MAD User Guide

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Porosity definitions used on ODP Leg 161 [From Revil et al., 1997 (10.2973/odp.proc.sr.161.274.1999)]

Introduction

Method Overview

Core specimens for moisture and density (MAD) analysis are extruded from a section half for:

- Mass: measured using a motion-compensating dual analytical balance system and a drying oven.
- Volume: measured using a caliper or by a helium displacement pycnometer that employs Archimedes' principle of fluid displacement to determine the volume of solid objects.

The MAD properties of interest are:

- Water content
- Bulk density
- Dry density
- Porosity
- Void ratio

These properties are calculated based on three out of four measured values:

- Wet mass
- Dry mass
- Wet volume
- Dry volume

Method Theory

Phase relationships of mineral density, porosity, void ratio, and water content are basic sediment and rock properties that are found most accurately through mass and volume determinations. The mass or volume of the bulk (wet) material, the dried material, and the extracted water (assumed to be interstitial pore fluid) is corrected for the mass and volume of salt evaporated during the drying process. The mass and volume of the evaporated pore water salts are calculated for standard seawater salinity, seawater density at laboratory conditions, and an average seawater salt density.

Soils can be either 2-phase or 3-phase compositions (i.e., completely dry or partially saturated). For MAD measurements the analyst determines whether the sample contains a 2- or 3-phase system:

- Completely dry soil contains 2 phases: solid soil particles and pore air.
- Fully saturated soil also contains 2 phases: solid soil particles and pore water.
- Partially saturated soil contains 3 phases: solid soil particles, pore water, and pore air.

MAD data provide a direct estimate of porosity and void ratio and the average density of constituent minerals. Porosity variations are controlled by consolidation and lithification, composition, alteration, and deformation of the sediments or rocks. MAD data can be used to calibrate high-resolution gamma ray attenuation (GRA) bulk density data, which are sampled at a much higher resolution than is possible with the MAD method. If mineral density can be defined with sufficient precision, GRA bulk density can be expressed as porosity.

Selecting the Appropriate Analysis Sub-method

The user needs to decide which sub-method (A, B, C, or D) should be used for the MAD analysis. The choice depends primarily on the type of sample material to be measured. In addition, Sub-methods A and B are **not** recommended from an analytical quality point of view. Therefore, the choice is generally limited to Sub-methods C and D based on the following criteria:

- Fine-grained, saturated sediments or fine-grained igneous material: Sub-method C
- Porous rocks (e.g., vesicular basalt and limestone) that cannot be confidently saturated: Sub-method D

Further information on sub-methods can be found in the '**Order of Actions by Method**' paragraph.

Saturation state of sample:	Saturated	Unsaturated	Soft, Saturated	Saturated
MAD analysis sub-method:	C	D	A	B
Is sub-method recommended?:	YES	YES	NO	NO
MAD Measurement (LIMS Analysis)				
Wet Volume (CALIPER)		X	X	
Wet Volume (PYC)				X
Wet Mass (MAD_MASS)	X		X	X
Dry Mass (MAD_MASS)	X	X	X	X
Dry Volume (PYC)	X	X		

Definition of LIMS Analyses

Analyses in the context of the Laboratory Information Management System (LIMS) are defined based on the data acquisition systems that provide sets of data. The MADMax software application captures the data from all three types of analysis. For the MAD group of analyses, these are as follows.

Caliper analyses (CALIPER): Volume is calculated after measuring the sample's geometric dimensions using micrometer calipers.

Pycnometer analysis (PYC): Sample volume is measured using a helium pycnometer.

MAD mass analysis (MAD_MASS): Wet or dry mass is measured using the motion-compensating dual balance system.

- "Wet" refers to the saturated (undrained) state of a sediment or rock sample
- "Dry" refers to the state after drying 24 hr at 105°C and holding in the desiccator 2–3 hr.

MAD analysis (MAD): This set of calculations is applied as appropriate for each sub-method.

Apparatus, Reagents, & Materials

- Dual balance system
- Hexapycnometer system
    - Helium supply and gauge
- Caliper
- Sample drying equipment
- Electronics and control systems
- Sampling tools and sampling containers

Hardware

Dual balance system

Two Mettler-Toledo XS204 analytical balances compensate for ship's motion while weighing samples (see the *Balances User Guide* for a detailed description of the mass determination system). In Figure 1 note that the left balance is the REFERENCE balance and the right balance is the UNKNOWN balance.
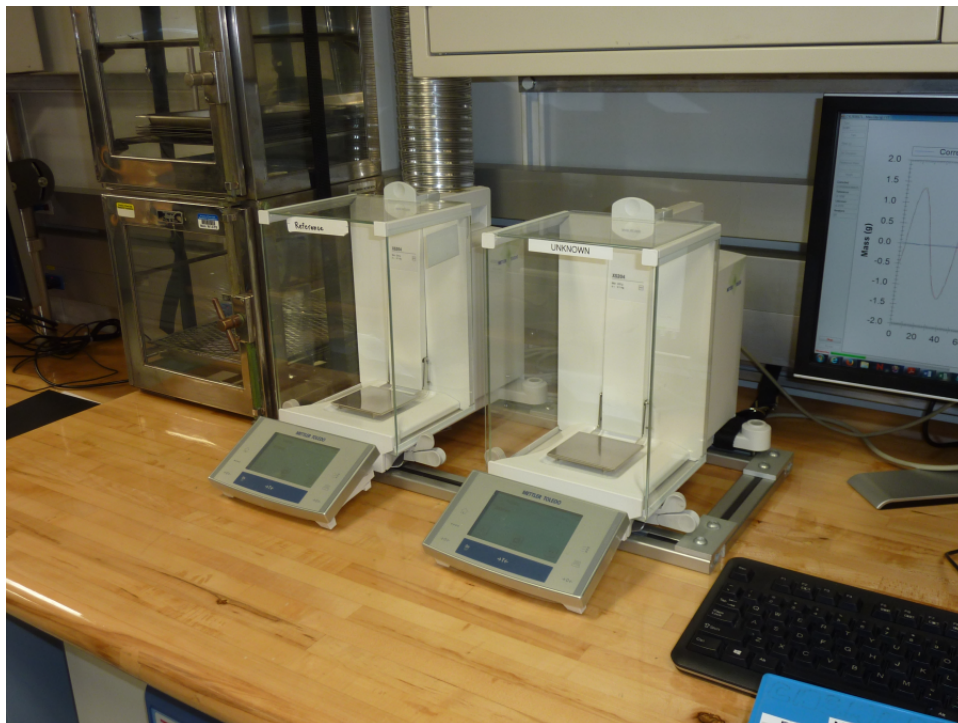

Figure 1. Dual Mettler Toledo XS-204 Analytical Balance System.

Hexapycnometer System

Six custom-configured Micromeretics AccuPyc 1330TC helium-displacement pycnometers can be run simultaneously (Figure 2a). The six cells are mounted in a chassis (Figure 2b) to protect the electronics and to help provide temperature stability. Although the cells are centrally controlled, they can be started and stopped independently.
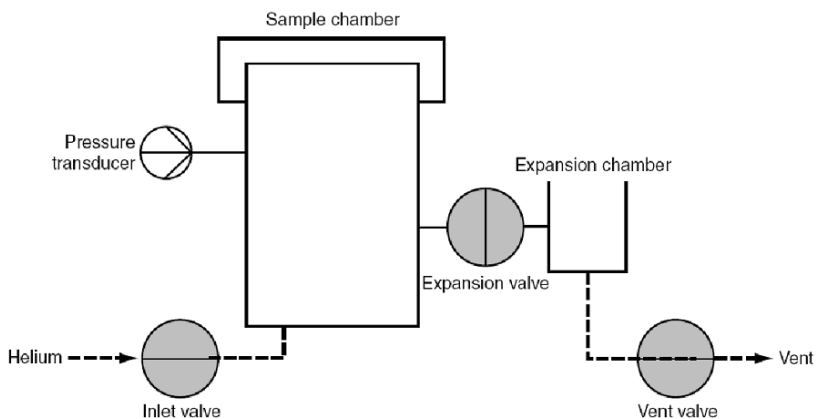

Figure 2a. Schematic of Pycnometer's Chambers, Valves (Solenoids), and Pressure Transducer.

Figure 2b. Helium-Displacement Hexapycnometer.

Helium Supply and Gauge

The system depends on a helium (He) reservoir in TBULK. The regulator at the TBULK gas bottle reservoir is set to 130 psi (Figure 3). Helium is delivered to the core lab through gas supply lines. A regulator and a pressure gauge (Figure 4) at the hexapycnometer are used to deliver low-pressure helium to the instrument. This regulator should be set to 18-20 psi (no higher than 20 psi) to deliver the proper helium pressure to the pycnometer cell gas inlets.

Pressure on the line

Pressure on the He bottle
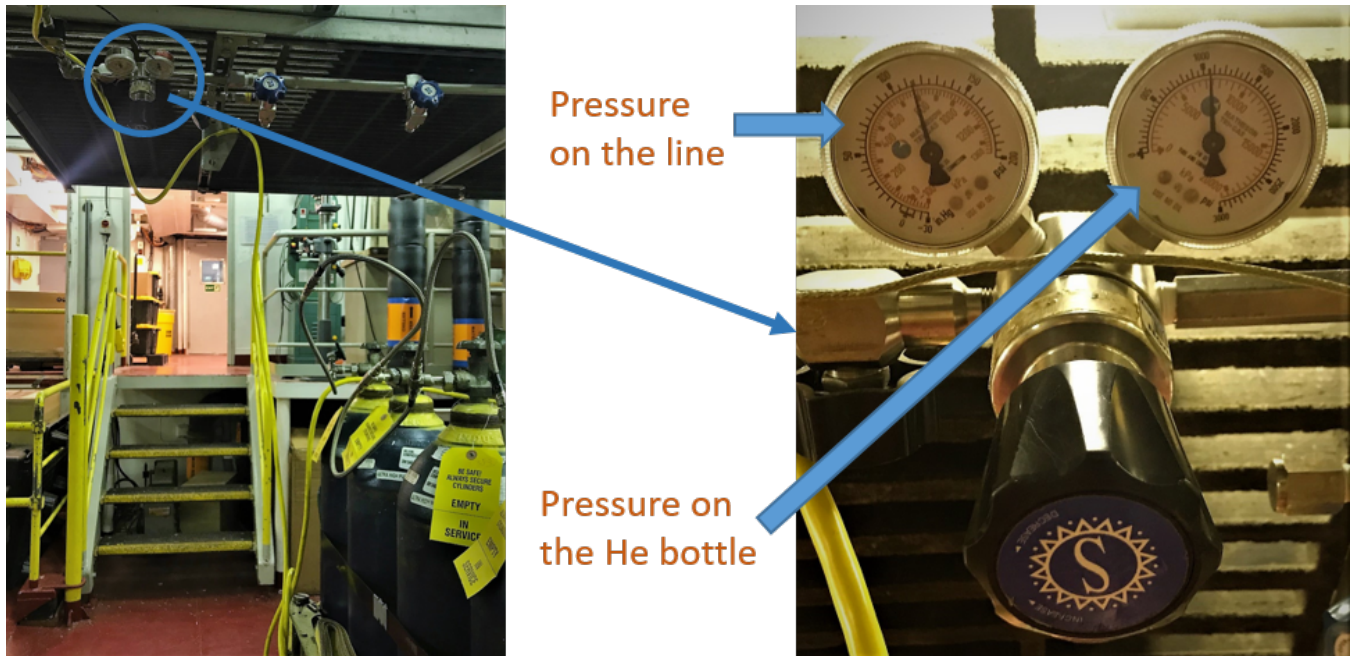
Figure 3. He regulator location in TBULK

Figure 4. Regulator behind pycnometer (set to 19 psi). Both valves, before and after the regulator, should be open.

If the helium pressure is low at the pycnometer, check that the valve in the ceiling above the sample table is OPEN (Figure 5) (Phys Props Lab). Also check the helium gauges in the Chem Lab (Figure 6). The chem lab helium pressure should be 80 psi. Do not adjust the regulators in the chem lab. Just note the pressure reading.

Figure 5. Ceiling panel with He shut-off valve.

Figure 6. Helium valve in the Chem Lab.

Note: The helium supply to cells is plumbed in parallel, which means that the user will notice fluctuations in pressure in the real-time monitoring windows. Each pycnometer measurement is done in isolation from the helium manifold, so this fluctuation has no effect on the results.

Precision Caliper

The caliper (Figure 7) is used to measure the dimensions of cylinder and rectangular prism-shaped samples. The information is entered in the MADMax software, which calculates the volumes of the solid samples.

Figure 7. Precision Digital Caliper for measuring dimensions of certain samples.

Thermo Scientific HERATHERM AP Oven and Desiccator Boxes

The HERATHERM AP oven (Figure 8) used in the moisture determination step is back-vented to the ship's fume hood system to carry away the moisture liberated from the samples as they dry. The mechanical convection oven has a 60 L capacity and is set to 105°C for the MAD process. The butterfly valve (Figure 9) should be kept vertical to allow oven to vent.

The desiccator boxes hold samples after they have been dried in order to prevent reintroduction of water weight. The Drierite used on the JR is indicating. The color should be blue, indicating that the desiccant is good. If the desiccant is purple it is close to being saturated and once it becomes pink it can be redried in the oven. See dessicant bottle for redrying time and temperature.

Figure 8. Thermo Scientific HERATHERM AP 60 L oven, mounted underneath the bench. Back-vented, the oven does not warm the benchtop and alter the results from the analytical balances.

Figure 9. Exhaust tubing and the butterfly valve control (circled in green). The desiccator boxes are to the left of the exhaust tubing.

Electronics and Control System

The software control system is a mixed language tool. MADMax is a C#/.NET application providing sample management, mass measurement, and methods computation. The C#/.NET component integrates with a LabVIEW component to manage volumetric measurement.

Masses are obtained via serial communication (RS232) with dual Mettler Toledo balances. The pycnometer control system supplies the control signals to manage helium flow for the duration of experiments and acquires the resulting data.

Sampling Tools and Sample Containers

Sampling Tools

- For soft materials, syringes/plugs are used to extract sediment samples with a nominal volume of 10 cm^3 from the section halves (Figures 10 a-b).
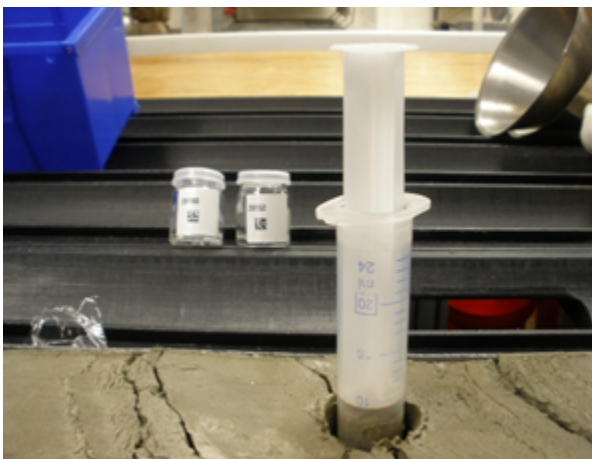

Figure 10a. Using cut-off syringe to get a 10ml sample. container, clean off debris around rim with a kim wipe.
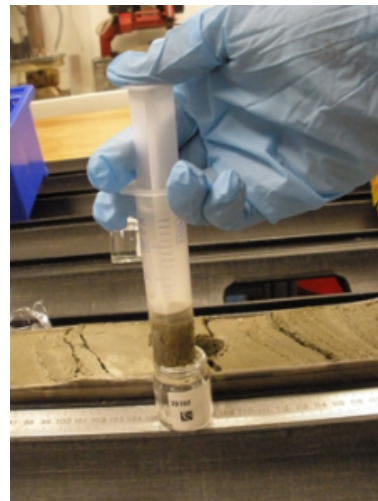
Figure 10b. Pushing sample out of syringe into assigned

- For hard materials, it is necessary to use drills and saws to cut cylindrical and rectangular prism-shaped rock samples; this is done in the core splitting room.

Sample Containers

Sample containers for the MAD analysis are either glass Wheaton vials or anodized aluminum cups (Figure 11). Each of them has a numeric identifier that is used to track the container and its sample throughout the process. Each container's mass and volume are recorded in the database and the MADMax program uses these values to subtract the container mass and volume from the sample values. Boxes of pre-weighed Wheaton vials are on the inventory list and must be checked out.

- Wheaton type 800 vials are used for soft to indurated sediment samples; basically if it fits in the glass vial, use a glass vial. The glass vials have a density of 2.48-2.50 g/cm^3 and their volume has been calculated from the mass determined on shore prior to shipment; their mass is approximately 21 grams and volume is about 8.3 cm^3.
- Anodized aluminum sample cups are used for igneous or consolidated sedimentary samples. The mass of each of the cups was determined experimentally (approximately 14.8 g) and the volume of 5.842 cm^3 was estimated from the dimensions and confirmed by experiment.

Figure 11. Sampling tool (left), aluminum cup, and Wheaton vial for MAD measurements.

MADMax Software and Procedure

MADMax is a C$^\#$ application that controls all of the measurements used in the MAD process. It can be found on the applications web page. Due to browser compatibility issues, MADMax must be installed from Microsoft Internet Explorer; any other browser is unlikely to install properly.

Login

Users must authenticate to the database in order to use the MADMax application. Upon starting the application, the user will see the login screen (Figure 12).
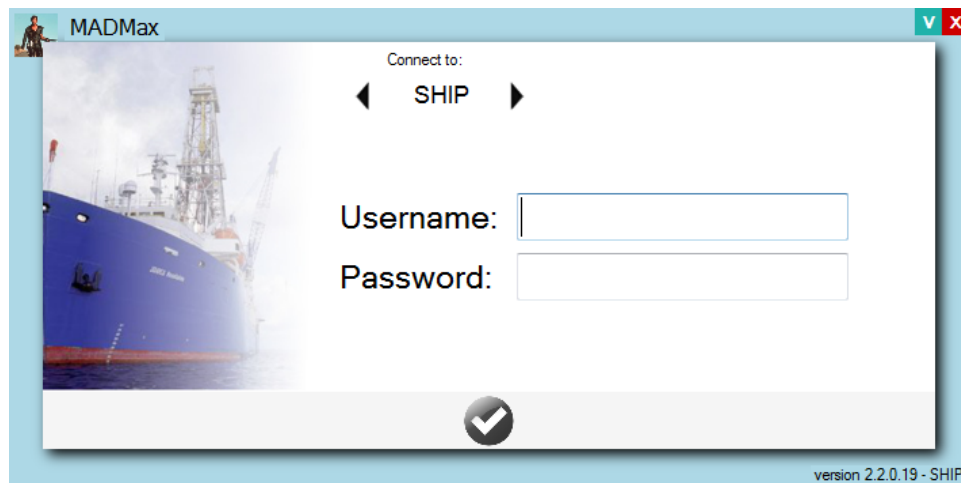
Figure 12. MADMax login screen. Note that the application is aimed at the SHIP database.

If the user cannot login, please see a technician to ensure that the appropriate database authorization has been granted to the user account.

Main Screen

The main screen of the application (Figure 13) is the central command center for the entire process. Various actions on this screen initiate the balance measurements, the pycnometer measurements, the entry of caliper data, and the calculation of the derived MAD results.

Figure 13. MADMax main screen. Note the pycnometer display screens below the main application window.

It is recommended to click the "Display ON" button to turn the live pycnometer monitoring off unless troubleshooting a problem. The live display is memory-intensive and will slow down the functioning of the software.

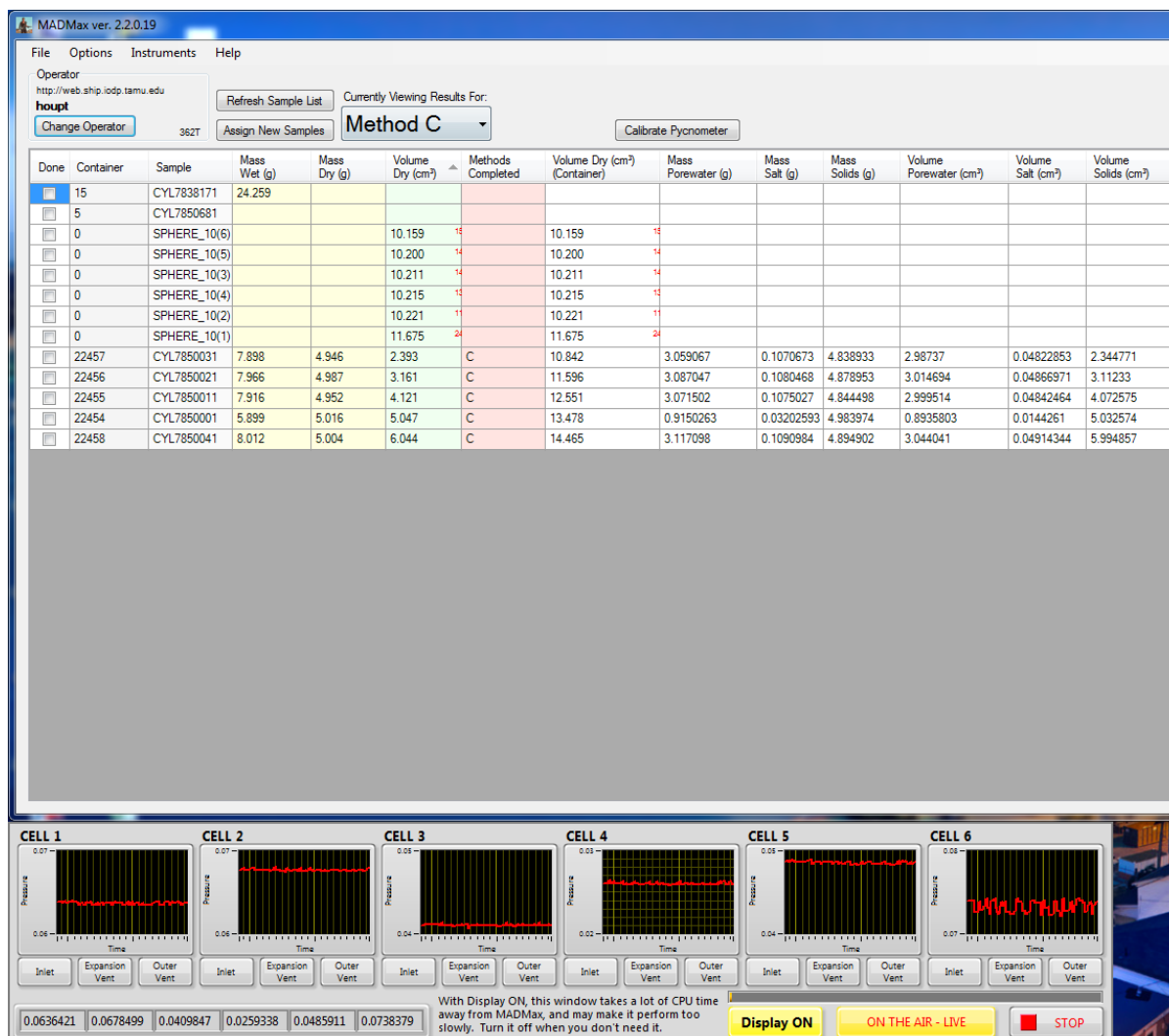Once a sample has been assigned to a MAD vial using the Sample Master program, it will be available to the MADMax application. Click the "Refresh Sample List" button to cause the sample to appear in the table.

> IMPORTANT! Note that once the samples are in the MAD vials, they are tracked solely by their container number. Care should be taken not to confuse the samples at any point in the process.

> **PRO TIP! Keep good logs!**

The main screen spreadsheet can be sorted by clicking on the column header, such as "Container". This will put all samples in order by container number.

MAD Method C is the most common one used on the *JOIDES Resolution*, so the MADMax application defaults to the "Method C" mode. The method indicator is a pull-down menu to switch between the four methods A, B, C, and D. Again, Method A and Method B are not recommended. Regardless of method, the user can make up to five discrete types of measurements:

- Wet mass determination by analytical balance
- Dry mass determination by analytical balance
- Dry volume determination by pycnometer
- Wet volume determination by pycnometer (not recommended)
- Wet or dry volume determination by precision caliper

Depending on the method selected, different actions will be available by double-clicking on the color-coded section of the main screen appropriate to the method. For example, to make a wet mass measurement on a sample, double-click on the left-hand yellow cell as shown in Figure 14. This action will invoke the balance control software portion of MADMax.
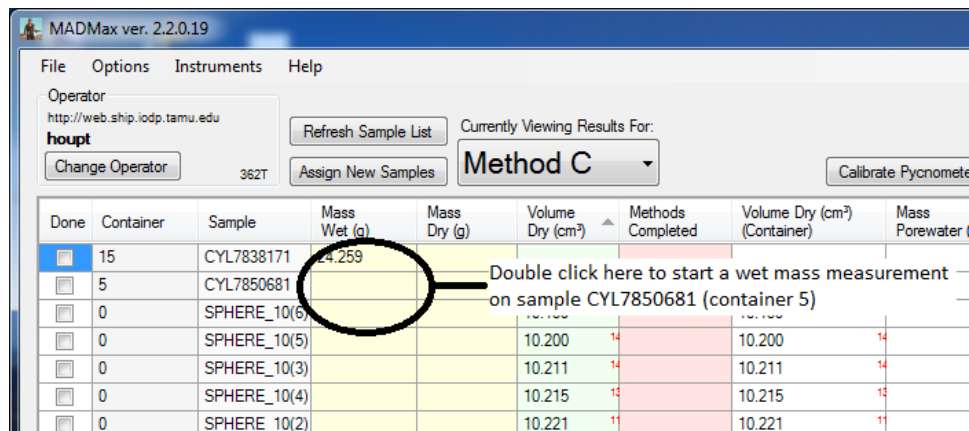
Figure 14. Activating a wet mass measurement.

Order of Actions by Method

For each method, the measurements should be done in a specific order, as given below. Note that the MADMax interface does not sort these columns by this order, but the columns can be rearranged to do so if the user wishes.

- Method A: Wet volume (caliper), wet mass (balance), dry mass (balance)
- Method B: Wet mass (balance), wet volume (pycnometer), dry mass (balance)
- Method C: Wet mass (balance), dry mass (balance), dry volume (pycnometer)
- Method D: Wet volume (caliper), dry mass (balance), dry volume (pycnometer)

In Method D's case, "wet volume" is better stated as "bulk volume," because the method is used only for samples with such high porosity that the water cannot be kept inside the sample (e.g., vesicular basalt or corals). Figure 15 shows all of the methods and the required measurements for each one.
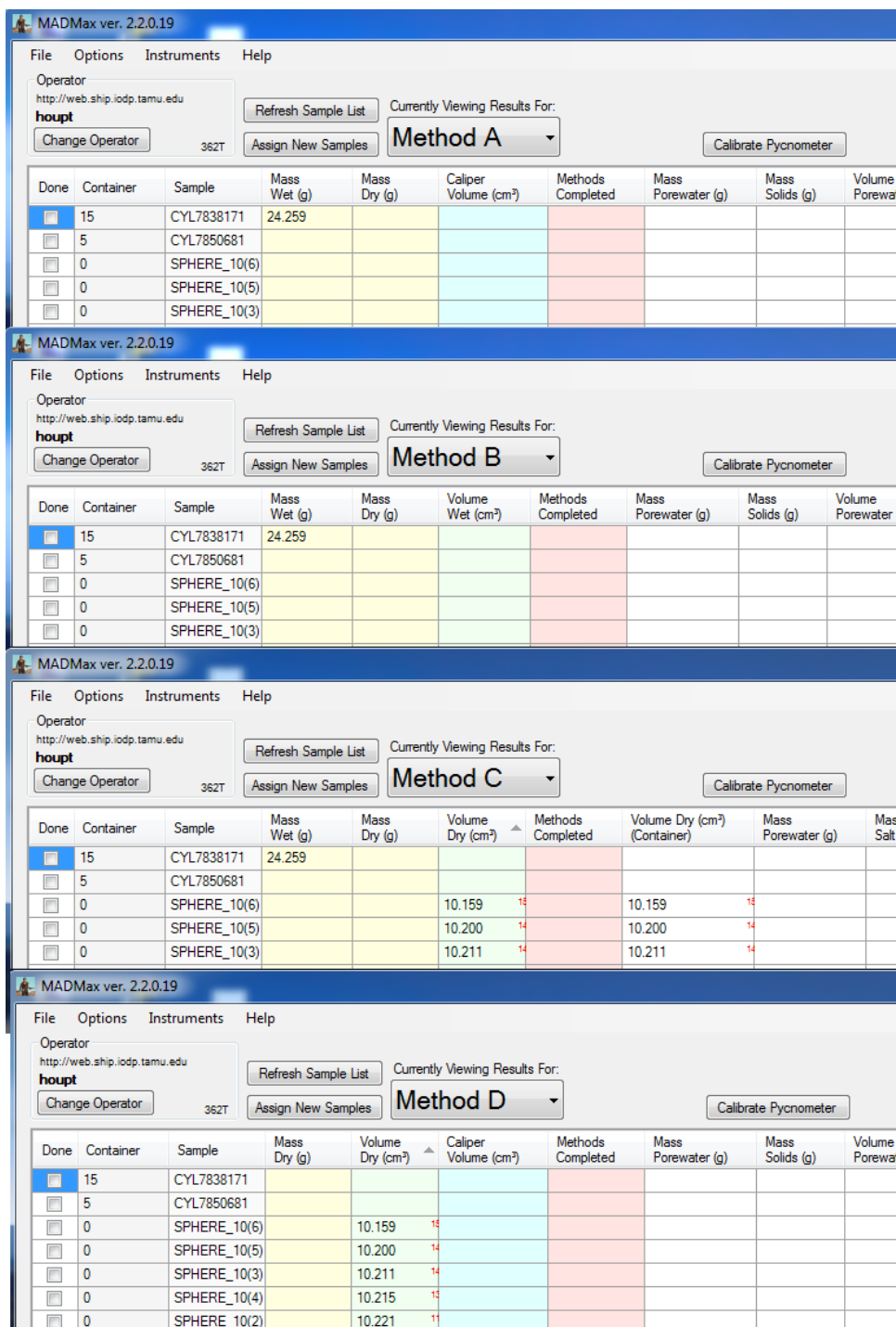
Figure 15. All of the MADMax methods shown in cascade style.

In Figure 15, note that the colored columns for the measurements (yellow for balance, green for pycnometer, and blue for caliper) are slightly different for each method. The pink Methods Completed column is the same for each method and will be discussed later in the MAD Calculations section.

Wet Mass Determination by Analytical Balance

As shown in Figure 14, above, double-click on the "**Mass Wet (g)**" column in the field adjacent to the sample to be analyzed. This will invoke the Balance Measurement dialog box as shown in Figure 16. The container number and the full Label ID of the sample will be displayed to ensure the correct sample was selected. Select the number of measurements to average (at least 300 is recommended, more if the sea state is high) and click "**Measure**." The measurement speed of the XS-204 balances is 5 Hz, so 300 measurements will take 60 seconds. The next screen will appear (Figure 17).

Figure 16. Balance Measurement dialog box. A typical expedition Label ID would be formatted as follows: 360-U1473A-21R-2-W 10/12-20127.

Taring the Balances

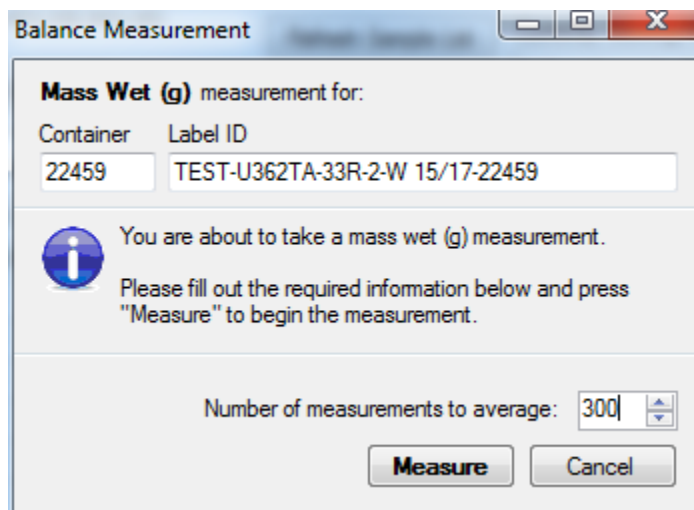The first step to be taken is to tare the balances; measure the tare with empty pans. This step measures the *differential* tare between the two balances and is used to create a motion-compensated tare value. Do not try to tare the balances by using the Tare button on the keypad; the balances **must** be tared using the software. The window header will show the Text ID of the sample, what measurement is being done (e.g, "Mass Wet (g)"), and the container number.

Activate the tare function by clicking the "**Tare**" button on the upper left-hand corner of the screen (circled in green) and the tare will begin. The reference balance trace is green, the unknown balance trace is red, and the corrected mass (in this case the tare value) is a blue trace.

The balances should be tared relatively often as many things can affect the balance reading. The most common causes of these changes are:

- Spilling something on the balance pan (and please don't leave it there!)
- Cleaning something that was spilled on the balance pan
- Temperature changes in the laboratory may affect the zero point of each balance

It is recommended that the user tare no *less* frequently than 6 hours, but certainly if the user suspects anything may have changed on either balance.

After a tare, check by placing a known weight (20g) on both balances and click "**Weigh**". The corrected value should read 20g. If not, take a new tare.
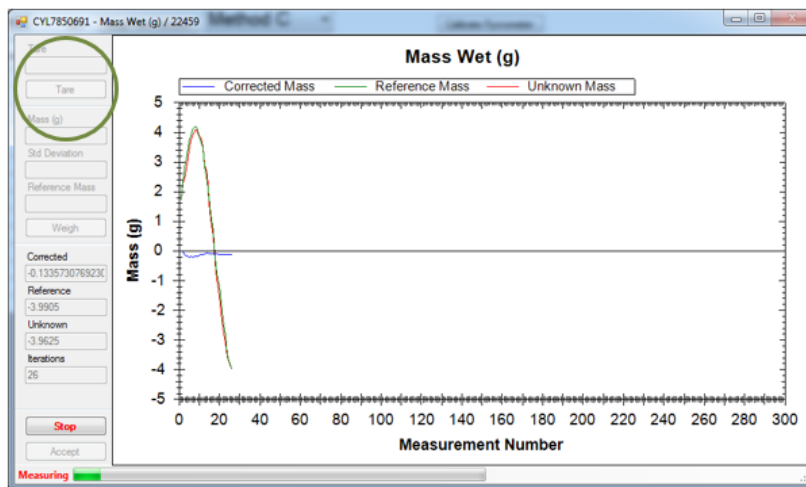

Figure 17. Taring the paired balances at 300 measurements.

Making the Measurement

Once the tare is complete, place the sample container on the unknown balance and close the sliding door. Place counterweights from the standard box as close to the mass of the unknown as possible and close the reference balance door. It may be useful to look at the balance LCD screens; the circle shows the rough load on the balance and masses can be roughly equalized by using them. It is important to get the masses within 5 grams. If the red and green traces are more than 5 grams apart after the measurement is started, then press the "Stop" button, add or subtract reference masses, and start the measurement over.

The "Reference Mass" field should be filled with the total mass of the reference masses before the measurement is started. Figure 18 shows the Reference Mass (circled in blue) and the traces of the two balances and their corrected result mass. Note that the reference mass balance began at a value of just under 19 grams and the unknown balance started close to 21 grams; the difference between them is 5 grams, so this measurement could be allowed to continue.
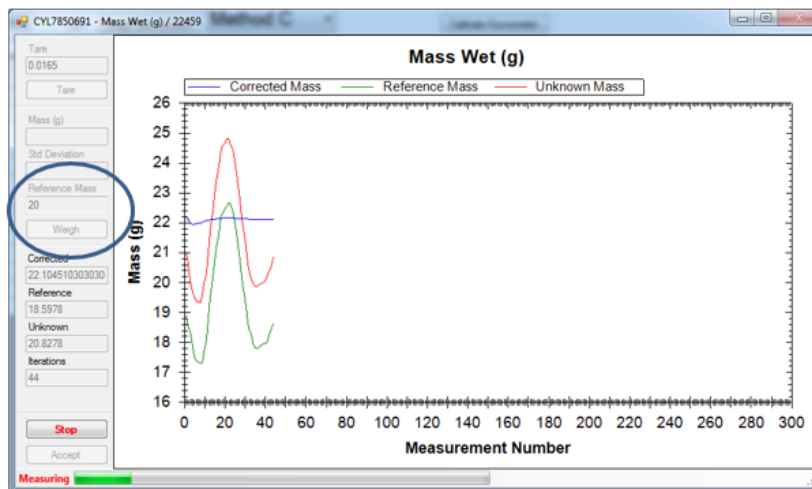
Figure 18. Mass measurement on a sample.

Once the analysis is completed (Figure 1), the mass determined by the measurement process will be displayed (in this case 22.120 grams). The "**Tare**" button is active again, but it not appropriate to use it at this time without removing the sample and reference masses. The user has three choices: accept the result and send it to the main screen display and the LIMS database by clicking the "**Accept**" button, reweigh the mass by clicking the "**Weigh**" button again, or completely cancel the measurement and discard all results by clicking the "**Cancel**" button.

The user should note that the instantaneous values of the balances varied by more than 20 grams in this example because of ship's heave, but the measured mass (22.110 grams of known masses) was accurate to within 0.010 grams.

> **Note**: At the time of writing this manual, the Std Deviation field is recording the standard deviation of the unknown mass measurement (which obviously varies highly). An upcoming upgrade will switch this to the corrected mass value.

Figure 19. Completed measurement.

Once the "**Accept**" button is pressed, the user is returned to the main MADMax screen.

Reassigning Results

The result should be in the appropriate field, but in case the user double-clicked the wrong mass measurement, MADMax provides the capability to switch the mass measurement from "wet" to "dry" and vice-versa. As shown in Figure 20, right-click the mass cell and select the "**Swap the result wi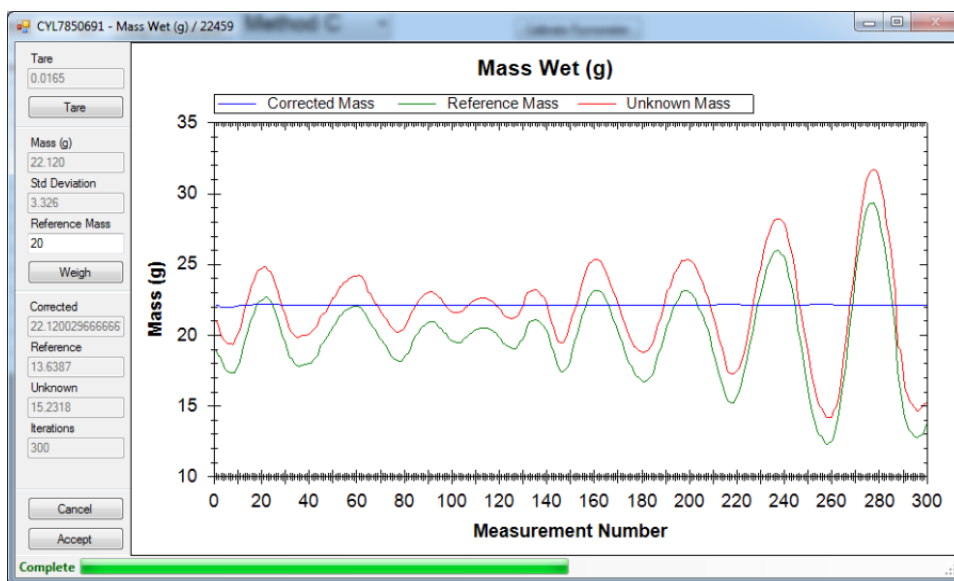th Mass Dry (g)**" option. If two masses are already present, this option will instead state "Swap Mass Dry (g) and Mass Wet (g)" to swap the mass measurements.

mple	Mass Wet (g)	Mass Dry (g)	Volume Dry (cm³)	Methods Completed	Vol (Co
L7838171	24.259				
L7850681					
HERE_10(6)			10.159		10.1
HERE_10(5)			10.200		10.2
HERE_10(3)			10.211		10.2
HERE_10(4)			10.215		10.2
HERE_10(2)			10.221		10.2
HERE_10(1)			11.675		11.6
L7850031	7.898			Cancel this mass_wet result	0.8
L7850021	7.966				1.5
L7850011	7.916			Review this result	2.5
L7850001	5.899			Swap this result with Mass Dry (g)	3.4
L7850041	8.012	5.004	6.044	C	14.4

Figure 20. Right-click options for the mass measurement.

Once the user clicks the "Swap" options, a window will pop up to confirm the action as shown in Figure 21; the window for moving a result from wet-to-dry or dry-to-wet is very similar.

Reassign Result

Swap results between Mass Wet (g) and Mass Dry (g)

You are about to swap measurement results between Mass Wet (g) and Mass Dry (g) for the sample:

TEST-U1473A-97R-4-W 45/47-22457

Pressing "Ok" will immediately cancel the original results, and create new results with the values swapped.

Do not press "Ok" unless this is what you want to do.

[Ok] [Cancel]
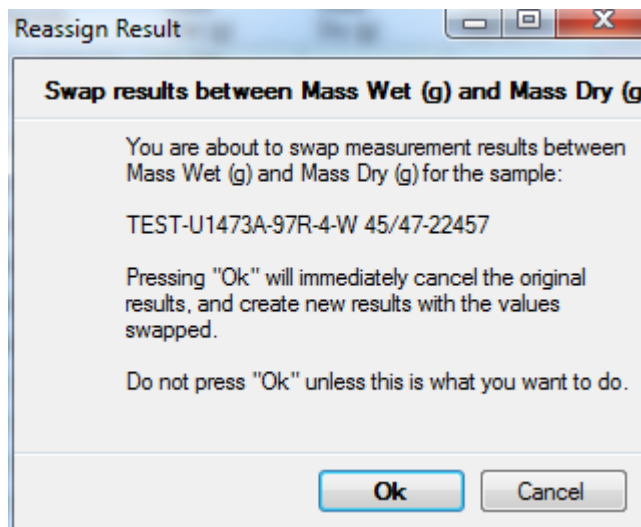
Figure 21. Reassign Result window. It is important to be able to do this as the drying

step is irreversible and the wet mass cannot be repeated without taking a new sample.

Cancel Mass Result

The user can also cancel a result using the same right-click option, with a confirmation window as shown in Figure 22. A developer or technician can uncancel the result if this was done in error.
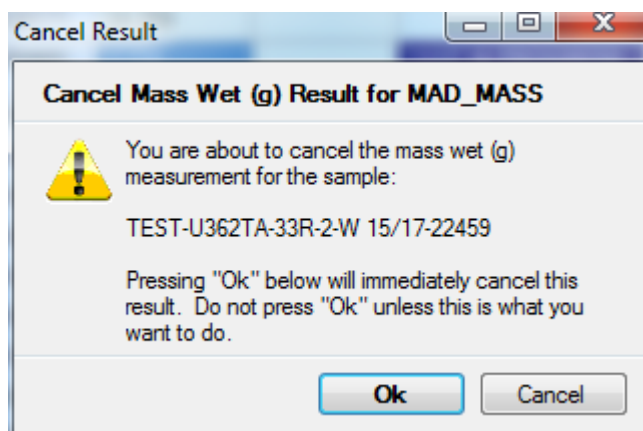
Figure 22. Cancel mass result pop-up window.

Review Results

If the user wishes, they can also look at the details of the mass results as shown in Figure 23. This is a summary of all of the parameters used to determine both the wet and dry masses with the parameters labeled by their database names.
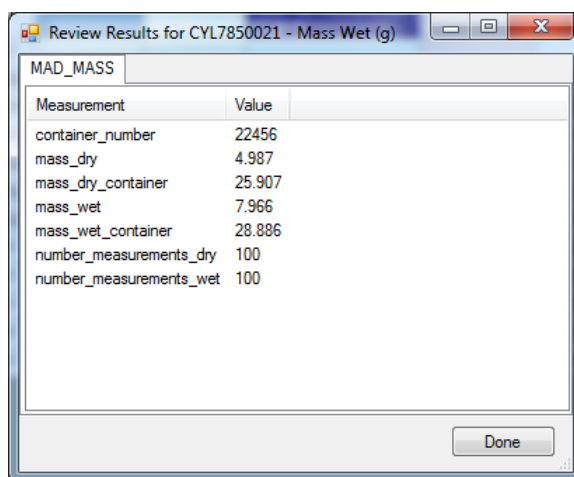
Figure 23. Review wet mass results window.

The mass_dry and mass_wet components are the mass of the sample. The mass_dry_container and mass_wet_container components are the mass of the sample and the container together.

Dry Mass Determination by Analytical Balance

Put samples in the oven, underlined, for at least 24 hours at 105°C . Let them cool to room temperature in the dessicator boxes for about 3 hours before placing inside pycnometer cells.

This step is performed the same way as the wet mass determination. Double-click on the "Mass Dry (g)" column in the field adjacent to the sample to be analyzed. Follow the rest of the instructions in Wet Mass Determination by Analytical Balance, except that it applies to the dry mass measurement.

Dry Volume Determination by Helium Pycnometer

Calibration of the Pycnometer Cells

Before any pycnometer measurements can be made, the pycnometer cells must be calibrated. On the main screen, click the "**Calibrate Pycnometer**" button on the right side of the main screen in order to invoke the calibration window as shown in Figure 24.
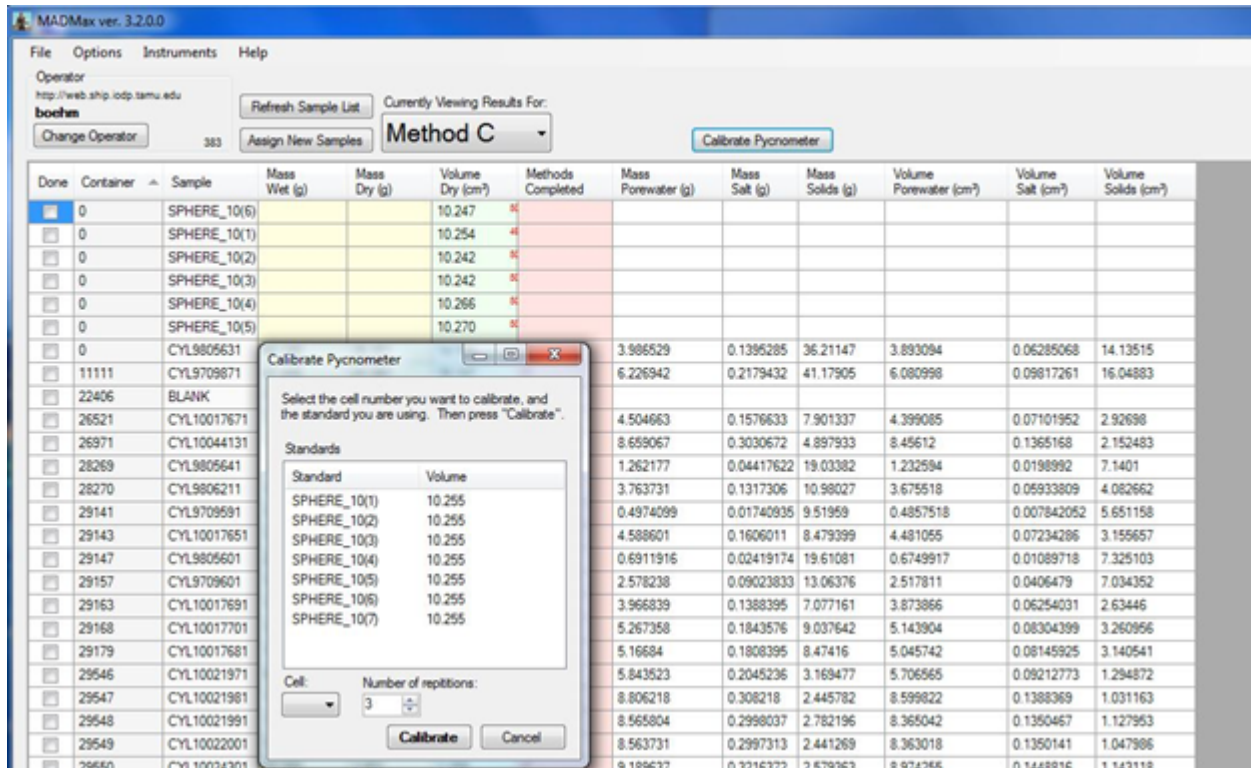
Figure 24. Calibrating the pycnometer cells.

First select the standard to be used in the calibration. For the 35 cm^3 inserts (Wheaton vials and aluminum cups), the best results are obtained by using "SPHERE_10," which is actually a 3 cm^3 and 7 cm^3 standard used together. Once the standard is selected (again, the normal standard used is "SPHERE_10"), assign it to a pycnometer cell using the pull-down selector as shown in Figure 25. Use at least 3 repetitions for good precision between measurements. Select SPHERE_10(1) and assign to Cell 1, SPHERE_10(2) assign to Cell 2, and so on.
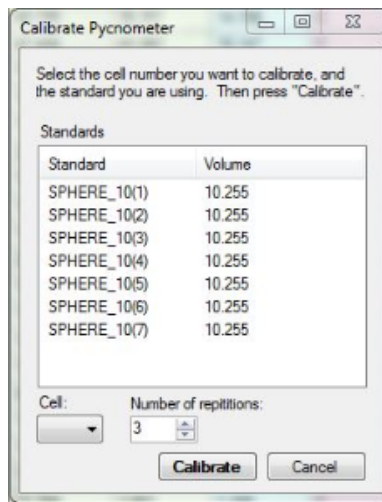
Figure 25. Selecting the standard and then assigning it to the cell.

Click "**Calibrate**" once the standard and the cell have been assigned and the number of replicates is chosen. A window will appear for the cell that was chosen as shown in Figure 26. The first calibration step is to measure the empty cell. Ensure that the cell is empty before clicking "**Done**". The measurement will start immediately. If calibrating multiple cells, click on Calibrate Pycnometer again to set up and start the next cell, all six can run simultaneously and at different steps in the measurement cycle. You will end up with 6 measurement windows on the screen.
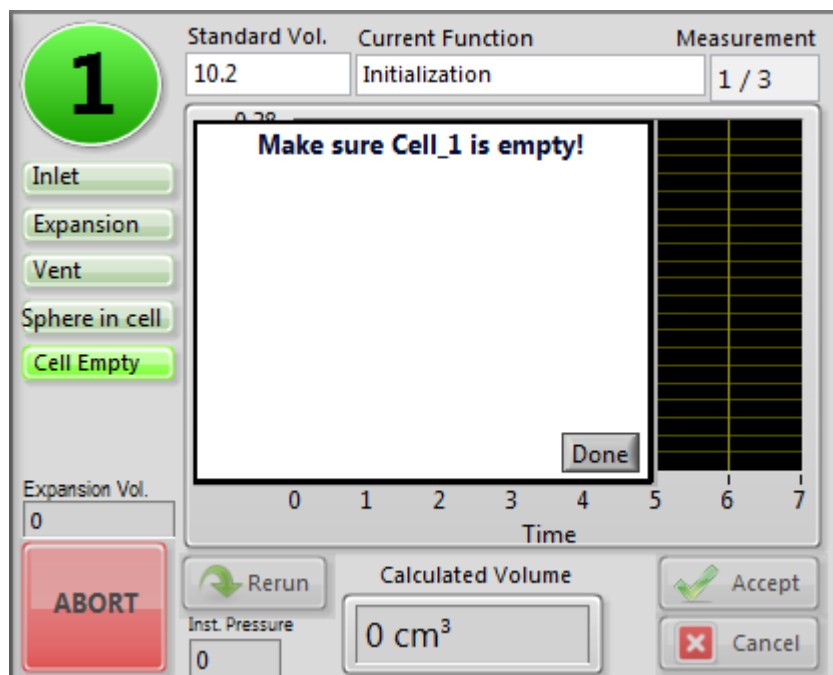
Figure 26. Cell 1 calibration window.

The pycnometer will step through its full measurement cycle once for each of the replicates selected (so three times if "3" was selected) as shown in Figure 27.
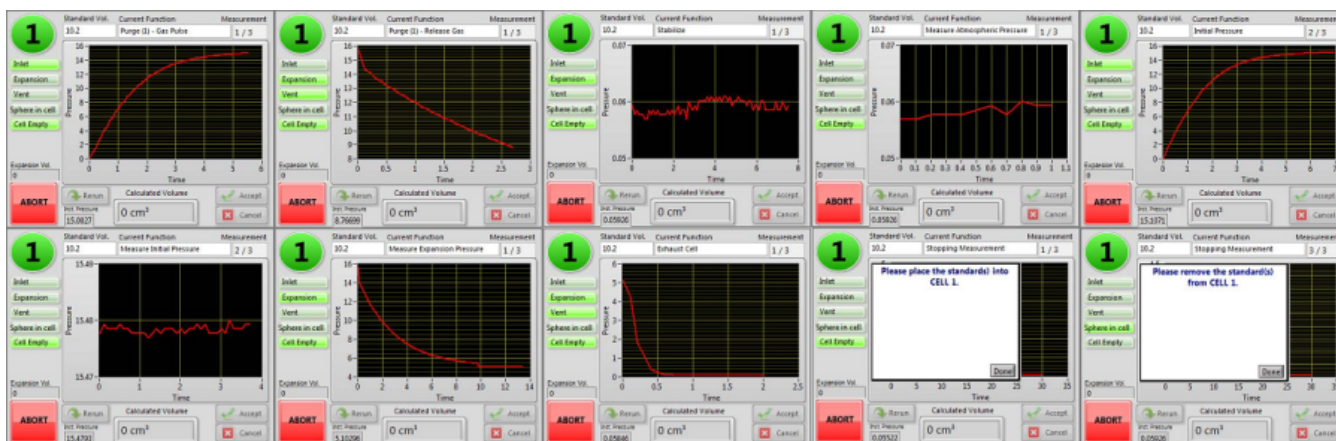
Figure 27. Pycnometer analysis steps.

The steps are detailed here:

1. The purge pulse and release step will repeat as many times as the number of replicates prior to moving on to the analytical steps below.
   a. Purge (no.) Gas Pulse—fills the main chamber with helium
   b. Purge (no.) Release Gas—empties the helium through both chambers
2. The following steps will repeat as many times as the number of replicates (without going back to the purge step).
   a. Stabilize—allows the chambers to come to equilibrium with each other after the purge step.
   b. Measure Atmospheric Pressure—measures the zero-point pressure in the analytical chamber (only).
   c. Initial Pressure—opens the helium inlet to fill the analytical chamber.
   d. Measure Initial Pressure—measures the initial pressure in the charged analytical chamber.
   e. Measure Expansion Pressure—opens the expansion valve and then measures the equilibrated pressure in the analytical plus expansion chambers.
   f. Exhaust Cell—releases the helium from both chambers.

Once the empty cell has been measured the specified number of times, the unit will prompt the user to add the standard(s) to the cell; the "SPHERE_10" standards are found in the wooden box shown in Figure 28. Open the pycnometer cell and place the standard ball(s) into the cell, then close securely. Clicking "**Done**" will trigger the above analytical steps, including purges, for the standard ball(s). After the replicate measurements on the steel ball(s) are done, the system will prompt the user to remove the standards from the cell. Pressing done at this point shows the expansion (red circle) and analytical cell (black circle) volumes determined by the calibration experiment as shown in Figure 29.

Figure 28. Six sets of steel balls are kept in this box along with the wire tool to extract them from the cells.

Note: Do not touch the spheres with bare fingers! Fingerprints don't significantly affect the volume measurement, but will cause the spheres to begin corroding. Wipe them thoroughly off with a Kim-Wipe if it is necessary to do so.
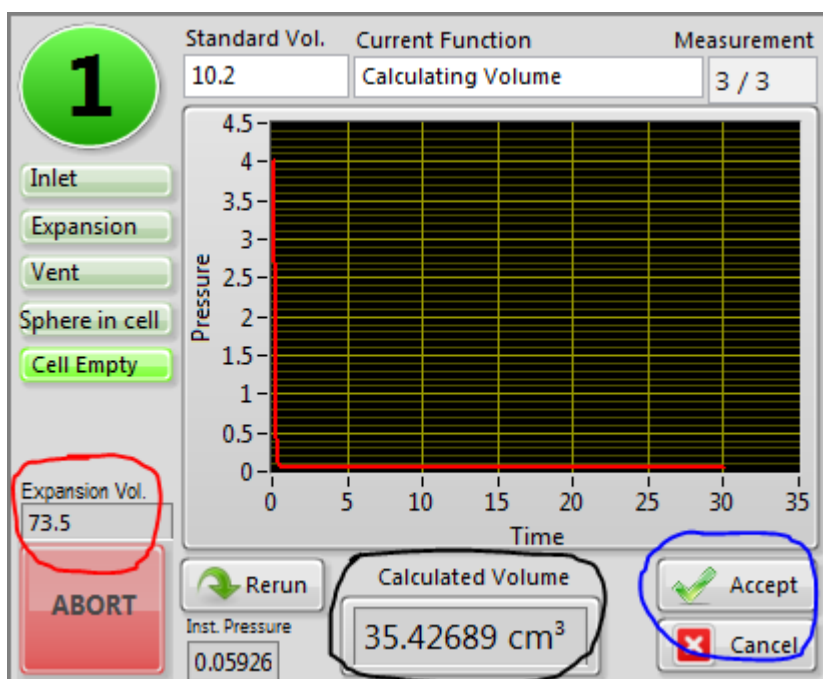
Figure 29. Final calibration screen for the pycnometer cell.

Click the "**Accept**" button (blue circle) to complete the calibration process for this cell. Once a cell's calibration is completed the pycnometer measurement on unknowns (i.e., samples) can be started independently of the other cells undergoing calibration.

To check the calibration, run the same set of standards as a sample. Double click on the spreadsheet in the green column ("Volume Dry") for the cell and sphere you want to test. You may need to click "options" and select "include complete MAD tests" to display all samples. The Calculated volume should be between 10.098-10.302. Enter the value in the calibration excel spreadsheet on the desktop. The spreadsheet is conditionally formatted where if the standard volume is out of this range it will turn red. That means something is wrong and the cell needs to be calibrated again.

Always use the standards that are labeled for the corresponding cells. The standard containers are labeled 1-6 to match up will cells 1-6.

Frequency of Calibration/Calibration Check Standards

The pycnometer has proven to be quite stable, so the standard practice is to calibrate the instrument at the beginning of an expedition. To ensure quality, each set of five samples should be accompanied by a standard rotated through the cells. So long as the standards continue to give an acceptable value (less than 0.5% deviation), recalibration is not necessary (NOTE: As of X375 the recommendation is to Calibrate the beginning of each shift, and then +/- 0.5% deviation).

Rotation of the check standards through the cells is important, however, to ensure on a continuing basis that the system is still stable and functioning properly. An example measurement plan is given here:

- Measure a sample in Cell 2, 3, 4, 5, and 6. Measure the 10.2 standard (3 cm^3 + 7 cm^3 balls) in Cell 1.
- Measure a sample in Cell 1, 3, 4, 5, and 6. Measure the 10.2 standard in Cell 2.
- Measure a sample in Cell 1, 2, 4, 5, and 6. Measure the 10.2 standard in Cell 3.
- Measure a sample in Cell 1, 2, 3, 5, and 6. Measure the 10.2 standard in Cell 4.
- Measure a sample in Cell 1, 2, 3, 4, and 6. Measure the 10.2 standard in Cell 5.
- Measure a sample in Cell 1, 2, 3, 4, and 5. Measure the 10.2 standard in Cell 6.
- Repeat pattern.

The SPHERE_10 standard fills the 35 cm^3 chamber to the fullest degree and will give the best precision in measurements. Using a smaller standard will result in a larger headspace and a lower pressure drop, and therefore lower precision.

Use the air hose behind the pycnometer to clean out any dust and debris that gets in the cells. Check o-ring on the cell lids for any damage or dirt.

Making a Pycnometer Measurement on a Sample

After calibration is complete, the user can begin analyzing samples and one standard per six runs as noted above. Double-click on the Volume Dry (cm^3) portion of the main screen next to the sample to be studied; normally the SPHERE_10 standards are also listed for the one cell in six to be dedicated for a standard measurement. A window will appear as shown in Figure 30 prompting the user to select a cell and to set the number of replicates. It is recommended to use the same number of replicates as the calibration.
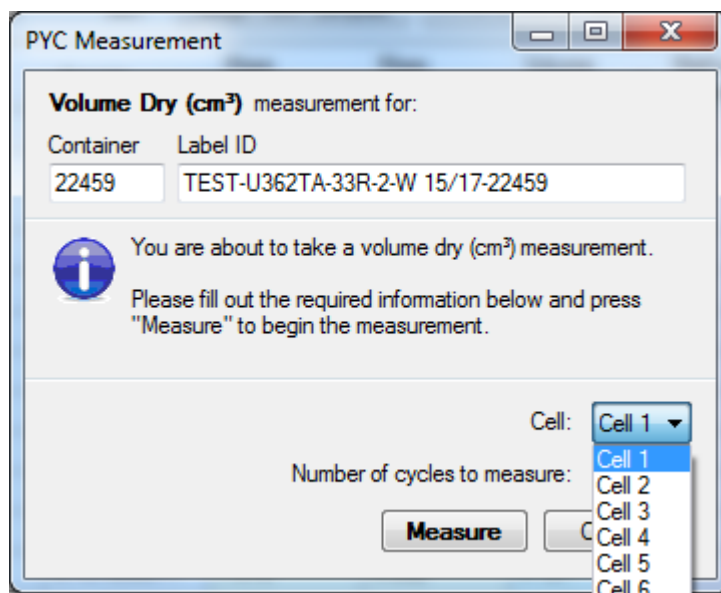
Figure 30. Pycnometer measurement window.

Important! Ensure that the sample is correct and select the cell to be used and the number of replicate measurements to be performed.

Once the user has selected the cell and number of cycles, click the "**Measure**" button, which will open the pycnometer cell control window (Figure 31). Place the sample container in the cell using long tweezers and do not put the cap on the container (Figure 32). Screw the lid back on the cell and then click "**Done**" followed by clicking the "**Start**" button (circled in blue). Unlike the calibration measurement, the experiment will not start until the "**Start**" button is clicked.
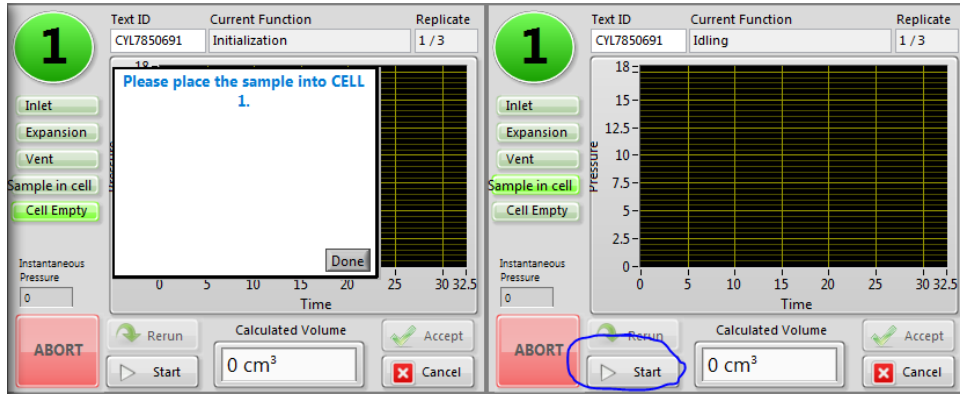
Figure 31. Starting a pycnometer measurement on a sample.
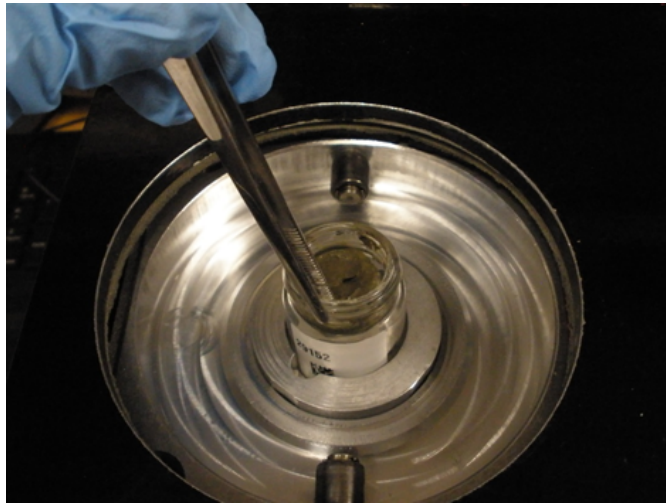
Figure 32. Loading the sample container into the cell.

The pycnometer will step through the same purge and measurement steps described in the calibration section and when it is finished, will show the screen shown in Figure 33.
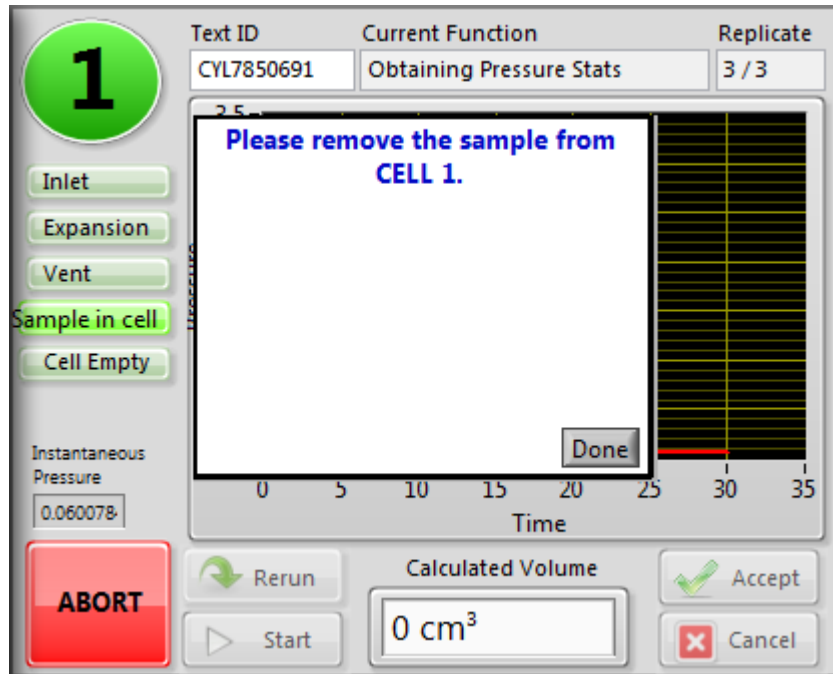
Figure 33. Sample completed indicator for pycnometer.

Once the "**Done**" button is clicked, the volume of the sample (including the volume of the container) will appear in the "**Calculated Volume**" window. At this point, the user may select "**Rerun**," which will redo the measurement, "**Cancel**" to reject the measurement, or "**Accept**" to keep the data and transfer it to the main screen.

Reassigning Results

The result should be in the appropriate field, but in case the user double-clicked the wrong volume measurement, MADMax provides the capability to switch the volume measurement from "wet" to "dry" and vice-versa. As shown in Figure 34, right-click the mass cell and select the "Send this result to the Volume Wet (cm^3)" option. If two volumes are already present, this option will instead state "Swap Volume Wet (cm^3) to Volume Dry (cm^3)."

Figure 34. Right-click options for the volume measurement.

Once the user clicks the "Swap" options, a window will pop up to confirm the action, similar to that for the swap mass measurements window.

Cancel Volume Result

The user can also cancel a result using the same right-click option, with a confirmation window similar to the one for canceling mass measurements mentioned earlier. A developer or technician can uncancel the result if this was done in error.

Review Results

If the user wishes, they can also look at a detail of the mass results as shown in Figure 35. This is a summary of all of the parameters used to determine the volume with the parameters labeled by their database names.
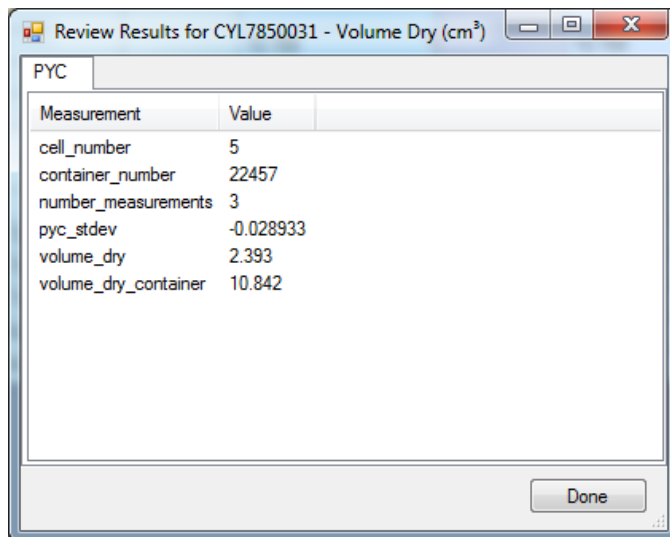
Figure 35. Review dry volume results window.

The volume_dry component is the volume of the sample. The volume_dry_container component is the volume of the sample and the container together. The pyc_stdev value is calculated from the individual measurements (in this case, three) done by the pycnometer.

Volume Determination by Caliper

Volume determination by caliper is best used on consolidated, high-porosity samples (e.g., coral or vesicular basalt) as part of Method D. It is not likely to give accurate results when used on soft sediments (Method A).

Double-click on the "Caliper Volume (cm^3)" column in the field adjacent to the sample to be analyzed. This will invoke the caliper measurement window as shown in Figure 36. The user should select cube or cylinder depending on the sample to be measured with the precision caliper.
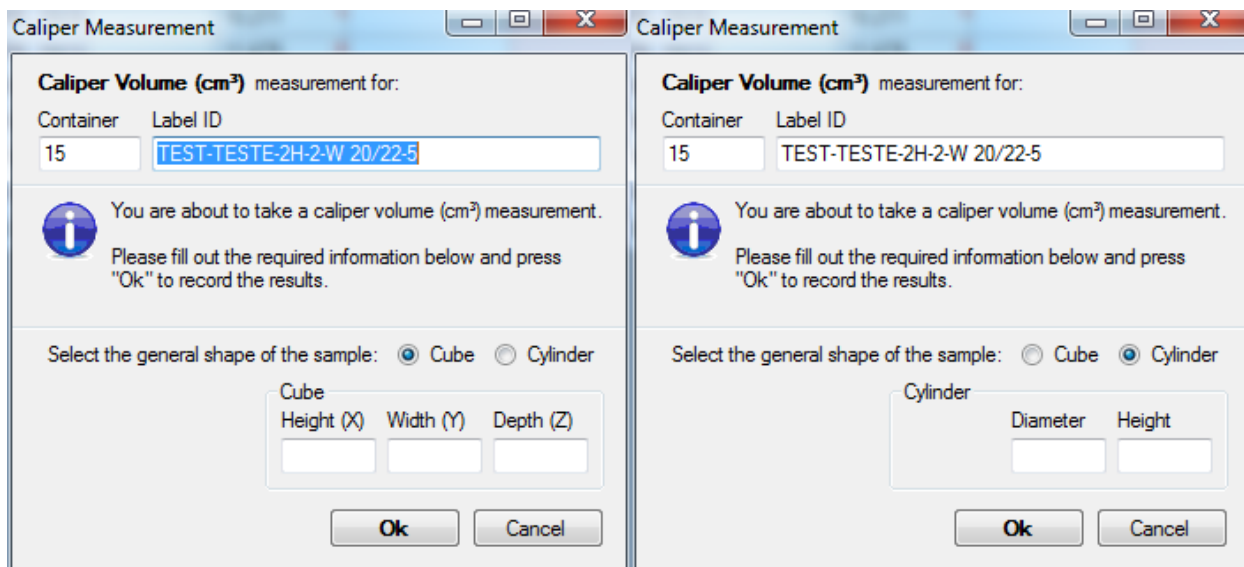
Figure 36. Caliper measurement entry screens.

Ensure that the sample being measured is the correct one and click either the "**Cube**" or "**Cylinder**" radio button. Make the measurements using the precision caliper (Figure 37). Enter the measurements for each dimension in centimeters. Once the measurements have been entered, click "**Ok**" and the result will be transferred to the main screen.

Figure 37. Digital caliper used for volume measurements.

Review Results

Right-clicking the caliper result will allow the user to select "Review Results" to see details of the measurement.

Cancel Result

The right-click window also gives the user the ability to cancel the result. A developer or technician can uncancel the result if this was done in error.

Wet Volume Determination by Helium Pycnometer

This method (part of MAD Method B) is not recommended as water vapor will be produced from the wet sample and negatively affect the data quality!

However, if the user wishes to perform this analysis, use the same guidelines as found in Dry Volume Determination by Helium Pycnometer, above.

Hard Rock Saturation Method

1. Take a sample cube from the working section half, it is common to use the same size dimensions as a p-mag cube (or to share the same sample as long as scientists understand the measurement is destructive).
2. Hard rock samples do not require a vial and therefore use container ID "0" when entered.
3. Place the sample cube into a snap cap vial and fill until sample is covered with seawater.
4. All hard rock samples need to be saturated completely before running them through the MAD measurements. To do this, all samples will need to be placed in a vacuum for at least ~12hrs, preferably 24hrs.
5. To set up the vacuum, the vacuum bowl is filled with seawater before setting up the remaining components.
6. Connect the bowl to a vacuum hose that runs through the drain flask, this keeps any water getting to the pump. Place the stopper with the copper tubing in the top of the flask before directly connecting with tubing to the pump.
7. Once this is set up, the pump can be turned on to pressurize the vacuum bowl, it is recommended to run the pump intermittently during saturation, about 30 minutes at a time every 4 hours.
8. Once the samples have been under vacuum for the allotted time, they can be weighed for wet mass and placed in the oven to dry at 110C.
9. After 24 hours, the samples are then weighed for dry mass.
10. The hard rock sample can be placed directly into the pycnometer for measurement.

If the container ID was not entered as "0" in SampleMaster the User can add the container ID directly in MADMax by double clicking in the container cell that directly correlates with the desired sample.

MAD Calculations

The MADMax software will calculate all of the other MAD parameters once any three of four measurements are completed: wet mass, dry mass, dry volume, and wet volume. Once the three measurements related to the chosen method (e.g., Method C: wet mass, dry mass, dry volume) are completed, double-click on the "Methods Completed" field on the line of the sample in question. This will invoke the MAD calculation window as shown in Figure 38.
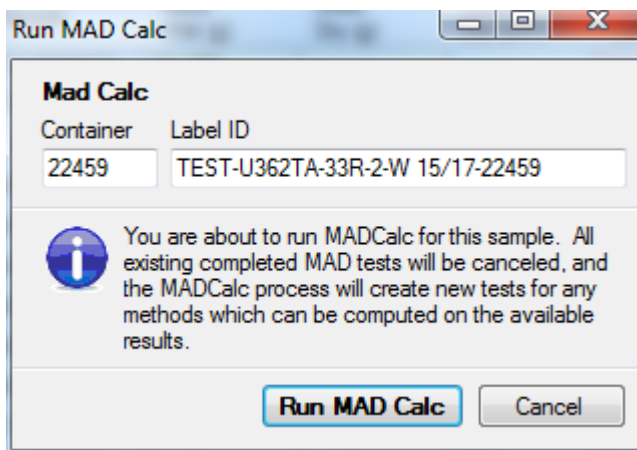
Figure 38. Run MAD Calc window. Ensure the proper sample is shown.

Click on the "Run MAD Calc" button and after a brief pause, the calculated parameters for the MAD analysis will appear on the main screen for that specific sample and will be updated in the LIMS database at the same time. See the "MAD Computations" section below for more information about the various parameters determined by the software.

Cleaning Up the Main Screen

After the MAD calculation is run, the user may wish to clean up the main screen list of samples. This is done by clicking in the "**Done**" boxes as shown in Figure 39. Once the samples to be removed are checked, click the "**Refresh Sample List**" button and the checked samples will disappear. (Any new samples will also appear.)

Note: Until this step is completed, two (and rarely three) lines will appear in the LIMS reports. Once this step is completed, the extra lines will be canceled.
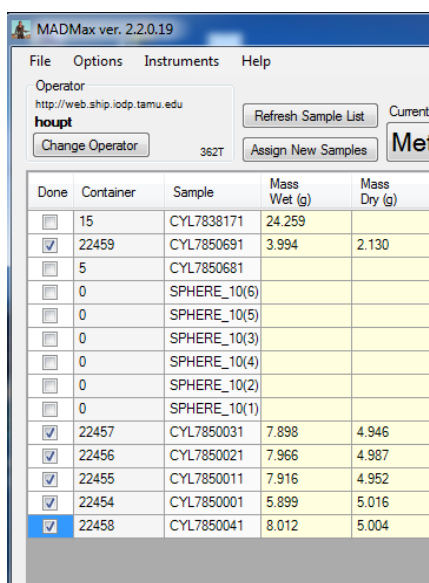
*Figure 39. Using the "**Done**" check boxes to clean up completed samples.*

Quality Assurance/Quality Control

QA/QC for MAD analyses consists of instrument calibration and continuing calibration verification for accuracy determination. The analytical balance calibration is checked by JRSO technical staff and verified by weighing reference masses. The pycnometer system is calibrated using precision ball bearings and verified with calibration verification experiments.

Analytical Batch

The user is encouraged to treat each group of five samples as an analytical batch and to carry a QC sample through the pycnometer process as the "sixth" sample. Thus, each pycnometer batch will consist of five samples and one calibration verification sample.

For the balance and caliper, the accuracy and precision of the equipment can easily be determined by measuring standards and confirming the system is giving proper numbers.

Precision and Accuracy

Each science party must define what error is acceptable for measurements, but the JRSO has some guidelines as to what can be expected.

Balance

For repeated mass measurements, we expect the data to be reproducible within 0.005 g; this will be significantly higher if the sea state is rough, the weights on the two balance pans are not roughly equal, or the number of measurements is too low. In other words, if the masses on the reference and unknown balances are within 5 grams of one another and the number of measurements is sufficient for the sea state, then we expect measurement of a reference mass to be within 0.005 grams.

Pycnometer

On dry samples or the steel ball bearings when the sample container is nearly full, we expect 1% or better precision between measurements. Reproducibility will go down severely if the sample container is not filled, and even more so if wet samples are measured. Accuracy is expected within 1% of the indicated volume if conditions are correct (dry sample, container nearly full).

Caliper

The precision caliper is capable of measurements accurate to 0.01 mm ± 0.01 mm. However, the source of error in this measurement is largely from the quality of the sample being measured. If the rectangular prism's sides are not end parallel to one another, or if the cylinder is not a right cylinder, the error can be significant.

Maintenance and Troubleshooting

1. If system components are turned on after a period of non-use, allow the system to warm up for at least 30 min before analyses are performed.

2. Keep the cap on the cell chamber except when actually inserting or removing a sample. If the chamber remains uncapped, temperature instability will occur, which could affect analysis results.

Greasing Chamber O-Ring

Use the greasing disk included in the accessory kit to grease the O-ring. Wear rubber gloves to prevent contaminating the grease with oil from your fingertips. Either too much or too little grease can cause problems. Too much grease may alter cell volume and too little grease may result in an imperfect seal and result in leaks.

1. Remove the chamber cap.
2. Using a fingertip, apply a thin layer of Dow Corning high vacuum grease to the greasing disk.
3. Insert the greasing disk into the chamber cap and turn slightly to apply a very light coating of grease to the O-ring.

Replacing the Chamber Cap O-Ring (PN: 004/25577/00)

Fine fibers and particles between the O-ring and its sealing surface can cause leaks, as can scratches or cuts in the O-ring or in the metal surfaces. To replace the O-ring:

1. Pry the O-ring from its groove by pushing a sharp tool into the ring. Be very careful not to scratch the metal surface.
2. Clean the groove in the chamber cap using a small brush or lint-free cloth and isopropyl alcohol.
3. Allow the chamber cap to dry.
4. Grease the O-ring with high vacuum grease.
5. Replace the O-ring.

Daily Maintenance

1. Keep the cell chamber and cap clean at all times. Particles on the cap seating surface, in the sample cup, under the sample, or clinging to the sample chamber wall may cause inaccurate results.

2. Inspect the cell and cap before each use, and use a lint-free cloth to remove any dust or particles.

Troubleshooting

Measurement problems generally manifest as either slow drift or random fluctuations of reported values.

Thermal Effects

A relatively massive analysis block (sample chamber + expansion chamber) with high thermal conductivity is one of the patented features of the gas pycnometer. Thermal equality between the sample, gas, and analysis block is achieved quickly with an extremely small change in system temperature. This is an important feature for stability because a gas pycnometer must sense pressures and must rely on constant and equal temperature.

A common example of thermal disruption is encountered when a hot sample is placed directly from an oven into the sample chamber. If the sample is not in thermal equilibrium with the sample chamber, then the output of the instrument will drift due to the expansion and associated pressure change of the gas.

Sample Outgassing during Analysis and the Effects of Vapor Pressure

A sample that contains volatile components outgases in the sample chamber of a gas pycnometer. The result is a varying pressure measurement reported as a varying volume or density over several sequential analyses.

It is often assumed that a volatile component such as water vapor contributes a significant volume and that its vapor pressure is insignificant. In fact, the opposite is true. The volume contributed by the presence of water is usually insignificant compared to sample volume but its vapor pressure is significant. The actual effect for a less-than-dry sample is increasing indicated sample volume and decreasing density as water is driven off.

As a general rule, liquids with vapor pressure equal to or less than that of water at room temperature can be measured with a loss of precision proportional to the excess pressure contributed by the liquid. The instrument specifications apply only to samples exhibiting essentially zero vapor pressure.

Leaks

If debris collects on the sealing surface of the cell lid, a leak is likely to ensue. Examine the lid each time it is installed. If the lid surface appears dirty, wipe it clean with a clean dry cloth.

Permeable Samples

Permeability effects are most commonly encountered with samples containing closed cells. Similar effects may be observed when using helium to analyze organic samples or some microporous carbons. Helium can permeate thin walls and even enter into the structure of some materials. The recourse may be to use another gas, perhaps nitrogen; however, this is not recommended for most IODP samples.

Improper Sample Quantity

The most common mistake is to use too little sample material. The sample and expansion chamber volumes are sized such that the maximum accuracy is achieved when the chamber volume is nearly filled with a sample.

If only a limited amount of sample is available, it is better to include a calibrating sphere with the sample and then manually subtract the sphere volume.

Other Common Issues

What Happened	Why	What to Do
Specified pressure not maintained	Chamber cap not closed	Turn cap clockwise to close
	Chamber cap contaminated with dust or debris	Clean chamber cap and rim of chamber cell with lint-free tissue
	O-ring not properly greased	Lightly grease O-ring (see Greasing Chamber O-Ring)
	O-ring not properly seated	Check O-ring for scratches or cuts
	O-ring is scratched or cut	Replace O-ring (see Replacing the Chamber Cap O-Ring (PN: 004/25577/00))
	Gas leaks in cell chambers	Check for leaks (see Leaks)
	Helium tank empty	Check tank for minimum 200 psig
	Cylinder valve closed	Check helium cylinder valve
	Pressure set incorrectly	Set pressure to 10–21 psig
	Zero offset of pressure transducer set too low	Open chamber cap to check; run zero offset if needed
He drained from tank	Leak in gas line connection	–Pressurize the system then open cylinder valve; if pressure gauge needle jumps, a leak may be indicated –Check all connections for leaks.

	Pycnometer valves open or chamber cap left off	Close valves and attach a new tank of helium
Will not equilibrate	Pycnometer valves open or chamber cap left off	Close valves and attach a new tank of helium
	Sample outgassing	Re-dry sample
	Pycnometer is leaking	Check O-ring and regrease or replace
	Valve seat leaking	–With cap on, open all valves –With He flowing, actuate expansion valve repeatedly for ~1 min to remove lint –Repeat process for vent valve with inlet and expansion valves open

Health, Safety, and Environment

Health

None of the materials used in the MAD process represent a significant risk to health.

Safety

Pressurized Helium Gas

Helium is a colorless, odorless, tasteless, non-toxic, inert monatomic chemical element that exists only as a gas except in extreme conditions. Neutral helium at standard conditions is non-toxic, plays no biological role and is found in trace amounts in human blood.

Pressurized cylinders represent a physical hazard and should be properly restrained at all times. When being transported, a cylinder must have its cap screwed on tightly.

The delivery pressure for the pycnometer is fairly low and does not present a significant hazard.

Electrical Hazard

The balances, oven, and pycnometer are all electrical devices and care should be taken not to spill liquids on or near them. No high-voltage components are accessible by the general user.

Hot Oven and Samples

The Thermo oven is set to 105°C and can burn the user if care is not taken. Likewise, the samples are very hot when first removed from the oven. Use proper protective equipment (insulated mittens).

Glass Containers

The Wheaton vials are borosilicate glass and somewhat resistant to breakage. If a vial breaks, the broken glass should be gathered up immediately and placed in a sharps container. Please see a technician or the ALO if this occurs for proper disposal instructions. Do NOT place sharp trash in the regular trash bins!

Pollution Prevention

The MAD method does not generate any hazardous waste.
Remaining samples are used for further analyses after oven-drying and volume determination.

Technical Information

Hardware Settings

Pressure transducer

- Output: 0–5 V DC
- Range: 0–25 psig
- Orifice: 0.04 in.
- Operational coil voltage: 11–13 V DC pulsed 30 ms ± 2 ms
- Service pressure: 30 psi differential bidirectional
- Valve type: latching solenoid

Solenoids

- Solenoid, HHE (each cell requires 1): Micromeritics PN: 133/60804/00
- Solenoid, IHE (each cell requires 2): Micromeritics PN: 133/60804/00
- Pin 1: brown: closed
- Pin 2: black: common
- Pin 3: orange: open
- Pin 4: key: key
- Pins 5 & 6: not used

Consumables

Frit: 0.5 in. diameter ´ 1/16 in. thick, 20 μm, 316 SS: Micromeritics PN: 004/27042/00

Greasing tools

- Cap O-ring greasing tool: Micromeritics PN: 133/25825/00
- High vacuum grease: Micromeritics PN: 008/16045/00

O-rings

- 015 70DURO BUNA-N: Micromeritics PN: 004/25076/00
- 014 70DURO BUNA-N: MIcromeritics PN: 003/25469/00
- 019 70DURO BUNA N: Micromeritics PN: 003/25575/00
- 033 70DURO BUNA-N: Micromeritics PN: 003/25577/00

Pressure transducer: Setra Systems (M/N 205-2): Micromeritics PN: 003/55609/01

Solenoids

- Solenoid, HHE (each cell requires 1): Micromeritics PN: 133/60804/00
- Solenoid, IHE (each cell requires 2): Micromeritics PN: 133/60804/00

Contact Information

Calibration Spheres

Precision Ball and Gage Co.

1417 Wimbledon Place

Springfield, OR, 97477

541.688.7268

541.689.6613 (fax)

Pycnometers

Micromeritics Instrument Corporation
One Micromeritics Drive
Norcross, GA, 30093-1877
770.662.3633
Micromeritics.com

Technical Documents

See Physical Properties Laboratory Notebook for more documents. Physical Properties Laboratory Notebook

- Thermo Scientific HERATHERM oven
- Micromeretics AccuPyc 1330TC helium pycnometer cells
- Mettler-Toledo XS-204 analytical balances
- Pycnometer Electronics Interface
- Setra pressure transducers (for pycnometer)
- National Instruments SCC-68 I/O connector block user guide
- National Instruments NI-622x specifications sheet
- Pycnometer hardware installation guide

LabVIEW structure

Software directory structure for LabVIEW

MAD Computations

MAD computations are performed when triggered by the user as described above in "MAD Calculations." Each time computation is triggered, the program will recalculate the results and update the database. (This may be necessary, for example, if the wet and dry mass measurements were switched.) The computations are performed for each completed submethod using the following formulas.

Formula or Condition	Action	LIMS Analysis	Formula with LIMS Components
Constants Used in Computations			
	get	MAD	density_water = 1 (g/cm3)
	get	MAD	density_porewater = 1.024 (g/cm3)
	get	MAD	density_salt = 2.22 (g/cm3)
	get	MAD	salinity = 35
	get	MAD	mass_ratio = 0.965
	get	MAD	volume_ratio = 0.988
Get Container Information: Submethods A, B, C, D			
	get	CONTAINER	container_number
	get	CONTAINER	material_type
	get	CONTAINER	mass
	get	CONTAINER	density
	get	CONTAINER	volume
Geometric Volume: Submethods A, D			
	select	CALIPER	container_number
	set	CALIPER	geometry = [cylinder; rectangular prism]
	measure	CALIPER	length
	measure	CALIPER	width
	measure	CALIPER	height
	measure	CALIPER	diameter
geometry = "rectangular prism"	calculate	CALIPER	volume = length * width * height
geometry = "cylinder"	calculate	CALIPER	volume = [diameter/2]^2 * pi * dimension_c_height
Wet Mass: Submethods A, B, C			
	select	MAD-MASS	container_number
	measure	MAD-MASS	mass_wet_container
	calculate	MAD-MASS	mass_wet = mass_wet_container – mass{CONTAINER}
Dry Mass: Submethods A, B, C, D			
	select	MAD-MASS	container_number
	measure	MAD-MASS	mass_dry_container
	calculate	MAD-MASS	mass_dry = mass_dry_container – mass{CONTAINER}

Formula or Condition	Action	LIMS Analysis	Formula with LIMS Components
Pycnometry Wet Volume: Submethod B			
	select	PYC	container_number
	select	PYC	cell_number
	measure	PYC	volume_wet_container

	calculate	PYC	volume_wet = volume_wet_container – volume{CONTAINER}
	count	PYC	number_measurements
	calculate	PYC	pyc_stdev
	measure	PYC	temperature
	enter	PYC	comment
Pycnometry Dry Volume: Submethods C and D			
	select	PYC	container_number
	select	PYC	cell_number
	measure	PYC	volume_dry_container
	calculate	PYC	volume_dry = volume_dry_container – volume{CONTAINER}
	count	PYC	number_measurements
	calculate	PYC	pyc_stdev
	measure	PYC	temperature
	enter	PYC	comment
Intermediary Computations: Submethod A			
	set	MAD	method = "A"
	get	MAD	volume_wet = volume{CALIPER}
	get	MAD	mass_wet = mass_wet{MAD_MASS}
	get	MAD	mass_dry = mass_dry{MAD_MASS}
Mpw = (Mt – Md)/rm	calculate	MAD	mass_porewater = [mass_wet – mass_dry]/mass_ratio
Vpw = Mpw/Dpw	calculate	MAD	volume_porewater = mass_porewater/density_porewater
Ms = Mt – Mpw	calculate	MAD	mass_solids = mass_wet – mass_porewater
Vs = Vt – Vpw	calculate	MAD	volume_solids = volume_wet – volume_porewater
Intermediary Computations: Submethod B			
	set	MAD	method = "B"
	get	MAD	mass_wet = mass_wet{MAD_MASS}
	get	MAD	mass_dry = mass_dry{MAD_MASS}
	get	MAD	volume_wet = volume_wet{PYC}
Mpw = (Mt – Md)/rm	calculate	MAD	mass_porewater = [mass_wet – mass_dry]/mass_ratio
Vpw = Mpw/Dpw	calculate	MAD	volume_porewater = mass_porewater/density_porewater
Ms = Mt – Mpw	calculate	MAD	mass_solids = mass_wet – mass_porewater
Vs = Vt – Vpw	calculate	MAD	volume_solids = volume_wet – volume_porewater

Formula or Condition	Action	LIMS Analysis	Formula with LIMS Components
Intermediary Computations: Submethod C			
	set	MAD	method = "C"
	get	MAD	mass_wet = mass_wet{MAD_MASS}
	get	MAD	mass_dry = mass_dry{MAD_MASS}
	get	MAD	volume_dry = volume_dry{PYC}
Mpw = (Mt – Md)/rm	calculate	MAD	mass_porewater = [mass_wet – mass_dry]/mass_ratio
Vpw = Mpw/Dpw	calculate	MAD	volume_porewater = mass_porewater/density_porewater
Ms = Mt – Mpw	calculate	MAD	mass_solids = mass_wet – mass_porewater
Msalt = Mpw – (Mt – Md)	calculate	MAD	mass_salt = mass_porewater – [mass_wet – mass_dry]
Vsalt = Msalt/Dsalt	calculate	MAD	volume_salt = mass_salt/density_salt
Vt = Vd – Vsalt + Vpw	calculate	MAD	volume_wet = volume_dry – volume_salt + volume_porewater
Vs = Vt – Vpw	calculate	MAD	volume_solids = volume_wet – volume_porewater

Intermediary Computations: Sub-Method D				
	set	MAD	method = "D"	
	get	MAD	volume_wet = volume{CALIPER}	
	get	MAD	mass_dry = mass_dry{MAD_MASS}	
	get	MAD	volume_dry = volume_dry{PYC}	
Mt = Md + Mw = Md + (Vt – Vd) * 1	calculate	MAD	mass_wet = mass_dry + [volume_wet – volume_dry] * density_water	
Vpw = Vw/r = (Vt – Vd)/rv	calculate	MAD	volume_porewater = [volume_wet – volume_dry]/volume_ratio	
Mpw = Vpw * Dpw	calculate	MAD	mass_porewater = [mass_wet – mass_dry]/[1 – salinity/1000]	
Ms = Mt(calc) – Mpw	calculate	MAD	mass_solids = mass_wet – mass_porewater	
Vs = Vt – Vpw	calculate	MAD	volume_solids = volume_wet – volume_porewater	
Final Computations: Submethods A, B, C, D				
WW = Mpw/Mt	calculate	MAD	moisture_rel_dry = mass_porewater/mass_wet	
WD = Mpw/Ms	calculate	MAD	moisture_rel_wet = mass_porewater/mass_solids	
BD = Mt/Vt	calculate	MAD	density_bulk = mass_wet/volume_wet	
DD = Ms/Vt	calculate	MAD	density_dry = mass_solids/volume_wet	
GD = Ms/Vs	calculate	MAD	density_grain = mass_solids/volume_solids	
PO = Vpw/Vt	calculate	MAD	porosity = volume_porewater/volume_wet	
VR = Vpw/Vs	calculate	MAD	void_ratio = volume_porewater/volume_solids	

LIMS Components and Definitions for MAD Analysis

CONTAINER Analysis: Sample Container

Analysis component	Definition	Unit	Explanation
container_number	Container number	—	Sample number for a specific container recorded in LIMS. The container number is supplied in the SAMPLE.NAME field. Sample number guarantees uniqueness. Written labels are reusable.
material_type	Container material		Material the container is made of.
mass	Container mass	g	Mass of container.
density	Container density	g /cm ^3	Density of container material used to compute the volume of container material.
volume	Container material volume	cm ^3	Volume of container material computed from its mass and density; NOT the volume the container contains.

MAD_MASS Analysis: Mass Determination for MAD Analysis

Analysis component	Definition	Unit	Explanation
mass_wet	Mass of wet sample	g	Mass of the bulk sample, including the mass of the porewater, solid material, and dissolved salt.
mass_wet_container	Mass of wet sample + container	g	Mass of the bulk sample, including the mass of the porewater, solid material, dissolved salt, and container. If one-way containers are used, this does not include the mass of the lid of the container.
mass_dry	Mass of dry sample	g	Mass of the dried sample, including mass of evaporated salt.
mass_dry_container	Mass of dry sample + container	g	Mass of the dried sample, including evaporated salt plus the mass of the container. If one-way containers are used, this does not include the mass of the lid of the container.
container_number	Container number	—	Sample number for a specific container recorded in LIMS. The container number is supplied in the SAMPLE.NAME field. Sample number guarantees uniqueness. Written labels are reusable.

CALIPER Analysis: Caliper Measurements for MAD Analysis

Analysis component	Definition	Unit	Explanation
volume	Sample volume	cm^3	Sample volume calculated from length, width, and height or height and diameter, depending on geometry.
geometry	Sample geometry	—	Geometry used to calculate volume: rectangular prism or cylinder.
length	Sample length	cm	Length (x) of a rectangular prism sample; measure orthogonally to the other dimensions.
width	Sample width	cm	Width (y) of a rectangular prism sample; measure orthogonally to the other dimensions.
height	Sample height	cm	Height (z) of a rectangular prism or cylindrical sample.
diameter	Sample diameter	cm	Diameter of a cylindrical sample.
container_number	Container number	—	Number inscribed on the container and logged into the LIMS.
comment	Comment	—	Comment about caliper measurement.

PYC Analysis: Pycnometry for MAD Analysis

Analysis component	Definition	Unit	Explanation
volume_wet	Volume of wet sample	cm^3	Volume of wet sample measured in pycnometer. NOTE: Wet volume measurement by pycnometer is not accurate and is not recommended.
volume_wet_container	Volume of wet sample with container	cm^3	Volume of wet sample and the container measured in pycnometer. NOTE: Wet volume measurement by pycnometer is not accurate and is not recommended.
volume_dry	Volume of dry sample	cm^3	Volume of dry sample including evaporated salt.
volume_dry_container	Volume of dry sample with container	cm^3	Volume of dry sample including evaporated salt and volume of container.
pyc_stdev	Standard deviation	cm^3	Standard deviation calculated from N measurement cycles in the pycnometer.
number_measurements	Number of measurements	—	Number of successive measurements used in determining the result value.
container_number	Container number	—	Sample number for a specific container recorded in LIMS. The container number is supplied in the SAMPLE.NAME field. Sample number guarantees uniqueness. Written labels are reusable.
volume_container	Volume of container material	cm^3	Volume of container material calculated from premeasured mass and material density of the container.
cell_number	Cell number	—	Number of the cell in the pycnometer used for this measurement.
temperature	Cell temperature	°C	Temperature of the pycnometer cell.
comment	Comment	—	Comment on pycnometer run.

PYC_QAQC Analysis: Pycnometry Calibration

Analysis component	Definition	Unit	Explanation
volume_cell_standard	Volume of cell with standard	cm^3	Volume of the cell as computed with the standard.
volume_cell_exp	Volume of expansion cell	cm^3	Expected volume of the cell (calibration).
cell_number	Cell number	—	Number of the cell in the pycnometer used for this measurement.
number_measurements	Number of measurements	—	Number of successive measurements used in determining the result value.
volume	Measured volume of standard	cm^3	Volume of standard used for calibration or calibration verification.
container_number	Container number	—	Serial number for a specific container recorded in LIMS. The container number is supplied in the SAMPLE.NAME field.
measurement_type	Calibration (verification)	—	Purpose of the measurement: calibration or calibration verification.
temperature	Cell temperature	°C	Temperature of the pycnometer cell.

comment	Comment	—	Comment on pycnometer run.

MAD Analysis: Computed Results for MAD Analysis

Analysis component	Definition	Unit	Explanation
moisture_rel_wet	Moisture content (wet)	% mass	Moisture content of sample in percentage mass of water/mass of bulk sample (includes moisture and dissolved salt).
moisture_rel_dry	Moisture content (dry)	% mass	Moisture content of sample in percentage mass of water/mass of dry sample (without evaporated salt).
density_bulk	Bulk density (MAD)	g /cm^3	Grams of mass/mL of volume of bulk sample.
density_dry	Dry density (MAD)	g /cm^3	Grams of mass of solids (including evaporated salt)/mL bulk sample.
density_grain	Grain density (MAD)	g /cm^3	Grams of mass of solids (without mass of evaporated salt)/mL solids (without volume of evaporated salt).
porosity	Porosity (MAD)	%vol	Volume of porewater/volume of solids (without volume of evaporated salt).
void_ratio	Void ratio (MAD)	—	Volume of porewater/volume of solids (without volume of evaporated salt).
method	MAD submethod	—	Submethod used to calculate MAD results: A, B, C, D.
mass_wet	Wet mass	g	Measured or calculated mass of the bulk sample (including mass of porewater, material, and dissolved salt).
mass_dry	Dry mass	g	Measured or calculated mass of the dried sample (including mass of evaporated salt).
volume_wet	Wet volume	cm^3	Measured or calculated volume of the bulk sample (including volume of porewater, material, and dissolved salt).
volume_dry	Dry volume	cm^3	Measured or calculated volume of dried sample (including evaporated salt).
mass_porewater	Mass of porewater	g	Mass of the porewater in the sample.
volume_porewater	Volume of porewater	mL	Volume of porewater in the bulk (raw) sample.
mass_salt	Mass of salt	g	Mass of evaporated salt in the sample.
volume_salt	Volume of salt	cm^3	Volume of dissolved salt in the bulk sample.
mass_solids	Mass of solids	g	Mass of the solid material in the sample (without mass of evaporated salt).
volume_solids	Volume of solids	cm^3	Volume of solid material in the sample (without volume of dissolved salt).

Credits

The wiki version of this document is based on the Exp. 362T document and was last edited on 6/27/2019 Exp 383. The 362T version is archived, below. Additional information from an older "Advanced User Guide" has been combined with this user guide by Susan Boehm on 383.

Archived Versions

- MAD_UG_v.362T.pdf: V362T D. Houpt, rev. M. Hastedt (1 March 2009)
- MAD Advanced User Guide 2014