SANDIA NATIONAL LABORATORIES
CIVILIAN RADIOACTIVE WASTE MANAGEMENT
TECHNICAL PROCEDURE (TP)

TP-064

VACUUM SATURATION OF GEOLOGIC CORE TO CONSTANT WEIGHT

Revision 04

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(Reviewer signatures above document the review and resolution of comments.)

REVISION HISTORY

Revision	Description
0	Initial issue
A	Minor revisions
1	Revised to incorporate QAIP 20-1 requirements and make other minor improvements.
2	TP-064 was deactivated during Audit BSC-ARC-01-010. It is now reactivated for additional work to be performed. No major technical revisions were required from the previous revision, only references to current procedures and other minor editorial revisions.
3	Minor modifications have been made to reflect actual procedural steps and to make it applicable to a wider range of sample sizes.
4	Minor modifications clarifying some of the procedural steps.

1.0 Scope and Objective

This procedure applies to the vacuum saturation of geologic core samples. This procedure is intended for implementation in a laboratory environment, in conjunction with work for the Yucca Mountain Project. The objective of this procedure is to define a process to vacuum saturate geological specimens to constant weight.

2.0 Prerequisites

Before performing work under this technical procedure, personnel must be trained by the Principal Investigator (PI) and demonstrate their proficiency in performing the work in this procedure. The PI has the responsibility for generating a record of the personnel proficiency training, as well as the responsibility that work is performed and documented in accordance with this procedure.

The personnel using this procedure are responsible for ensuring that a controlled copy of this procedure is available and used for performing the work in this procedure.

3.0 Description of Activity

Samples are weighed, submerged in water with an applied vacuum, then left submerged under ambient pressure. Air in the sample is removed by the vacuum and replaced by the water. A sequence of saturating and weighing is repeated until the sample reaches constant weight. In this procedure, the criterion for a constant weight is defined as the condition when a sample's weight changes by no more than a specified amount, between successive vacuum saturation cycles.

4.0 Operations

4.1 Specimen Identification and Chain-of-Custody Activities

Samples will be handled in accordance with QAIP 20-03, *Sample Control*. Specimen identification (ID) will appear on the specimen or its container and on the corresponding specimen Chain-of-Custody Form (see QAIP 20-03, Appendix B). If the specimen ID marking becomes illegible, it should be written on a portion of the specimen where it would not be removed during any subsequent process. In any case, only one unmarked specimen should be outside of its container at any one time.

4.2 Equipment

The following pieces of equipment are necessary to vacuum saturate a sample:

4.2.1 Sample Container: The container must be large enough for the sample to be completed submerged in water.

4.2.2 Vacuum Chamber: The vacuum chamber must be large enough to hold the container and be capable of safely maintaining an absolute pressure of 250 Torr, or lower. (Note: The container and the chamber may be one and the same.)

4.2.3 Vacuum Pump: The vacuum pump must be capable of evacuating and maintaining the vacuum chamber to an absolute pressure of 250 Torr or lower.

4.2.4 Vacuum Gage: There should be a vacuum gage and/or a corresponding pressure indicator capable of reading between ambient pressure and 100 Torr, with a minimum of 25 Torr (0.5 psi) resolution. The gage and/or corresponding pressure indicator will be marked with the following notation:

"For Indication Only"

4.2.5 Temperature-Indicating Device: The temperature-indicating device should have a range of 18 to 30° C (inclusive) and be readable to 1° C. The temperature-indicating device will be marked with the following notation:

"For Indication Only"

4.2.6 Balance: The balance must have a range greater than the expected weight of the saturated sample with an accuracy greater than, or equal to, 0.2% of the pre-vacuum saturation sample weight. For samples of low mass (less than 50 g), the accuracy is at the discretion of the PI.

The manufacturer/make, model, serial number, date of last calibration, and calibration due date of the balance will be documented on the Vacuum Saturation Data Sheet (Appendix A). If the make, model, and/or serial number are not available, the user will define an identifier in accordance to AP-12.1Q, *Control of Measuring and Test Equipment and Calibration Standards*.

4.2.7 Water: A sufficient quantity of water to cover the sample in the container and maintain coverage as the sample saturates. The PI will define the type, purity, or other requirements regarding source and/or origin of the water for each study.

4.3 Vacuum Saturation Procedure

4.3.1 Verify that the following pre-requisites have been met:

- The most current revision of this TP is being used.
- The sample identification requirements given in Section 4.1 have been met.
- The laboratory temperature is between 18 and 30° C.
- The items listed in Section 4.2 are ready and available.

4.3.2 Part 1 of the Vacuum Saturation Data Sheet (VSDS)

Part 1 of the VSDS (Appendix A) provides documentation of initial conditions. Data recording may consist of one or more pages of the VSDS.

The following information should be recorded on the first page of single or multiple data sheets:

- Test Plan identifier and revision
- Sample identification (ID)
- Make/manufacturer, model, and serial number of balance
- Verification of calibration of the balance, consisting of date of last calibration and the due date of the next calibration.

Only the sample ID needs to be recorded in Part 1 of subsequent pages when multiple data sheets are generated for a sample.

4.3.3 Part 2 of the VSDS: Step-by-Step Operating Procedures

Note: More than one sample may be saturated at a time.

1. Place the sample to be saturated in its container.
2. Fill the container with enough ambient temperature water to completely cover the sample, anticipating that some water will be lost by evaporation and some will be imbibed into the sample during the vacuum saturation process.
3. Place the container (with water and sample) in the vacuum chamber.
4. Close the vacuum chamber and the vacuum chamber bleed valve.
5. Apply vacuum by slowly opening the regulating valve from the closed position. This will probably cause the water to bubble, so the vacuum must be applied slowly to prevent water from entering the vacuum lines.
6. Record the vacuum saturation cycle number (Line A).
7. If this is a second or subsequent saturation cycle, then record the mean sample weight from the conclusion of the previous cycle (i.e., Enter on Line B the quantity from Line I on the previous page.)
8. Record the date and time at which the vacuum is applied (Line C).
9. Vacuum saturate at least 20 hours. Record nominal vacuum pressure reading (Line D). (Note: Pressure should be checked periodically to assure proper vacuum pump operation.)
10. Close the regulating valve.

11. Open the bleed valve, allowing laboratory air to enter the vacuum chamber. Verify that the sample is still completely submerged in water.

12. Record the date and time at which the vacuum is removed (Line E).

Note: If any part of the sample is not completely submerged, note this on the VSDS, then go back to Step 2.

13. Keep the sample submerged at ambient pressure for at least 16 hours.

14. Record the date and time at which ambient pressure phase ends (Line F).

15. Remove the sample from its container in the vacuum chamber.

16. Weigh the sample using one of the following methods:

    a. Weigh in Air: Within 15 seconds of sample removal, blot the sample with a damp, lint-free paper or cloth towel. Weigh the sample within 15 seconds of blotting.

    b. Weigh under Water: Have the balance set up such that the specimen can be suspended from it in a wire container submerged in water. Tare the balance with the suspension system in place (wire container is submerged). Place the specimen in the wire container and submerge in water. Lightly tap the specimen until no air bubbles rise from it. Suspend the wire container from the balance to obtain the submerged weight. The total mass is equal to the submerged weight plus the mass of the water displaced (specimen volume multiplied by water density).

Note: The weights can be taken using the gram (g) or the pound (lb) unit, and the appropriate units will be circled on the form.

17. Record all weights to the maximum resolution of the balance (Line G).

18. Return the sample to its container and cover completely with water, anticipating that some water will be lost by evaporation and imbibed into the sample during the vacuum saturation process. Keep the sample in the water for between 5 to 15 minutes before performing Step 19.

19. Weigh the sample again, per Steps 15 and 16. Record the results on Line H.

20. Calculate and record the mean value of the weights recorded during Steps 17 and 19. Round the mean weight to the same decimal point that the weights were recorded. Numbers less than 5 are rounded to the next lowest number. Numbers greater than, or equal to, 5 are rounded up (Line I).

Note: If these data are from the first vacuum saturation cycle, go back to Step 1; otherwise, go on to step 21.

21. Calculate the weight change, i.e., the change in sample weight between the vacuum saturation cycles (Line J).

22. Determine if the weight change is within the specifications. If the weight change is ≤ the larger of 0.5% or 0.05 g, the process has met the specification and vacuum saturation of the sample is terminated. If the weight change is > the larger of 0.5% or 0.05 g, vacuum saturation should continue until the weight change specification has been met. (Line K).

Note: If the sample is "Out of Specification" then go back to Step 1; otherwise, go on to step 23.

Note: Ultimately, the accuracy requirement is the responsibility of the PI. The above accuracy is assumed unless the PI states otherwise.

23. For each sample meeting the specification:

- Remove from the vacuum chamber and store in water.
- Sequentially number multiple vacuum saturation data sheets used (i.e., page 1 of 2, page 2 of 2, etc.).
- Verify that all parts of the VSDS (i.e., Parts 1, 2 and 3) are complete

23. Discard the water from the container/vessel.

4.3.4 Post-requisites

When the last sample has been completed, verify that all samples are stored appropriately and all equipment is turned off.

5.0 Safety

There are no special safety hazards, only the normal hazards of the equipment. Operations will be in accordance with safety requirements of the facility where the work is being performed and that of the employer of person(s) performing the work.

6.0 Nonconformances, Deviations, and Corrective Actions

Any nonconformances or deviations must be reported to the PI as soon as possible. Deviations, deficiencies and corrective actions must be determined and documented in accordance with AP-16.1Q, *Condition Reporting and Resolution*.

7.0 QA Records

QA records, and any corrections or changes thereto, generated as a result of implementing this procedure will be prepared and submitted as inclusionary QA records (QA:QA) by the PI in accordance with AP-17.1Q, *Records Management*. These records include:

- Proficiency training records (Section 2.0)
- Calibration records
- Vacuum saturation data sheets (VSDS) (Section 4.3)

8.0 References

AP-12.1Q, *Control of Measuring and Test Equipment and Calibration Standards*

AP-16.1Q, *Condition Reporting and Resolution*

AP-17.1Q, *Records Management*

QAIP 20-03, *Sample Control*

Appendix A Cycle # _____ Page ____ of ___

VACUUM SATURATION DATA SHEET (VSDS)

Part 1

Sample ID _____ Type of Water Used_____

Test Plan and Rev. _____

Make/manufacturer of balance _____

Model and serial number of balance _____

Date of last balance calibration _____Due Date _____

Sample Weight _____g/lb Repeat of Sample Weight _____g/lb

Mean value of pre-vacuum-saturation sample weights _____g/lb

Part 2

A. The data below is for vacuum saturation cycle number: _____

Note: If this is the beginning of the first cycle, skip to step C. Any other cycle, fill in step B.

B. Mean sample weight from Line I (or Ib) of previous page: _____g/lb

C. Date and time vacuum saturation begins: Date _____Time_____

D. Nominal vacuum pressure reading: _____

E. Vacuum saturation ends/ambient pressure phase begins: Date _____Time_____

F. Ambient pressure phase ends: Date _____Time_____

G. First sample weight: _____g/lb

H. Second sample weight: _____g/lb

I. Mean sample weight from Lines G & H: _____g/lb

Note: If this is the end of the first vacuum saturation cycle, then continue data on the next page, if this is the end of any later cycle, then complete Lines J and K.

Note: This section to be used when weighing sample underwater.

a. Sample volume or weight of displaced water _____

b. Total sample weight: (Line I + weight determined from line Ia) _____g/lb

J. Sample weight change [100 X (Line I (or Ib) – Line B)]/Line B = _____%

K. Sample Disposition (check one):

In Specification _____ (i.e., weight change is $\leq$ 0.5% or 0.05 g)

Out of Specification _____ (i.e., weight change is not $\leq$ 0.5% or 0.05 g)

Note: If the sample is "In Specification", the vacuum saturation process is completed. If the sample is "Out of Specification", then continue to the next vacuum saturation cycle and record the data on another VSDS.

Comments:

_____

_____

Part 3

Work performed by: _____

Printed Signed Date

Company/Division: _____ Location of Work: _____