Moisture and Density (Method C): Quick Start Guide

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Introduction

Core specimens for moisture and density (MAD) analysis are extruded from a section half for mass and volume. The MAD properties of interest include water content, bulk density, dry density, porosity, and void ratio, which are calculated based on the measured values of wet mass, dry mass, wet volume, and/or dry volume. MAD Method C is most often applied to fine-grained, saturated sediments or fine-grained igneous material, and wet mass and dry mass are measured by balance, and dry volume is measured by pyncnometer.

Method C QuickStart Procedure

1.	Collect sample.
2.	Weigh wet sample + container.
3.	Dry sample + container.
4.	Weigh dry sample + container.
5.	Measure volume of dry sample + container by pycnometer.

Apparatus, Reagents, & Materials

–Dual balance system

–Hexapycnometer system

–Helium gas

–Controlling software (see the MADMax Quick Start Guide)

–Water circulating bath

–Sample drying equipment (oven)

–Sampling tools (syringes/plugs to extract soft-sediment of volume of 10 cm^3 from the section halves)

–Sampling containers (Wheaton glass vials (type 800): volume = ~8 cm^3; density = 2.48 g/cm^3)

Sample Preparation and Analysis

Samples are collected directly from the working section halves in the Core Laboratory. The typical weight of sample to be used in MAD analysis (~10 mL with density of >~1.6 g/mL) is a minimum of ~16 g.

The definition of a routine MAD sampling plan or selection of samples based on visual observations or core logging data should take the following into consideration:

Consider other analyses that should/could be made on the same material (e.g., magnetic properties or *P*-wave velocity). Subsamples for carbonate content or XRD analyses should be taken from or adjacent to the MAD sample.

If core recovery is high (≥3000 m), MAD sampling generally consists of 1 specimen (volume ~12 mL) per section.

Sampling

1. After a core section has been split, flag the location where the MAD sample is to be taken in the working half of the core and enter it into the log sheet.

   Note: Confirm that no critical sediment or rock interval will be destroyed or depleted.

2. Insert a syringe or minipiston corer into the soft sediment sample and extract a cylindrical sample.
3. Place the sample in a glass container identified with a unique container number; clean the lip of the container if necessary. Place a lid on the container to prevent (retard) evaporation of pore fluid.
4. Place a styrofoam plug into the hole where the sample was taken.
5. Log the collected sample into LIMS and assign the MADC test, which contains MAD_MASS and PYC analyses.
6. Once a batch of 3–7 samples is ready, transfer the samples to the Petrophysics Lab for analysis.

Mass Determination

For additional information on using the MADMax Software Interface, see the *MADMax Quick Start Guide*.

1. On the *MADMax User Interface*, click the dropdown arrow in **Currently Viewing Results For** field and choose the method for determining MAD values (for this QSG, choose Method C).
2. Measuring **Mass Wet:** in the tabular data sheet find the sample to be analyzed and double click the **Mass Wet** cell for the particular sample.
3. Ensure the sample being measured and the container number are correct on the screen. Further down there is the prompt for the number of mass measurements to be averaged (generally 300; if sea state increases increase number of measurements for better accuracy).
4. Click **Measure**. Expand the measurement window to the desired size.
5. If this is the first measurement using the balances (or if no measurements have been taken within the last hour) the balances will need to be Tared.

   Remove any objects from the balances and click on the **Tare** button. After taring the two balances, measure a "Known Weight" against a "Known Weight" (e.g., 20 g) on both balances to cross-check precision and accuracy of the balance systems.
6. Enter the **Reference Mass** (known mass of the reference weight). Use a weight similar to the sample + container (20–30 g). Place the reference mass on the '*Known*' balance and the sample on the '*Unknown*' balance, and click **Weigh**.
7. Once the measurement has been completed, click **Accept** to populate the **Mass Wet** field.
8. After **Mass Wet** has been measured, place sample in oven to dry for 24 hr (see Sample Drying, below).
9. After drying the sample, measure the **Mass Dry**. Double click the **Mass Dry** column in the correct sample row. Verify the sample ID and container number, then click **Measure**. From here *repeat* steps 5–7 listed above. Make sure to write the data also into a corresponding log sheet (same with data for the drying and pycnometer working steps).

Sample Drying

Important: Double check the proper operation of the oven and its power plug. Sometimes the power plug can unlock itself and cause an error due to power failure! Of course, an oven also always presents a fire hazard.

1. After wet mass analysis, place the container on a sample tray and place in the drying oven at 105°C.
2. Leave the sample in the oven for 24 hr.
3. Remove the sample tray from the oven and place it in the desiccator for at least 1 hr to return the sample to room temperature with minimum uptake of moisture.
4. Remove sample from the desiccator.
5. Determine dry mass on the sample (see Mass Determination).

Measuring Volume

1. Double-click on the desired sample cell under **Volume Dry**. Check to ensure Sample ID and Container number are correct.
2. Place the sample in the selected pycnometer cell and tighten the lid. Enter the number of measurement cycles (generally 3 measurements). Once all parameters are set as desired and the sample has been entered into the chamber, click **Measure.**
3. The **Volume Dry** measurement interface will appear; to begin measurement click **Start**. This initiates the pycnometer analysis; the process automatically runs through the number of cycles selected in Step 2.
4. Once the measurement is complete the **Calculated Volume** interface will appear. If this volume is acceptable click **Accept;** otherwise the sample can be reanalyzed by choosing **Rerun**.
5. Note the **Volume Dry** field is now populated with the newly calculated volume.
6. Under the **Methods Completed** column, double-click the cell for the particular sample run to bring up a prompt for *MAD Calc.* Ensure that container number and sample ID are correct and click **Run MAD Calc**. This calculates the remaining data for the sample being measured (i.e., Mass Pore water, Mass Salt, Mass Solids, Volume Pore water, etc.).

Calibration/Verification

The balance and pycnometer systems must be calibrated and verified. Calibration verification is run on a routine basis to ensure that the original instrument calibration is still valid. If calibration verification shows a problem in the system, the instrument(s) should be recalibrated and any samples measured on that instrument since the last acceptable calibration verification reanalyzed.

Pycnometer Calibration

The pycnometer cells need to be calibrated by a staff member or a trained visiting scientist when one of the following occurs:

–A cell insert is removed, added, or exchanged

–Helium inlet pressure is changed

–Water bath temperature is changed

–A cell is reinstalled after troubleshooting, repair, or replacement

–A certain time period has elapsed without calibration (current recommendation = 1 day)

–A calibration verification measurement exceeds control limits

Each pycnometer cell is calibrated individually. The calibration action first analyzes the cell empty, and the second action analyzes the cell containing the standard sphere(s) in the sample chamber. The software prompts when it is time to place the standard sphere inside the sample chamber.

1. Ensure cell to be calibrated is equipped with the desired insert and otherwise empty.
2. Ensure the cell lid is properly sealed.
3. Click the Calibrate Pycnometer button at the top of the MADMax user interface.
4. Follow the instructions in MADMax to complete calibration of each cell.
5. Log calibration data in the Excel sheet that is either on the desktop or the server. It is imperative to log this data and keep track of the calibration to ensure high-quality measurements throughout the expedition.

Calibration Verification

To perform a calibration check, run an analysis with a calibration sphere as a sample. The deviation of the results from 5 cyclic measurements should be within ±1% of the known value. If the result is outside the 1% control limit, recalibration of that cell is necessary.