Turn off the magnet main power switch (at the bottom left of the magnet power supply)

- tall rack in the corner to the right).
- e) Close the cooling water valve (just to the right of the table on the wall).
- 8) Turn off the Teslameter:
 - a) Set the toggle switch to Off (right side of the Teslameter panel in the electronics rack).
 - 9) Turn off the Vibration Control:
 - a) Set the toggle switch to On (right side of the Vibration Control panel in the electronics rack).
 - b) When the sample is in place, set the Vibration toggle switch to On (middle of the Vibration Control panel).
 - 10) Turn off the Lock-In Amplifier:
 - a) Set the toggle switch to Off (right side of the Lock-In Amplifier panel in the electronics rack).
 - 11) Turn off the Thermocouple Sensors:
 - a) Set the toggle switch to Off (right side of Thermocouple panel in the electronics rack).
 - 12) Turn off the Temperature Controller:
 - a) Set the toggle switch to Off (right side of Temperature Controller panel in the electronics rack).
 - 13) Close the air valve on the right wall near the magnet power supply.
 - 14) Turn off the computer: Type **bye** to park the heads, and then turn off the red switch on the power strip on the end of the table.
 - 15) Close the Roughing Valve (#6), and open both the Experiment Valve (#2) and the Oven Valve (#1) to let air into the furnace jacket.
 - 16) Turn off the mechanical vacuum pump by turning the Main Power switch to Off (left side of the blue power box on the bottom of the pump cart).
 - 17) Disconnect the furnace power supply and thermocouple lead wires from the furnace.
 - 18) Disconnect the vacuum system white plastic tubing from the furnace.
 - 19) Remove the furnace by unscrewing the threaded cap, and then gently pulling the furnace down until its O-ring seal releases. Hang it up on the wall.
 - 20) Replace the sample-rod guide by pushing it up until its O-ring seals. Then screw the threaded cap on until firm.
 - 21) Reload the normal sample rod into the VSM from the top, making sure all keyways pass on the way down. The threaded end of the rod should screw in many turns at the end, after the final keyway prevents the rod from rotating.

22) Replace the VSM head dust cap.

PART 3. HYSTERESIS LOOPS AT LOW TEMPERATURE

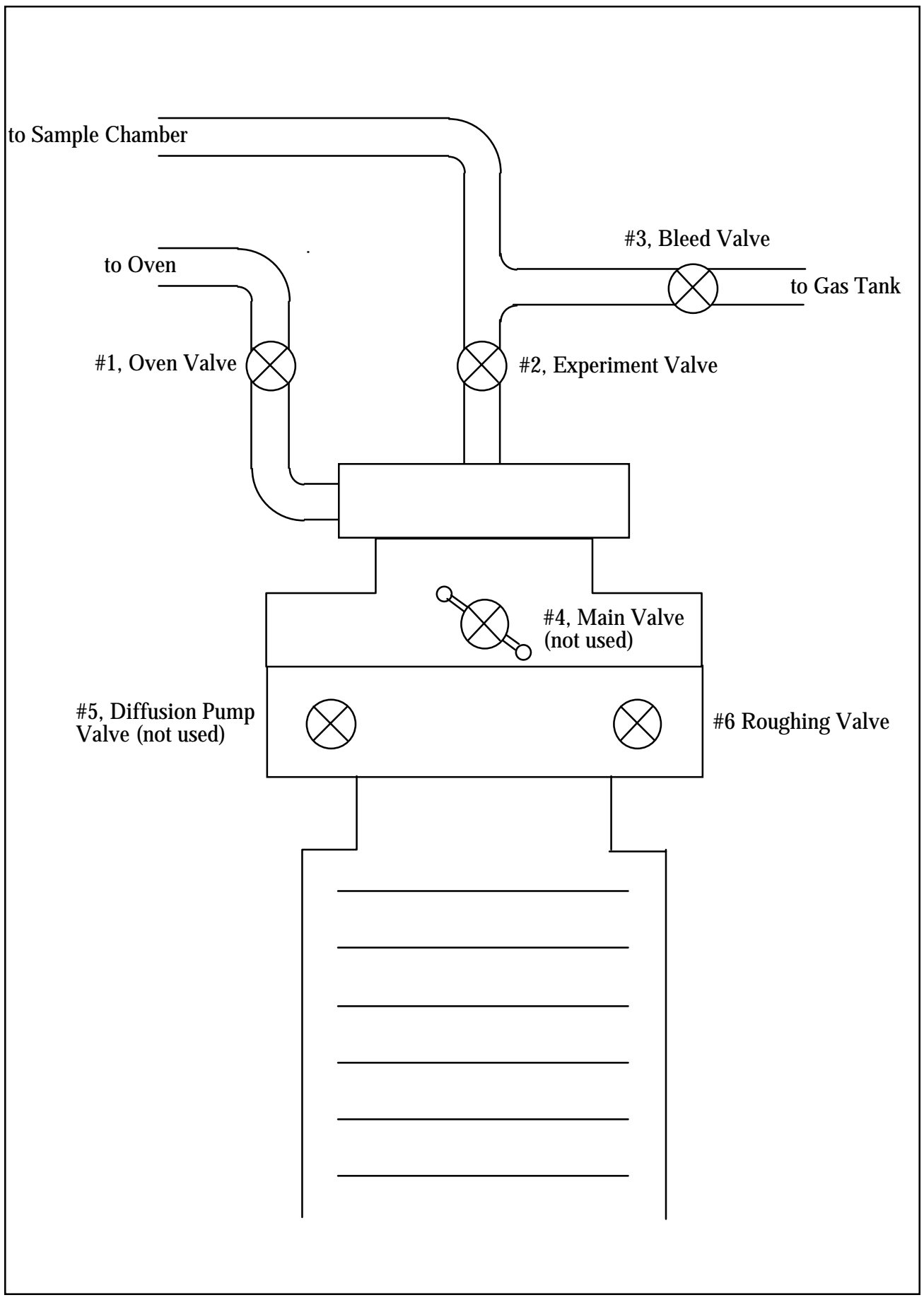
I. INSTRUMENT MODIFICATION

- 1) Remove the VSM head dust cap from the top of the VSM head.
- 2) Remove the sample rod by unscrewing its threaded end at the top of the VSM head, and pulling it all the way up and out.
- 3) Remove the sample-rod guide by unscrewing the threaded cap under the VSM head near the z-direction translation screw, and pulling the guide gently straight down until its O-ring seal releases. Hang it up on the wall also.
- 4) Attach the low-temperature dewar (in the cabinet above the sink) by guiding it between the pole pieces of the magnet, and then pushing it up until its O-ring seals. Then screw the threaded cap on until firm.
- 5) Attach the lead wires from the low-temperature controller to the mating connector on the dewar.
- 6) Attach the liquid nitrogen (LN₂) boiler lead wires from the power supply to the mating connector on the dewar.

II. PREPARING THE LN₂ DEWAR

- 1) Attach the white plastic tubing that comes from the vacuum system to the mating piece of copper tubing on the dewar. After they are made finger tight, the nuts should be given another quarter turn with the crescent wrenches (on the narrow shelf behind the VSM) to insure a good vacuum seal.
- 2) With all vacuum system valves closed [refer to the schematic diagram on the next page and on the wall behind the vacuum system], turn on the mechanical vacuum pump:
 - a) Turn the Main Power switch to On (left side of the blue power box on the bottom of the pump cart).
 - b) Turn the Mech. Pump switch to On (middle of the blue power box on the bottom of the pump cart).
 - c) Wait for about 10–15 seconds until the pump settles down and stops gurgling, then open the Roughing Valve (#6).
- 3) Open the “Oven Valve” (#1), which here leads to the dewar vacuum jacket, and then open the valve on the dewar.
- 4) After pumping on the dewar vacuum jacket for at least half an hour, close the dewar valve, the “Oven Valve” (#1), and the Roughing Valve (#6).
- 5) Turn off the mechanical vacuum pump by turning the Main Power switch to Off (left side of the blue power box on the bottom of the pump cart).

6) Disconnect the vacuum system white plastic tubing from the dewar.



- 7) Take the white LN₂ jug (often in the lower cabinets in room 242) downstairs to the LN₂ tank in the room next to the mail room (102) near the southwest building door. Record the tank weight on the log sheet, fill the jug by opening the valve on the tank [USE GLOVES!], and record the tank weight again. On the log sheet, also enter an appropriate billing number (see an *IRM* Staff member for a number if you don't have one already), and any other information requested.
- 8) Back at the VSM, remove both corks from the (vertical) ports on the top of the dewar, and put the plastic funnel (on the shelf with the dewar) into one of the ports. Seal the connection with masking tape.
- 9) Slowly pour LN₂ into the funnel to fill the dewar [USE GLOVES!]. You may need two jugs full. When LN₂ spurts out of the other port and out of the (horizontal) vent, the dewar is full.
- 10) When the ports are thawed, remove the funnel and replace the corks in both ports. Nitrogen vapor should be flowing gently from the vent.
- 11) Refill the dewar as necessary (at least once a day after initial cooling). Be sure there is LN₂ in the dewar whenever it is in use; otherwise, things burn up!

III. SAMPLE PREPARATION

- 1) Samples must fit into the plastic sample holder for the dewar (with the screw cap) which lives in the table drawer (< 4 mm in diameter, 1–2 cm long; usually about 100 mg).
 - a) Rock chips should be weighed on the balance which is on the counter near the door. They should then be wrapped in silver foil to aid thermal contact and to prevent contamination of the sample holder. Silver foil can be found near the balance.
 - b) Unconsolidated samples should be packed into silver foil pellets. Make a small cylindrical pellet wrapper out of the silver foil by wrapping the foil around the end of one of the tools in the plastic container near the balance. Fold the edges over to seal one end, remove the shell, and weigh it on the balance. Fill the shell with your sample, tamping often to get as tight a packing as possible. Seal the other end and weigh the full pellet. Finally subtract the mass of the empty shell from that of the full pellet to get your sample mass.
- 2) Pack your sample into the end of the plastic sample rod, and screw it on until snug. Use fiberfrax (spun alumina) from the table drawer as cushioning, if needed. Take time with this step to make sure your sample is very snug and secure, because thermal cycling and vibration try hard to loosen your sample and degrade your signal.
- 3) Load the sample rod into the VSM from the top, making sure all keyways pass on the way down. The threaded end of the rod should screw in many turns at the end, after the final keyway prevents the rod from rotating.

IV. COMPUTER SETUP

- 1) Turn on the red switch on the power strip on the end of the table to power up the computer and the control electronics
- 2) After the computer boots, get into your data directory:
type **guest** to get to the guest directory (C:\usr\guest)
or type **cd\usr\xxxx** where xxxx is the name of any other user directory.
- 3) Type **magsi** to start the control program. Follow the instructions on the screen for setting up the machine, which are also given below:

V. INSTRUMENT SETUP

- 1) Turn on the Magnet:
 - a) Open the cooling water valve (just to the right of the table on the wall).
 - b) Turn on the magnet main power switch (at the bottom left of the magnet power supply = tall rack in the corner to the right). [The orange “ready” light should come on (left side of the tan Bipolar Power Supply panel in the smaller rack to the right of the table).]
 - c) Make sure the Mode Control knob is set to Off (middle of the Bipolar Power Supply panel).
 - d) Press the On button (left side of the bipolar power supply panel). [The “on” light should come on (left side of the Bipolar Power Supply panel) after two loud clicks from the magnet power supply.]
 - e) Set the Mode Control knob to Remote. [This allows the computer to control the magnetic field.]
 - f) The other switches in the Bipolar Power Supply panel are not used.

Note: Never turn off the magnet when current is flowing (as read on the meter in the Bipolar Power Supply panel)—this blows fuses, blasts amplifiers, and otherwise causes headaches for the *IRM* staff!

- 2) Turn on the Teslameter:
 - a) Set the toggle switch to On (right side of the Teslameter panel in the electronics rack). [This allows the computer to measure the field at the Hall probe between the pole pieces of the magnet.]
 - b) Check that the left-hand knob in the panel is set to “measure,” and the right-hand knob is set to “computer control”.
- 3) Turn on the Vibration Control:
 - a) Set the toggle switch to On (right side of the Vibration Control panel in the electronics rack).
 - b) When the sample is in place, set the Vibration toggle switch to On (middle of the Vibration Control panel).
- 4) Turn on the Lock-In Amplifier:
 - a) Set the toggle switch to On (right side of the Lock-In Amplifier panel in the electronics rack).

- b) Press the Remote Enable button so that its red light comes on (middle of the Lock-In Amplifier panel). [This allows the computer to control signal detection.]
 - c) The other buttons in the Lock-In Amplifier panel are not used.
- 5) Turn on the LN₂ Boiler Power Supply
- a) Set the toggle switch to On (middle of Lambda Power Supply on the table).
 - b) Check that the voltage is set to 18 V maximum (left-hand knob) and that the current is limited to 3 A (right-hand knob).
 - c) Be sure there is LN₂ in the dewar when the boiler is on; otherwise things burn up.
- Turn off the LN₂ Boiler Power Supply when not in use!
- 6) Turn on the Temperature Controller
- a) Set the push button to On (right side of the Lakeshore Temperature Controller on the table). [The temperature should settle at about 77 K when the temperature is set below 77 K.]
 - b) Set the desired temperature with the thumb wheels (left side of the Lakeshore Temperature Controller). [Temperature settles at about 5 K lower than the set point.]
 - c) Set the Heater Power push button to Lo for the range 77–200 K; set it to Hi for the range 200–300 K.
 - d) Other switches and knobs are not used.

VI. RUNNING THE CONTROL PROGRAM

- 1) Back at the computer, press any key to bring up the *Main Menu*. Type **loop** to run a hysteresis loop. The program first tests the magnet and communication with the Lock-In Amplifier.
- 2) At the prompts, enter the appropriate information about your sample:
 - a) Enter your sample name (all characters are OK).
 - b) Enter sample mass in mg.
 - c) Enter sample description (any 30 characters are OK).
 - d) Enter filename for data storage (8 letters and numbers, no extension). The program will not add an extension to files with the raw data, but it will add a .par or .dia extension to files with corrected data later.
 - e) Enter h to H_{cr} measured. Entering **h** will make the VSM do a step-wise DC demagnetization of a saturation IRM after it finishes the loop. This procedure usually takes about 15 extra minutes. [*Recommended.*]
 - f) Enter i for initial suscept curve. Entering **i** will make the VSM start its loop at zero field, but requires that you demagnetize your sample after centering it below. [*Not recommended.*]
 - g) Enter the maximum field (in mTesla) for the loop.
- 3) The program will then set up the Lock-In Amplifier (choosing sensitivity, phase, time constant, etc.). The phase (lower right meter in the Lock-In Amplifier panel) should read in the low thirties. If it does not, check to make sure that the sample is securely mounted to the rod, and that the sample is not touching the magnet or the sensors.

- 4) When the program prompts you, center the sample with the three translation screws near the head of the VSM so that you get a minimum reading on the Output meter (right side of the Lock-In Amplifier panel in the electronics rack) when adjusting the y direction (side-to-side: use the screw on the right), and maximum readings when adjusting the x direction (front-to-back: use the screw at the front) and the z direction (up-and-down: use the screw underneath the head). Press any key when satisfied.
- 5) The program will double-check the Lock-In Amplifier settings, prompt you to press any key one last time, and then run the hysteresis loop, plotting the data in real time as it goes.
- 6) If your sample falls off, or the loop is otherwise unsatisfactory, press **alt-s** to stop the loop.
- 7) When finished, the program will ask if you want to make a paramagnetic or a diamagnetic correction to the data in order to remove the high-field slope. Entering **p** or **d** as appropriate will create a new datafile which contains the corrected data. It will be denoted with a .par or .dia extension, as appropriate. Pressing any other key will save only the uncorrected data. The program will then return you to the *Main Menu*.

VII. PLOTTING YOUR DATA

- 1) Turn on the pen plotter on the table by pressing its power switch (rear-left button in the bank of buttons). Align a piece of paper with the white line and press the paper-grabber button (left-middle) to load the paper.
- 2) From the *Main Menu*, type **plot** to get to the *Plot Menu*. Then type **loop** to call up the plotting program.
- 3) When prompted for a filename, press **enter** for choices, or type in the filename you want. The plotter should start plotting immediately. You can plot a second loop (the corrected data, for instance) on the same piece of paper by following the prompts.
- 4) Type **exit** to return to the *Main Menu*.
- 5) When the plotter finishes, remove the plot by pressing the paper-grabber button again.

VIII. CHANGING TEMPERATURES

- 1) Set the thumbs screws on the Lakeshore Temperature Controller to a new temperature. Allow time for system to reach a new thermal equilibrium. Then repeat sections VI and VII above to do another loop.
- 2) Repeat sections VI through VIII until finished with your sample.

IX. CHANGING SAMPLES

- 1) Set the Vibration toggle switch to Off (middle of the Vibration Control panel).
- 2) Remove the sample rod from the vertical shaft and change your sample.
- 3) Replace the sample rod on the vertical shaft.
- 4) Set the Vibration toggle switch to On (middle of the Vibration Control panel).
- 5) Repeat sections VI through IX above to measure another set of low-temperature loops.

X. INSTRUMENT SHUT DOWN

- 1) Set the Vibration toggle switch to Off (middle of the Vibration Control panel).
- 2) Remove the sample rod by unscrewing the threaded end and pulling the sample rod straight up, again making sure all keyways pass on the way up. Remove your sample.
- 3) From the *Main Menu*, type **exit** to quit the program.
- 4) Turn off the Magnet:
 - a) Check that the magnet current is zero (meter in the middle of the Bipolar Power Supply panel).

Note: Never turn off the magnet when current is flowing (as read on the meter in the Bipolar Power Supply panel)—this blows fuses, blasts amplifiers, and otherwise causes headaches for the IRM staff!

- b) Set the Mode Control knob to Off (middle of the Bipolar Power Supply panel).
 - c) Press the Off button (left side of the Bipolar Power Supply panel). [The “on” light should go off (left side of the Bipolar Power Supply panel) after one loud click from the magnet power supply.]
 - d) Turn off the magnet main power switch (at the bottom left of the magnet power supply = tall rack in the corner to the right).
 - e) Close the cooling water valve (just to the right of the table on the wall).
- 5) Turn off the Teslameter:
 - a) Set the toggle switch to Off (right side of the Teslameter panel in the electronics rack).
- 6) Turn off the Vibration Control:
 - a) Set the toggle switch to On (right side of the Vibration Control panel in the electronics rack).
 - b) When the sample is in place, set the Vibration toggle switch to On (middle of the Vibration Control panel).
- 7) Turn off the Lock-In Amplifier:
 - a) Set the toggle switch to Off (right side of the Lock-In Amplifier panel in the electronics rack).

- 8) Turn off the Thermocouple Sensors:
 - a) Set the toggle switch to Off (right side of Thermocouple panel in the electronics rack).
- 9) Turn off the LN₂ Boiler Power Supply by setting its toggle switch to Off (middle of Lambda Power Supply).
- 10) Turn off the Temperature Controller by setting its push button to Off (right side of the Lakeshore Temperature Controller on the table).
- 11) Turn off the computer: Type **bye** to park the heads, and then turn off the red switch on the power strip on the end of the table.
- 12) Disconnect the lead wires from the low-temperature controller from the dewar.
- 13) Disconnect the liquid nitrogen (LN₂) boiler lead wires from the power supply from the dewar.
- 14) Remove the dewar by unscrewing the threaded cap, and then gently pulling the dewar down until its O-ring seal releases. Empty it of any remaining LN₂ by turning it over to pour the LN₂ on the floor, being careful not to spill it on yourself. [USE GLOVES!]
- 15) Replace the sample-rod guide by pushing it up until its O-ring seals. Then screw the threaded cap on until firm.
- 16) Reload the sample rod into the VSM from the top, making sure all keyways pass on the way down. The threaded end of the rod should screw in many turns at the end, after the final keyway prevents the rod from rotating.
- 17) Replace the VSM head dust cap.
- 18) When the dewar has thawed, put it and the funnel back in the cabinet above the sink.

PART 4. HYSTERESIS LOOPS AT HIGH TEMPERATURE

Alternating Gradient Force Magnetometer (MicroMag)

[Princeton Measurements]—Room 242 (*MicroMag*)

sensitivity 1×10^{-11} A m² [~10 min/sample for hysteresis loops (= 100 mg chip or powder)]

See *IRM Quarterly*, Spring 1992, Vol. 2, No. 1

See *IRM Quarterly*, Fall 1993, Vol. 3, No. 3

See *IRM Quarterly*, Spring 1994, Vol. 4, No. 1 (magnetic quantities and units)

The MicroMag consists of a magnet/electronics/power supply/sample assembly, a computer, a pen plotter, three sample holders in a box, a sample-holder tray, and an ultrasonic bath, various small tools, supplies, and solvents (see Chemical Hygiene Plan for proper use of chemicals).

I. SAMPLE PREPARATION

For routine measurements, use one of the plastic sample holders. The sample-holder glass rods are extremely delicate—please check with IRM staff before using.

- 1) Prepare the sample for measurement: Small rock chips can be mounted as is; powders can be mixed with vaseline and put into a sandwich of plastic wrap.
- 2) Place the sample-holder in the tray. Clean the sample-holder with isopropyl alcohol or solvent in the ultrasonic bath, bracing the sample-holder on the lip.
- 3) Then put a small amount of vaseline (or vacuum grease for really big samples that tend to droop) on the plate surface with a toothpick.
- 4) Affix the sample to the sample-holder with tweezers using the vaseline as an adhesive.

II. INSTRUMENT PREPARATION

- 1) Turn on the master switch (on the power strip on the desk) to power up the computer, the cooling water, and the controller power supply. Wait for the computer to finish booting and to begin the control program. When the initial control program screen appears, turn on the Applied Magnetics magnet power supply switch on the panel below the magnet [lower left toggle].

Note: The upper right toggle (controller power supply) should always be left on. The other small toggles on the magnet power supply panel should always be set to: run, normal, remote, and amps. The coarse and fine adjustment knobs are not used.

- 2) With the stage and sample-holder locks locked, attach the sample-holder to the stage by aligning the pins (white dot out), and pushing up firmly until the pins are seated. Secure the sample-holder with the velcro tab.
- 3) Unlock the sample-holder by pulling out the sample-holder lock so that the holder swings freely (square knob at the front of the sample-holder). Steady it with your fingers at the four corners of the assembly if necessary.
- 4) With your thumb under the stage to provide support, unlock the stage by setting the left-side level to the down position, and slide the stage down gently, watching to be sure of sufficient clearance, until the sample is approximately centered between the gradient coils.

III. COMPUTER SETUP

The computer should come up in the MicroMag program. If not, from DOS, type **measure** to start the program. In general, use the **arrow** keys to select an item, press **enter** to move down a menu level, and **escape** to move up a menu level. To alter settings, select an item, press **enter**, type the new value, then press **enter** again to register the change.

- 1) From the *Main Menu*, select the *AGM Measurements* menu.
- 2) Select *Autofunctions* and press **F1** for an initial, coarse tuning. When this finishes, in about a minute, you should have a resonant frequency between about 200 and 500 Hz, phase angle in the -80 s degrees, and a Q of several tens (the bigger the better). If it is OK, press *Enter* to accept the values, and then *Escape* to return to the *AGM Measurements* menu. Among other things, this procedure sets the sensitivity based on the magnetization at the maximum field value.
- 3) Select *Optimize* to electronically center the sample. Adjust the position of the sample with the three translation screws so that you get a minimum reading in the x direction (side-to-side), and maximum readings in the y and z directions (front-to-back and up-and-down, respectively). Press *Escape* when finished to return to the *AGM Measurements* menu. If the green bar goes off scale, press **F1** to reset it to zero.
- 4) Just before starting your measurements, select *Autotune* again, and press **F2** to make an incremental, fine tuning. Once again, if it is OK, press *Enter* to accept the values, and then *Escape* to return to the *AGM Measurements* menu.

IV. DAILY CALIBRATION

The MicroMag should be calibrated against the Ni standard at the beginning of each day it is used.

- 1) Mount the nickel standard on the sample-holder and follow the steps outlined in sections I through III above.
- 2) Under the *AGM Measurements* menu, select *Magnetization Curves*. Press **F2** to do a single-scan full hysteresis loop.
- 3) When this is done in about a minute, press **F5** and then **enter** to adjust the high-field slope to zero.
- 4) Then press **F6** to set the probe factor. Enter $378e-9$ ($A\ m^2$) in SI units, or $378e-6$ (emu) in cgs units, as the known saturation moment of the nickel standard.
- 5) Press **escape** twice (to get to the *Main Menu*), then select *Save/Retrieve Configuration* and press **F1** to save the probe factor settings for the day.
- 6) Remove the nickel standard, following the steps in section VII.

VI. TYPICAL FULL HYSTERESIS LOOP MEASUREMENT

- 1) Mount your sample on the sample-holder and follow the steps outlined in sections I through III above.
- 2) ~~In the *AGM Measurements* menu, select *Remanence* to measure H_{cr} . Note: If you do not know~~

what H_{cr} to expect, try 50 mT maximum field with a spacing of 10 mT as a rough guess. Then redo the procedure with a reasonable maximum field (i.e., $\sim 2H_{cr}$) and a spacing that gives you 10 or 20 points for accuracy.

- a) Enter an appropriate saturating field.
 - b) Enter a maximum field that is greater than the expected H_{cr} .
 - c) Enter a spacing that gives you 10 or 20 points.
 - d) Select *DC Demagnetization Remanence* to determine H_{cr} .
 - e) If the result is indeterminate, increase the maximum field until the remanence curve crosses the axis. (You may wish to change the sensitivity back in *AGM Measurements* menu for better results.)
 - f) Record the value of H_{cr} by hand to be entered in the loop description later.
 - g) Press escape to return to the *AGM Measurements* menu.
- 2) Under the *AGM Measurements* menu, select *Magnetization Curves*. Press **F2** to do a single-scan full hysteresis loop.
 - 3) When this is done in about a minute, press **F5** and then **enter** to adjust the high-field slope to zero. This insures that the values for loops parameters are accurate.
 - 4) Manipulate your data with the various function keys:
 - a) Press **F1** to add a sample description. Include the value of H_{cr} recorded earlier.
 - b) Press **F7** to expand the loop, if necessary.
 - c) Press **space** and **F1** to subtract a previously stored loop from the data, if desired.
 - d) Normalize your data by mass, volume, or saturation moment by pressing **space** and **F4**, **F5**, or **F6** respectively.
 - e) The raw data can always be recovered by pressing **F9**.
 - f) Etc.
 - 5) When satisfied, press **F2** to save your data by entering `\usr\guest\filename`, for example.

VI. PLOTTING YOUR DATA

- 1) Turn on the pen plotter on the table by pressing its power switch (rear-left button in the bank of buttons). Align a piece of paper with the white line and press the paper-grabber button (left-middle) to load the paper.
- 2) From the *Magnetization Curves* menu with your processed data on the screen, type **F3** (plot) and then **F1** (Plot All). You can leave the paper in place (if you wish to overlay subsequent plots), or press **F6** to eject the page.
- 4) Press **escape** to return to the *AGM Measurements* menu.
- 5) When the plotter finishes, remove the plot by pressing the paper-grabber button again.

VII. SAMPLE REMOVAL

- 1) Leaving the sample-holder swinging freely, support the stage as you unlock the stage. Raise the stage and gently lock it in the up position. Lock the sample-holder, and remove the sample-holder by grasping it near the top [use both hands!] and pushing up against the stage with your thumbs to ease the pins out. Be careful not to snap off the glass rods when the pins come out!
- 2) Place the sample-holder in the tray and remove the sample with tweezers.
- 3) Repeat sections V through VII until finished.

VIII. SHUTTING DOWN

- 1) Put the sample-holder into its case and close it.
- 2) Press **escape** enough times to get to the *Main Menu*.
- 3) Type **alt-x** to quit the MicroMag program
- 4) Turn off the power strip switch on the wall.
- 5) Shut off the magnet power supply with its toggle switch.

Superconducting Susceptometer (MPMS)

[Quantum Design]—Room 242 (MPMS)

sensitivity 1×10^{-11} A m²; applied fields from 0 T to 5 T; temperatures from 2 K to 400 K [~1 hour/sample minimum depending on measurement program (= 200 mg chip or powder)]

See *IRM Quarterly*, Spring 1992, Vol. 2, No. 1

See *IRM Quarterly*, Fall 1993, Vol. 3, No. 3

See *IRM Quarterly*, Spring 1994, Vol. 4, No. 1 (magnetic quantities and units)

The MPMS consists of a instrument station, a control/electronics rack/station, a computer, a ThinkJet printer, a sample holder rod, various small tools, and supplies

I. SAMPLE PREPARATION

- 1) Prepare the sample for the drinking straw holder:
 - a) Gel-cap method: First, put in some fiberfrax or bits of kimwipe in the ends of a gel-cap (to keep the sample snug). After placing the sample in the gel-cap and closing it until it snaps shut, seal the side of the gel-cap with enough Capton tape to make a snug fit in the drinking straw.
 - b) Second straw method: First, place the sample chip in a short piece of drinking straw. Then, insert this sideways into the main straw. Recommended only for single chips.
- 2) Position the sample in the straw so that it is about one-third of the way up from the bottom.

- 3) Prevent the sample from falling out of the straw by placing a small piece of Capton tape over the bottom of the straw. Then, make a hole in the tape with a paper clip to allow air to pass. Make small holes in the side of the drinking straw above the sample to allow air to pass.
- 4) Slide the drinking straw onto the sample rod over the tape wad to fit snugly. Tape the straw to the tape wad. Slide the sheath down to cover the straw.
- 5) With the bracket hanging over the edge of the table but up against it, loosen the knurled knob and move the sample rod until your sample is centered on the mark on the table, then tighten the knurled knob again. This aids in the sample centering procedure later.

II. SAMPLE INSTALLATION

- 1) With the blue plug in, crack the sample chamber valve and close it again.
- 2) Loosen the sample locks and remove the blue plug.
- 3) Insert the prepared sample rod assembly with the white dot facing forward, and tighten the sample locks.
- 4) Push the “purge air lock” button. The “venting” light will go on and off for about 30 seconds. Watch the sample through the window to make sure it does not move* or fall out of the straw**.
 - *If the sample moves only a little (<few mm), it is still OK. If it moves up or down too much, it will not be possible to center it. In this case, wait for the green “ready” light, open the sample chamber valve, then close it again, loosen the sample locks, and remove the sample rod assembly. Adjust the sample in your straw (using more tape for a snugger fit), and start again at step 3.
 - **If the sample falls out of the straw, remove the sample rod assembly as above (*), then refer to the MPMS manual for how to retrieve wayward samples.
- 5) When the green “ready” light comes on, open the sample chamber valve.
- 6) Push the sample rod assembly down, fitting the bracket under the thumb screws. Tighten the thumb screws.

III. SAMPLE CENTERING

- 1) Go to the *Sample Register* menu [F2] and make sure the *Auto Sample Tracking* is on.
- 2) Run the *Initialize Sample Transport* procedure to prepare for auto tracking.
- 3) Run the *Perform DC Centering Scan* procedure. A SQUID response curve will be displayed in about 30 seconds. The response curve should be symmetric with the large central bump (up for positive fields or moments) centered on the plot. See example on the wall.

- 4) Select *Adjust Sample Position* to verify that the *Sample Location* is at the middle of your scan (3.00 cm for 6-cm scans, 2.00 cm for 4-cm scans). If it is not, run the *Adjust Position Automatically* procedure, or enter the position of the measured peak in the SQUID response curve. If the sample is too far away from being centered, you will have to run the *Adjust Position Manually* procedure, which involves loosening and tightening the knurled nut on the sample rod.
- 5) Rerun the *Perform DC Centering Scan* procedure to make sure your sample is really centered.

IV. RUNNING A LOW-TEMPERATURE SIRM THERMAL DEMAGNETIZATION

- 1) In the *Register Sample* menu [F2], enter sample, name, mass, etc. for the header of the data file.
- 2) In the *Set Parameters* menu [F4], choose *Data File Name*, and enter your **filename**. Also verify that the *Magnet Charging Mode* is set to “No Overshoot.”
- 3) In the *Editor/File Control* menu [F9], choose *File Control*, and the *Data Directory*, and enter **c:\usr\guest**. Then press **escape** once.
- 4) In the *Sequence Control* menu [F8], choose *Sequence File Name*, and then select *SIRMWARM*. The following should now appear on the screen:

Sequence Type	programmed
Sequence File Name	sirmwarm
Start at Record #	1
- 5) Select *Execute Sequence*, and press **enter** to start the experiment.
- 6) In the *Graph* menu [F5], choose *Display Graph* and then *EMU vs. Temp* to view your data as it is taken. Choose *Modify Graph* to adjust the plot parameters.
- 7) For other experiments or sequences, see an *IRM* Staff member for help.

V. SAMPLE REMOVAL

- 1) Loosen the thumb screws and pull the sample rod assembly up until the sample is visible in the window.
- 2) Close the sample chamber valve. The “venting” light will go on for a few seconds.
- 3) When the light goes out, loosen the sample locks and remove the sample rod assembly.
- 4) Replace the blue plug and tighten the sample locks.

VI. HELIUM TRANSFER

⊗ This is a two-person job ♦

- 1) Call Ron Fremstad (x6-2263) in Physics room 77 to reserve a dewar of liquid helium (about 60 liters).
- 2) Collect the following near the MPMS:
 - liquid helium dewar (from Physics)
 - dewar exit valve adapter (in the top left drawer behind the door)
 - helium gas tank (in the corner behind the door)
 - helium transfer tube (on the back wall)
 - two pair of gloves (in the cabinets)
 - heat gun (in the cabinets).
- 3) Attach the dewar exit valve adapter to the dewar. Purge both the hose from the helium gas tank and the connector to the dewar with helium gas, then connect the hose to the dewar. Set the regulator for about 1 psi, but leave the inlet valve on the dewar closed.
- 4) Bleed the 0.5 psi relief valve on the dewar, then open the valve at the top of the dewar. Insert the long end of the transfer tube, making sure to seat the outer sleeve of the tube in the dewar adapter. Push in slowly.
- 5) Close the 0.5 psi relief valve and open the inlet valve to start gas flow into dewar.
- 6) Bleed off the pressure in the MPMS relief valve, then remove the cap when ready to insert the short end of the transfer tube [See next step].
- 7) When the sound of the gas plume coming out of the transfer tube starts to change (indicating liquid is nearing), insert the short end of the transfer tube into the MPMS. Do this quickly to avoid freeze-up, but with finesse to negotiate around all the internal baffles. Again make sure that the outer sleeve of the transfer tube seats in the connector to the MPMS.
- 8) Adjust the regulator to get about 1 psi for transfer pressure.
- 9) In the *Graph* menu [F5], plot He-level vs. time to watch the transfer proceed. The whole transfer should take about 20 minutes.
- 10) Set up the heat gun so that it thaws the connector to the MPMS
- 11) Close the regulator when the MPMS is about 95% full. Pull the transfer tube out of the MPMS when the helium-level jumps up to 98% full. CAUTION! The tube is dangerously cold! USE GLOVES! Pull the other end of the transfer tube out of the dewar. USE GLOVES!
- 12) Replace the cap on the MPMS. Close the inlet valve on the dewar and open the 0.5 psi relief valve. Measure the liquid helium left in the dewar if possible, then close the dewar exit valve, and remove the adapter
- 13) Stop the graph, and verify that the He-level meter is now off under the “diagnostics” menu [F7] in “gas controls.” ~~The gas flow on the MPMS meters should drop back to below 100 cc/min~~

in a few minutes.

14) Put everything away, and return the dewar to Physics room 77.

Magneto-Optic Kerr Effect (MOKE) System

[Leitz, Hamamatsu, Sony, and Buehler]—Room 298 (*MOKE*)

applied fields from 0 T to 0.1 T; image manipulating software; sample preparation facilities [~4 hours/sample (5 cm² polished grains or thin sections)]

See *IRM Quarterly*, Summer 1992, Vol. 2, No. 2

See *IRM Quarterly*, Winter 1993–94, Vol. 3, No. 4

See *IRM Quarterly*, Spring 1994, Vol. 4, No. 1 (magnetic quantities and units)

The MOKE consists of a microscope assembly with an attached video camera, a stage magnet, digital image-processing electronics, a DeskJet printer, and a computer.

Magnetic Force Microscope (MFM)

[Digital Instruments]—Room 298 (*MFM*)

magnetic and topographic resolution ~20 nm [several hours/sample (1 cm² polished single grains, thin sections, or thin films)]

See *IRM Quarterly*, Winter 1992–93, Vol. 2, No. 4

See *IRM Quarterly*, Winter 1993–94, Vol. 3, No. 4

See *IRM Quarterly*, Spring 1994, Vol. 4, No. 1 (magnetic quantities and units)

The MFM consists of a microscope assembly with an attached video camera, digital image-processing electronics, external magnetic field coils, and a computer.

Mössbauer Spectrometer

[Ranger Scientific]—Room 256 (*Moss*)

temperatures from 2 K to ambient; zero applied field (external field up to 5 T not yet available)

[several hours/sample (= 200 mg chip or powder)]

See *IRM Quarterly*, Fall 1992, Vol. 2, No. 3

See *IRM Quarterly*, Winter 1993–94, Vol. 3, No. 4

See *IRM Quarterly*, Spring 1994, Vol. 4, No. 1 (magnetic quantities and units)

Authorized Personnel Only!

**Only people who have completed a Radiation Safety Course
are permitted in the Mössbauer Lab**

Film badges must be worn at all times in the Mössbauer Lab!

The Mössbauer consists of a lead-shielded spectrometer, a sample rod, electronics boxes, and a computer.

I. SAMPLE PREPARATION

- 1) The standard sample holder for the Mössbauer is a small disk with a 12.7 ± 0.1 mm well for the sample. Although the diameter is fixed at 12.7 mm the thickness of the sample can be up to about 0.5 cm, however, thicker samples can cause problems by being a “shield” between the source and detector. There is a program that can be used to calculate the optimum thickness of a sample given its iron content and diameter. If the sample fills the sample holder than a piece of cryo tape is required to keep the sample from falling out. If the sample needs to be fixed in place than either double faced tape or evaporating acetone off the sample can be used. Other methods of adhesion can be used as long as they are tested and don't interfere with the signal. Powdered samples can be dispersed in Boron Nitride Calcium Carbonate or petroleum jelly if necessary.

II. COMPUTER SET-UP

- 1) Turn on the computer and spectrometer with the orange switch on the power strip.
- 2) Type **mr**un to start the control program.
- 3) Get into the *Waveform Setup* menu by typing **W**.
- 4) Use a waveform saved to the disk by typing **L**. Choose from :
 - T158A.WAV for velocity scale of 15.8 mm/sec
 - T100A.WAV for velocity scale of 10.0 mm/sec
 - T600A.WAV for velocity scale of 6.0 mm/sec
- 5) Send the setup to half 1 of the MS-1200.
- 6) Turn the grabbing on (**G**) and zero the memory (**Z**).
- 7) Type **512** to show the center channel.
- 8) On the Spectrometer box push the PHA (pulse height analysis) button. After a few seconds of counting, a peak should appear which needs to be centered on the 512 channel. Adjust the gain pot on the spectrometer box to center the channel. Zero the memory (**Z**) and wait for the peak to appear. Repeat this until the peak is centered on the 512 channel. (Note: the display scale can be changed using the up and down arrows.)
- 9) Push the MB (Mössbauer) button on the spectrometer box and zero the memory (**Z**). You are now ready to collect data.

III. LOADING SAMPLES

- 1) In the copper sample holder, place the plastic sample holder in the well on the removable piece. Sandwich the sample between the removable copper piece and the piece fixed on the end of the rod. Use 2 bolts on a diagonal to secure pieces together. Carefully, not touching the copper wire wound around the rod, insert the sample into the sample chamber with the black marks on the top of the sample rod and the sample chamber lined up. Slowly let it down until the sample rod is all the way in and seated on the seal.
- 2) On the computer zero the memory (**Z**) and save (**S**). Use \samples*(filename)*. The necessary .ADT extension will be added.

IV. SHUT DOWN

Type **Q**, answer **Yes**. When you see the C:\> prompt, shut off the power by turning the switch on the power strip off.

Mössbauer Spectrometer Radiation Safety

Early work on radioactivity was done by Marie Curie (1867–1935); ionizing radiation (x-rays [machine-produced] or γ -rays [natural]) was investigated by Wilhelm Konrad Röntgen (1845–1923). These people therefore got units named after them. To wit:

quantity	units	definition
radiation	roentgen [>W. K. Röntgen]	amount of ionizing radiation which produces 1 esu of electric charge (positive or negative) in 0.001293 gm (1.000 cm ³) of dry air at 0°C and 760 mm Hg (STP).
dose	rad [>radiation]	amount of energy absorbed per unit mass, with 1 rad = 100 ergs gm ⁻¹ [10 ⁻² J kg ⁻¹].
adjusted dose	rem (R) [>roentgen equivalent in man]	biological effect equivalent to 1 roentgen of x-rays or γ rays. The (somewhat-arbitrarily-defined) limit is 500 mR yr ⁻¹ with no more than 100 mR yr ⁻¹ of this from class-related work. Nevertheless, the ALAP (as low as possible) principle applies!
activity, A	Curie (Ci) [>M. Curie]	number of nuclear disintegrations per unit time, with 1 Ci = 3.70 \times 10 ¹⁰ disint sec ⁻¹ .
halflife, τ	second (sec)	time required for half of a population of radioactive nuclei to decay, as given by $N(t) = N_0 e^{-t/\tau}$, where $N(t)$ is the number of nuclei after time t , and N_0 is the original number of nuclei.
intensity, I	watts/square meter (W m ⁻²)	power (energy per time) absorbed per unit area, as given by $I = P/4\pi r^2$, where P is the power produced by the source and r is the distance from the source.

Ionizing radiation comes in three forms: alpha (α) particles, which are helium nuclei [⁴He²⁺]; beta (β) particles, which are high-energy electrons [e⁻] or positrons [e⁺]; and gamma (γ) rays, which are high-energy photons [electromagnetic waves or light]. For α -particles and β -particles, the energy E is given by $E^2 = m_0^2 c^4 + p^2 c^2$, where m_0 is the rest mass of the particle, c is the speed of light, and p is the relativistic momentum of the particle. For γ -rays, the energy E is given by $E = h\nu$, where h is Planck's constant [= 6.63 \times 10⁻³⁴ J s], and ν is the frequency of the photon.

Our Mössbauer spectrometer uses sealed ⁵⁷Co radioactive sources which initially have an activity of 50 mCi. ⁵⁷Co has a halflife of 270 days, and produces β particle and γ ray radiation. The ⁵⁷Co

source undergoes β -decay to an excited form of ^{57}Fe , which then relaxes by emitting γ -rays of various energies (primarily at 122.1 keV, 136.5 keV, and 14.4 keV, the last being the “Mössbauer” γ -ray). An unshielded 50-mCi ^{57}Co source would produce an exposure rate of about 36 mR hr⁻¹ at a distance of one foot, or about 1 mR hr⁻¹ at the computer work station 6 feet away. But both the β -particles and the γ -rays are effectively stopped [i.e., their number is reduced by a factor of at least 5 or 10] by about 1/16” of lead shielding. The shielding found surrounding the source in the spectrometer (three layers at 1/4” each???) or in the corner of the room where extra or used sources are temporarily stored (2”) is thus considered to be ample, and Geiger-counter surveys have always shown only background levels in room 256, except right at the machine where they average twice???) the background. Nevertheless, caution and care should always be taken when using this instrument!

Film badges must be worn at all times in the Mössbauer lab!

DATA MANIPULATION

Introduction

Instruments control-computers are all DOS machines. They are turned on with either a power strip switch or a switch on the computer. Entries are typed in (except when using Windows on the *MOKE* or *292plot* computers where a mouse can be used), and are generally registered by pressing **return** or **enter**, which will not be noted. The **escape** key usually will get you out of something you don't want to do. Control-break will get you out of most programs. ****As a last resort**** In case of lock-up, push the **reset** button or press **control-alternate-delete** to reboot the computer. Do not simply turn off and turn on the computer; it's too brutal. Type **bye** to park the heads before turning off a computer with a switch. This utility is not needed on the newer computers, but it's safer to try anyway—it can't hurt.

Minimum computer (IBM or Mac) skills:

- how to safely turn on a computer
- how to type in data from a keyboard
- how to press **return** or **enter** to register entries
- how to use the **escape** key
- how to use the arrow keys to move around menus and select items
- how to use the reset button (IBM or Mac) or **control-alternate-delete** (IBM) to reboot
- how to safely turn off a computer

Minimum DOS commands:

dir	directory	lists the contents of a directory
dir/p	directory/page	lists the contents of a directory one screenful at a time
dir/w	directory/wide	lists the contents of a directory in wide view format
cd	change directory	changes the current directory
copy	copy	copies files
del	delete	deletes the filename
mkdir	make directory	creates a new directory
rmdir	remove directory	erases an empty directory
format	format	formats a new disk for DOS

Handy DOS utilities:

dr		sorts and views directories and files
co		copies files in chunks
win	windows	runs Windows on the <i>MOKE</i> or <i>292plot</i> computers

Minimum Mac or Windows skills:

- how to point with the mouse
- how to click with the mouse to select items
- how to double-click to open or start files, folders, or applications
- how to drag to move or copy files

how to delete with the Trash on the Mac
how to format disks for the Mac

If any of these things is unfamiliar, get help from the *IRM* staff.

Using the IBM computer network

Turn on any IBM computers you need to get to using the Lantastic network. Most computers come up with the network software loaded. If it doesn't load, type **startnet** to do so. As an example, log on to the *Gateway* computer from any other computer:

From any IBM computer, type **net**. [If you get an error message saying xxxx is not loaded, type **startnet** to load the network software.] Then follow the following steps:

- 1) logon name = **user**, password = press **return**, clock set = press **return**
- 2) At the prompt select **network drives and printers** with the up/down arrow keys, and press **return**
- 3) choose a drive to be redirected to Gateway, e.g., **G:**
- 4) select **Gate** from the list, and press **return**
- 5) Choose the **C:-drive**, and press **return**
- 6) Press **escape** twice to get back to the DOS prompt
- 7) The (virtual) **G:-drive** on the computer you are using is now the (physical) **C:-drive** on the *Gateway* computer.

Moving and Translating files from the IBM machines to the Macs

- 1) The *Gateway* computer in room 288–290 should be on and “Running...” in Lantastic for PC/Mac. If it is not, follow the instructions taped to the computer. If that fails, find someone to help.
 - 2) Turn on the *Main* computer in room 288–290
 - 3) Log on to the *Gateway* computer by redirecting drive G:, for instance [see using the IBM computer network]
 - 4) Turn on and log onto any other IBM computers you need to get to
 - 5) Copy data to be transferred into the C:\Transfer directory on the *Gateway* computer
 - 6) On the Mac, open the *IRMGATE* icon and copy the files by dragging them to the desired destination
 - 7) To translate the files to Mac format (for KaleidaGraph 3.0, e.g.), open the IRM Chris icon. (Excel 4.0 can then read the IBM files directly [Select open from within Excel].)
 - 8) Open the Converters folder
 - 9) Click on **Maclink/Desktop** (which opens MacLink Plus/PC with the appropriate settings).
These settings are:
 - 1) Set Mode: MacLink
 - 2) Set Communications: N/A
 - 3) Set Translators: [IBM] Text to [Mac] Text
 - 10) Under **Select Files**, find your IBM files with **Drive** and **Open**, and set your destination on the Mac
 - 11) Click on **Convert**
 - 12) Click on **Done** when finished
 - 13) Clean up the Transfer directory on Gateway by putting files in the trash (Mac) or by deleting them over the IBM network from Main (for instance, C:\> **del G:\transfer\filename**, or G:\transfer\> **del filename**)
- To be polite, cancel the redirected drives on the *Main* computer by typing **net....**

Exporting IBM Quattro files into Kaleidagraph (or any other Mac application)

In Quattro on the IBM you will save your spreadsheet as a plain text file:

Temporarily change the printer:

Options

Hardware

Printers

Default

2nd Printer (should be Epson 800/850/950)

Select the portion of spreadsheet you want to use:

Print

Block A1..H10 [for example]

Adjust some details:

Print

Layout

Break Pages

No

Setup String

(none)

Save the selected portion of the spreadsheet to a new filename:

Print

Destination

File

C:\>Newname [for example]

Spreadsheet Print

Your Quattro file is now a text file in IBM format.

On the Mac, follow the instructions for grabbing/translating files from the IBM computers.

APPENDICES

Appendix 1. Units and Conversions

term	symbol	SI units	cgs units	conversion
magnetic induction	B	(T)	(G)	$1 \text{ (T)} = 10^4 \text{ (G)}$
magnetic field strength	H	(A m ⁻¹)	(Oe)	$1 \text{ (A m}^{-1}\text{)} =$
magnetic moment	m	(A m ²)	(emu)	$1 \text{ (A m}^2\text{)} = 10^3 \text{ (emu)}$
volume magnetization	M	(A m ⁻¹)	(emu cm ⁻³)	$1 \text{ (A m}^{-1}\text{)} = 10^{-3} \text{ (emu cm}^3\text{)}$
mass magnetization	J or σ	(A m ² kg ⁻¹)	(emu g ⁻¹)	$1 \text{ (A m}^2 \text{ kg}^{-1}\text{)} = 1 \text{ (emu g}^{-1}\text{)}$
volume susceptibility	k	(dimensionless)	(emu Oe ⁻¹ cm ⁻³)*	$1 \text{ (SI)} =$
mass susceptibility	χ	(m ³ kg ⁻¹)	(emu Oe ⁻¹ g ⁻¹)	$1 \text{ (m}^3 \text{ kg}^{-1}\text{)} =$

*This is also dimensionless

term	symbol	to SI from cgs	to cgs from SI
magnetic induction	B	[SI value] = (10^{-4}) [cgs value]	[cgs value] = (10^4) [SI value]
magnetic field strength	H	[SI value] = [cgs value]	[cgs value] = [SI value]
magnetic moment	m	[SI value] = (10^{-3}) [cgs value]	[cgs value] = (10^3) [SI value]
volume magnetization	M	[SI value] = (10^3) [cgs value]	[cgs value] = (10^{-3}) [SI value]
mass magnetization	J or σ	[SI value] = (1) [cgs value]	[cgs value] = (1) [SI value]
volume susceptibility	k	[SI value] = (4π) [cgs value]	[cgs value] = [SI value]
mass susceptibility	χ	[SI value] = [cgs value]	[cgs value] = [SI value]

(A) = Ampere

(cm) = centimeter

(emu) = electromagnetic unit

(γ) = gamma

(G) = Gauss

(g) = gram

(kg) = kilogram

(m) = meter

(Oe) = Oersted

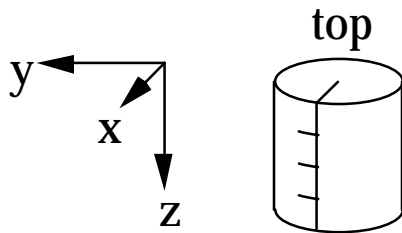
(T) = Tesla

In the SI system, we have $B = \mu_0(H + M)$, where μ_0 is the permeability of free space ($4\pi \times 10^{-7}$ Henry m^{-1}), B is in Tesla, and H and M are in A m^{-1} . But in the cgs system, we have $B = H + 4\pi M$, where B is in Gauss, H is in Oersted, and M is in emu cm^{-3} . The Earth's magnetic field in Minneapolis has an approximate value of $B \cong 5 \times 10^{-5}$ (T) = 50 (μT) = 0.5 (G) = 5×10^4 (γ)

For more information on relating the SI and cgs systems, see *IRM Quarterly*, Spring 1994, vol. 4, no. 1, p. 6; B. M. Moskowitz, *Handbook of Physical Constants*, American Geophysical Union, Washington, DC, in press, 1994; P. N. Shive, *Eos Trans. Am. Geophys. Union*, 67, 25, 1986; or M. A. Payne (1981), *Phys. Earth Planet. Inter.*, 26, P10–P16, with errata (1981), *Phys. Earth Planet. Inter.*, 27, 233.

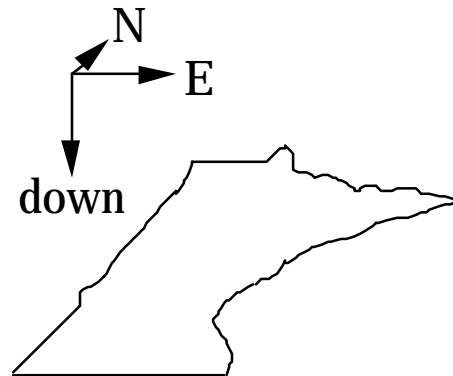
Appendix 2. The *IRM*'s Orientation Scheme

Sample Coordinate System



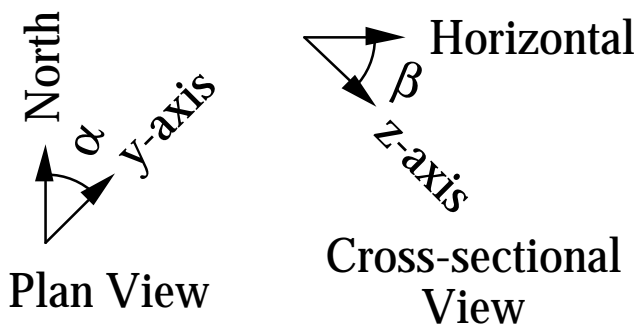
In the field, the y-axis of the sample is chosen so as to be in the horizontal plane, so that the x- and z-axes lie in the same vertical plane. If the sample is unoriented or semi-oriented, the unknown axes can be assigned arbitrarily in the lab.

Earth Coordinate System



The standard right-handed earth coordinate system used in paleo- and rock magnetism consists of the directions north, east, and down.

Coordinate System Relations Alpha (α) and Beta (β)

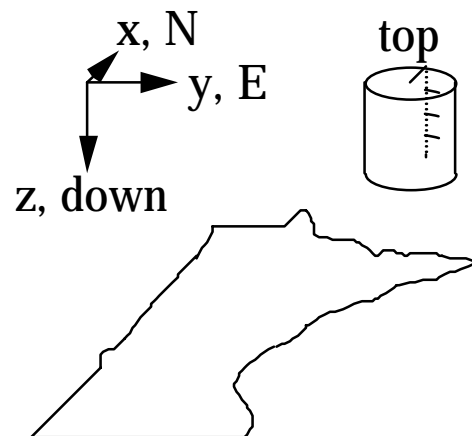


In the field, the two coordinate systems can be related through a pair of angles, alpha (α) and beta (β), which are defined as follows:

α = trend of y-axis (measured in degrees clockwise from north, $0^\circ < \alpha < 360^\circ$)

β = plunge of z-axis (measured in degrees downwards from horizontal, $-90^\circ < \beta < 90^\circ$)

Default Coordinate System Relation $\alpha = \beta = 90^\circ$



The default conventional relationship between the coordinate systems (used for unoriented samples as well) is to set both alpha and beta equal to ninety degrees. This results in the x-axis pointing north, the y-axis east, and the z-axis down:

Appendix 3. Sample Types

Sample Type	Holder Example	Default Volume
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P1		5.28 cm ³
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Cylinder		11.00 cm ³
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Cube		7.65 cm ³
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Arbitrary	[special shapes]	[entered by hand]
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Appendix 4. IRM Equipment List

SUSCEPTIBILITY EQUIPMENT

Susceptibility Bridges [Bartington Instruments]

sensitivity 1.2×10^{-5} SI; frequencies 470 Hz and 4700 Hz [~ 10 sec/sample (8 cm^3)]

Susceptibility Anisotropy Bridge [in-house]

sensitivity 1.2×10^{-6} SI; frequency 680 Hz [~ 2 min/sample (8 cm^3)]

High-Temperature Susceptometer [Kappa]

sensitivity 4×10^{-8} SI; frequency 920 Hz; temperatures from ambient to 1000 K [several hours/sample (= 200 mg chip or powder)]

Low-Temperature AC Susceptometer [Lake-Shore Cryotronics]

sensitivity 2×10^{-8} SI; frequencies 1 Hz to 10,000 Hz, real and imaginary components plus harmonics; temperatures 15 K to 325 K [several hours/sample (= 200 mg chip or powder)]

PALEOMAGNETIC EQUIPMENT

Spinner Magnetometer [Schonstedt]

sensitivity 1×10^{-10} A m² [~ 3 min/sample (8 cm^3)]

Superconducting Rock Magnetometer (SRM) [2G Corporation]

sensitivity 2×10^{-11} A m² [~ 1 min/sample (8 cm^3)]

Alternating Field (AF) Demagnetizers [Schonstedt]

peak applied fields from 0 to 100 mT; biasing fields from 0 mT to 0.2 mT; ARM and partial ARM capability [~ 1 min/demagnetization step or ARM (8 cm^3)]

Thermal Demagnetizer [Schonstedt]

temperatures from ambient to 1100 K; optional applied fields from 0 mT to 0.2 mT [~ 1 hour/demagnetization step or TRM (8 cm^3 , 8 samples per batch)]

ROCK-MAGNETIC EQUIPMENT

Vibrating Sample Magnetometer (VSM) [Princeton Applied Research]

sensitivity 2×10^{-8} A m²; applied fields from 0 T to 1.7 T; temperatures from 77 K to 1200 K [~ 30 min/sample for hysteresis loops (8 cm^3) and ~ 2 hours/sample for Curie points (= 200 mg chip or powder)]

Alternating Gradient Force Magnetometer (MicroMag) [Princeton Measurements]

sensitivity 1×10^{-11} A m² [~ 10 min/sample for hysteresis loops (= 100 mg chip or powder)]

Superconducting Susceptometer (MPMS) [Quantum Design]

sensitivity 1×10^{-11} A m²; applied fields from 0 T to 5 T; temperatures from 2 K to 400 K [~ 1 hour/sample minimum depending on measurement program (= 200 mg chip or powder)]

SPECIALTY EQUIPMENT

Magneto-Optic Kerr Effect (MOKE) System [Leitz, Hamamatsu, Sony, and Buehler]

applied fields from 0 T to 0.1 T; image manipulating software; sample preparation facilities [\sim 4 hours/sample (5 cm² polished grains or thin sections)]

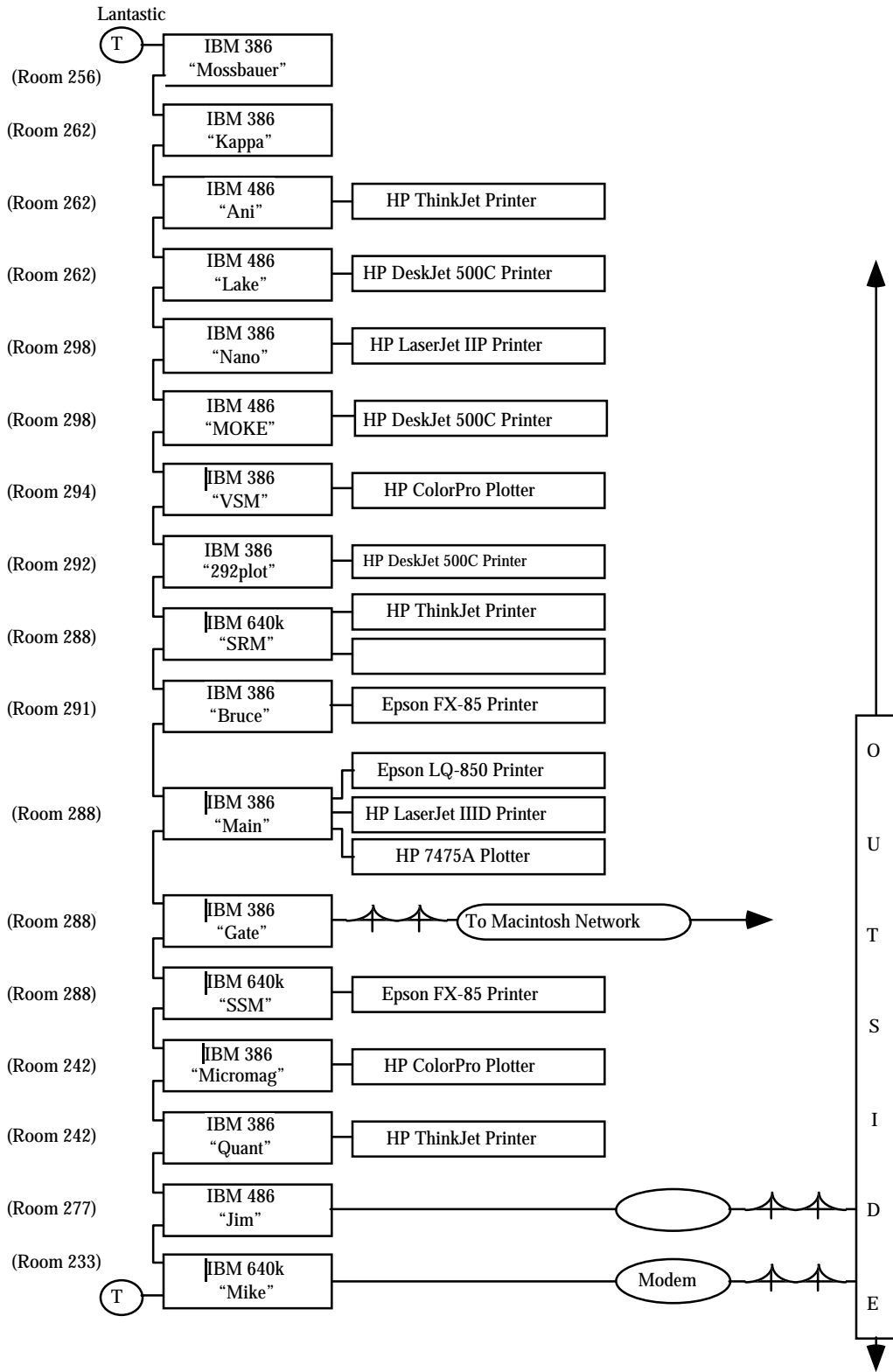
Magnetic Force Microscope (MFM) [Digital Instruments]

magnetic and topographic resolution \sim 20 nm [several hours/sample (1 cm² polished single grains, thin sections, or thin films)]

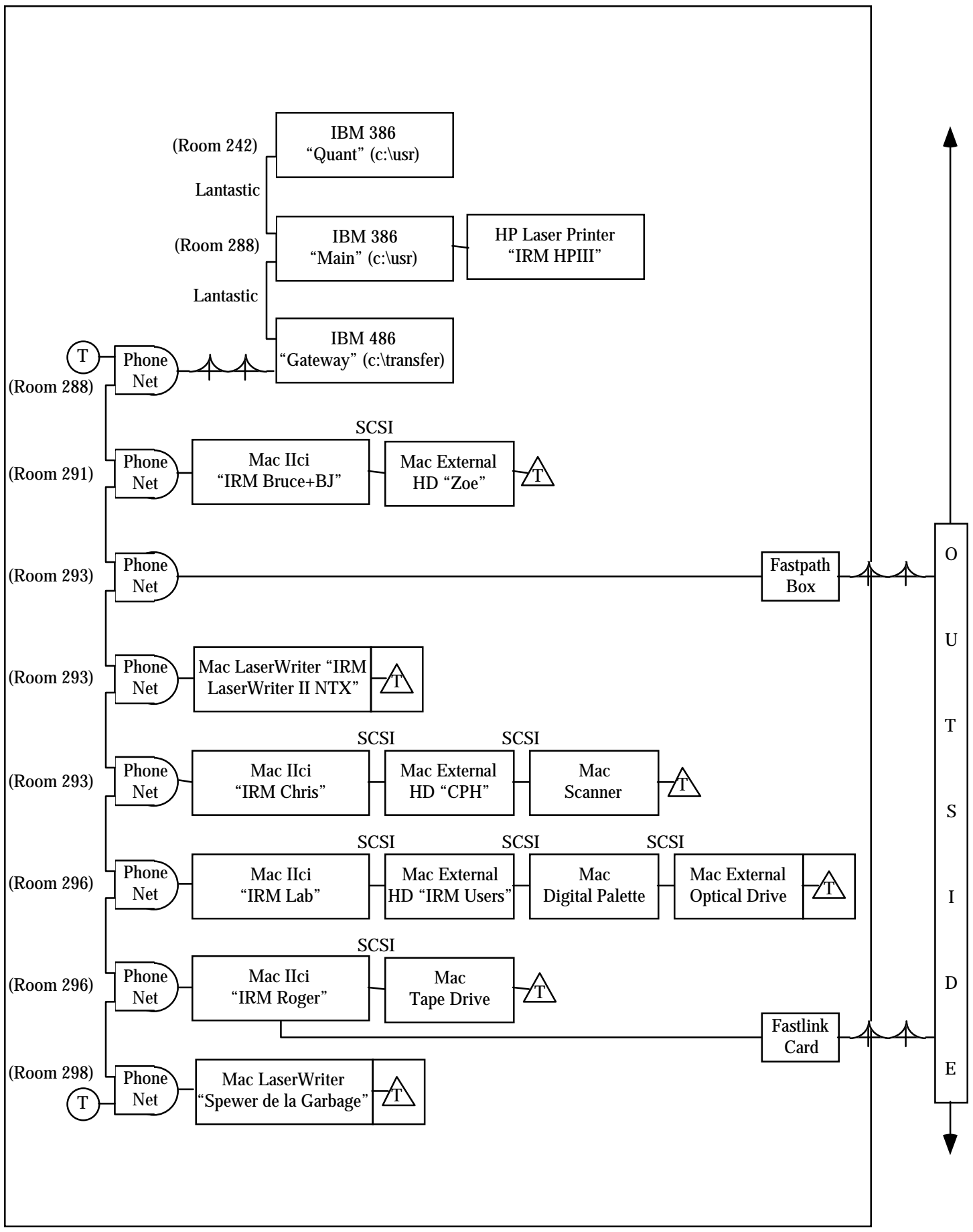
Mössbauer Spectrometer [Ranger Scientific]

temperatures from 2 K to ambient; zero applied field (external field up to 5 T not yet available) [several hours/sample (= 200 mg chip or powder)]

Appendix 6. IRM IBM Network



Appendix 7. IRM Macintosh Network



Appendix 8. The *IRM Quarterly*