Physical Property Lab
Moisture/Density (MAD) Cookbook

The MAD station consists of two main parts: a Cahn C29 Electro Balance and a Quantachrome Penta-pycnometer. ODP procedure for calculating volume, moisture content, bulk density, and grain density with this equipment involves measuring both wet and dry masses as well as the dry volume of a sample.

Before You Leave Port

Calibrating the Mass Balance

This station must be calibrated while still tied to the dock to ensure the most accurate set of measurements. This is done using the mass standards found in the Physical Properties Lab drawer labeled "Balance, Oven, and Sampling Tools."

To begin, open the MAD program by double clicking on the icon on the Windows screen of the MAD PC.
This will bring up the main MAD screen (Figure 1).

DO NOT USE THE MAD UPGRADE ICON OR PROGRAM.

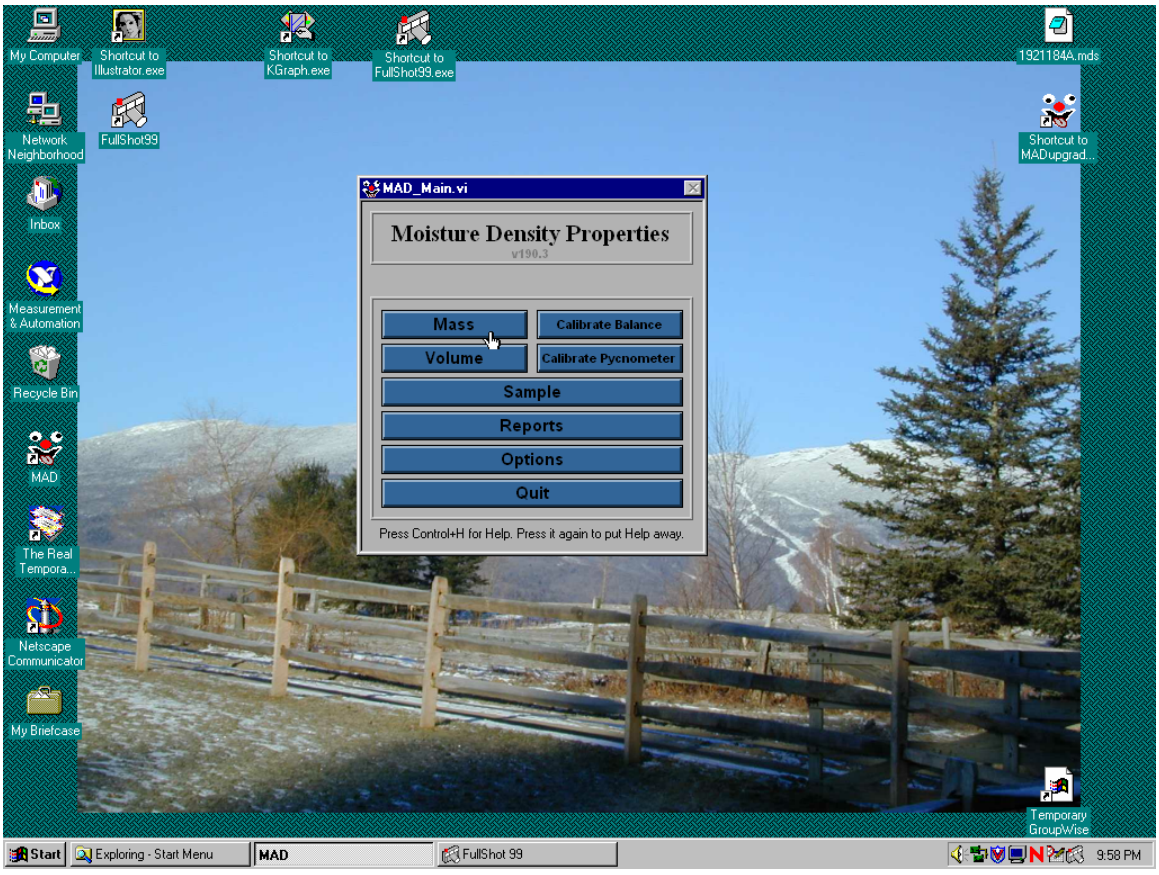


Figure 1. Main MAD Screen.

Click on CALIBRATE BALANCE to bring up the next screen (Figure 2).

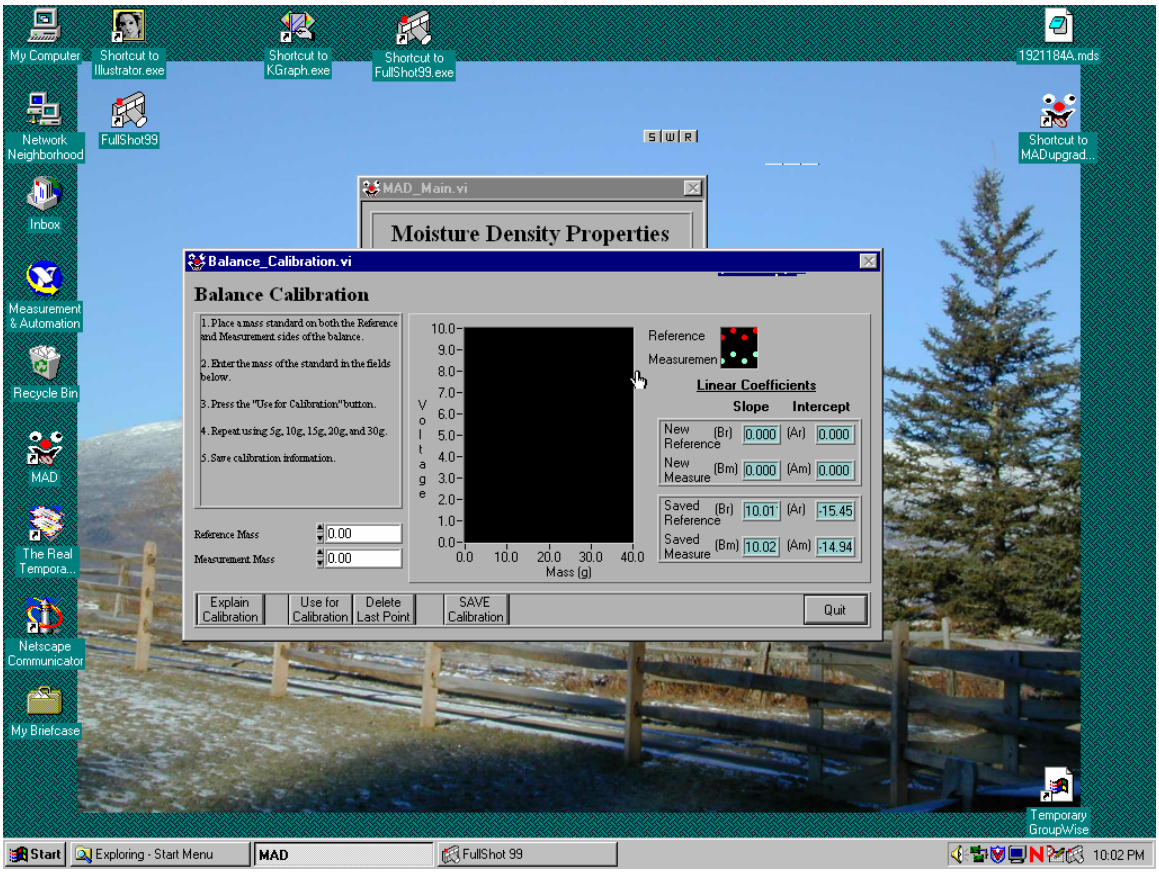


Figure 2. Balance Calibration.

There are two balances in the wooden box located between the pycnometer and the MAD PC. One is labeled REFERENCE and the other MEASURE. Follow the directions on the screen to complete the calibration procedures. Once you have finished the measurements, be sure to SAVE CALIBRATION and then QUIT to return to the main menu.

Also, before you leave port you will want to make sure the helium bottle used for the pycnometer is full and ready to go. The bottle is located on the other side of the wall next to the pycnometer. It is attached by copper tubing to a filter system, which in turn is attached to the pycnometer. We use copper tubing instead of plastic tubing since it is not affected by minor pressure changes; also Quantachrome Corp. recommends it for best results. We run the pressure setting to the pycnometer at 22 psi, and at 200 psi the helium bottle should be changed, as it seems there is not enough pressure to consistently recharge the pycnometer.

NOTE: There are no air-conditioning vents around the pycnometer station purposely to avoid air flow and possible pressure changes as these greatly affect the measuring abilities of the pycnometer. Open doors, ceiling repairs, ventilation work will all affect the running of the pycnometer so be aware of that.

There are three pycnometers on board, two considered “old” and one “new.” Use the two old ones as back-ups should the situation arise. Check with the Tech reports from previously legs to see what the latest condition of the three Pycnometers are.

At Sea

Some of these procedures may be completed before you leave port but if you don’t have time, it’s no problem to do them once underway.

Calibrating the Pycnometer

Be sure that you have the correct pycnometer (old vs. new) selected on the admin options screen. To do this, on the main MAD screen select OPTIONS then ADMIN OPTIONS (Figure 3), enter MAD as the admin password to access the Admin Options screen (Figure 4), and choose the pycnometer you will be using. Then QUIT to return to the main screen. The “New” Pycnometer will be used for the newest Pycnometer. ONLY.

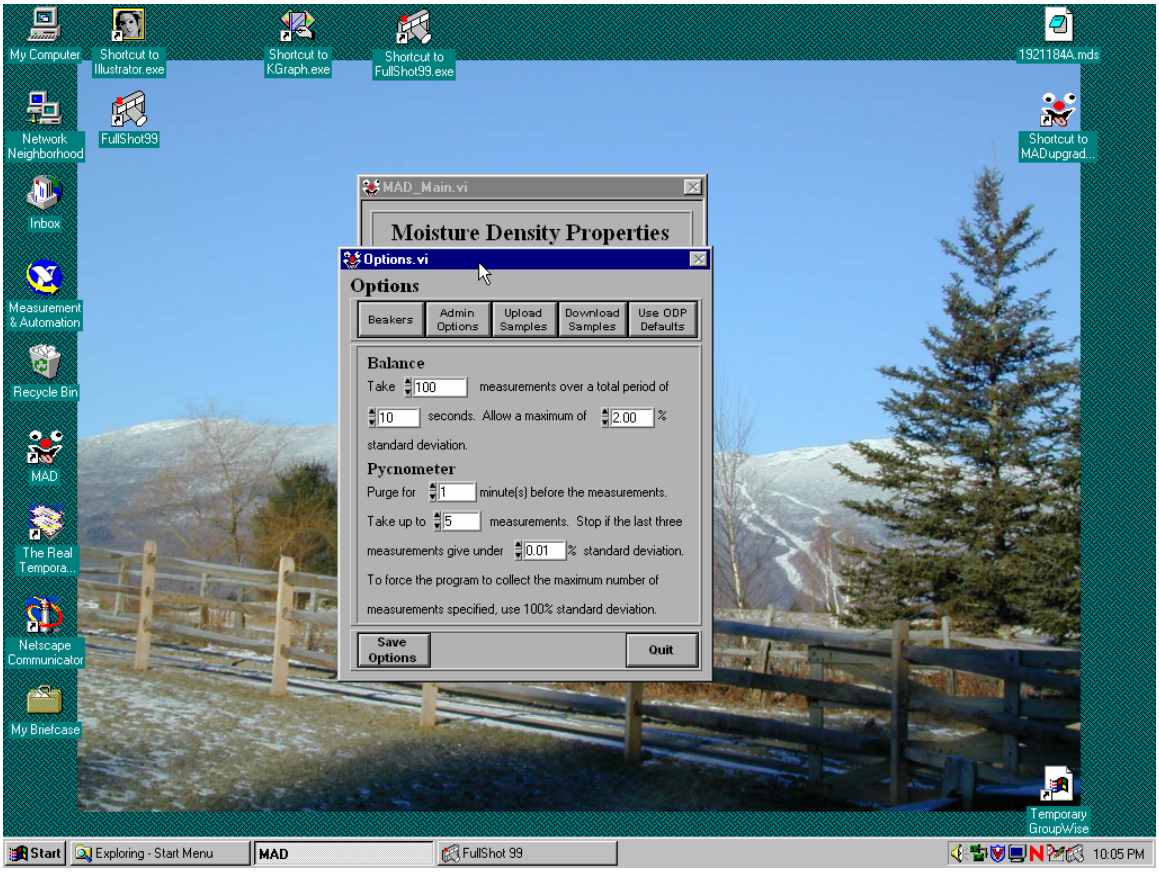


Figure 3. Main MAD Options.

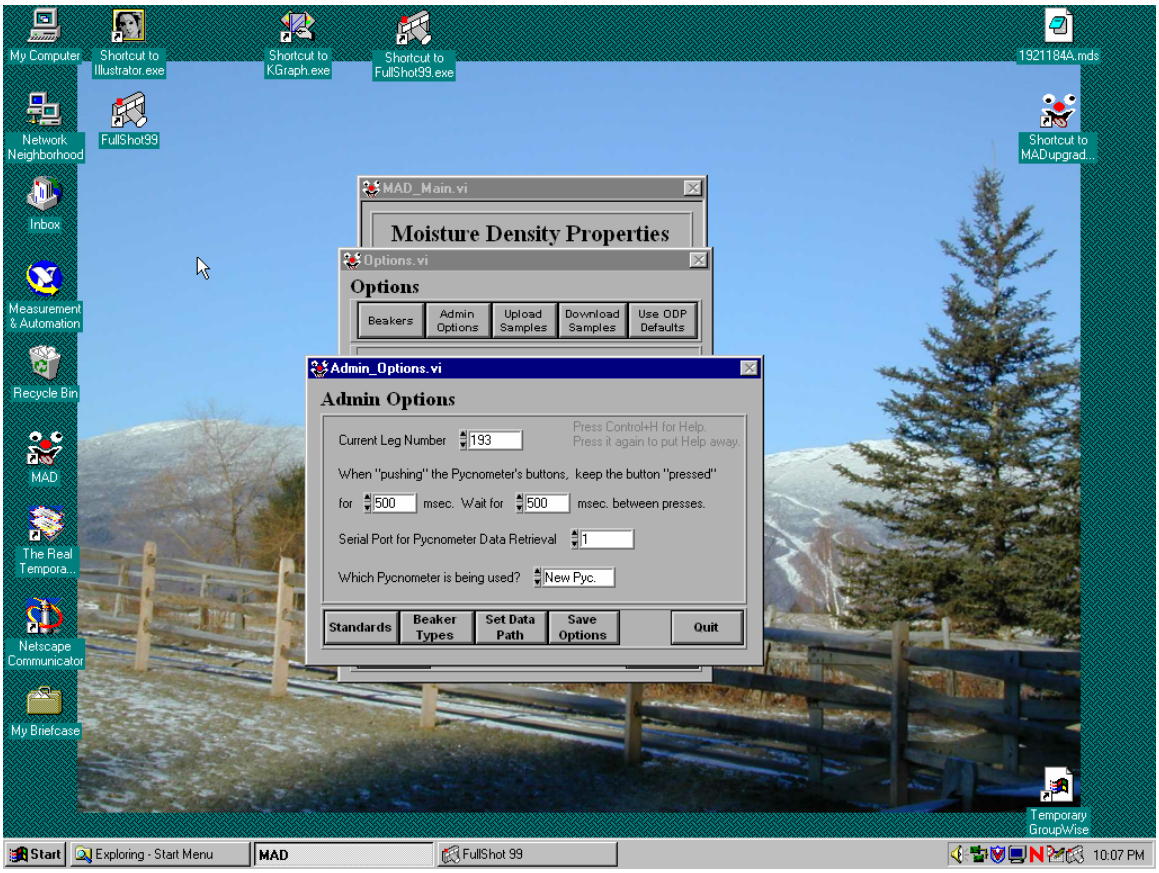


Figure 4. MAD Admin Options.

To calibrate the pycnometer, click on PYCNOMETER CALIBRATION on the main MAD screen to bring up Figure 5.

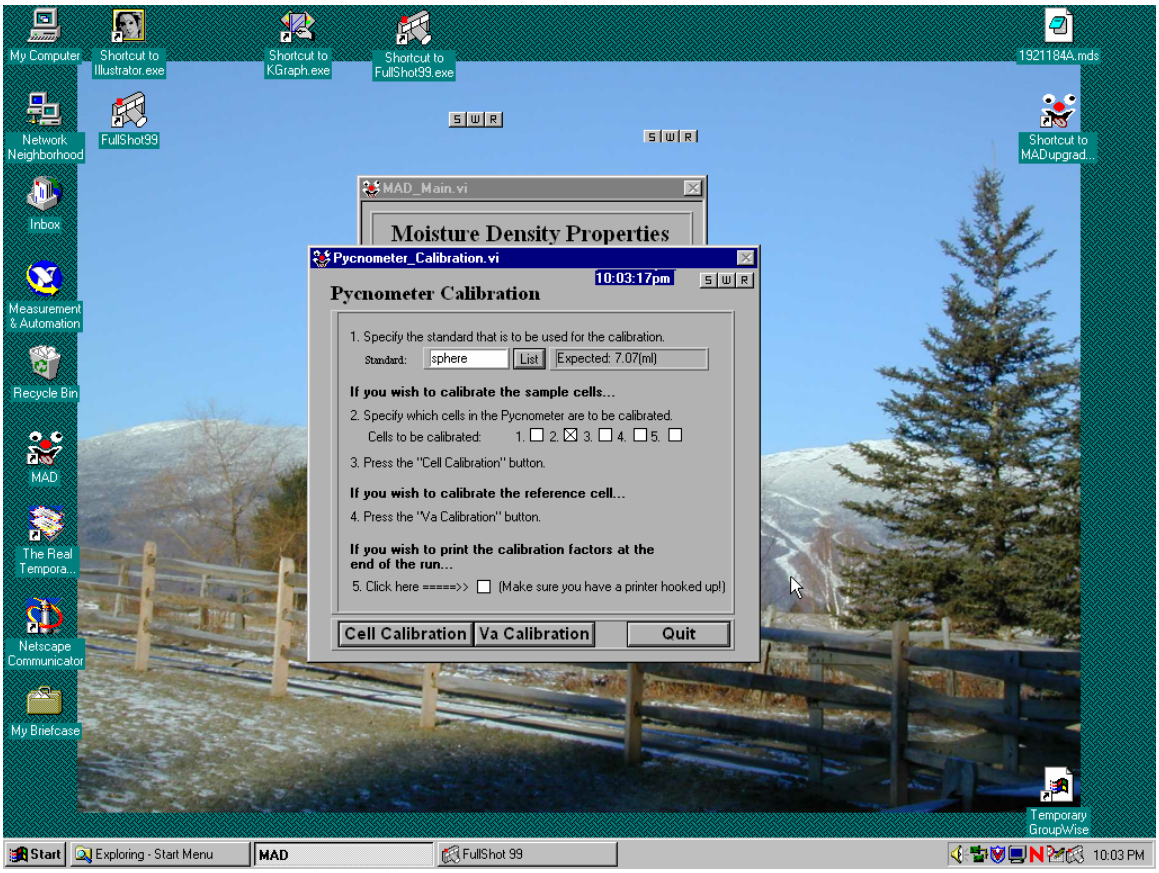


Figure 5. Pycnometer Calibration.

To choose the standard to be used for calibrating the pycnometer, select LIST from section 1. We usually use the sphere; pay attention to the labels on the containers because there are new and old spheres (old ones will tend to be more battered, which result in having less accurate readings).

The first calibration to be done is the Va calibration or calibration of the internal reference cell. This should only be done at the beginning of a leg and after the helium bottle has been changed, or if there has been a change in gas pressure to the pycnometer for any other reason (such as disconnecting the gas lines for cleaning). For the Va calibration, only the standard to be used must be selected; do not select specific cells to be done. Click on Va CALIBRATION and follow the instructions on the screen. Basically, the pycnometer will do a run with all cells empty and when finished, will prompt you to put the standard sphere in Cell #4 and click ENTER to continue. When completed, the menu screen on the pycnometer itself will display a run completed message.

Once you have completed the Va calibration, you must calibrate each cell individually. Under section 2, select the cell(s) to be calibrated. Click on CELL CALIBRATION and follow the on-screen instructions. Be sure the standard sphere is in the specified cell. Repeat for each cell.

WHEN CALIBRATING EITHER INDIVIDUAL CELLS OR THE REFERENCE CELL, ALL CELL LIDS MUST BE SECURELY CLOSED (FINGER TIGHT – DO NOT FORCE).

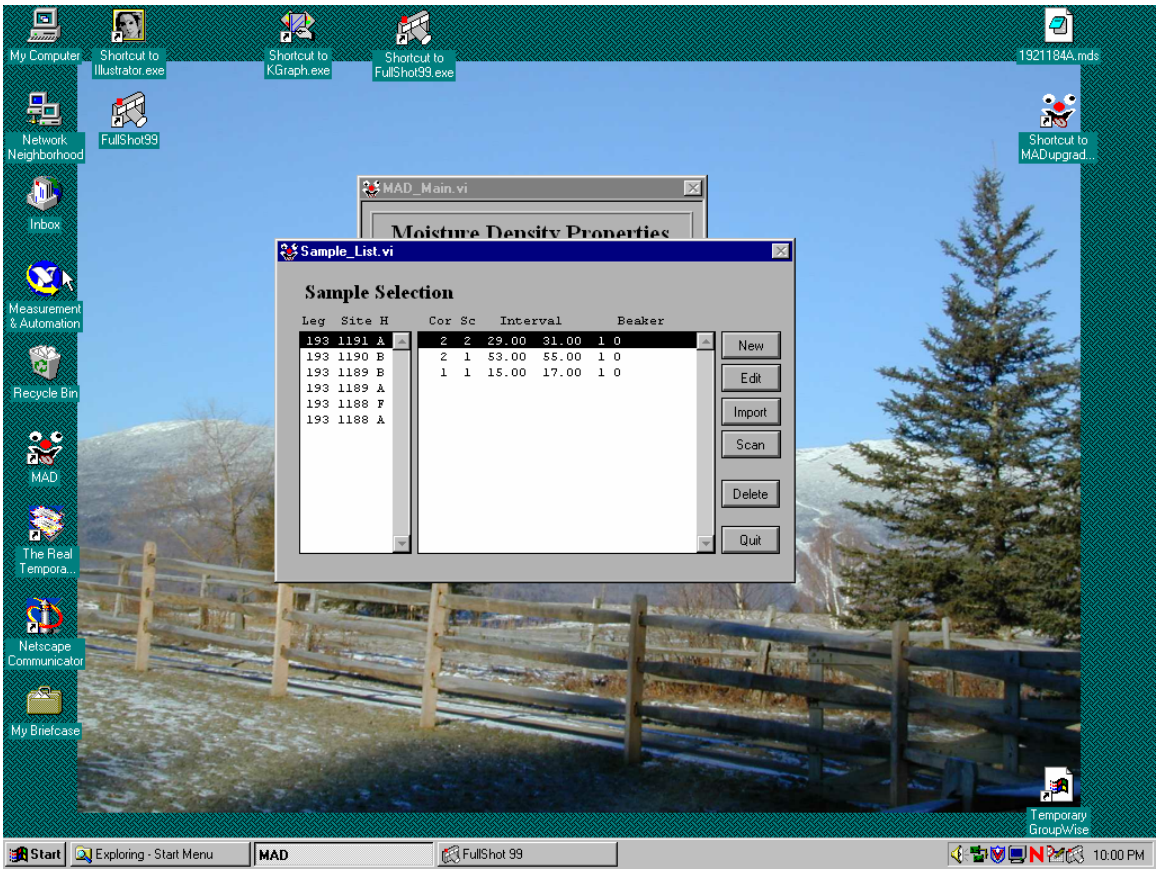
Running Samples

Both hard rock and sediment samples may be run through the MAD station. When choosing samples, aim for homogeneous representative pieces or sediments from each (if possible) section. Notes on how to sample and measure hard rock minicores (i.e. what's different from sediment samples) are at the end of this section.

Sediment cores are run using 10 ml beakers. A special sampling tool resembling a cylindrical plunger is located (usually) in the PP Lab drawer labeled "Sampling Tools." This tool allows a 2cm cylindrical interval to be cored which fits perfectly into the 10 ml beakers. Much debate has been ongoing about whether or not it matters if the sediments get compressed or packed into the beakers; Staff Scientist Peter Blum has stated that it does NOT matter. However, for peace of mind it is suggested that the scientists do their best not to squish the samples into the beakers. Using spatulas and other tools is permissible to get the sample out of the section.

Once the sample has been taken, record all relevant information on the Index Properties log sheet. This will include the core, core type, section, interval (Use only 2-cm intervals in whole integer values because of software glitches), and beaker number. Each beaker is then covered with Parafilm to prevent further moisture loss before it is weighed. Once the core has been sampled, the log sheet information is entered into Janus via the curatorial/sampling computer. Ask the curator or MCS if you have questions concerning how to run Janus. After the samples are entered clicking on save will print out a barcode sticker for that sample that will label the sample bag later. By printing out the stickers immediately, it is easy to keep track of what has been entered.

Moving to the Mad station, from the main menu click on SAMPLE to bring up the sample screen (Figure 6).



Leg	Site	H	Cor	Sc	Interval		Beaker	
193	1191	A	2	2	29.00	31.00	1	0
193	1190	B	2	1	53.00	55.00	1	0
193	1189	B	1	1	15.00	17.00	1	0
193	1189	A						
193	1188	F						
193	1188	A						

New
Edit
Import
Scan
Delete
Quit

Figure 6. Sample List.

Click on IMPORT to upload the sample information you just entered into Janus (Figure 7). Choose the correct site and hole (VERY IMPORTANT) and click GET SAMPLES.

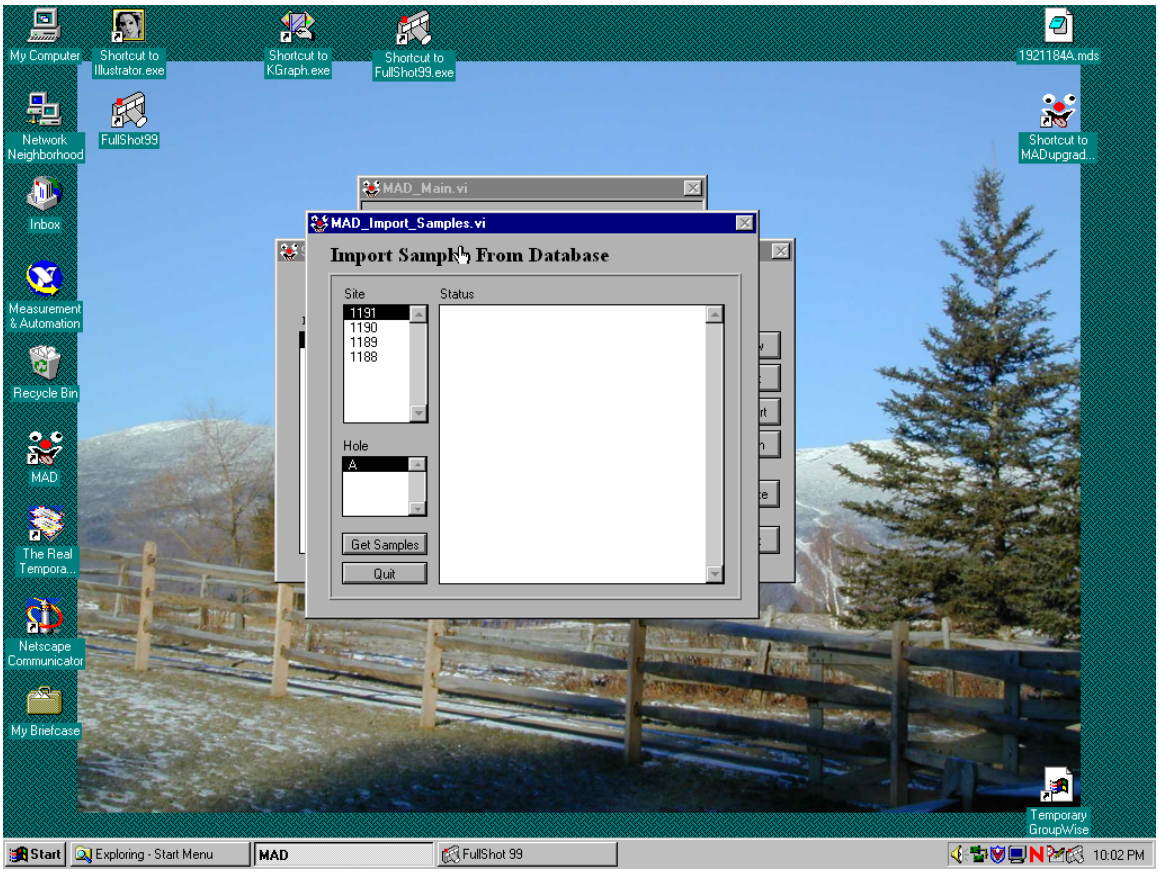


Figure 7. Import Samples.

The software will connect to the database and upload any recently entered samples. The raw data file is put in a folder labeled *LEG/SITE/HOLE* in the **c:/ODP/MAD/DATA** folder. Once completed, click QUIT to return to the Sample List screen. All of the sample information may now be viewed and/or edited individually on the sample ID screen (Figure 8), which can be accessed by double clicking on the desired sample or clicking on EDIT.

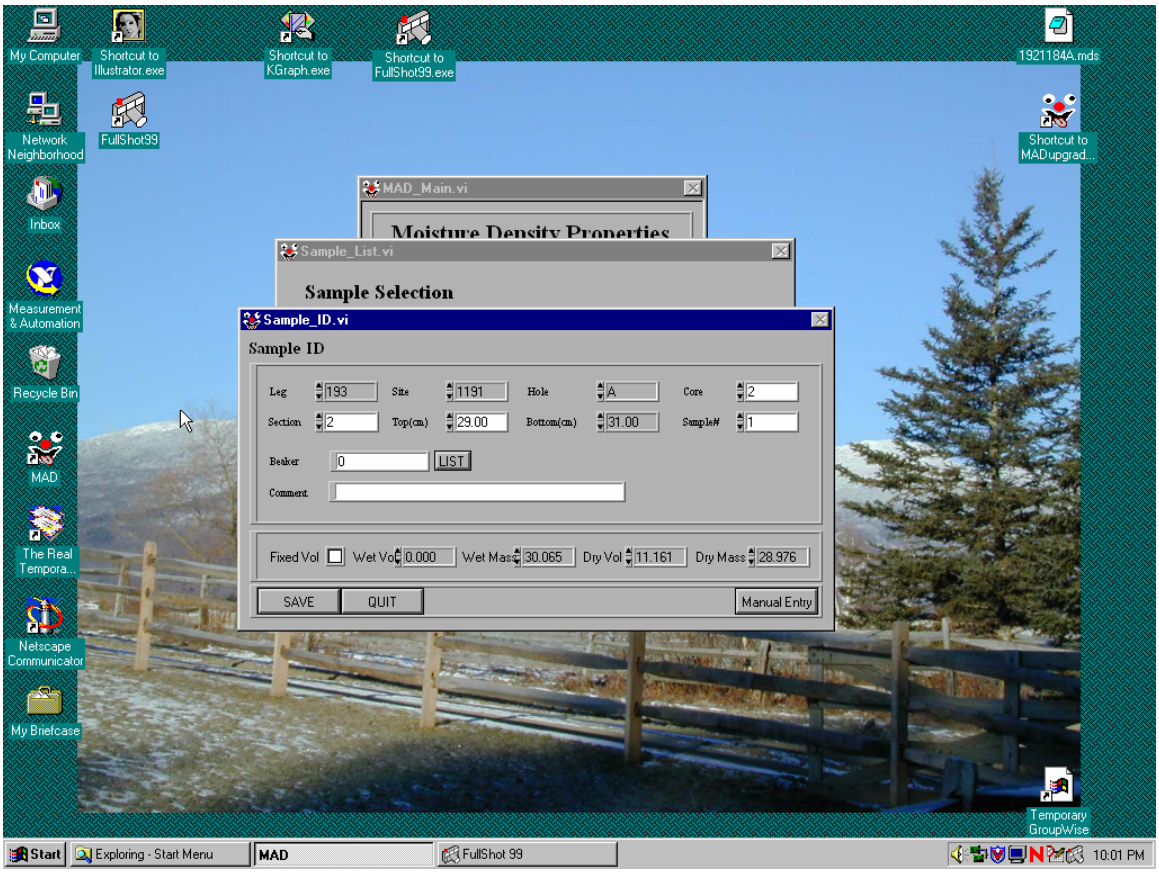


Figure 8. Sample ID.

This screen allows you to manually edit information, a highly useful tool should information be lost or entered incorrectly. Click MANUAL ENTRY to access the volume and mass. Now that the details have been entered into the MAD program, you can begin running the samples.

Wet and Dry Masses

On the main screen, click on MASS to bring up the balance window (Figure 9). Place a reference mass, usually 20 grams, onto the Reference balance and enter it into the REF. MASS(g) window. Select a sample and enter the beaker number in the appropriate window. If you have used the beaker before during the leg, be sure the correct sample ID shows up in the window. If not, click on LIST which will take you to the SAMPLE LIST menu and you may manually select the correct ID by either double clicking or highlighting it and clicking SELECT. In the TYPE window, enter WET. Remove the parafilm from the beaker and place beaker and sample on the Measure balance, ensuring that the outside of the beaker is clean of all residue (excess will dry and fall off later, affecting the moisture content calculations) and that the sample is not extending above the rim of the beaker.

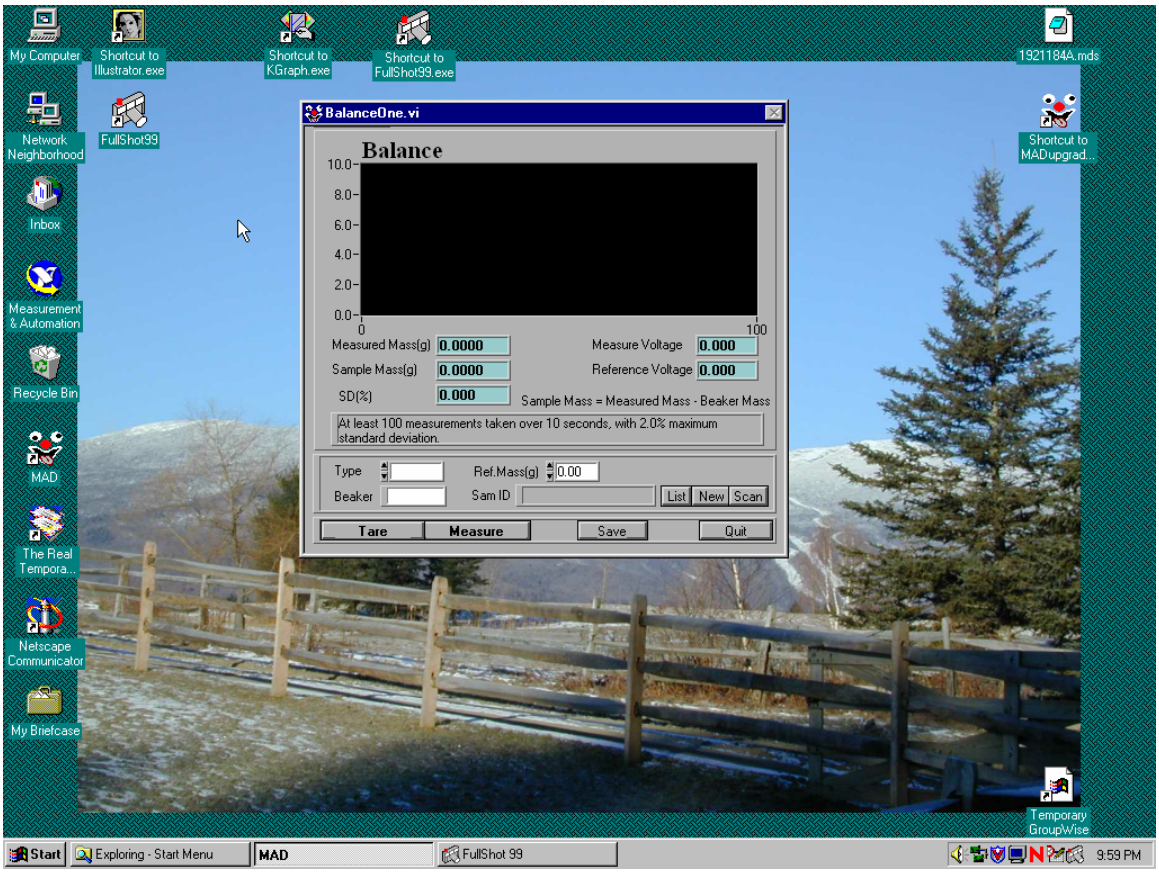


Figure 9. Mass Balance.

Click MEASURE. When the mass has been taken, a dialogue box will appear stating that fact and asking if you wish to save the data at this point. Click OKAY to save data. Remove the sample from the balance and place on a metal tray. On the log sheet, record the MEASURED MASS and NOT the sample mass. On the Sample ID screen, the information recorded is for the total mass of sample and beaker; the beaker information will be removed in later calculations by the software. Once all of the samples have been measured, place the tray in the oven and record core ID, time, and date on a sheet so that you can keep track of when they need to be taken out.

NOTE: DO NOT PUT THE PARAFILM BACK OVER THE SAMPLES BEFORE PUTTING THEM IN THE OVEN!!!!

Samples remain in the oven to dry for 24 hours. When the samples have dried for 24 hours, remove the tray and place directly into the desiccator. Let cool to room temperature, which usually takes about an hour. Follow the same procedures as for wet mass measurements but enter DRY in the TYPE window. Again, record MEASURED MASS on the log sheet.

Return the beaker to the desiccator until you are ready to run the sample through the pycnometer.

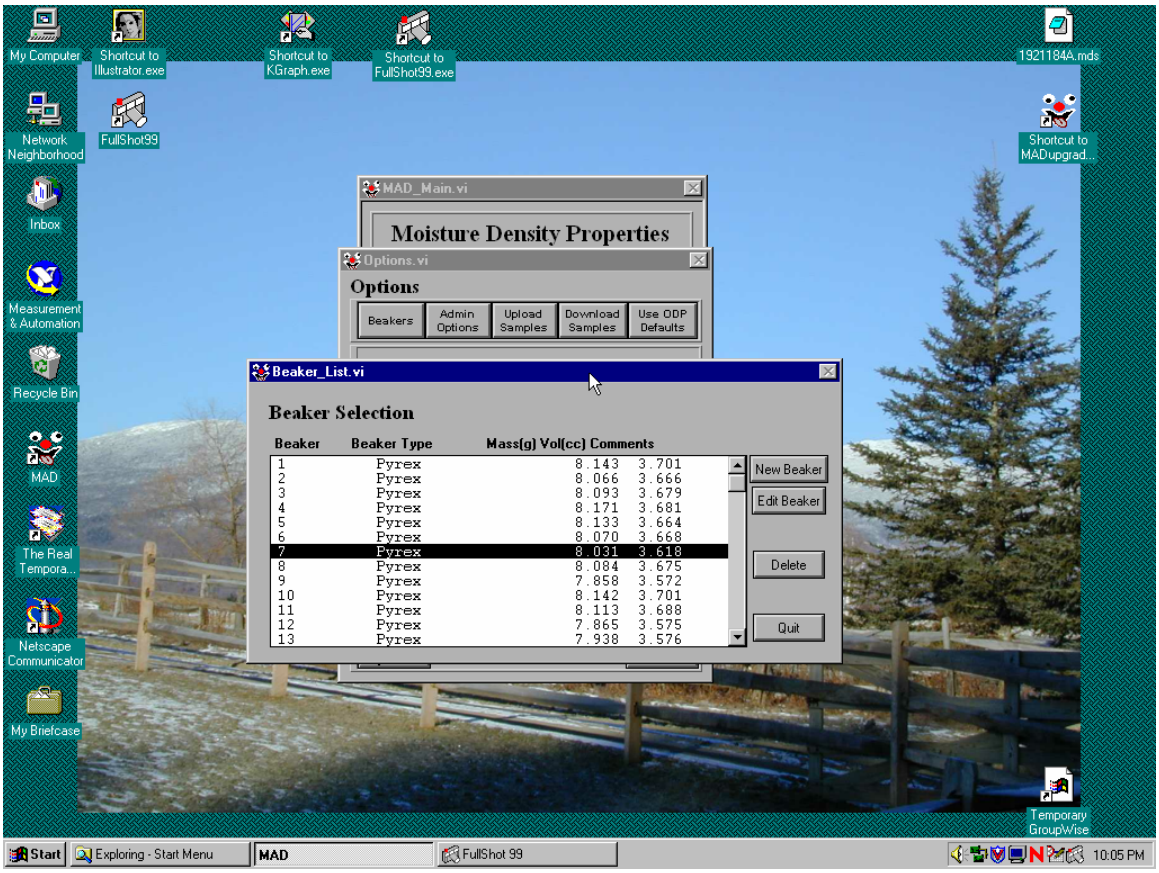


Figure 10. Beaker List.

Beaker information (mass and volume) is stored in a raw file and is accessible (and changeable) by clicking on OPTIONS on the main screen, then clicking on BEAKERS (Figure 10). You may add new beakers from here (be sure the beaker number you use isn't already being used on another beaker) by following the instructions when you click on NEW BEAKER (Figure 11). All beakers used are made of Pyrex (known properties) so the only thing you need to do when entering a new beaker ID is to weigh it; the software will calculate the volume automatically.

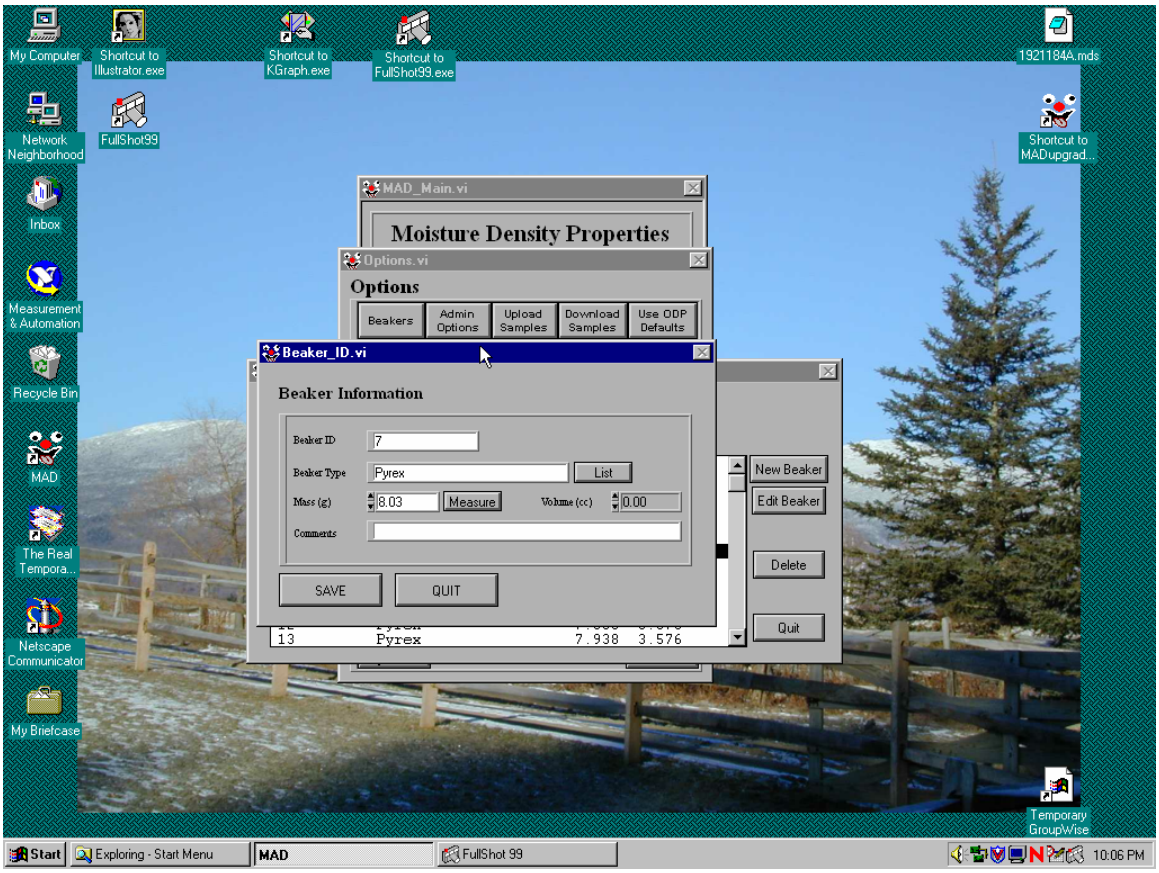


Figure 11. Beaker ID.

Volume

Click on VOLUME on the main menu to bring up the pycnometer screen (Figure 12).

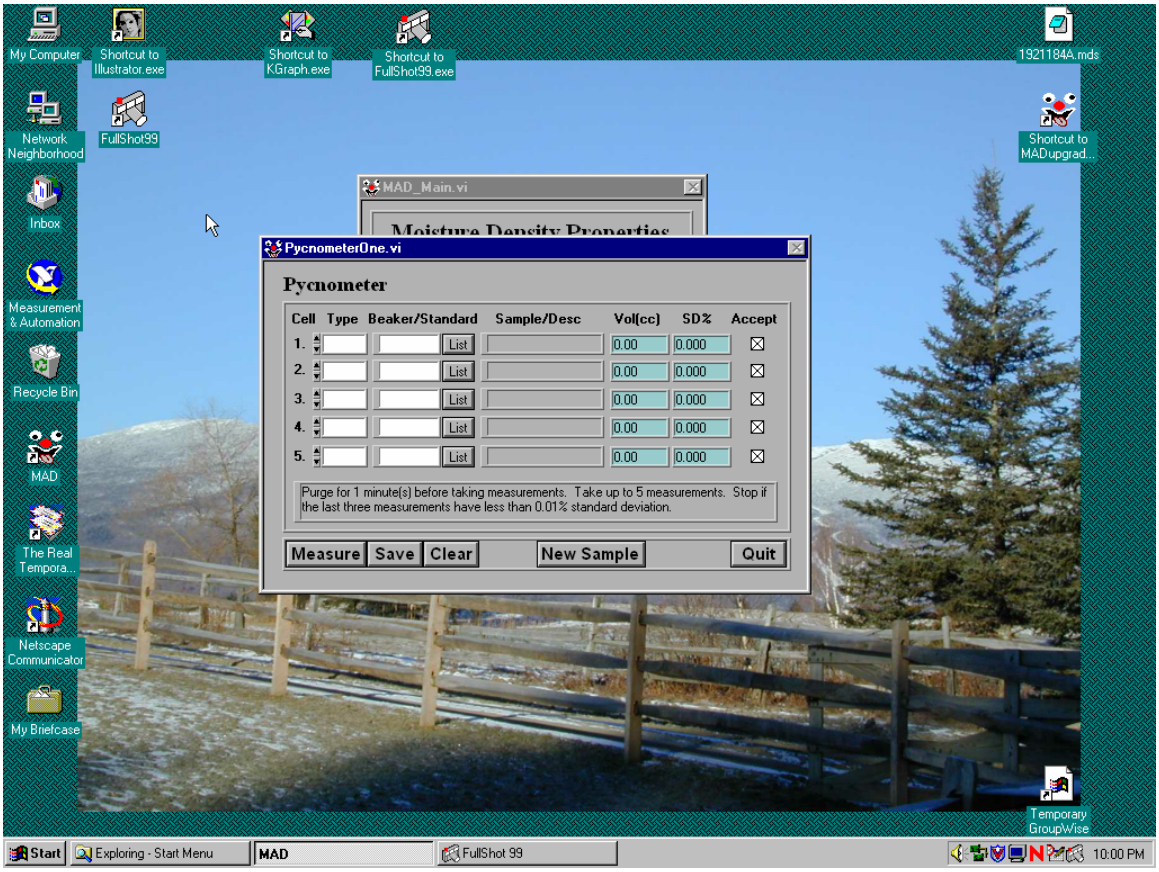


Figure 12. Pycnometer.

Under TYPE you will select either DRY (sample) or CONTROL (sphere standard). To run the samples, place a beaker in each cell, noting the beaker number before you close the lid. Type in the beaker number and click ENTER on the keyboard or select the appropriate sample using the LIST option (click LIST to bring up the sphere option if TYPE is CONTROL). The VOL (cc) column ***WILL NOT*** delete between runs unless you hit CLEAR. Make sure the numbers you are copying down are for the correct run and not the previous run. The ACCEPT column allows you to check or uncheck the cells you wish to run (see NOTE below).

Normally you will run four samples and one standard sphere for each run, moving the sphere from cell to cell in numerical order. This allows each cell to be checked for accuracy at least once every five runs. The allowable drift for the sphere (normally a volume of 7.07 cc) is 0.02. Thus a range of 7.05-7.09 is acceptable. If the volume measurement is outside of this range, the individual cell must be recalibrated using the procedure described above for CELL CALIBRATION. After recalibrating, the sphere may then be moved to the next cell and a new run of samples begun.

When the cells are ready to go, click MEASURE. The pycnometer will run through its menu, taking the information from the computer. You'll hear a clicking noise from the transfer box on the top shelf.

Once the data have been input, a dialogue box will appear on the screen asking if you wish to proceed. Click OK. Don't forget this or it will sit there, not running, until you notice it. Once it's running, it should take about 25 minutes to run through all five cells. If it hangs, the menu on the pycnometer will not change for sometime, or all the lights may light up and not click off after a minute. If this happens, you must click STOP to get the run to quit. You may have to click the RESET button on the pycnometer if the machine won't clear its screen. Once the pycnometer has completed its measurements, the only notification you'll get is a RUN COMPLETE message on the *pycnometer* screen, **NOT** the computer screen. Click on RETRIEVE on the computer screen to download the volumes (this will take about three or four minutes so be patient). Once the values are in the computer, a dialogue box will appear asking if you wish to save the data. Click OK and a "VALUES SAVED OK" message will appear. Click OK and copy the volume values onto the logsheet.

NOTES: Hard rock cores are usually run as 2 cm minicores without beakers (enter beaker number zero and select the correct sample ID with the LIST feature). A few things that are done differently include soaking the minicores in seawater for 24 hours before weighing them and storing them in snap-top plastic vials instead of sample bags. The labels adhere to the outside of the vials and the samples can soak individually in salt water in each vial. The minicores are place directly into the pycnometer without beakers.

Things to remember about the MAD station:

- keep inflowing helium pressure at 22 psi
- do not tighten cell lids more than finger tight
- ONLY use silicon-based lubricant on O-rings inside cell lids; petroleum-based products will break down the rubber
- dot-matrix printer on top shelf MUST be turned on in order for the pycnometer to run
- be sure the Scientech 202s Electronic balances for the measure and reference balances are turned on (red light on)
- make sure there is desiccant (found in Hold Reefer if there isn't any in the lab) in the desiccators; either replace or dry in oven periodically
- ovens set to 105 degrees Celsius (check thermometer inside oven)
- samples must NOT protrude above the lip of the beakers or the cell lids will not close properly, usually resulting in the breakage of the beaker
- be sure the minicores are cut with parallel ends so that they may be used for PWS3 measurements; also, so that there aren't any pieces sticking out that won't fit in the pycnometer
- don't forget the reference mass (usually 20g)
- balances are only accurate to around 35g; pay attention to this when running minicores
- use the sonicator to wash beakers; dry in ovens after rinsing with distilled water
- under OPTIONS on main menu, standard settings are: Balance: 100 measurements over 10 seconds with 2% SD; Pycnometer: 1 minute purge, 5 measurements, 0.01% SD
- enter ALL data on log sheets
- minicores must be a 2cm interval when entered (as of this writing, ALL intervals must be 2cm but you can get around this default by changing the data in the raw data file) and have a beaker number of zero