

# 2. MOISTURE AND DENSITY (BY MASS AND VOLUME)

## 2.1. Principles

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### PHYSICAL BACKGROUND

Moisture content and mineral density are basic sediment and rock properties that are determined most accurately through mass and volume determinations. Core specimens of approximately 8 cm<sup>3</sup> are extruded from the working-half core for this purpose. Moisture content is determined by measuring the specimen's mass before and after removal of interstitial pore fluid through drying. The drying method is the most critical part of the entire procedure. At present, a convection oven is used for this purpose for 24 hours at temperatures varying from 100° to 110°C. This method is suspected to remove a substantial portion of the interlayer (hydrated) water from clays such as smectite in addition to interstitial water, which may result in porosity errors of up to 20%. Alternative methods such as microwave or freeze-drying have other potential problems and have not replaced the convection oven.

Moisture content, porosity, and void ratio are defined by the mass or volume of extracted water (assumed to be interstitial pore fluid), corrected for the mass and volume of salt evaporated during the drying process (see also ASTM, 1990). The mass and volume of the evaporated pore-water salts are calculated for a standard seawater salinity (35), seawater density at laboratory conditions (1.024 g/cm<sup>3</sup>), and an average seawater salt density (2.20 g/cm<sup>3</sup>). Any gases that may be present are allowed to escape during core retrieval, core splitting, and specimen extrusion.

The volume of a specimen can be measured in three ways:

- method A: wet-bulk volume measured with special volume sampler,
- method B: wet-bulk volume measured by gas pycnometry, and
- method C: dry volume measured by gas pycnometry

Method A is the least standardized method. The device to be used depends on user preference and can be a simple steel ring ("fixed volume," available on the ship) or some sort of syringe (volume is measured after the sample has been taken). The advantage of method A, according to some users, is that a larger number of specimens can be measured than with gas pycnometry in a given time. The disadvantages are (1) the method works only in soft, non-sticky sediment (mainly homogenous carbonate oozes to a depth of about 200 mbsf), (2) the volume measured includes potential cracks or other spaces filled with air, (3) there is no precision estimate for this method, and (4) there is no standard for this method.

This method should therefore be used only if there is ample justification, and measurements must be “calibrated” with an appropriate number of pycnometry results.

Methods B and C use the same gas pycnometer. The measurement principle of this device is briefly described in the following. Gas pycnometry works with pressure ratios of an ideal gas (helium), which are sensitive to contamination with partial pressures of other fluids. The material to be measured should therefore be dry. The ODP database contains thousands of examples from specimens measured with both method B and method C. A systematic error is clearly discernible in comparing calculated results, with bulk densities 1%–5% too high and grain densities about 5%–10% too high for method B. It is therefore recommended that only method C be used.

The following relationships can be computed from two mass measurements and one or two volume measurements. First, if methods B or C are used, the beaker mass and volume, which are determined periodically and stored in the program’s lookup table, are subtracted from the measured total mass and volume measurements. If method A is used, only the beaker mass is subtracted (the user must specify the use of method A in the program). This results in the following directly measured values:

- $M_b$ : bulk mass,
- $M_d$ : dry mass (mass of solids,  $M_s$ , plus mass of evaporated salt),
- $V_{b(A \text{ or } B)}$ : bulk volume, method A or method B, and
- $V_{d(C)}$ : dry volume = volume of solids,  $V_{s(C)}$ , plus volume of evaporated salt,  $V_{salt}$ .

Variations in pore-water salinity,  $s$  ( $s = S/1000$ ), and density,  $\rho_{pw}$ , that typically occur in marine sediments do not affect the calculations significantly, and standard seawater values at laboratory conditions are used:

$$s = 0.035 \quad (1)$$

$$\rho_{pw} = 1.024. \quad (2)$$

Pore-water mass,  $M_{pw}$ , mass of solids,  $M_s$ , and pore-water volume,  $V_{pw}$ , can then be calculated:

$$M_{pw} = (M_b - M_d) / (1 - s) \quad (3)$$

$$M_s = M_b - M_{pw} = (M_d - s M_b) / (1 - s) \quad (4)$$

$$V_{pw} = M_{pw} / \rho_{pw} = (M_b - M_d) / [(1 - s) \rho_{pw}]. \quad (5)$$

Additional parameters required are the mass and volume of salt ( $M_{salt}$  and  $V_{salt}$ , respectively) to account for the phase change of pore-water salt during drying. It should be kept in mind that for practical purposes the mass of salt is the same in solution or as precipitate, whereas the volume of the salt in solution is negligible.

$$M_{salt} = M_{pw} - (M_b - M_d) = (M_b - M_d) s / (1 - s) \quad (6)$$

$$V_{salt} = M_{salt} / \rho_{salt} = [(M_b - M_d) s / (1 - s)] / \rho_{salt}, \quad (7)$$

where the salt density value  $\rho_{salt} = 2.20 \text{ g/cm}^3$  is a value calculated for an average composition of seawater salt (Lyman and Fleming, 1940; Weast et al., 1985).

Moisture content is the pore water mass expressed either as percentage of wet bulk mass or as percentage of the mass of salt-corrected solids:

$$W_b = M_{pw} / M_b = (M_b - M_d) / M_b (1 - s) \quad (8)$$

$$W_s = M_{pw} / M_s = (M_b - M_d) / (M_d - s M_b). \quad (9)$$

Calculation of the bulk volume for method C and volume of solids depend on the volume measurement method used:

$$V_{s(A \text{ or } B)} = V_{b(A \text{ or } B)} - V_{pw} \quad (10)$$

$$V_{s(C)} = V_{d(C)} - V_{salt} \quad (11)$$

$$V_{b(C)} = V_{s(C)} + V_{pw}. \quad (12)$$

Bulk density,  $\rho_b$ , density of solids or grain density,  $\rho_s$ , dry density,  $\rho_d$ , porosity,  $P$ , and void ratio,  $e$ , are then calculated accordingly for each method:

$$\rho_{b(A,B,C)} = M_b / V_{b(A,B,C)} \quad (13)$$

$$\rho_{s(A,B,C)} = M_s / V_{s(A,B,C)} \quad (14)$$

$$\rho_{d(A,B,C)} = M_s / V_{b(A,B,C)} \quad (15)$$

$$P_{(A,B,C)} = V_{pw} / V_{b(A,B,C)} \quad (16)$$

$$e_{(A,B,C)} = V_{pw} / V_{s(A,B,C)}. \quad (17)$$

## ENVIRONMENTAL EFFECTS

### *Core Expansion*

Cores, particularly sediment cores from a few hundred meters below the seafloor, expand upon recovery for a number of reasons, which include

- elastic recovery,
- gas expansion, and
- mechanical stretching.

Expansions of solids can be neglected. Pore water expands by about 4% for every 1000 bar (100 MPa) pressure release. This is what the pore water of a seafloor sample from about 10,000-m water depth would experience, or in ocean drilling terms, what a sample buried by about 2000 m of water and about 3000 m of sediment would experience. For the bulk of ODP cores, pore-water expansion is in the order of 1% and therefore negligible compared with the analytical error.

Free gas expands by orders of magnitude, according to the simple relationship  $P_1V_1 = P_2V_2$ . A few percent of free gas in the sediment can produce an explosive sediment-gas mixture that has torn apart plastic core liners on the ship on several occasions. Most gas escapes before the cores are analyzed and can produce microfractures, which appear as porosity with methods based on core unit-volume measurements, such as the gamma-ray attenuation bulk density method.

Mechanical stretching may also cause microfracturing. The MAD method measures the mass and volume of the solid and liquid phases only and is therefore not affected by this type of artificial porosity. The original contribution of the gas to in situ porosity cannot be measured with our routine core analysis program.

### *Composition of Seawater*

Different water masses of the world oceans have different chemical compositions and physical properties. For the purpose of correcting oven-dried sediment specimens for the evaporated salt from the pore water, the standard compositions

after Lyman and Fleming (1940) and salt densities after Weast et al. (1985) are used (Table 2—1).

Table 2—1 Composition of sea water.

Salt	Mass fraction <sup>a</sup> ( $\times 10^3$ )	Density <sup>b</sup> ( $\text{g}/\text{cm}^{-3}$ )
NaCl	23.476	2.165
MgCl <sub>2</sub>	4.981	2.316-2.33
Na <sub>2</sub> SO <sub>4</sub>	3.917	1.46 (monocl.) 2.68 (orthorh.)
CaCl <sub>2</sub>	1.102	2.15
KCl	0.664	1.984
NaHCO <sub>3</sub>	0.192	2.159
KBr	0.096	2.75
H <sub>3</sub> BO <sub>3</sub>	0.026	
SrCl <sub>2</sub>	0.024	2.671 (leaf)
SrCl <sub>2</sub> ·2H <sub>2</sub> O		3.052 (cub.)
NaF	0.003	2.08
<b>Total</b>	34.481	
	<b>Weighted average</b>	2.10-2.24

<sup>a</sup>Lyman and Fleming (1940).

<sup>b</sup>Weast et al. (1985).

Given the uncertainty in regard to the crystalline structure of some evaporated components, the average density of the standard seawater salt is between 2.10 and 2.24  $\text{g}/\text{cm}^3$ . A value of 2.20  $\text{g}/\text{cm}^3$  is used routinely for the MAD calculations.

### Density of Pore water

Density of pore water is a function of temperature ( $T$ ), salinity ( $S$ ), and pressure ( $P$ ). Equations of state for seawater (Millero et al., 1980; Millero and Poisson, 1981) can be used to illustrate the variability of pore-water density as a function of these three parameters (Figure on page 5).

Typical salinity values for pore waters are 30 to 40, although more extreme values exist. At laboratory pressure and temperature, this range of salinity change affects pore-water density change of less than 1%, which is negligible compared with the analytical uncertainty. We therefore use a standard value of 35 for all MAD calculations and leave it up to the user to apply corrections if warranted.

The typical temperature change experienced by nonlithified sediment upon recovery is from about 100°C at depth to few degrees at the seafloor to about 22°C in the laboratory. At standard salinity and laboratory pressure, a 100°C change results in about a 2% change in seawater density. These effect is not figured in our MAD calculations because it is close to the uncertainty.

The effect of pressure change on density is of a similar magnitude. For a high-porosity mud sample (for example, from 100 mbsf) at a water depth of 3000 m, the pressure release is about 320 bar (32 MPa). According to Figure 2—1, if the in situ

temperature is about the same as laboratory temperature and salinity is 35, pore-water density decreases by about 1% upon recovery.

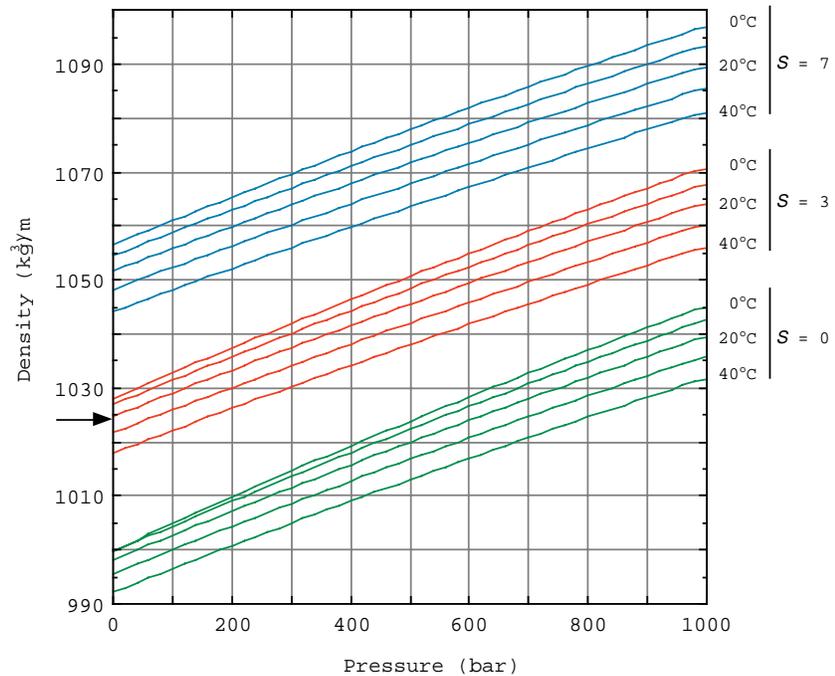


Figure 2—1 Density of seawater as a function of pressure, salinity, and temperature, using equations from Millero and Poisson (1981). The pressure range from 0 to 1000 bar covers most ODP situations. Standard salinity of 35 and two extreme salinities (0 and 70) are plotted as a function of a temperature between 0° to 40°C (experimental temperature range of Poisson and Millero, 1981). The arrow indicates standard laboratory conditions.

## USE OF MAD DATA

MAD data are the only data that provide a direct estimate of porosity and void ratio and the average density of the 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 the high-resolution gamma-ray attenuation bulk density data sampled automatically at much smaller intervals than would be possible for MAD data. If mineral density can be defined with sufficient precision, GRA bulk density can be expressed as porosity.

## 2.2. Moisture and Density System

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### EQUIPMENT

#### *Balance*

Mass is determined with two Scientech 202 electronic balances to compensate for the ship's motion. A set of mass standards ranging from 1 to 20 g is used for calibration and on the reference balance during measurements.

#### *Gas Pycnometer*

The helium displacement pycnometer with five cells (penta-pycnometer), manufactured by Quantachrome Corp., employs Archimedes' principle of fluid displacement to determine the volume of solid objects. The five measurement cells contain custom-fabricated inserts that reduce the chamber to a cylindrical space that holds exactly one 10-mL Pyrex beaker. The measurement chamber must contain as little air space as possible to maximize measurement precision. (The user should also ensure that the Pyrex beakers are filled as completely as possible with core material.)

Each sample cell of volume  $V_C$  has an input valve (from the gas tank) and an output valve (to the pressure transducer). An additional reference cell of volume  $V_A$  is located downvent of the sample cells, with an input valve (which separates  $V_A$  from the pressure transducer) and a vent valve (Figure on page 7). All cell volumes must be calibrated periodically (see calibration section). The following is the operation sequence of the pycnometer during the measurement of a specimen.

The specimen to be measured is placed in a cell of known volume,  $V_C$ . It is pressurized, using helium, to an exactly measured pressure of about 18 psi (~120 kPa). The solenoid valve between sample cell and the reference cell of known volume  $V_A$  is opened and the helium from the pressurized chamber is ported to the reference cell. The subsequent pressure in the system is measured. Using the ideal gas law, the sample volume can be calculated from the pressure ratio. The following is the sequence of operation (Figure 2—2).

1. Gas input valves to all five cells are closed (corresponding light-emitting displays [LEDs] on pycnometer are unlit). The five sample cell output valves, the reference cell input valve, and the vent valve are open, ensuring that all cells are at the ambient pressure,  $P_a$ .
2. For all cells in use all valves are opened and cells are purged in parallel (for a 1-min minimum). Cells not being used (not identified by the user) are isolated by closing the input and output valves.
3. At the end of the purge period, processing begins on the first cell to be used. (Cells are run in ascending numerical order regardless of the order in which they were specified). When a stable ambient pressure is reached, the vent valve of the reference cell closes and the pycnometer acquires and stores a zero pressure value.
4. The reference cell input valve is closed to isolate  $V_A$  from the cell. Approximately 6 s later, the current sample cell input valve opens and the cell is pressurized to approximately 17 psi or until 3 min elapse.
5. When the cell pressurization is complete, the current cell input valve closes.

The pycnometer waits until a stable pressure is detected and then acquires and stores the pressure  $P_1$  (LED display: pressure A).

6. The  $V_A$  input valve opens. This will cause a pressure drop in the sample cell that is proportional to the change in volume because of the introduction of  $V_A$ . When the pressure stabilizes, the system acquires and stores the cell pressure  $P_2$  (LED display: pressure B).
7. The vent valve is opened to return the cell to ambient pressure. After a short vent period, the instrument begins processing the next specified cell (if any) by venting the cell to ambient pressure.
8. After all cells defined for use have been processed, samples may be removed. The pycnometer indicates this by displaying “<RUN COMPLETED>”.

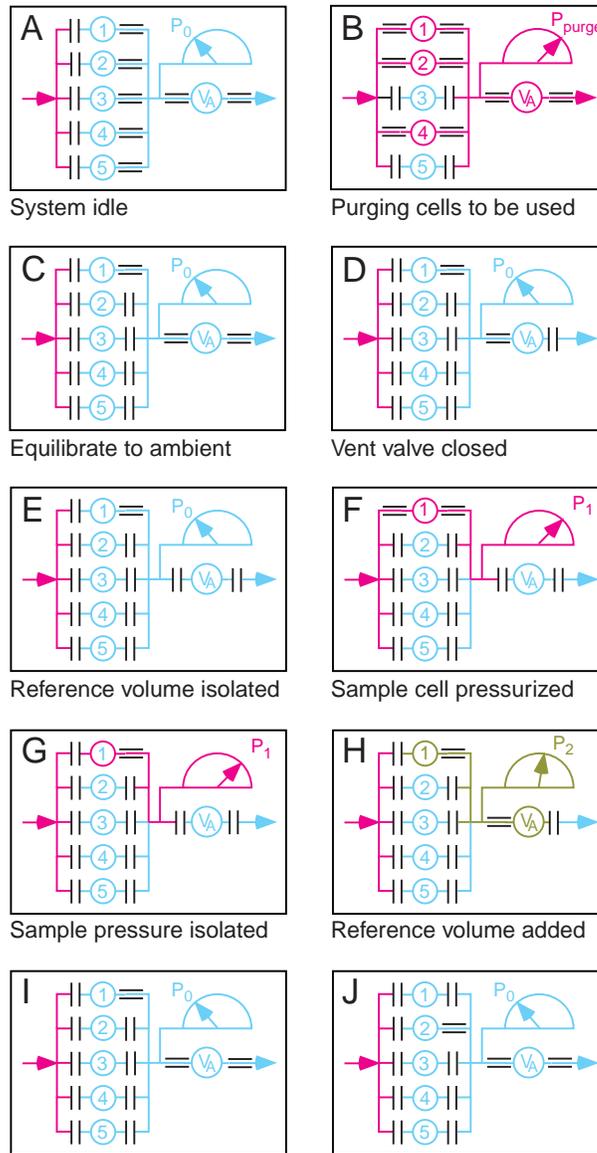


Figure 2—2 Operating sequence of the Quantachrome penta-pycnometer. Blue lines and cells are under ambient pressure  $P_a$  ( $P_a = P_0$ ). Red lines and cells are

under system pressure  $P_1$  (about 17 psi). Green lines and cells are under a reduced system pressure  $P_2$ .

The sample volume can be calculated using the ideal gas law. By opening the solenoid valves on one sample cell with volume  $V_C$ , the system is brought to ambient pressure  $P_a$  after being purged with helium. The state of the system is then defined as

$$P_a V_C = n R T_a, \quad (18)$$

where  $n$  is the moles of gas occupying volume  $V_C$  at pressure  $P_a$ ,  $R$  is the gas constant, and  $T_a$  is the ambient temperature in degrees Kelvin.

When the solid sample of volume  $V_S$  is placed in the sample cell, the equation can be written as

$$P_a (V_C - V_S) = n_0 R T_a. \quad (19)$$

After pressurizing to about 17 psi above ambient pressure, the state of the system is given by

$$P_1 (V_C - V_S) = n_1 R T_a. \quad (20)$$

Here,  $P_1$  indicates a pressure above ambient and  $n_1$  represents the total moles of gas contained in the sample cell. When the solenoid valve is opened to connect the added volume  $V_A$  to that of the cell  $V_C$ , the pressure falls to the lower value  $P_2$  given by

$$P_2 (V_C - V_S + V_A) = n_1 R T_a + n_A R T_a, \quad (21)$$

where  $n_A$  is the moles of gas contained in the added volume when at ambient pressure.

The term  $P_a V_A$  can be used in place of  $n_A R T_a$  in Equation on page 8 yielding

$$P_2 (V_C - V_S + V_A) = n_1 R T_a + P_a V_A. \quad (22)$$

Substituting  $P_1 (V_C - V_S)$  from Equation on page 8 for  $n_1 R T_a$ :

$$P_2 (V_C - V_S + V_A) = P_1 (V_C - V_S) + P_a V_A \quad (23)$$

$$(P_2 - P_1) (V_C - V_S) = (P_a - P_2) V_A \quad (24)$$

$$V_C - V_S = (P_a - P_2) / (P_2 - P_1) V_A. \quad (25)$$

Equation on page 8 is further reduced by adding and subtracting  $P_a$  from  $P_2$  and  $P_1$  in the denominator, giving

$$V_S = V_C - \{[(P_a - P_2) V_A / [(P_2 - P_a) - (P_1 - P_a)]]\} \quad (26)$$

$$= V_C + V_A / \{1 - [(P_1 - P_a) / (P_2 - P_a)]\}. \quad (27)$$

Because  $P_a$  is zeroed prior to pressurizing:

$$V_S = V_C + V_A / [1 - (P_1 / P_2)]. \quad (28)$$

This is the working equation employed by the penta-pycnometer.

## Convection Oven

The convection oven can maintain  $105^\circ \pm 5^\circ\text{C}$ .

This drying process has two main problems: (1) clay mineral interlayer water is largely lost in addition to interstitial water and (2) specimens dried in a convection oven become brick hard and are rarely useful for any other analyses that require substantial sample volumes. Use of freeze-drying would partly eliminate these problems. In particular, stable isotope analyses on foraminifers would be possible from freeze-dried samples, but not from oven-dried samples. The convection oven is used based on advice from the relevant JOIDES advisory panel, because drying at  $105^\circ \pm 5^\circ\text{C}$  for 24 hr is a well-established soil science standard.

## CALIBRATION

### *Beaker Mass and Volume*

Beaker mass must be measured and entered into the MAD program for all beakers to be used on a leg. Beaker volume is not convenient to measure because the low material volume to void ratio in the pycnometer cell gives inaccurate values. We have therefore determined the density of the Pyrex beaker material accurately by filling a beaker with chips of other beakers, measuring its mass and volume, and calculating its density. The density of  $2.2 \text{ g/cm}^3$  is stored in the MAD program, which returns the volume corresponding to each beaker mass.

Custom-made aluminum beakers were used until Leg 168. These beakers were difficult to clean, corroded with time, and were expensive to manufacture. For historical data migration purposes, those beaker materials had a density of  $2.78 \text{ g/cm}^3$  (determined by P. Blum, 1996).

### *Balance Calibration*

The ship is an environment of cyclically changing gravity, and the measured weight  $W$  of a mass  $M$  is significantly affected by the ship's motion. If  $W$  is measured over a period of time several times the periodicity of the ship's acceleration  $a$ , the average can be related to  $M$ . By using two balances, mass determination can be significantly accelerated. The following two equations can be written for two balances:

$$F_s = M_s \times a(t) = A_s + B_s \times V_{s(t)} \quad (29)$$

$$F_r = M_r \times a(t) = A_r + B_r \times V_{r(t)}, \quad (30)$$

where  $F_s$  and  $F_r$  are average measured weights and  $M_s$  and  $M_r$  are known mass standards on the sample and the reference balance, respectively,  $a$  is the ship's average acceleration,  $V$  is the average voltage measured, and  $A$  and  $B$  are constants characteristic for the balances. The calibration principle is to measure multiple standards (typically 1, 5, 10, 20, and 30 g) to determine  $A$  and  $B$  for each balance. For the calibration, measuring time should be at least 30 s to cover several of the 7–8 heave cycles of *JOIDES Resolution*.

Equations on page 9 and on page 9 can be solved for  $a(t)$ , which is assumed to be equal for both balances:

$$M_s = [A_s + B_s \times V_{s(t)}] \times M_r / [A_r + B_r \times V_{r(t)}], \quad (31)$$

which is identical to

$$M_s (\text{unknown}) / M_s (\text{calculated}) = M_r (\text{known}) / M_r (\text{calculated}). \quad (32)$$

The first right-hand term in Equation on page 9 is the first approximation to the calculated sample mass. This value is uncorrected for motion and is returned instead of 0 if the user sets  $M_r$  (*known*) = 0. The second right-hand term in Equation on page 9 uses the ratio between a known mass  $M_r$  on the reference balance and its corresponding calculated value to correct the first term for the ship's motion.

The MAD program performs the linear regression for multiple standards and stores the coefficients until a new calibration is performed. A balance calibration takes up to 15 min. It is recommended that a few control measurements be taken after a calibration to verify the correct mean value and a percent standard deviation of less than 1% for 100 or more measurements taken over approximately 30 s.

### *Pycnometer Calibration*

The pycnometer has an internal calibration procedure. The user is guided through the procedure step by step by the program. First, cell 4 must be used to calibrate the reference volume (pressure)  $V_A$ . Then, the calibration sphere is cycled through all five cells to determine the empty cell volume (pressure). The calibration values are stored in the pycnometer and used until a new calibration is performed. A pycnometer calibration takes up to 30 min.

The instrument calibrates  $V_A$  by performing two pressurizations, once with the sample cell empty ( $V_S = 0$ ) and once with the calibration standard of volume  $V_{std}$  in the same sample cell.

Equation on page 8 derived previously for a sample measurement for these two conditions can be written as

$$V_S = 0 = V_C - V_A / [(P'_1/P'_2) - 1] \quad (33)$$

and

$$V_S = V_{std} = V_C - V_A / [(P_1/P_2) - 1]. \quad (34)$$

Combining these two equations yields

$$V_A = V_{std} / \{ [1/(P'_1/P'_2) - 1] - [1/(P_1/P_2) - 1] \}. \quad (35)$$

The instrument calibrates the volume  $V_C$  of each cell with one pressurization of each cell holding the appropriate sample holder and the calibration standard.

Equation on page 10 is then used and can be written as:

$$V_S = V_{std} = V_C + V_A / [1 - (P_1/P_2)] \quad (36)$$

$$V_C = V_A \{ 1 / [(P_1/P_2) - 1] \}. \quad (37)$$

## PERFORMANCE

### *Precision*

Standards of 1 and 20 g are measured to confirm balance calibration, and the readings should be within 1 mg, or better than 0.1%. Repeatability of specimen mass at sea should also be within 0.1%.

For the pycnometer, a standard sphere is measured (e.g., 7.0699 cm<sup>3</sup>) and precision should be within 0.1% (0.005 cm<sup>3</sup> for the sphere mentioned). Repeat measurements on sediment samples yield a precision of about 1%, probably

resulting from changes in ambient pressure and temperature and the material during handling.

### *Accuracy*

Mass error: 0.1%.

Volume error: 1%.

## **MEASUREMENT**

The user is guided through data entry by the MAD program, which controls the balance as well as the pycnometer. The sample ID needs to be entered only once for the entire process. The pycnometer key pad is not used during measurement. The following is the general measurement protocol:

1. Typical sampling frequency for MAD measurements is two per section. One per section is considered a minimum; more than two per section on medium- to high-recovery legs is rather demanding with the present staff assignments.
2. Fill a numbered 10-mL Pyrex beaker with sediment to about 3 mm below the rim so that material is not lost during handling of the beaker. The largest errors in MAD measurements probably stem from lost material during the process and from volume measurements with incompletely filled beakers. It is the operator's responsibility to find the optimum. Place a special PP Styrofoam plug into the hole left from where the sample was taken from the working-half core.
3. Enter the sample and beaker number into the Sample program at the sampling table. This information will then be in the database; only the beaker number is used at the MAD station to select samples.
4. Measure the mass. Do not let the sample stand without covering it with plastic film, being careful not to lose material.
5. Optionally, measure the wet volume in the pycnometer. However, this is not necessary and years of experience have shown that wet volume measurements (method B) appear to have a large error.
6. Place the sample in the oven at  $105^{\circ} \pm 5^{\circ}\text{C}$  for 24 hr. Place the sample in a desiccator after it is removed it from the oven.
7. Measure the mass and volume of the dry sample and beaker.
8. Place the residue in a sample bag, attach a completed label, seal, and box.
9. Clean the beaker.

# DATA SPECIFICATIONS

## Database model

Table 2—2 MAD database model.

Sample	MAD sample data	MAD control data	MAD beaker history
sample_id [PK1] location [PK2]	sample_id [PK1] [FK] location [PK2] [FK]	mad_control_id [PK1] run_date_time	mad_beaker_id [PK1] beaker_date_time [PK2]
sam_section_id . section_id sam_archive_working top_interval bottom_interval piece sub_piece beaker_id . mad_beaker_id volume entered_by sample_depth sample_comment sam_repository . repository s_c_leg . leg s_c_sam_code . sam_code sam_sample_code_lab . s_c_l	mad_beaker_id beaker_date_time fixed_volume mass_wet_and_beaker mass_dry_and_beaker vol_wet_and_beaker vol_wet_and_beaker_stdev vol_wet_and_beaker_n vol_wet_and_beaker_cell vol_dry_and_beaker vol_dry_and_beaker_stdev vol_dry_and_beaker_n vol_dry_and_beaker_cell comments sample_date_time	ctrl_standard_id control_type expected_value pyc_cell_no measured_value measured_stdev	beaker_number beaker_type beaker_mass beaker_volume
			MAD beaker mad_beaker_id [PK1]
			MAD calibration history mad_calibratin_id [PK1] calibration_date_time calibration_type

Notes: The Sample table is used for all ODP core samples. MAD samples are identified by sampling code; the ODP standard designation is linked through the beaker\_id. If method A is used the "fixed\_volume" must be set to be TRUE. The MAD calibration history table is a log of calibrations but does not hold the calibration data.

## Standard Queries

Table 2—3 MAD query A (results).

Short description	Description	Database
Sample ID	ODP standard sample designation	Link through [Sample]sample_id
Depth	User-selected depth type	Link through [Sample]sample_id
Wb	Water content, relative to bulk mass	see MAD Query B
Ws	Water content, relative to solid mass	see MAD Query B
<b>Calculations depend on the volume measurement method used: A, B, or C</b>		
Bulk density	Bulk density, method A, B, or C	see MAD Query B
Dry density	Dry density, method A or B	see MAD Query B
Grain density)	Grain density, method A or B	see MAD Query B
Porosity	Porosity, method A or B	see MAD Query B
Void ratio	Void ratio, method A or B	see MAD Query B

Table 2—4 MAD query B (results, measurements, and parameters) (to be implemented).

Short description	Description	Database
Sample ID	ODP standard sample designation	Link through [Sample]sample_id
Depth	User-selected depth type	Link through [Sample]sample_id
Method A	Indicates if method A was used	[MAD Sample Data] fixed_volume
Mb+beak	Bulk mass of sample + beaker	[MAD Sample Data] mass_wet_and_beaker
Md+beak	Dry mass of sample + beaker	[MAD Sample Data] mass_dry_and_beaker
Vb+beak	Bulk volume of sample (+ beaker for B)	[MAD Sample Data] vol_wet_and_beaker
sd(Vb+beak)	Std. dev. of n vol. measurements (for B)	[MAD Sample Data] vol_wet_and_bkr_sd
n(Vb+beak)	No. of vol. measurements (for B)	[MAD Sample Data] vol_wet_and_bkr_n
c(Vb+beak)	Cell no. used for vol. measurement (for B)	[MAD Sample Data] vol_wet_and_bkr_cell
Vd+beak	Dry volume of sample + beaker	[MAD Sample Data] vol_wet_and_beaker

Table 2—4 MAD query B (results, measurements, and parameters) (to be implemented).

sd(Vd+beak)	Std. dev. of n vol. measurements	[MAD Sample Data] vol_wet_and_bkr_sd
n(Vd+beak)	No. of vol. measurements	[MAD Sample Data] vol_wet_and_bkr_n
c(Vd+beak)	Cell no. used for vol. measurement.	[MAD Sample Data] vol_wet_and_bkr_cell
Comments	Comments	comments
Date/Time	Date and time of measurement	sample_date_time
Beaker	Beaker number	[MAD Beaker History] beaker_number
Mbeak	Mass of beaker	[MAD Beaker History] beaker_mass
Vbeak	Volume of beaker	[MAD Beaker History] beaker_volume
Mb	Bulk mass	= (Mb+beak) - Mbeak
Md	Dry mass (includes evaporated salt)	= (Md+beak) - Mbeak
Mpw	Mass of porewater	= (Mb - Md) / 0.965
Ms	Mass of solids (salt-corrected)	= (Md - 0.035*Mb) / 0.965
Vpw	Volume of porewater	= Mpw / 1.024
Msalt	Mass of evaporated salt	= Mpw - (Mb - Md)
Vsalt	Volume of evaporated salt	= Msalt / 2.20
Wb	Water content relative to bulk mass	= Mpw / Mb
Ws	Water content relative to solid mass	= Mpw / Ms
<b>For volume method A</b>		
Vb(A)	Bulk volume (method A)	= (Vb+beak)
Vs(A)	Volume of solids (methods A)	= Vb(A) - Vpw
<b>For volume method B</b>		
Vb(B)	Bulk volume (method B)	= (Vb+beak) - Vbeak
Vs(B)	Volume of solids (methods B)	= Vb(B) - Vpw
<b>For volume method C</b>		
Vd(C)	Dry volume (method C)	= (Vd+beak) - Vbeak
Vs(C)	Volume of solids (method C)	= Vd(C) - Vsalt
Vb(C)	Bulk volume (method C)	= Vs(C) + Vpw
<b>For volume method A or B</b>		
Bulk density	Bulk density, method A or B	= Mb / Vb(A,B)
Dry density	Dry density, method A or B	= Ms / Vb(A,B)
Grain density)	Grain density, method A or B	= Ms / Vs(A,B)
Porosity	Porosity, method A or B	= Vpw / Vb(A,B)
Void ratio	Void ratio, method A or B	= Vpw / Vs(A,B)
<b>For volume method C</b>		
Bulk density	Bulk density, method C	= Mb / Vb(C)
Dry density	Dry density, method C	= Ms / Vb(C)
Grain density)	Grain density, method C	= Ms / Vs(C)
Porosity	Porosity, method C	= Vpw / Vb(C)
Void ratio	Void ratio, method C	= Vpw / Vs(C)

Table 2—5 MAD query C (control measurements) (to be implemented).

Short description	Description	Database
Date/Time	Date/time of control measurement.	[MAD Control Data] run_date_time
Standard	Standard identification	[MAD Control Data] ctrl_standard_id
Type	Type of control meas. (mass or vol.)	[MAD Control Data] control_type
Expected	Expected value	[MAD Control Data] expected_value
Cell	If pycnometer, cell number used	[MAD Control Data] pyc_cell_no
Measured	Measured value	[MAD Control Data] measured_value
Stdev.	Std. dev. of multiple vol. meas.	[MAD Control Data] measured_stdev

Table 2—6 MAD query D (beaker data) (to be implemented).

Short description	Description	Database
Date/Time	Data/time of beaker meas.	[MAD Beaker History] beaker_date_time

Table 2—6 MAD query D (beaker data) (to be implemented).

Beaker	Beaker number	[MAD Beaker History] beaker_number
Type	Type of beaker (e.g., Pyrex 10 mL)	[MAD Beaker History] beaker_type
Mbeak	Measured mass of beaker	[MAD Beaker History] beaker_mass
Vbeak	Calculated volume of beaker	[MAD Beaker History] beaker_volume

Table 2—7 MAD query E (calibration log) (to be implemented).

<i>Short description</i>	<i>Description</i>	<i>Database</i>
Date/Time	Date/time of calibration	calibration_date_time
Type	Type of calibration (mass or vol.)	calibration_type

