

USGS TECHNICAL PROCEDURE GP-14

Measurement of Dry Bulk Rock Densities From Paleomagnetic Samples

1.0 PURPOSE.

- 1.1 To assure the accuracy, validity, and applicability of the methods used to to measure the dry bulk density of 2.5 cm diameter core samples originally gathered for paleomagnetic study, this procedure provides a guide for USGS personnel and their contractors to perform the described work. From this procedure the Department of Energy (DOE) and the Nuclear Regulatory Commission (NRC) can evaluate these activities for meeting requirements of the NNWSI repository.
- 1.2 The procedure documents the responsibilities for QA training and enforcement, the processes and authority for procedure modification or revision, and to whom the procedure applies.
- 1.3 The procedure describes the components of the work, the principles of the methods used, and their limits. It also describes the detailed methods to be used, where applicable, for system checkout and maintenance, calibration, operation and performance verification. In addition, it defines the requirements for data acceptance, documentation, and control; and it provides a means of data traceability.

2.0 SCOPE OF COMPLIANCE.

- 2.1 This procedure applies to all USGS personnel and other persons assigned by the USGS to perform work referred to in Section 1.1, or use data from this procedure if it is deemed within the NNWSI Project to potentially affect public health and safety as related to a nuclear waste repository.
- 2.2 All data derived from this procedure that are presented to support licensing the NNWSI Project, and any calibrations or recalibrations that may be required shall be in accordance with this technical procedure and will vary from it only if and when this procedure is formally revised, or modified as described in Section 8.

3.0 PERSONNEL REQUIREMENTS AND RESPONSIBILITIES. The PI shall require that all personnel assigned to the activities and responsibilities of this procedure shall have the necessary technical training, experience, and personal skills, to adequately perform this procedure; and they shall have a working knowledge of the USGS QA Manual.

- 3.1 The USGS NNWSI Project Coordinator shall have overall responsibility for assuring that the activities described are performed in accordance with this procedure.

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- 3.2 The PI using this procedure shall have direct responsibility for the management and quality of this procedure's use including measurements, descriptions, mapping, sample collection and maintenance, and interpretations conducted by the USGS for the NNWSI Project.
 - 3.3 As a delegate of the PI, contributing investigators performing the work described in this procedure for the NNWSI Project shall have the immediate responsibility for complying with the procedure.
 - 3.4 The USGS QA Office shall have direct responsibility for the appropriate quality-assurance training for all personnel assigned to conduct work described in this procedure.
- 4.0 **DETAILED PROCEDURE.** This procedure provides a quick access method for obtaining dry bulk density measurements of rock core samples. It will be used if the densities of paleomagnetic samples are desired, but that the complete suite of other rock properties tests as determined by NWM-USGS-GPP-10, RO "Rock Property Analysis of Yucca Mountain Core Samples" are not required.

- 4.1 **Objective:** This procedure provides a basis for the consistency and reproducibility of dry bulk density data obtained from paleomagnetic samples using the Sartorius 1475MP8 balance or an equivalent precision balance.
- 4.2 **Basis of the Procedure:** The procedure involves drying and then sealing the samples against moisture. Density measurement using the Sartorius balance is performed following these steps.
- 4.3 **Methods Used:** The procedure involves two main steps: sample preparation; and density measurement.

4.3.1 **Sample preparation**

The paleomagnetic sample to be measured must be thoroughly dried. Drying the samples in an oven at about 105°C for several hours is sufficient. Only samples from which all relevant paleomagnetic data has already been obtained should be used. Samples which are fractured or broken are glued with epoxy prior to waterproofing.

The dried samples are sprayed with a silicone water repellent on all surfaces and allowed to dry-to-touch. Any commercially available repellent for use on tents, etc. will suffice. Following spraying, the samples are placed in an oven at about 105°C for at least 6 hours or left at ambient temperature for 24 hours to allow any excess propellant to evaporate. Tests show that the extra mass from the residual dried spray on the sample does not increase its weight in excess of one milligram. Densities are calculated based on the buoyancy of the samples in water. Waterproofing serves to minimize the permeability variations of different samples over the brief (approximately 5 seconds) period of their immersion.

When the samples have cooled to room temperature, they are ready to be measured.

4.3.2 Density measurement

The instrument used for the density measurements is a high precision Sartorius 1475MP8 balance, or its equivalent. Use of an external density (DEN) keyboard, is optional (Attachment 1).

The scale is placed on a sturdy box such that the "weighing-below hook" is situated over a hole in the top of the box. A hook and basket assembly is hung from this fitting into the box, which has one open side for access. The basket is suspended into a beaker of distilled water at room temperature so that a sample placed in the basket will be completely submerged. The actual balance "pan" is located within the glass cylinder. A small diameter glass sample bottle is placed on the "pan" so that it extends through the hole in the upper metal draft protection lid without touching it. This provides a stand for the samples during weighing.

When the balance has been set up, the tare button (T) on the scale is pushed to zero the display. Then the appropriate water density is input to the fourth decimal place by typing it in on the external data input keyboard and pressing the " ρ f1" button. For example, the density of pure water at 22°C is 0.9978.

The sample to be measured is next placed on the glass bottle and, when the "g" symbol appears on the display, the "Wa" keyboard button is pressed. The sample is then placed in the submerged basket. When the "g" again appears, the "Wf1" keyboard button is pushed. The display then reads the density of the sample to four decimal places. This density is recorded on the data sheet (Attachment 2).

After the sample is removed from the basket the " ρ /g" keyboard button is pushed. If the display does not zero, then the tare button on the balance is pushed again. The next sample is then measured using the same procedure.

- 4.4 Alternative Methods Applicable: There are many different instruments and procedures which could be used to obtain the same result for the dry bulk density of a paleomagnetic sample. Too many alternative methods exist to be detailed here. Data from any other procedure employing a similar high-precision balance could be considered equivalent. Procedure NWM-USGS-GPP-10, RO "Rock Property Analysis of Yucca Mountain Core Samples" is specifically singled out as an alternative to this procedure.

4.5 Materials/Equipment Required:

- (1) Paleomagnetic samples.
- (2) Oven for drying samples.
- (3) Silicone water-repelling spray.
- (4) Sartorius 1475MP8 balance (or equivalent high-precision balance); external density keyboard (optional).
- (5) Supporting box.
- (6) Sample basket.
- (7) One liter beaker of distilled water.
- (8) Data sheets.
- (9) Epoxy.

4.6 Assumptions:

As this is an entirely hands-on technique the only assumption which effects the data is that the silicone spray seals the samples and that the resulting measurement is a true dry bulk density. The effectiveness of the sealant depends largely on the surface porosity of the sample: the more porous, the less effective the water barrier. Samples with high porosity are weighted as quickly as possible. No procedural changes are required for samples containing lithophysal cavities. Testing has demonstrated that, given the short period of immersion, the error involved in high porosity samples and samples containing lithophysal cavities, is within acceptable limits (beyond the third decimal place of the density value).

4.7 Data Information: The data generated is illustrated in Attachment 2.

4.8 Limitations: In general, the use of the procedure is limited only by operator error, and the degree to which samples can be successfully waterproofed. The Sartorius balance, correctly calibrated, is deemed to exceed the accuracy requirements for the data.

5.0 CALIBRATION REQUIREMENTS. Calibration is an important part of this technical procedure. When calibrations are required, it is understood that all instruments, and methods when applicable, will be calibrated in compliance with the Instrument Calibration Procedure (NNWSI-USGS-QMP-12.01) prior to obtaining data that will be cited to support licensing the NNWSI Project.

- 5.1 Calibration Responsibility: The responsibility for timely conduct and maintenance of the calibrations required by this procedure lies with the PI. Performance of the calibration in accordance with procedures as described or referenced in Section 5.2 and maintenance of all pertinent calibration records as described in Section 5.3 may be done by a contributing investigator under the direct supervision of the PI.
- 5.2 Calibration Procedure: The Sartorius 1475MP8 precision balance is the only equipment requiring calibration in this procedure. The procedure for calibration is covered in the pamphlet "Installation and Operating Instructions" which comes with this balance. The calibration shall be conducted every 6 months, or any time the balance needs other service work.
- 5.3 Calibration Records: Calibration data will be entered in a notebook or other organized documentation. A field notebook will be used if the test equipment is used in the field. These notebooks or other documents shall be maintained as described in the Document Control Procedure (NNWSI-USGS-QMP-6.01) and stored in accordance with the Records Control Procedure (NNWSI-USGS-QMP-17.01). They shall contain calibration data, recalibration data, and any pertinent observations. Minimum data will include instrument type, its identification and location, and the calibration procedure used, its date, the standard used, its range and accuracy, recalibration due date, responsible division subunit, and the name of the person calibrating the instrument. Calibration entries shall be signed and dated by the person performing the calibration and filed with the QA Office.
- 5.4 Labeling of Equipment Calibration Status: In compliance with NNWSI-USGS-QMP-12.01, a sticker will be affixed to each piece of equipment used in this procedure denoting the calibration status according to one of the following three categories:
1. Showing equipment identification, date calibrated, date recalibration is due, procedure number and calibrator.
 2. Indicating the equipment identification, "OPERATOR TO CALIBRATE", and the procedure number.
 3. Showing the equipment identification and "NO CALIBRATION REQUIRED".

6.0 IDENTIFICATION AND CONTROL OF SAMPLES.

6.1 No samples are taken under this procedure. The samples used are labeled according to QA procedure NWM-USGS-GPP-06, R0, and are designated as such on the data sheets (Attachment 2).

7.0 QUALITY ASSURANCE RECORDS. It is acknowledged that all information collected and recorded under this procedure that will be used in support of the NNWSI Project licensing process are required to be a part of the

official USGS record. Input needed to process the information as a record includes: title or description, subject, originator, date of the document, and if it is an original, a revision or an addendum.

7.1 Notebooks and log books will be prepared by the PI or a contributing investigator as necessary to record data from this procedure and shall include any information considered by the originator to be pertinent. When data are kept in loose-leaf form, each page will be numbered consecutively and chronologically. These documents will be signed or initialed and dated by the investigator on a daily basis when entries are made. Any revisions will be lined out, initialed, and dated.

7.2 All data collected and the applicability of methods used in this procedure will be reviewed and cosigned by a peer of the investigator knowledgeable with the objectives of this procedure in accordance with NNWSI-USGS-QMP-6.01, Section 4.2.2; and as such are acknowledged by both the investigator and the reviewer to be acceptable and meaningful data that meet appropriate quantitative and qualitative acceptance criteria.

8.0 MODIFICATIONS. Significant procedural changes shall be fully documented in subsequent revisions of this technical procedure, and will be subject to QA, peer review, and signature approval.

8.1 If it is necessary to deviate from this approved procedure the PI shall be informed; and the procedure changes shall be documented before proceeding.

8.2 Documentation concerning procedural changes shall describe the modifications, give the sections of the procedure affected, and they shall be signed and dated by both the PI and the Branch Chief, or his delegate.

9.0 REFERENCES CITED.

9.1 Installation and operating instructions for Sartorius electronic toploader 1475MP8 balance.

10.0 ATTACHMENTS. The following attachments are included with this Technical Procedure for the purpose of examples as described.

1. Schematic of Sartorius Toploader 1475MP8 Balance.
2. Data Sheet for Density Data for Paleomagnetic Specimens.

11.0 APPROVAL. This technical procedure shall become effective upon its approval as noted by completion of all the following signatures and dates.

K. R. Sternlof
Prepared by: K. R. Sternlof

5/21/85
Date

J. G. Rosenbaum
Technical Reviewer: J. G. Rosenbaum

5/22/85
Date

K. A. Sargent
Branch Chief: K. A. Sargent

5/23/85
Date

W. W. Dudley, Jr.
Chief, Branch of NNWSI: W. W. Dudley, Jr.

4/25/86
Date

J. R. Willmon
Quality Assurance: J. R. Willmon

4/25/86
Date

