1.0
1.1
1.25
1.4
1.6
1.8
2.0
2.2
2.5
2.8
3.2
3.6
4.0
4.5
5.0
5.6

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Yucca Mountain Site Characterization Project

An Experimental Comparison of Laboratory Techniques in Determining Bulk Properties of Tuffaceous Rocks

P. J. Boyd, R. J. Martin III, R. H. Price

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An Experimental Comparison of Laboratory Techniques in Determining Bulk Properties of Tuffaceous Rocks

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ABSTRACT

Samples of tuffaceous rock were studied as part of the site characterization for a potential nuclear waste repository at Yucca Mountain in southern Nevada. These efforts were scoping in nature, and their results, along with those of other investigations, are being used to develop suitable procedures for determining bulk properties of tuffaceous rock in support of thermal and mechanical properties evaluations. Comparisons were made between various sample preparation, handling, and measurement techniques for both zeolitized and nonzeolitized tuff in order to assess their effects on bulk property determinations. Laboratory tests included extensive drying regimens to evaluate dehydration behavior, the acquisition of data derived from both gas and water pycnometers to compare their suitability in determining grain densities, a comparison of particle size effects, and a set of experiments to evaluate whole core saturation methods. The results affirm the added complexity of these types of measurements where there is a zeolite component in the sample mineralogy. Absolute values for the bulk properties of zeolitized tuff are immeasurable due to the complex nature of their dehydration behavior. However, the results of the techniques that were investigated provide a basis for the development of preferred, consistent methods for determining the grain density, dry and saturated bulk densities, and porosity of tuffaceous rock, including zeolitic tuff in support of themal and mechanical properties evaluations.

This report was prepared under the Yucca Mountain Project WBS number 1.2.3.2.7.1.3. The data in this report was developed subject to QA controls in QAGR S1232713; the data is not qualified and is not to be used for licensing.

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1.0 INTRODUCTION

This report summarizes the results of experimental studies on representative tuffaceous rock samples collected from the potential nuclear waste repository site at Yucca Mountain in southern Nevada. The laboratory bulk properties to be determined are dry bulk density, saturated bulk density, grain density, and porosity (matrix and intact). The methods summarized in this report will be evaluated to determine which techniques are most suited to determining these properties for tuffaceous rock in support of thermal and mechanical properties evaluations. The results of this, as well as previous studies, will be utilized in the development of technical procedures for bulk property measurements in support of the Yucca Mountain Site Characterization Project (YMP).

Previous studies have shown that the results of bulk property measurements are significantly affected by numerous factors, particularly when zeolitic tuffs are being investigated (Schwartz, 1985; Martin et al., 1991). Variations in preparation, drying, saturation, and/or handling of the samples significantly influence bulk property determinations. Existing standard procedures (e.g., ASTM C 135, D 854, and C 604) do not provide sufficient control over these factors for the testing of hygroscopic samples. The objectives of this investigation were to examine the techniques, to collect data on representative samples, and to assess which methods may be most appropriate for the tuffs at Yucca Mountain. Of particular interest were the methods to determine grain densities and to dry samples (particularly zeolitic samples). In addition, test specimen particle size and saturation method may have significant effects on bulk property determinations. All of these factors have been investigated by implementing an integrated approach to determining preferable preparation, handling, and measurement techniques.

Two methods are frequently used to determine the average grain density of porous rock: water pycnometry and gas pycnometry. Schwartz (1985) compared the two methods and concluded that, in general, either method provides adequate results for tuff, but the use of water pycnometers can allow for more precise and faster processing of the samples. Martin et al. (1991), however, concluded that gas pycnometry is the preferred method for the testing of zeolitic samples because they felt the effects of moisture adsorption could be adequately accounted for, whereas they felt that would not be the case for the water pycnometry method. The variability in results is due to complex interactions between sample mineralogy (commonly zeolites) and test gases, or fluids. The results of this investigation confirm that for nonzeolitized rock either technique is adequate, but as the zeolite content increases, the difference in the results derived by the two methods increases accordingly, with water pycnometry providing progressively higher grain densities relative to those arrived at with the gas pycnometer. The maximum difference in grain densities in this study was 3.4% for the most zeolitized sample. Martin et al. (1991) report a maximum difference of 3.9% for tuffs having a similar zeolite content (approximately 80%

clinoptilolite). A similar trend was reported by Schwartz (1985), although the relationship was not as consistent and the maximum difference in grain densities for the most zeolitized sample was only approximately 1%. Many factors contribute to this relationship and the results of this study indicate that water pycnometry is the preferable method for determining grain densities of tuff samples that may have a zeolite component in their mineralogy. Because residence time under laboratory atmospheric conditions is limited by this method, it allows for better control on the moisture content of the samples at the time of testing, thereby reducing the unquantifiable effects of rehydration. This, along with the unstable test gas-zeolite interactions, explains why water pycnometry is consistently more precise than gas pycnometry throughout all of the laboratory investigations. Consistency is important because the relative proportions of the water types associated with zeolites are not easily defined or determined and will vary with zeolite type and abundance.

Previous bulk property measurements on tuff from Yucca Mountain (see Nimick and Schwartz, 1987 for a summary) have employed a number of different drying methodologies. For example, some specify that the samples be dried at a constant temperature (usually 110°C) for a minimum time period, while others specify that the samples be dried to an "equilibrated" weight, that is, the variation in mass for successive weighings at 24- to 36-hour intervals be less than or equal to 0.05% (Nimick and Schwartz, 1987, Appendix A). Based on these studies, a drying procedure (TP-65, Rev. B, entitled "Drying Geologic Samples to Constant Weight") was developed at Sandia National Laboratories (SNL) that specifies the duration of the drying cycle (120 hours) and the maximum acceptable change in sample mass for successive cycles (0.05%). It is important to note that different drying regimens produce variability in the sample mass and consequently in the bulk properties. This makes comparisons between studies difficult, particularly when zeolitic specimens are involved. This problem was studied by carrying out an extensive drying program and determining grain densities at various levels of dryness. It was hoped that the three commonly defined types of water associated with zeolites (external, loosely bound, and tightly bound (Knowlton et al., 1981)) could be differentiated in some way during these drying sequences. However, the desorption of water from zeolitized samples was continuous, with no definite trends that would allow for the characterization of the types and amounts of water remaining in the samples. As a result, no method has been developed to accurately determine the moisture condition of zeolitized samples, particularly in terms of defining exactly the types and proportions of the water associated with them. Also, there is no clear definition of which of the water types should be included as part of the mineral density. This necessitates that a convenient drying method be applied that will dry the samples to a consistent level without removing substantial amounts of water associated with the zeolite mineral structures. The grain density results can then be evaluated and utilized on a common basis. The procedure outlined by TP-65 is considered acceptable, as the drying temperature of 110°C is

substantially less than the loosely to tightly bound water transition temperature of 170°C defined by Knowlton et al. (1981). This procedure is also followed for other test purposes in the YMP, so its use in the characterization of the bulk properties of tuffaceous rock would provide consistency throughout the rock property measurement studies.

The effects of changes in particle size of tuff specimens were integrated into the investigation of grain density determinations, which also impacts the determination of matrix porosity. A range of particle sizes (0.050 mm powder to 13 cm^3 cylinders) was investigated to assess the contribution of occluded porosity (isolated pore spaces that are inaccessible to fluids) to the grain volume measurements. The objective was to maximize access to the pore spaces of the tuff while maintaining a workable particle size. There appears to be little effect on grain density results caused by the particle size variation investigated here. In fact, differences between the grain densities determined by gas pycnometry of powdered samples and whole cores were less than 0.5% for each of the three sample types tested. Ease of preparation and handling makes particle sizes on the order of 0.5 mm to 1.0 mm preferable.

The determination of dry and saturated bulk densities is dependent on weight and volume measurements. Their values will in turn impact the intact porosity determinations to varying degrees, depending on the nature of occluded porosity in the specimens. The volume of a sample is often measured by the buoyancy method (e.g., ISRM "Suggested Method for Porosity/Density Determination Using Saturation and Buoyancy Techniques," 1981). This measurement is dependent on the saturated weight, which is in turn strongly dependent on the saturation level of the sample. Previous studies (Schwartz, 1990, pp 10-14) have utilized several different methods to achieve sample saturation. These ranged from submersion in water at ambient pressure to pressure saturation at an undefined level. In an attempt to standardize the technique, a procedure that utilizes a vacuum saturation technique (TP-64, Rev. 0, entitled "Procedure for Vacuum Saturation of Geologic Core Samples") was developed. The procedure specifies the duration of the saturation cycle (>16 hours) and the maximum acceptable change in sample mass for successive cycles (0.05%). As a part of the study presented here, whole core samples were saturated in a manner consistent with TP-64 and by pressure saturation. Pressure saturation more fully saturates whole cores, which provides more accurate sample volume and saturated bulk density results. Pressure saturation is faster (a greater level of saturation can be achieved in one hour under pressure than can be achieved by the entire vacuum saturation procedure) and can increase saturation level by as much as 15% over that achievable by vacuum saturation methods, depending on lithology.

The results of this study will direct the development of laboratory procedures that can easily be implemented with a high degree of confidence in the results. The integrated approach to these investigations has allowed for direct cause and effect comparisons, closely emulating the entire bulk property measurement process.

The data collected in this study were accumulated for the purposes of developing procedures and should not be used in any QA analyses for the YMP. The samples were machined from core taken in a drillhole at Yucca Mountain (USW G-1) and an outcrop on the southeast flank of Busted Butte to the southeast of Yucca Mountain (see Figure 1-1).

2.0 AVERAGE GRAIN DENSITY

Three major factors influence average grain density determinations. One involves the method of measurement and the other two are related to sample preparation. Two common methods for determining grain density involve the use of water or gas pycnometry. Extensive grain density determinations of tuff and quartz samples were performed utilizing each of the two methods. Two sample preparation factors were also evaluated: particle size and sample dryness. To examine the influence of sample preparation, samples of the same material were prepared to different particle sizes (0.050 mm powders to 13 cm^3 cylinders) and were measured at three levels of dryness (room dry, dried at 110°C, and dried at 200°C).

2.1 Water Pycnometry versus Gas Pycnometry in the Determination of Grain Volumes

The comparison between the two pycnometer methods was carried out by using both methods on the same samples. The samples were first measured with the gas pycnometer and then with the water pycnometers. The procedures for each of these methods are based on ASTM standard procedures D 854-83 and C 604-86 for water and gas pycnometry, respectively. The only significant deviation from the standard procedures was the use of argon instead of helium in the gas pycnometer for one aspect of the study in order to evaluate argon's behavior with the zeolitized tuff specimens.

2.1.1 Water Pycnometry

Water pycnometers are used to measure the volume of a sample with known dry weight by determining the amount of water the sample displaces after it has been saturated in the pycnometer. The average grain density is then calculated by dividing the dry weight of the sample by its volume. The volume of each pycnometer was calibrated, and the grain densities of high purity quartz specimens were determined to assess the accuracy of the method.

A major advantage of water pycnometry over gas pycnometry is that many samples can be processed simultaneously. The number is limited only by the size of the vacuum chamber. Furthermore, since the water temperature is measured and accounted for with

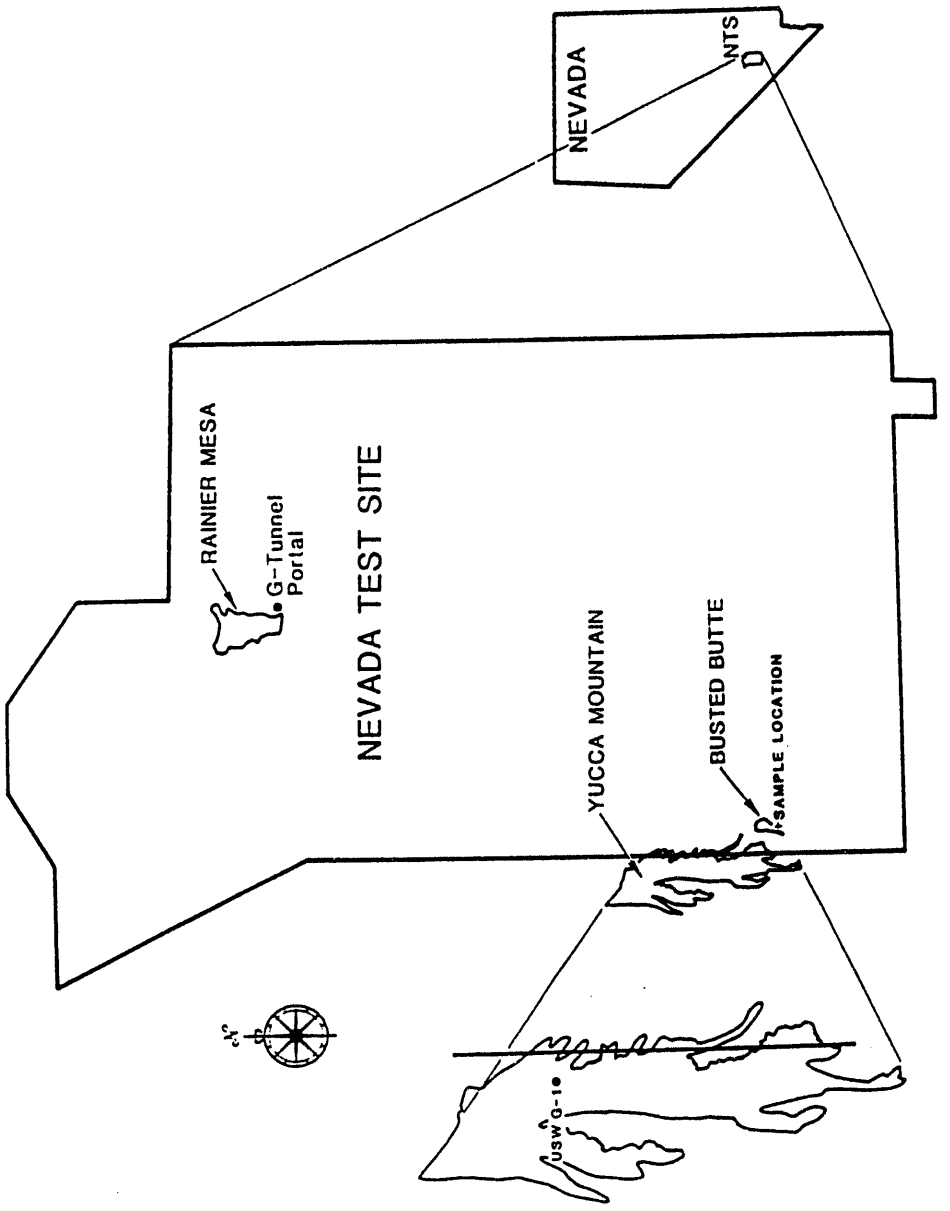

Figure 1-1. Sampling sites at Yucca Mountain and Busted Butte. Samples were selected from the USW G-1 borehole on Yucca Mountain and from outcrop at Busted Butte in southern Nevada.

each measurement, variations in temperature have no effect on the results.

Apparatus--The water pycnometers used in these investigations were nominally 100 ml volumetric flasks. All masses were determined with an equal arm balance accurate to ± 0.1 mg. Vacuum saturation was achieved in a vacuum chamber with an active vacuum supplied by a pump capable of developing 0.667 Pa. Water temperature was measured with a liquid-in-glass thermometer readable to 0.1°C. Auxiliary items included a funnel for transferring the powder, cotton swabs for drying the inside of the pycnometers, and lint-free wipes for wiping the exteriors of the pycnometers clean and dry.

Calibration Techniques--Prior to the grain volume measurements, each water pycnometer was calibrated by a procedure very similar to ASTM D 854-83 (see Appendix I). However, whereas D 854-83 specifies that the mass of the pycnometer with water versus temperature is tabulated, the procedure employed here was to determine the volume of the pycnometer as a function of temperature. This procedure also deviates from that reported by Schwartz (1985), where calibrations were carried out at a single temperature. Multiple temperatures were used because the pycnometer volumes will differ due to expansion and contraction. In this way, it is not necessary to maintain a specific temperature when making grain volume determinations.

In order to evaluate the overall accuracy of the water pycnometer technique, grain densities of quartz specimens were determined. Two specimens were used in this study: a very fine (140 mesh, or less than 0.106 mm), 99.5% pure sample and a coarser (20 to 30 mesh, or less than 0.850 to 0.500 mm) specimen of lesser, but unknown purity. Both specimens are expected to have a grain density very close to the accepted value for quartz of 2.647 g/cc (ASTM C 604-86). Any value that deviated from 2.647 g/cc by more than 1% was unacceptable and the calibrated volume of the pycnometer would be rechecked to investigate the cause of the problem. The maximum deviation from the accepted value in this study was 0.34% . The average density for five determinations was 2.646 g/cc with the fine grained specimen (Table 2-1), a deviation of 0.04% from the accepted value.

Operational Procedure--The procedure for determining grain volumes with a water pycnometer is straightforward. However, there are a number of operations involved that inherently incorporate a certain amount of operator subjectivity. Therefore, it is advantageous to have the same operator perform all the measurements, including the pycnometer calibrations, in order to reduce operator variation. All of the measurements for this work were performed by a single operator.

The procedure followed in this study, as outlined in Appendix I, conforms to ASTM D 854-83 and the procedure used by Schwartz (1985), except for the manner in which the samples are dried and the volume calculated. For this study the samples were either dried in an oven only before testing with the gas pycnometer, or they were dried both before and

Table 2-1. GRAIN DENSITY MEASUREMENTS

I: Measured Grain Density (g/cc) of Tuff Specimens

A: Finer Part.

Sample Sizes:	Max Part. Size	-Dried to 110°C- Helium Pyc.	Water Pyc.	-Dried to 200°C- Helium Pyc.	Argon Pyc.	Water Pyc.
BB-11-C-SNL-A	0.050 mm	2.571	2.609	2.557	N/A	2.566
BB-11-C-SNL-B	0.200 mm	2.570	2.608	N/A	2.579	2.563
BB-11-C-SNL-C	0.100 mm	2.566	2.603	N/A	2.601	2.578
Mean Value:		2.569	2.607			2.569
Standard Deviation:		0.0026	0.0032			0.0079
G1-1361.9-SNL-A	0.200 mm	2.344	2.382	2.364	N/A	2.358
G1-1361.9-SNL-B	0.100 mm	2.355	2.383	N/A	2.483	2.367
G1-1361.9-SNL-C	0.050 mm	2.345	2.387	N/A	2.426	2.368
Mean Value:		2.348	2.384			2.364
Standard Deviation:		0.0061	0.0026			0.0055
G1-1391.3-SNL-A	0.200 mm	2.302	2.378	2.283	N/A	2.334
G1-1391.3-SNL-B	0.100 mm	2.307	2.375	N/A	>5	2.395
G1-1391.3-SNL-C	0.050 mm	2.294	2.385	N/A	>5	2.379
Mean Value:		2.301	2.379			2.369
Standard Deviation:		0.0066	0.0051			0.0316

B: Coarser Part.

Sample Sizes:	Max Part. Size	-Dried to 110°C- Helium Pyc.	Water Pyc.	-Room Dry- Helium Pyc.
BB-11-C-SNL-D-1-b	0.500 mm	2.599	2.600	N/A
BB-11-C-SNL-D-1-c	1.000 mm	2.594	2.607	2.597
G1-1361.9-SNL-D-1-b	0.500 mm	2.372	2.374	N/A
G1-1361.9-SNL-D-1-c	1.000 mm	2.374	2.386	N/A
G1-1391.3-SNL-D-1-b	0.500 mm	2.314	2.363	N/A
G1-1391.3-SNL-D-1-c	1.000 mm	2.322	2.356	2.308

II: Measured Grain Density (g/cc) of Quartz Specimens

Sample	Max Part. Size	Helium Pyc.	Mean Value	Std. Dev.	Water Pyc.	Mean Value	Std. Dev.
NER-A	0.106 mm	2.648			2.646		
NER-B	0.106 mm	2.647			2.642		
NER-C	0.106 mm	2.645			2.645		
NER-D	0.106 mm	2.628			2.652		
NER-E	0.106 mm	2.633	2.640	0.0053	2.644	2.646	0.0038
SNL-A	0.850 mm	2.646			2.647		
SNL-B	0.850 mm	2.648			2.649		
SNL-C	0.850 mm	2.645	2.646	0.0016	2.638	2.645	0.0059

III: Measured Grain Density (g/cc) via the Helium Gas Pycnometer, and Porosity (%) of Whole Cores

Sample	Grain Density	Porosity[1]	Porosity[2]	Porosity[3]	Porosity[4]
BB-11-C-SNL-D-2	2.575	12.19	11.99	13.26	12.55
G1-1361.9-SNL-D-2	2.352	17.77	17.63	18.89	17.23
G1-1391.3-SNL-D-2	2.301	32.29	32.29	34.52	33.76

1) Porosity = 1-(Dry Bulk Density / Whole Core Grain Density via Helium Pycnometer)
2) Porosity = 1-(Dry Bulk Density / Average Grain Density of Sample Powders via Helium Pycnometer) *Note: Fine particle size data only.
3) Porosity = 1-(Dry Bulk Density / Average Grain Density of Sample Powders via Water Pycnometer) *Note: Fine particle size data only.
4) Porosity = [(Saturated Weight - Dry Weight) / Water Density] / Sample Volume

after testing with the gas pycnometer. Gas pycnometry preceded the water pycnometry measurements. In either case, the samples were considered dry prior to testing with the water pycnometers, and it was not necessary that they be dried while in the pycnometers prior to weighing. In addition, the drying regimen followed prior to determining grain densities was much stricter than that prescribed in the referenced procedure, as it conformed to TP-65 for drying geologic samples to an equilibrated weight.

A major potential source of error in this method is the way in which the meniscus is read by the operator. Turbidity of the water due to suspended sample and differences in the form the meniscus may take make accurate readings difficult. In addition, there is a certain amount of operator subjectivity involved in judging the height of the meniscus relative to the scribe line. These factors lead to variations on the order of one drop from a pipette in volume or approximately $0.07cm^3$, which represents errors as large as $\pm$ 0.02 g/cc (0.8%) in the average grain density measurements made in this study.

2.1.2 Gas Pycnometry

Gas pycnometers operate on principles similar to those of water pycnometry, except that the displaced volume is gas rather than water. The sample is measured while dry, eliminating the need for a saturation regimen. Once the sample volume is determined, its average grain density is calculated by simply dividing the dry weight by the volume. The gas pycnometer is calibrated and quartz specimens are measured to check its accuracy.

Helium is the preferred test gas. Helium behaves very much like an ideal gas, which is crucial to the operation of the pycnometer because the technique is based on the ideal gas law. Other gases, such as argon, can be used, but may introduce some error due to differences in their adsorptive characteristics with respect to zeolites. In the course of this study it became apparent that there was some interaction between the zeolitic samples and helium. Therefore, argon was introduced as a test gas in order to determine whether or not it would produce better results on the zeolitized samples.

Schwartz (1985) compared water pycnometry to gas pycnometry using a gas pycnometer (Micromeritics Model 1303) that operated in a significantly different manner than the one used in this study (Micromeritics Model 1305). The major difference between the two models is the property of gas that is measured. For the Model 1303, the volume of helium required to reach a specified pressure is measured, whereas for the Model 1305 only pressure is measured, and changes in pressure are used to determine sample volumes. The Model 1305 provides highly objective results because of its digital readout and its straightforward procedure, and because pressure can be measured more easily than volume. Also, because of the time-dependent gas adsorption phenomena associated with zeolites, the ability to monitor changes in pressure with time with the Model 1305 is advantageous. Two disadvantages of the Model 1303 are that it does not utilize purging,

and that the vacuum procedure does not ensure that helium will totally permeate the sample, particularly for the zeolitized samples where it is evident that the permeation of the samples is very time-dependent. Where the Model 1303 was considered to be operating properly when an accuracy of ± 1.5% was achieved for a calibration check, the Model 1305 is expected to be accurate to ± 0.2%, and preferably to ± 0.1%.

Apparatus--The gas pycnometer used in this investigation was a Micromeritics Model 1305. Helium is the preferred gas, but others (e.g., argon) can be used as long as their suitability is confirmed. A schematic of the device is shown in Figure 2-1. The internal volumes of the device are known and can be verified by a calibration routine. A sample is placed into the sample chamber and after several purging cycles, the pressure in the chamber is stabilized. After the stabilized pressure is recorded, the sample chamber is connected to an expansion volume (adjustable for various sample sizes, see Figure 2-1), allowing the gas to expand and the pressure to respond accordingly. The change in pressure is related to the sample volume by a simple equation based on Boyle's Law, where the more volume the sample takes up in the sample chamber, the greater the pressure drop due to the reduced gas capacity. The sample dry weight is then divided by this volume in order to determine average grain density.

The general equation for computing the sample volume is

$$V_{samp} = V_{cell} - V_{exp} / [(P_1/P_2) - 1]$$

where V_{samp} = the sample volume to be found, V_{cell} = the volume of the sample cell with the empty cup in place, V_{exp} = the expansion volume, P_1 = the charge pressure, and P_2 = the pressure after expansion.

Calibration Techniques--The gas pycnometer was calibrated according to the procedure outlined by the manufacturer. A complete recalibration of the unit is necessary only when checks of the calibration are unacceptable. The calibration determines the volumes of the internal chambers of the unit by running it with an empty sample cup, and then again with the calibration volume (i.e., a precision ball bearing). The pressures during the runs are compiled and used to calculate the internal volumes per the calibration sheet in Appendix II. Each run is made according to the operating procedure outlined in Appendix II.

During the grain density determinations, two checks are made prior to a series of measurements to determine if the unit needs to be recalibrated. One checks the zero offset and the other checks the scale factor error. The zero check consists of running an empty sample cup and calculating the result (see Appendix II). The scale factor check consists of measuring the volume of the calibration specimen. For either check the volume error has to be less than ± 0.2% of the full-scale volume to be acceptable.

In addition to the calibration checks, the grain densities of the standard quartz specimens described in Section 2.1.3 were determined in order to evaluate the accuracy of

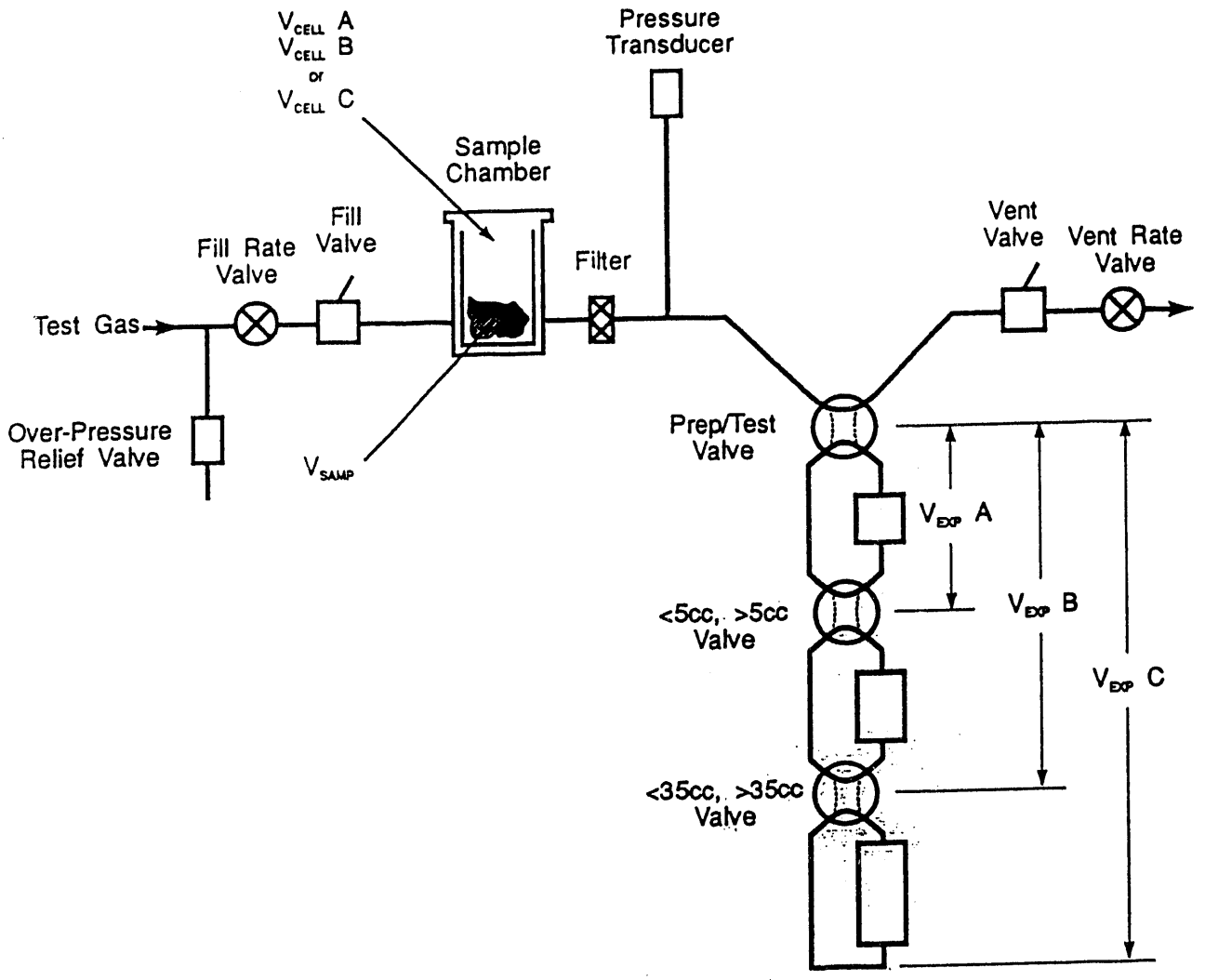

Figure 2-1. Multivolume Pycnometer 1305 schematic. A, B, and C represent device volumes used for samples having volumes of less than 5cc, 5cc to 35cc, and 35cc to 150cc, respectively.

the instrument and the overall accuracy of gas pycnometry as compared to that of water pycnometry. Any results that deviated from the accepted value of 2.647 g/cc by more than 1% were considered unacceptable and the calibration checks would be run to check the operation of the device. No single result deviated from the accepted value by more than 0.72%. The average of five determinations was 2.640 g/cc for the fine grained specimen (Table 2-1), or a deviation of 0.27% from the accepted value.

Operational Procedure--The operation of the gas pycnometer used in this study conveniently lends itself to specific step-by-step instructions. The procedure used conformed to that prescribed by the manufacturer (Micromeritics Instruction Manual, 1987) and ASTM C 604-86. The procedure is outlined in Appendix II. Because the device is operated by following very specific instructions, there is little opportunity for operator bias. There are, however, a number of factors that must be approached with care. Specifically, it has been observed that rock specimens, depending on their mineralogy (particularly zeolite content), behave differently in the manner in which the test gas permeates the sample. The operator must be aware of this and perform the measurements accordingly. The rates at which the test gas is introduced into and removed from the system must be closely regulated. Sample handling, particularly exposure time to ambient conditions, must be performed in such a way as to minimize variability in gas and moisture contents throughout the measurement process.

The most important factors not addressed by the operational procedure are 1) a method for cooling samples after drying, 2) purging of the sample with test gas, 3) the optimum number of cycles (procedure runs) for each sample, and 4) at what point the pressures can be considered stable. These factors become most important when zeolitized samples are being characterized. The complex behavior of the zeolitic samples caused by their hygroscopic nature and interaction with gases, makes them particularly susceptible to the performance of these four factors.

All of the samples were observed to rehydrate to varying degrees, depending on the zeolite content, when cooled from the drying temperature. For this study, two different methods were used to cool the dried samples to ambient temperature so that accurate results could be obtained with the pycnometer. One method was to apply an active vacuum to the warm oven, then allow it to cool to ambient. Samples were removed one at a time, while the vacuum was reapplied to the remaining samples. The second, and preferable, method was to remove a sample from the drying oven and immediately place it into a vacuum chamber, apply an active vacuum for a minimum of 30 minutes to allow the sample to cool, and then release the vacuum while backfilling with the test gas. Ideally, backfilling with the test gas would prevent the atmospheric air from partially rehydrating the sample. Irrespective of the method that was employed, the samples (particularly zeolitized samples) universally rehydrated in the process (on the order of 0.1% weight gain for welded tuff and

0.8% for 80% zeolitized tuff). This has the potential of leading to systematically low determinations of grain densities, on the order of 0.2% (0.005 g/cc) for welded tuff and 1.0% (0.024 g/cc) for 80% zeolitized tuff.

Purging of atmospheric air from the sample chamber with the test gas is suggested by the manufacturer, but a universally satisfactory number of purging cycles is not feasible because of inconsistent adsorption between sample types. This is particularly true where zeolitic samples are concerned, because of varied interactive effects between the samples and the test gas. Therefore, as the study progressed, it became evident that a preferable method was to record each pressurization cycle from the start and continue to record each cycle until the calculated volumes averaged to a stable range of values (see Step 17 of the operational procedure given in Appendix II) from which an average volume can be calculated.

Optimally, the pressures recorded during measurements with the gas pycnometer will stabilize quickly (within fifteen seconds) and can be recorded with confidence. This was generally true for the samples of welded tuff and quartz, where no zeolites were present. When zeolites were present, there was an apparent interaction between the zeolites and the test gases. The interactions seemed to reduce the gas permeability of the samples. Therefore, the rate at which gas would fully permeate the samples when it was introduced into the sample chamber and the rate of gas flow back out of the samples when the volume was increased (reduced pressure) were significantly lower for the zeolitic samples. This caused the pressure to drop quickly at first, as with the non-zeolitic samples, but then drop more slowly with time as the samples became more saturated with the gases. When the expansion chamber volume was added and the pressure dropped, it also would not stabilize quickly and would rise as the gases came back out of the samples. The pressure changes were generally greatest for the most highly zeolitized samples and were taking as long as ten minutes to stabilize to a point where a satisfactory pressure could be recorded. For those types of samples, it was particularly necessary to continue the measurement cycle until consistent, stabilized sample volumes were obtained.

When the pressures stabilized quickly at a definite value, the data was recorded with a high degree of confidence. With the more difficult zeolitic samples, the operator was required to judge when a stabilized pressure was reached, based on the rate of change. Typically, time intervals were chosen that would allow a rate of change on the order of 2×10^{-5} MPa min^{-1}. The time intervals were determined for each sample based on the behavior of the pressures for the first three or four cycles and were followed consistently thereafter.

2.1.3 Sample Descriptions

Three tuff specimens, having significantly different mineralogies, and two quartz specimens were utilized in this study. Specimens G1-1361.9-SNL and G1-1391.3-SNL were recovered from drill holes at Yucca Mountain, Nevada, and BB-11-C-SNL was acquired from an outcrop at nearby Busted Butte. These specimens were prepared (see Section 2.2.1) to the specifications presented in Table 2.1, Section I. In addition to the tuff specimens, the quartz standard materials were measured to check pycnometer calibrations and provide data to evaluate and compare the accuracy of the two pycnometry methods. The specimens are listed below.

BB-11-C-SNL: Welded, devitrified Topopah Spring Member tuff.
Thermal/Mechanical unit: TSw2.

G1-1361.9-SNL: Nonwelded, 50% vitric, 50% zeolitized Calico Hills tuff.
Thermal/Mechanical unit: CHnlv.

G1-1391.3-SNL: Nonwelded, 80% zeolitized Calico Hills tuff.
Thermal/Mechanical unit: CHnlz.

NER: Very fine grained, 140 mesh (greater than 98% of grains are less than 0.106 mm), 99.5% pure SiO_2 silica powder.

SNL: Medium grained, 20 to 30 mesh (0.850 to 0.600 mm) quartz grains of unknown purity.

2.1.4 Discussion of Results

A definite relationship exists between the degree to which tuff samples have been zeolitized and the differences between average grain density results acquired with the gas pycnometer using helium and those acquired with water pycnometers. Typically, the water pycnometry method gave higher grain density values than the helium gas pycnometer method. (Note that the fine particle size powders were redried between methods, see Section 2.3.1. This developed somewhat different moisture conditions than for the coarse particle size measurements.) This relationship became more apparent with increased zeolite content. For the welded tuff specimen (BB-11-C-SNL) the variation was not large (1.5%), but for sample G1-1391.3-SNL the variation was more significant (3.4%) (see Table 2.1, Section I). Further, the grain densities determined for the quartz specimens were virtually identical for both methods (Table 2.1, Section II). There was very good agreement for grain densities acquired with either method for nonzeolitized samples. However, as the zeolite content increases, water pycnometry provided progressively higher values of average grain density than helium gas pycnometry.

This relationship is apparently due to the openness of the zeolite structure and the reactivity of zeolites with gas molecules. This interaction was evident during the gas

pycnometer measurements (see Section 2.1.2). Numerous pressurization (purging) cycles were required to fully saturate the zeolitic samples with helium. As the purging process increased the degree of saturation, the calculated volumes grew progressively larger. The helium may have been cumulatively adsorbed within the zeolitic structure and added to the apparent volume of the entire sample. This effect produced inconsistent results with the helium gas pycnometer and became more pronounced as zeolite content increased. Water pycnometry appears to provide more accurate results for zeolitic materials, at the level of dryness at which they were measured, because the water is able to fully permeate the samples due to their hygroscopic nature. There could also be some effect due to non-ideal behavior of the air in the sample chamber prior to purging with helium, but weights taken before placing the samples into the pycnometer were identical to those taken on removing them after testing. This indicates that no detectable amount of moisture was gained or lost during the measurement process.

The possibility that a test gas other than helium might behave differently and provide better results for the zeolitic samples was considered. Argon is suggested by the manufacturer as a substitute for helium. Therefore argon was utilized for the measurements of six tuff samples (see Table 2.1, Section I). The results indicate that the use of argon significantly worsens the test gas-zeolite interaction and leads to very spurious results where zeolites are present. Data from the measurements on the most zeolitized sample (G1-1391.3-SNL) were virtually useless. Note that the values for the welded tuff samples (BB-11-C-SNL) are nearly the same as those obtained with helium, which also indicates that the inconsistent results are due to test gas-zeolite interactions. Argon should not be considered for use in the gas pycnometer where the presence of zeolites is possible.

On the positive side, water pycnometry provides the capability of processing numerous samples simultaneously. Because the water temperature is measured and corrected for with each measurement, variations in temperature have no effect on the results. Also, because the samples do not need to be cooled before beginning measurements, there is little time for rehydration between removal from the drying oven and weighing of the samples. The ability to control the level of dryness may be the most significant advantage of water pycnometry. The disadvantages of water pycnometry are the potential for operator bias in the interpretation of the meniscus, and the inability to conveniently measure whole core samples.

There are two major advantages of gas pycnometry. First, there is less opportunity for operator subjectivity due to the manner in which the measured parameters are determined, as the pressure levels are displayed on a digital readout and can be accurately observed and recorded. Second, because permeability to a gas is higher than to water, the gas more completely permeates (saturates) the samples, and therefore can provide reasonably accurate results where large particle size samples, or even whole core samples, are being measured. This allows for direct determinations of average grain densities (and

subsequently intact porosities) of whole, undisturbed samples (see Section 2.2.2, and Table 2-1, Section III). This is particularly important if the rocks are to be used in other investigations (i.e., mechanical and thermal properties testing).

Disadvantages encountered while using the gas pycnometer were mostly related to the presence of zeolites in the samples. Weight changes due to rehydration of the samples prior to weighing were directly related to zeolite content; the degree to which the samples rehydrate is very difficult to control or quantify. Furthermore, as the zeolitic content increased, the time required to perform a measurement sequence increased due to the interaction with the test gas. A disadvantage unrelated to sample behavior is the amount of time required to make periodic calibration checks of the internal volumes. Also, although use of the gas pycnometer removes the need for a saturation procedure, it is offset in that it does require a good deal of time to measure each sample. Therefore, if a large number of samples can be processed simultaneously by water pycnometry, the time per measurement can be greatly reduced.

For the quartz specimens, very similar results were achieved with water and helium gas pycnometry. This indicates that both methods are capable of determining grain volumes/grain densities accurately to within 0.3% (0.007 g/cc). The results of measurements on welded, nonzeolitized tuff were also very similar with either method. Significant variations occurred as zeolite content increased; more consistent results are obtained by water pycnometry for these samples.

The results of this work indicate that water pycnometry is the preferable method for determining grain densities of tuff. The problems associated with test gas-zeolite interactions are avoided and better control over the moisture condition of the samples is possible, which leads to more precise results. The results are also more accurate at specific moisture levels. Gas pycnometry can be used to accurately determine grain densities of whole cores, as long as its limitations are understood.

2.2 Particle Size Dependency

Typically, rocks have some porosity that is inaccessible to fluids, caused either by isolated pores or by capillary forces that inhibit fluid from passing through small pore throats. Measurement of the grain volume of whole samples would therefore include these void spaces as part of the total grain volume and lead to low grain densities. To overcome this problem, rock specimens are usually processed by grinding them to powders having small particle sizes. In this way, the majority of the void spaces are exposed at the surface of the grains (i.e., the majority of void spaces accessible to the test fluid).

As a part of this study, several different particle sizes were prepared to evaluate the effects of particle size on grain density determinations and to select the most suitable particle size for the subject tuffs. The maximum particle sizes ranged from 0.050 to 1.0 mm. Right circular cylinders (25.4 mm diameter by 25.4 mm long) were also studied.

Table 2-1 presents the relevant data in Sections I and III.

2.2.1 Sample Preparation

Initially, three samples were prepared from each specimen with an automatic mortar and pestle at the University of New Mexico, Albuquerque, NM. This equipment was used because it is capable of grinding rock fragments to fine powders with very narrow particle size distributions. Samples with maximum particle sizes of approximately 0.050, 0.100, and 0.200 mm were prepared with this equipment. These very fine particle powders presented some difficulties in sample handling during the measurements. Grain densities were determined for each of these samples. There were no discernible effects from particle size variation; therefore, the study was expanded to include coarser particle samples.

The coarser particle samples were prepared at NER. Very simple methods were employed. First, a piece of each specimen was crushed to a powder in a tool steel mortar and pestle. The crushing was continued until the entire sample was able to be passed through the sieves for 0.5 mm and 1.0 mm particle sizes. This way no mineralogy was preferentially acquired or lost because of variations in hardness.

For the whole core, direct porosity measurements, a 25.4 mm diameter by 25.4 mm long right circular cylinder was taken from each of the three specimens. These samples were cylindrically ground and the ends were ground flat and parallel. Their dimensions were accurately measured and the bulk volume of each was calculated accordingly.

2.2.2 Comparison of Results

Average grain density measurements for the powdered samples, using both water and gas pycnometry, showed no significant differences because of variations in particle size (see Table 2-1, Sections I and III). For each technique, on each of the three compositions, the results were very similar, with no consistent trend with respect to particle size. Grain densities of the coarser particle samples (Table 2-1, Section IB) were generally higher with gas pycnometry, and lower with water pycnometry than the finer particle samples, but the differences may be attributed to variability within the measurement/handling techniques (particularly those affecting moisture level) as much as particle size. The greatest observed difference in grain density was 1.2% for the most zeolitic specimen.

The whole core analyses with the helium gas pycnometer also indicate that particle size has a limited affect on grain density results. The results (see Table 2-1, Section III) are virtually identical to those acquired when using the helium pycnometer to measure the fine particle powders prepared from the same specimens. Although these results indicate that there may be no need to grind the samples, it should be noted that results may differ for samples with greater occluded porosity. Because there is no indication that grain density

results from measurements on tuff are affected by variations in particle size, it is not necessary to prepare ultra-fine particle size samples. The use of water pycnometry necessitates that the samples must be prepared to a particle size that can be placed into the pycnometers. Also, because occluded porosity can be present, some reduction in particle size is desirable. Therefore, in order to address these issues, while maintaining a workable particle size for ease of preparation/handling, a particle size of 0.5 mm to 1.0 mm is considered appropriate.

2.3 Sensitivity to Level of Dryness

In order to evaluate the effect that differences in moisture content has on the determination of grain density, the samples described in Section 2.1.3 were measured with both water and gas pycnometry while at various levels of dryness. It is generally assumed that the drier the sample, the higher the average grain density value will be. The differences seen in this study are generally small and may be exceeded by the inaccuracy imparted by the variability in handling procedures.

Of particular significance in the work performed on tuff from Yucca Mountain is the affinity of the zeolitized tuffs to adsorb moisture. Therefore, it is essential that the effects of drying the samples be well characterized.

2.3.1 Methods

Due to the integrated nature of this study, the data utilized in this portion was acquired in the course of performing work also relevant to the other aspects. The samples studied were described in Section 2.1.3. Drying was carried out as explained in Section 3.0, and the grain densities were determined with both gas and water pycnometry.

Care was taken to minimize the potential for the samples to rehydrate following their drying regimens. This was of greatest significance with the zeolitized samples that readily adsorb moisture on exposure to ambient conditions. Detailed explanations of the cooling methods used follow. They differ because other aspects of the study were being considered and their requirements addressed.

Fine Particle Samples--For the measurements carried out on the finer particle samples, dried at 110°C, a vacuum was applied to the drying oven on completion of the drying procedure. The oven was then allowed to cool to ambient temperature under the vacuum. Samples were removed from the oven one at a time, weighed, and placed into the gas pycnometer, where purging with helium was begun immediately. The samples were exposed to ambient conditions for less than one minute in this sequence, yet they still weighed more (as much as 0.3% for the most zeolitized sample) then when drying was completed and they were still at 110°C. After completion of the gas pycnometer

measurements, the sample was removed, reweighed to monitor changes in weight during testing, and placed back into the drying oven. Another sample was simultaneously removed and the vacuum was reapplied. There was no weight change of the samples during the gas pycnometer measuring process. When all of the measurements with the gas pycnometer were completed, the oven temperature was increased to 110°C and the samples were redried per TP-65. They were then removed from the oven and placed into the water pycnometers while still at 110°C. The pycnometers, with samples, were immediately weighed, allowing for very limited exposure of the samples to ambient conditions.

When the fine particle samples were dried at 200°C, a somewhat different sequence was carried out. After an extensive drying procedure that culminated at a maximum temperature of 200°C, the samples were removed one at a time from the oven, weighed, and then placed into a small vacuum chamber where a vacuum was applied and maintained for a minimum of thirty minutes. The remaining samples were left in the oven at 200°C. Once the sample had cooled to ambient temperature, the vacuum was released while the chamber was backfilled with test gas instead of air. This procedure was adopted to minimize the amount of moisture adsorption by the sample. When the sample was removed from the vacuum chamber, it was weighed to detect any weight change. The samples did gain weight (as much as 0.8% for the most zeolitized sample) during this procedure and to a greater level than that for the samples dried at 110°C. This is most likely a result of the greater affinity to moisture imparted in the zeolites due to being more extensively dried (activated). As soon as the cooled sample had been weighed, it was placed into the gas pycnometer and the measurement process was initiated. Weights taken before and after the measurements with the gas pycnometer indicated that no moisture increase was occurring during the measurements. After the sample was removed from the gas pycnometer and weighed, it was placed into a water pycnometer, which was immediately weighed in order to minimize residence time at ambient conditions. The measurement process with the water pycnometers was then performed on these samples without going through another drying sequence. This was appropriate due to the lack of rehydration during the gas pycnometry process, so that the samples were at the same moisture level for both methods, and because a major objective of the study was a comparison of the two pycnometry methods. Furthermore, another extensive drying process would have been required at 200°C, with no certainty that the same level of dryness would be reached.

There were two significant differences in the way the samples dried at 110°C and those dried at 200°C were handled. First, samples dried at 110°C were dried between pycnometry methods and were not allowed to cool (i.e., minimizing adsorbed moisture) before they were placed into the water pycnometers. In contrast, samples dried at 200°C were not redried following the gas pycnometer measurements. This may have imparted some bias as to the sample's moisture content; any moisture adsorbed during the cooling

process would have been present and affected the results of both pycnometry methods. Second, helium was not the only gas used in the gas pycnometer for the samples dried at 200°C. Six of the nine were measured using argon as the test gas (see Section 2.1.4). Because argon produces unacceptable results, a comprehensive comparison of the density values determined by gas pycnometry is not possible. These differences must be considered when analyzing the test results.

Coarse Particle Samples--The coarser particle samples (Table 2-1, Section 1B) were also measured at two levels of dryness. In this case, however, they were measured after drying at 110°C and without any elevated temperature treatment (room-dry). Limited measurements on room-dry samples were made as an auxiliary activity to the comparison of pycnometer methods and particle size effects. This data allows for some comparison of grain density determinations at these two levels of dryness.

Initially, two of the coarser particle size samples were measured in a room-dry condition with the helium gas pycnometer. The intent of this was to evaluate the effect a relatively high moisture content would have on behavior of the pressures recorded during the measurements. The results of these measurements offer valid, though limited, data for comparison with results from dried specimens.

After completing the helium pycnometer measurements, all of the room-dry samples were dried at 110°C per TP-65. Cooling was carried out by applying a vacuum to individual samples and allowing them to cool to ambient temperature. The vacuum was then released while backfilling with helium. The sample was then immediately placed into the helium gas pycnometer, where purging was begun and the measurements made. After completing the gas pycnometer measurements, the samples were placed directly into the water pycnometers where that procedure was completed. As was the case for the finer particle samples, moisture adsorption occurred during the cooling process, but there was no change in the moisture content during the gas pycnometer measurements.

2.3.2 Comparison of Results

Variable moisture contents affect grain density determinations. The data indicates that adsorption during the cooling process leads to noticeable variations in the observed average grain density values. This implies that the drier the sample, the higher the observed density will be. A comparison between the three drying temperature levels is somewhat obscured by differences in handling procedures between grain volume measurement techniques, which affected the levels of dryness (see Section 2.3.1). However, this does allow for a characterization of the effects of moisture variation on samples dried to the same level and then rehydrated to different degrees. Also, the most valid comparisons of the effects of dryness that can be made are between data obtained by the same pycnometry method.

The data (Table 2-1) show that the average grain densities determined with the helium

gas pycnometer for the coarser particle samples average slightly higher than those for the finer particle samples. The variation is greatest (1.09%) for the nonzeolitized tuff, which is contrary to what would be expected where there is more moisture adsorption in the zeolitic tuffs. This may be more attributable to the higher level of dryness of the coarser samples due to the manner in which they were cooled (one at a time; see Section 2.3.1) than to effects of particle size differences. However, in the extreme cases, where there either was no drying (room-dry) or drying occurred at a temperature as high as 200°C, this relationship is not maintained. Note that the room-dry gas pycnometry measurements (Table 2-1, Section IB) resulted in grain density determinations greater than those for the dried, fine particle samples, which are expected to have been allowed to adsorb more moisture prior to testing. The samples dried to 200°C were expected to have provided the highest grain density results, but this was true only for the moderately zeolitized sample. For both of these extreme cases the data is limited, and for all of the helium pycnometry results there is the issue of gas-sample interactions, a factor that makes these comparisons somewhat tenuous.

Two drying temperature levels can be compared for the water pycnometry results, no room-dry measurements were performed. The most illustrative comparison can be made between the fine particle samples redried at 110°C prior to measurements by water pycnometry and the coarse particle samples that were not redried between pycnometry techniques (assuming the particle size effects are negligible). The fine particle samples (Table 2-1, Section IA) are expected to have been drier when the measurements were begun, as they were not allowed time to adsorb moisture. These samples provided consistently higher grain density results than those that had been cooled prior to testing (Table 2-1, Section IB). The variation is greatest for the most zeolitized sample (0.80%) and least for the nonzeolitized sample (0.12%). This is consistent with expectations that the more zeolitized samples would show the greatest change in moisture level over time and hence the greatest effect on the grain density determinations.

After initial examination of the results of the water pycnometry measurements on the samples dried at 200°C, however, this relationship does not appear to hold. In fact, the grain densities determined were consistently lower than for the same samples dried at only 110°C. One reason for the difference may be the treatment of the samples prior to their measurements with the water pycnometers (see Section 2.3.1). Because the samples dried at 200°C were allowed to cool and were measured with the gas pycnometer first, they were given time to adsorb moisture. Also, due to the nature of zeolites, they were more thoroughly activated at 200°C, and therefore, were more capable of fast adsorption of relatively large amounts of moisture. It is not as obvious why the nonzeolitized sample not only had the same relationship, but had the greatest deviation (1.46%) between the results of the measurements taken after drying at 110°C and those after drying at 200°C. There is considerable uncertainty imparted into these measurements when cooling and moisture

adsorption are allowed to occur, and the variation in results approaches that inherently present in measurements such as these because of such factors as sample variability and the imprecision of the measurements.

3.0 POWDERED SAMPLE DRYING

As discussed in the preceding section, the presence of zeolites in some of the tuff horizons at Yucca Mountain adds complexity to the manner in which samples should be dried because of their anomalous dehydration behavior. There are three different types of water associated with zeolites and they are generally classified as external water, loosely bound water, and tightly bound water. The strength of the bonds between the zeolite and water is dependent on its position within the zeolitic structure. It is felt that, because of the variation in bonding energy, the three types of water can be differentiated by temperature and heats of dehydration (Knowlton et al., 1981).

Knowlton and his colleagues investigated the dehydration behavior of clinoptilolite, a major zeolite component of the zeolitic tuffs at Yucca Mountain. Their results indicate that changes in the proportions of external to loosely bound water and loosely to tightly bound water occur at approximately 75°C and 171°C, respectively. Their investigation involved continuous monitoring of weight change while increasing temperature at a constant rate. They did not incorporate the effect of time on their results.

The intent of this portion of the study was to attempt to define convenient laboratory methods of drying zeolitic samples so that their moisture content could be defined and the types of water remaining in the sample could be identified or at least a consistent state achieved in all samples tested. This would allow for the determination of average grain densities of samples at a preferred moisture content. The effects of time of exposure to different temperatures was felt to be an important consideration; therefore, drying methods were chosen that would address it.

3.1 Operational Procedure

Moisture loss behavior as a function of temperature and time of exposure was investigated over two temperature ranges. The ranges were chosen based on the existing SNL technical procedure for drying samples (TP-65) and the results of previous studies. Therefore, the temperature ranges were from ambient to 110°C (drying temperature per TP-65) and from 110°C to 200°C (in excess of the transition temperature of 171°C determined by Knowlton et al., 1981). The coarse and fine particle samples were utilized in the low temperature and high temperature ranges, respectively. The duration (120 hours) of the drying cycles was chosen to correspond with that specified in TP-65 for consistency and consists of repeated five-day periods at the desired temperature until the sample weight

equilibrates. This method contrasts with that generally followed, where weight is continuously monitored by thermogravimetric analysis as temperature is constantly increased. By following the procedure outlined above, the element of time is addressed and can be accounted for.

Because moisture adsorption is a significant variant in the samples investigated, it was important that its occurrence was minimized. The procedure followed during these investigations was to weigh the samples with an electronic digital balance immediately after their removal from the drying oven. This allows for only minimal residence time at ambient temperature. The oven was kept closed while each sample was weighed, and the sample was immediately returned to the oven after weighing.

If after at least two five-day drying cycles at a given temperature, the sample weight had met the criterion of TP-65 (i.e., $\leq$0.05% weight change), the oven was raised to the next temperature level. Where the criteria was not met, the oven temperature was left at the same level for additional five-day cycles until the criterion was met.

The samples were placed into aluminum drying dishes for the drying activities. The weight of the dish and sample was measured, and the dish weight was deducted from the total weight in order to arrive at the sample weight.

3.2 Comparison of Results

The anomalous desorption behavior of zeolites is clearly evident in the results of this study. Whereas the nonzeolitic tuff had a low (approximately 0.5%) moisture content at equilibrated ambient conditions and reaches a "dry" condition at low temperature (on the order of 110°C), the zeolitized samples had discernible moisture contents as high as 4% by weight at ambient conditions and the moisture is desorbed continuously as temperature is increased. The relative amount and types of the zeolites in the tuff will determine its drying behavior. Not only is it difficult to delineate desorption transition temperatures, but it is also not possible (with common laboratory means) to quantify relative amounts of the three types of water associated with zeolites. A summary of the results is presented in Table 3-1.

3.2.1 High Temperature (Up to 200°C)

The main purpose of monitoring moisture content as a function of temperature up to 200°C was to attempt to identify temperatures at which the transition from the desorption of loosely bound to tightly bound water occurs for the zeolitic samples. It was felt that the ideal equilibrated moisture content might be the point at which all external and loosely bound water are desorbed and only the tightly bound, structural water remained. To this end a temperature in excess of that believed by Knowlton et al. (1981) to be the critical temperature for the desorption of tightly bound water (171°C) was finally achieved for this study. The transition temperature needed to be obvious and result in samples having

Table 3-1. POWDERED SAMPLE DRYING RESULTS

High Temperature Range

Equilibrated Sample Weight/Percent Change From Previous (g/%) vs Temperature

Sample	Room Temp.	23 Days at 110°C	10 Days at 125°C	15 Days at 150°C	20 Days at 175°C	15 Days at 200°C
BB-11-C-SNL-A	18.599	18.570/0.16	18.569/0.01	18.567/0.01	18.554/0.07	18.504/0.27
BB-11-C-SNL-B	19.653	19.620/0.17	19.621/0.01	19.621/0.00	19.619/0.01	19.578/0.21
BB-11-C-SNL-C	32.258	32.215/0.13	32.206/0.03	32.202/0.01	32.189/0.04	32.124/0.20
G1-1361.9-SNL-A	27.327	27.105/0.81	27.089/0.06	27.036/0.20	26.938/0.36	26.776/0.60
G1-1361.9-SNL-B	32.225	31.995/0.71	31.973/0.07	31.897/0.24	31.785/0.35	31.475/0.98
G1-1361.9-SNL-C	31.093	30.845/0.80	30.799/0.15	30.710/0.29	30.571/0.45	30.417/0.50
G1-1391.3-SNL-A	23.801	23.358/1.86	23.289/0.30	23.249/0.17	23.136/0.49	22.829/1.37
G1-1391.3-SNL-B	27.973	27.504/1.71	27.427/0.28	27.344/0.30	27.240/0.38	26.882/1.31
G1-1391.3-SNL-C	25.238	24.718/2.06	24.642/0.31	24.557/0.35	24.387/0.69	24.283/0.43

Percent Weight Change From Previous in Each Rock Type (Average Of Three Samples)

BB-11-C-SNL	N/A	0.15	0.02	0.01	0.01	0.23
G1-1361.9-SNL	N/A	0.77	0.09	0.24	0.39	0.69
G1-1391.3-SNL	N/A	1.88	0.30	0.27	0.52	1.04

Percent Water Removed Where Total = Cumulative Loss After 200°C Cycle

BB-11-C-SNL	0.00	64.26	67.10	69.73	74.56	100.00
G1-1361.9-SNL	0.00	38.27	42.77	54.46	72.57	100.00
G1-1391.3-SNL	0.00	50.95	58.38	65.27	77.74	100.00

Low Temperature Range

Equilibrated Sample Weight/Percent Change From Previous (g/%) vs Temperature

Sample	Room Temp.	10 Days at 60°C	10 Days at 70°C	10 Days at 80°C	10 Days at 110°C
BB-11-C-SNL-D-1-b	29.495	29.452 / 0.15	29.449 / 0.01	29.444 / 0.02	29.433 / 0.04
BB-11-C-SNL-D-1-c	40.122	40.077 / 0.11	40.066 / 0.03	40.060 / 0.02	40.040 / 0.05
G1-1361.9-SNL-D-1-b	50.199	49.716 / 0.96	49.648 / 0.14	49.619 / 0.06	49.495 / 0.25
G1-1361.9-SNL-D-1-c	43.666	43.202 / 1.06	43.130 / 0.17	43.087 / 0.10	42.965 / 0.28
G1-1391.3-SNL-D-1-b	44.087	43.173 / 2.07	43.029 / 0.33	42.940 / 0.21	42.656 / 0.66
G1-1391.3-SNL-D-1-c	37.370	36.703 / 1.79	36.609 / 0.26	36.495 / 0.31	36.295 / 0.55

moisture content changes that significantly affected grain density measurements in order to be deemed critical to the bulk property determinations for the YMP. Also, any process developed to attempt to consistently attain the desired moisture levels must be conveniently carried out with typical laboratory means.

As the drying regimen progressed, the nonzeolitic sample (BB-11-C-SNL) had little moisture loss after equilibrating at 110°C until the 200°C level was reached. At that point there was a sharp weight loss, indicating a transition to the desorption of another type of water from the sample. The source of this water is unclear, but it may be associated with clay mineralogy or, more likely, micropores within this tuff. The zeolitized samples also desorbed a large amount of their moisture at 110°C, but continued to desorb it in significant portions as the drying temperatures were increased. Again, a major transition apparently occurred between 175°C and 200°C, where the percentage weight loss increased markedly. A less significant weight loss was also observed between 150°C and 175°C. These relationships are best illustrated in the second portion of the high temperature range section of Table 3-1, where average percent weight changes are presented. Note the relatively consistent weight loss for all sample types between 110°C to 200°C. At 200°C the degree of desorption is obviously elevated.

A transition temperature was encountered during this process. Although it cannot be precisely determined from this data, it seems to be in the range between 175°C to 200°C. This would be comparable to the results presented by Knowlton et al. (1981) and corresponds to the point at which the desorption of tightly bound zeolitic water from clinoptilolite begins.

3.2.2 Low Temperature (Up to 110°C)

The drying series at lower temperature levels (60°C, 70°C, 80°C, and 110°C) was carried out in order to determine whether or not the external to loosely bound water desorption transition could be satisfactorily delineated with the methods used in this study. It was hoped that by defining this transition temperature level, a more accurate characterization of the moisture content of the tuff specimens could be made.

Knowlton et al., (1981) delineated a transition from external to loosely bound water desorption at 75 $\pm$10°C. However, no transition could be delineated from the results of this study (Table 3-1, Low Temperature Range). The weight losses are generally consistent from one temperature level to another for each rock type. Also, more of the total desorbed water was removed after completing the 60°C sequence than for all of the remaining sequences combined.

There is no evidence that drying at temperatures less than 110°C provides for any control over the types of water remaining in the zeolitic mineral component of tuff. There is no discernible desorption transition through these lower temperature levels; therefore, the

definition of critical temperatures is not possible. Similar to the drying at high temperature levels, the amount of moisture loss is greatest for the most zeolitized samples. This again illustrates the variability in drying behavior as a function of the nature of the zeolite component of the tuff.

4.0 SATURATION

Saturation of rock samples is typically performed through a vacuum procedure. Samples submerged in a fluid are subjected to cycles of vacuum and ambient pressure conditions in order to remove as much air as possible from the sample and replace it with fluid. This method provides satisfactory results for most purposes, but requires a lot of time to complete and does not result in a maximum level of saturation. A faster and more thorough method is to saturate the samples by applying an elevated pressure to them while they are submerged in the saturating fluid.

The two methods were compared as a part of this study in order to evaluate their relative utility and applicability in determining bulk properties of tuffaceous rock. In all cases, distilled water was the saturation fluid. Samples were vacuum-saturated per SNL TP-64, pressure-saturated with argon gas as the source of pressure, and pressure-saturated with a gas-free system (water as the source of pressure).

Experiments were carried out to first determine a preferable pressure at which to perform the saturations. Then more detailed analyses were performed in order to evaluate the effects of time, both at pressure and at ambient conditions following the pressurization sequence. The levels of saturation after completing pressure saturation procedures were also compared to the saturation levels achieved by the vacuum method. In this way, relative benefits of the two methods can be compared and preferable procedures can be determined.

4.1 Apparatus

The vacuum saturation procedure complied with the specifications of TP-64. The apparatus consisted of a vacuum chamber, within which the samples were submerged in a container of distilled water, and a vacuum pump.

The application of the pressure saturation procedure was carried out by two methods. The first was to place a container of distilled water, within which the samples were submerged, into a pressure vessel. The pressure was then applied by introducing argon gas directly from a pressurized cylinder. This was a simple means of applying the pressure and was easily controlled with valves. After it was determined that there were shortcomings to this method, a second approach was developed that avoided the introduction of any gas into the water/sample system. This was achieved by placing the samples directly into a pressure vessel, where water pressure was applied and held constant for the appropriate time interval.

The rock specimens used for this portion of the study are from the same samples described in Section 2.1.3.

4.2 Operational Procedure

The pressure saturation procedure is very simple and can be easily performed satisfactorily by a number of methods, depending on available apparatus. The main concern is to apply a preferred pressure to a rock sample/water system and maintain that pressure at a constant level. Because of its simplicity, the procedure could easily be automated. The vacuum saturation procedure complies with TP-64.

4.2.1 Argon Pressure Generation

For the initial series of pressurization cycles, the pressure was applied to the system by introducing argon gas into a pressure vessel within which a container holding the submerged samples was placed. A cylinder of argon was connected to the pressure vessel with a gas regulator placed in the line. The argon was introduced into the vessel and the desired pressure was applied by adjusting the regulator. Each pressurization cycle lasted approximately 24 hours. At the end of each cycle, the container was removed from the vessel and the samples were weighed. It was quickly determined that after returning the container to ambient pressure condition, there was a great deal of gas evolving from the water and samples. As a result, the samples were allowed to equilibrate approximately 24 hours before weighing for subsequent cycles. This procedure was repeated at each pressure level until the $\pm 0.05\%$ weight change tolerance was met (per TP-64).

4.2.2 Water Pressure Generation

Because there was obviously a great deal of argon being dissolved in the water at elevated pressures, the use of a gas to generate the pressure was abandoned. Instead, a system was developed that pressurized water only, thereby minimizing the amount of dissolved gas within the water.

For the water pressurization system, the procedure followed is similar to that for the gas pressure generation system. In this case, however, the samples were placed directly into the pressure vessel, which was then filled with distilled water. A hand pump was used to increase the water pressure to the desired level and was then isolated from the system. The force on the loading piston entering the pressure vessel was held constant by the servo-control system so that it would maintain the desired pressure in the vessel. Once again the pressure was maintained for 24-hour periods before the samples were removed. On removal from the vessel, the samples were weighed immediately and than again after

equilibrating at ambient pressure while submerged for approximately 24 hours. Other equilibration durations were used as the study progressed to evaluate the saturation level as a function of equilibration time. The results are discussed in Section 4.3.

4.3 Comparison of Results

Saturating rock cores with the pressure saturation method increases the degree of saturation and achieves saturation faster than vacuum saturation methods. Schwartz (1990) reports saturation increases resulting from pressure saturation as great as 9.5% over vacuum saturation levels. During the study reported on here an increase in saturation of 16.9% over that achieved by vacuum saturation was observed for a welded tuff sample (BB-10AE-52Y-SNL) due to pressure saturation. Therefore, the improvements in saturation level are significant and allow for more accurate determinations of saturated bulk densities and porosities. For example, the saturated bulk density of BB-10AE-52Y-SNL is either 2.378 g/cc or 2.395 g/cc and its porosity varies from 9.7% to 11.3%, depending on whether the vacuum saturated weight or pressure saturated weight is used.

Pressure saturation is a simple, convenient method for attaining saturation levels greater than those achievable by vacuum saturation methods, and saturation can be performed more rapidly as well.

4.3.1 Argon Pressure Generation

The initial pressure saturation experiments were designed to determine the pressure level that generates the most thorough saturation results with readily available laboratory apparatus. These experiments utilized the argon pressure generation method because of its convenience.

Pressures of 5.0, 7.5, and 10.0 MPa were used. It was desirable to keep the pressures in this range in order to simplify the apparatus needed to attain them. These pressures were also found to be sufficient to significantly improve the rapidity and completeness of saturation.

After one sequence at 5.0 MPa, the welded tuff sample had a 0.40% weight gain, whereas the zeolitic tuff samples actually lost as much as 0.10% of their post-vacuum saturation weight (Table 4-1). This results in porosities via the saturation method of 12.4, 18.2 and 34.0% for the welded, 50% zeolitized and 80% zeolitized samples, respectively. This compares to porosities of 11.4, 18.4, and 34.2% determined from vacuum saturation data for the same samples. A second sequence at a pressure of 5.0 MPa indicated that one pressurization sequence can be sufficient to produce an equilibrated saturation level in the welded tuff. The succeeding pressurization, at 7.5 MPa, increased the saturation of the welded tuff only slightly (0.07% weight gain) and reduced that of the zeolitic samples by as much as 0.67% (Table 4-1). After pressurizing to 10 MPa, the weights of the three

Table 4-1. ARGON PRESSURE SATURATION SUMMARY

	Weight (g) / Percent Change From Previous		
Saturation Condition	(Welded) BB-11-C-SNL-D-2	(50% Zeolitized) G1-1361.9-SNL-D-2	(80% Zeolitized) G1-1391.3-SNL-D-2
Dry	29.158g	25.920g	21.584g
Vac. Sat.	30.628 / 5.0	28.324 / 9.3	25.903 / 20.0
1st 5.0 MPa	30.749 / 0.40	28.295 / (-0.10)	25.889 / (-0.05)
2nd 5.0 MPa	30.759 / 0.03	28.291 / (-0.01)	25.879 / (-0.04)
7.5 MPa	30.780 / 0.07	28.222 / (-0.24)	25.707 / (-0.67)
10.0 MPa	30.765 / (-0.05)	28.128 / (-0.33)	25.683 / (-0.09)
After 24-Hour Vac.	30.769 / 0.01	28.162 / 0.12	25.835 / 0.59

samples decreased. The zeolitic samples decreased most markedly, but the welded tuff also weighed significantly less (Table 4-1). Apparently, the amount of argon dissolved in the water at this pressure was sufficient to affect the welded tuff in a manner similar to the way the lower pressures affected the zeolitic samples.

Weight loss in the zeolitic samples was a significant feature of this method of pressure saturation and became evident early on in these initial experiments. This characteristic is consistent with the results obtained when using argon as the test gas in the gas pycnometer while measuring the volume of zeolitic samples (Section 2.1.4). Apparently, dissolved argon enters the zeolitic structure and when the pressure is released it expands and displaces water within the zeolites and remains in them, thereby reducing the weights of the zeolitic samples. As the pressure increased, the magnitude of weight loss (water displacement) increased as well, because of the larger amount of argon going into solution. A 24 hour equilibration period was allotted each time the pressure was removed from the system in order to allow most of the argon to evolve from the water.

These initial experiments indicated that pressure saturation methods that allow gas to dissolve into the saturation fluid are not desirable. They also indicate that pressures in excess of 5 MPa and less than 10 MPa are sufficient to achieve a degree of saturation that easily exceeds that which can be achieved by vacuum saturation only.

4.3.2 Water Pressure Generation

Once the feasibility of the pressure saturation method was determined, the issues of residence time at pressure, number of cycles, and post-pressurization equilibration time were then addressed. Because the use of gas to generate the pressure was proven to have significant drawbacks (Section 4.3.1), a system involving only the pressurization of water was developed. A pressure of 7.5 MPa was chosen based on the results presented in Section 4.3.1.

Pressure Saturation of Vacuum Saturated Samples--The initial experiments were designed to evaluate the increase in saturation caused by use of the pressurization method rather than the vacuum saturation method, and to determine the number of cycles necessary to reach an equilibrated weight ($\leq$0.05% tolerance). The samples were presaturated under the vacuum saturation procedure prior to the initiation of the pressurization sequences. These were carried out on samples BB-10AE-52Y-SNL and UE25A#1-1413.8-SNL (Table 4-2).

Results of these initial experiments show that pressure saturation procedures can increase the water content of previously vacuum-saturated welded tuff by as much as 16.9%. In this case, as opposed to the results using argon (Section 4.3.1), the saturation level of the zeolitic samples also increased, although to a lesser degree. This may be

Table 4-2. WATER PRESSURE SATURATION SUMMARY

I: Pressure Saturation vs Vacuum Saturation (Cycles 1 through 5 at 7.5 MPa)

	Vac. Sat. Prior to Pressurization		Press. Sat. Starting From Room Dry	
Sample:	BB-10AE-52Y-SNL	UE25A#1-1413.8-SNL	BB-10AE-24X-SNL	UE25A#1-1448.2-SNL
Pre-Vac. Sat. Wt:	471.01g	336.73g	N/A	N/A
Vac. Sat. Wt/ % Change:	491.05 / 4.25	373.45 / 10.91	N/A	N/A
Pre-Press. Sat. Wt/ % Change:	491.46 / 0.08	373.83 / 0.10	457.09g	304.44g
1st Press. Cycle Wt/ % Change:	494.40 / 0.60	374.26 / 0.12	482.16 / 5.49	349.65 / 14.85
2nd Press. Cycle Wt/ % Change:	494.43 / 0.01	374.29 / 0.01	483.10 / 0.20	351.24 / 0.46
3rd Press. Cycle Wt/ % Change:	N/A	N/A	483.70 / 0.12	351.56 / 0.09
4th Press. Cycle Wt/ % Change:	N/A	N/A	484.19 / 0.10	351.54 / (-0.01)
5th Press. Cycle Wt/ % Change:	N/A	N/A	484.33 / 0.03	N/A
6th Press. Cycle (at 15.0 MPa) Wt/ % Change:	N/A	N/A	485.08 / 0.16	N/A
Water Content Increase Over Vac. Sat.:	3.38g / 16.9%	0.84g / 2.3%	N/A	N/A
Total Wt Increase Over Room Dry Wt:	23.42g / 4.97%	37.56g / 11.15%	27.99g / 6.12%	47.10g / 15.47%

II: Postpressurization Monitoring

BB-10AE-24X-SNL (At end of 5th Cycle)

Time from Removal from Vessel:	Weight/% Change from Previous:
0	485.28g
1 hour	484.67 / 0.13
2 hours	484.60 / 0.01
3 hours	484.51 / 0.02
4 hours	484.52 / +0.002
5 hours	484.47 / 0.01
6 hours	484.46 / 0.002
24 hours	484.33 / 0.03

BB-10AE-24X-SNL (End of 15.0 MPa Cycle)

Time from Removal from Vessel:	Weight/% Change from Previous:
0	485.66g
15 minutes	485.49 / 0.04
30 minutes	485.47 / 0.004
45 minutes	485.44 / 0.01
1 hour	485.36 / 0.02
2 hours	485.34 / 0.004
3 hours	485.28 / 0.01
4 hours	485.26 / 0.004
24 hours	485.08 / 0.04
48 hours	484.95 / 0.03
144 hours	484.84 / 0.02

III: Short-Term Pressurization Cycles

Sample: BB-10AE-52Y-SNL Room Dry Wt: 471.01g (Pre-Pressurization) ; Vac. Sat. Wt: 491.05g

Time at 7.5 MPa:	Wt / % Change from Previous:	Time at 7.5 MPa:	Wt / % Change from Previous:
1 hour	493.26g / 4.72	4 hours	493.63g / 0.00
2 hours	493.50 / 0.05	5 hours	493.76 / 0.03
3 hours	493.63 / 0.03	6 hours	493.74 / (-0.004)

Note: BB-10AE-24X-SNL and BB-10AE-52Y-SNL are from the TSw2 T/M unit (welded tuff).
UE25A#1-1413.8-SNL and UE25A#1-1448.2-SNL are from the CHn1z T/M unit (zeolitized tuff).
Samples of the same T/M unit have nominally equal dimensions (volumes).

because of the greater affinity of the zeolites to water, which would improve the efficiency of the vacuum saturation procedure, allowing for a more complete saturation of that type of sample. The negative effects observed as a result of using gas to generate the pressure are avoided by using a system that pressurizes water only.

Pressure Saturation of Dry Samples--A second experiment was performed in order to evaluate the proficiency of pressure-saturating samples that are not previously vacuum-saturated. Two samples of the same thermal/mechanical units as were used for the initial experiments were used here as well so that the effects of lithology would be minimal. The tolerance of $\leq$0.05% weight change was used to determine the point at which the sample was "saturated". The number of pressurization cycles necessary to achieve that level of saturation was also of concern in this investigation.

The results (Table 4-2, Section I, samples BB-10AE-24X-SNL and UE25A#1-1448.2-SNL) indicate that both rock types become well saturated by this method. The zeolitic sample met the tolerance in one less cycle than the welded tuff, which may be because of either its greater effective porosity or its hygroscopic mineralogy. It should be noted that although the total weight increases of these samples is greater than for those used in the initial experiments (BB-10AE-52Y-SNL and UE25A#1-1413.8-SNL), they also have a higher porosity (compare dry weights). Therefore, it is likely that similar levels of saturation were actually achieved for both experiments. Note that although it took five pressurization cycles to attain an equilibrated weight for the welded tuff, the level of saturation that would have been achieved by the vacuum saturation method was most likely exceeded after just the first cycle (a 5.5% weight change versus 4.3% for the vacuum-saturated BB-10AE-52Y-SNL).

For the welded tuff sample, one cycle at 15 MPa was performed in order to acquire information on the magnitude of improvement that would be achieved by greatly increasing the saturation pressure. The increase of 0.16% in weight is somewhat significant, in that it does exceed the 0.05% tolerance; however, the benefit of attaining this slightly higher level of saturation (2.7% increase) may not warrant a saturation procedure that requires a pressure of 15.0 MPa.

Postpressurization Equilibration Effects--It was observed that there was a substantial variation in sample weights depending on their residence time at ambient pressure while submerged in water. The weights were consistently lower as the residence time increased. In order to evaluate the magnitude of this effect, two monitoring sequences were performed (Table 4-2, Section II). Throughout both sequences, the sample was periodically weighed by removing it from its water-filled container and handling it as per TP-64.

For the initial sequence, intervals of one-hour duration were used between weighings for the first 6 hours, then a final weight was measured after 24 hours at ambient pressure.

These measurements were made following the fifth pressurization cycle of BB-10AE-24X-SNL at 7.5 MPa. Note that the major weight loss occurred during the first hour (Table 4-2, Section II). The total weight loss after 24 hours at ambient pressure reached 0.95 g, or 0.20%.

The second monitoring sequence was carried out on the same sample, but after it had gone through the 15 MPa pressurization cycle. This sequence was performed in order to monitor weight changes over shorter time intervals so as to have a better idea of the rapidity of the weight loss during the first hour at ambient pressure. The sample was also monitored over a term of 144 hours to determine the long-term effects of equilibration.

Following the 15 MPa cycle, the weight loss during the first hour was less than during the same period after the final 7.5 MPa cycle (0.06% vs 0.13%). The reason for this is not totally understood. The highest weight loss actually occurred over the first 15-minute period, which indicates that this phenomenon occurs rather rapidly and may be related to the permeability of the sample. This rate of weight loss corresponds closely to the rate at which water is emplaced into these samples during the pressurization cycles, further confirming its dependence on permeability.

Weight loss was continuous over the 144-hour monitoring period. Over the first 48 hours the total loss was 0.15% (0.05% less than the loss after 24 hours for the initial sequence) and it was only 0.17% after an additional 96 hours. The greatest weight loss occurred during the first 24 hours (0.12%). The rate dropped significantly after that.

One reason for the substantial postpressurization weight loss may be gas (in these cases air) expansion when the pressure is reduced. This would force water back out of the pore spaces at rates and volumes controlled by the pore geometries. The welded tuff, for instance, has four size classes of pores (Price et al., 1985). They range from large (a few millimeters to several centimeters) to submicroscopic. This may explain the time dependence of the weight loss: gas expansion in larger pores rapidly forces water out while there is transient flow from smaller pore sizes over time. This phenomenon is also indicated by the continuous, but diminishing, weight gain with repeated pressurization cycles (Table 4.2, Section III). This would seem to indicate that the transient flow phenomenon is of the same magnitude for both the pressurization and depressurization cycles.

Short-Term Pressurization Cycles--Because the rate at which the samples seemed to be saturating under pressure was so great, an experiment was performed to more accurately quantify it. A welded tuff sample (BB-10AE-52Y-SNL) was tested by repeatedly subjecting it to 1-hour pressurization cycles at 7.5 MPa. At the end of each hour the pressure was released and the sample was immediately weighed. The results are presented in Table 4-2, Section III.

It is obvious from these results that the rate at which water is forced into the samples is very fast. After just one hour under pressure, the weight gain was already greater than that

achieved by a complete vacuum saturation procedure per TP-64 (4.72% vs 4.25%, see Table 4-2, Section I). In addition, the total weight gain after 6 hours was only slightly less than that for the same sample after vacuum saturation and two pressure saturation cycles (4.83% vs 4.97%). For these experiments, no equilibration time at ambient pressure was allowed. This has been shown to reduce the weight gains somewhat.

This experiment clearly shows that these pressure saturation procedures can rapidly saturate the tuff samples. Although saturation increased with continuous pressurization, most pore spaces are filled with water after only one hour. It also shows that after only six hours, the rate at which water is emplaced slows significantly. This is consistent with the observations from the postpressurization equilibration effects investigation discussed above.

4.4 Conclusions

Saturation of rock samples by a pressure saturation method clearly improves the rate and level of saturation over that attainable by vacuum saturation methods without significantly adding to the complexity of the saturation procedure. There also is no advantage to vacuum saturating prior to pressure saturating. Although it is desirable to achieve 100% saturation of all pore spaces within rock samples to be characterized, an exact measure of the pore volume is difficult, and determination of the percentage of that volume that is filled by fluid is dependent on that knowledge. The error attributable to this deficiency would have negligible impact on most bulk property applications. Therefore, as for TP-64, a somewhat arbitrary measure must be used to judge the point at which the saturation procedure can be deemed complete and further saturation would be of little value. Although no definite, complete saturation level was achieved by pressure saturation, the improvement over vacuum saturation was such that any further increase would not be substantial and would likely not significantly affect bulk property calculations. Therefore, higher pressures are probably not warranted.

It became obvious during the early experiments that no gas should be allowed to dissolve in the saturation fluid, particularly for zeolitized samples. Pressure saturation systems should be designed so that only the saturation fluid is pressurized, thereby minimizing the possibility of dissolved gases in the fluid and, as a result, lower actual saturation than desired.

Earlier studies have shown that the mechanical and thermal properties are highly dependent on porosity. The densities collected by the techniques determined in this study will be used to calculate porosities, which will support the analysis of mechanical and thermal property data collected in the future.

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Appendix I

Water Pycnometry Procedure

WATER PYCNOMETER CALIBRATION PROCEDURE

All weights and temperature measurements must be recorded on the water pycnometer calibration sheet. A number of pycnometers may be calibrated simultaneously.

1. Weigh a clean, dry, and numbered 100 ml pycnometer to ± 0.0001g.

2. Add distilled water to the pycnometer so that its level is just below the scribe line.

3. Place the pycnometer with water, and a clean beaker of distilled water into an active vacuum for a minimum of 24 hours.

4. Remove the pycnometer from the vacuum.

5. Place the pycnometer into a water bath at a temperature of 20° ± 0.5°C, and allow the water in the pycnometer to equilibrate (15 minutes minimum) to this temperature until it also reaches 20° ± 0.5°C.

6. Using a pipette, fill the pycnometer with the extra de-aired water until the bottom of the meniscus is equal in height to the scribe line.

7. Using a cotton swab, wipe the inside of the neck of the pycnometer dry.

8. Using a lint free wipe, clean and dry the outside of the bottle.

9. Weigh the pycnometer with water to ± 0.0001g.

10. Using a liquid-in-glass thermometer, measure the temperature of the water in the pycnometer, and record it to the nearest 0.2°C.

11. Repeat steps 5 through 10 at temperatures of 22°, 24°, 26°, and 28°C (± 0.5°C).

12. Using the calibration sheet, calculate the pycnometer's volume at the five temperatures. Water densities are determined from standard properties tables.

Bulk Properties
Yucca Mountain Project
WATER PYCNOMETER CALIBRATION
per TP-_____

PYCNOMETER ID: _____

Operator: Print/Sign: _____

Date/Time: _____

Dry Bottle Weight (g)_____

Water Temp. (°C)	Wt. Pyc. + Water (g)	Wt. Water Only (g)	Water Density (g/cc)	Pyc. Volume (cc) [Wt. Water x (1/Density)]
_____	_____	_____	0.99	_____
_____	_____	_____	0.99	_____
_____	_____	_____	0.99	_____
_____	_____	_____	0.99	_____
_____	_____	_____	0.99	_____

Average Volume: _____

Absolute Density of Water

(From Lange's Handbook of Chemistry, edited by J. Dean, 11th Edition, Sect. 10-127)

Temp °C	Density	Temp °C	Density	Temp °C	Density	Temp °C	Density
18.0	0.998595	20.8	0.998035	23.6	0.997394	26.4	0.996676
18.2	0.998553	21.0	0.997992	23.8	0.997345	26.6	0.996621
18.4	0.998520	21.2	0.997948	24.0	0.997296	26.8	0.996567
18.6	0.998482	21.4	0.997904	24.2	0.997246	27.0	0.996512
18.8	0.998444	21.6	0.997850	24.4	0.997196	27.2	0.996457
19.0	0.998405	21.8	0.997815	24.6	0.997146	27.4	0.996401
19.2	0.998365	22.0	0.997770	24.8	0.997095	27.6	0.996345
19.4	0.998325	22.2	0.997724	25.0	0.997044	27.8	0.996289
19.6	0.998285	22.4	0.997678	25.2	0.996992	28.0	0.996232
19.8	0.998244	22.6	0.997632	25.4	0.996941	28.2	0.996175
20.0	0.998203	22.8	0.997585	25.6	0.996888	28.4	0.996118
20.2	0.998162	23.0	0.997535	25.8	0.996835	28.6	0.996060
20.4	0.998120	23.2	0.997490	26.0	0.996783	28.8	0.996002
20.6	0.998078	23.4	0.997442	26.2	0.996729	29.0	0.995944

PROCEDURE TO DETERMINE GRAIN DENSITIES WITH A WATER PYCNOMETER

All weights and temperature measurements must be recorded on the "Water Pycnometer Grain Density Measurement " data sheet. A number of pycnometers may be used simultaneously.

1. Weigh a clean, dry, and numbered 100ml pycnometer to ± 0.0001g.

2. Add 15g to 30g of dry sample powder to the pycnometer by pouring it through a clean, dry funnel.

3. Immediately weigh the pycnometer, with dry sample to ± 0.0001g.

4. Add 50 to 60ml of distilled water to the pycnometer, and swirl it to moisten all of the sample powder.

5. Place the pycnometer, with sample and water, and a clean beaker of distilled water into an active vacuum for a minimum of 24 hours. For the first one or two hours the pycnometer must be watched closely to ensure that the boiling action does not displace some of the sample out of the pycnometer. The vacuum should be regulated accordingly. Also, after approximately one hour the vacuum shall be released, and the pycnometer swirled again to help dislodge air bubbles.

6. Remove the pycnometer from the vacuum.

7. Pour water down the neck of the pycnometer from the beaker until the water level is just below the scribe line. Pouring the water down the neck reduces the likelihood of it forming bubbles, and helps to keep the sample from going into suspension.

8. Using a pipette, add more water until the bottom of the meniscus is at the height of the scribe line. It may be necessary to raise the water level higher than the scribe line so as to wet the sides of the pycnometer for suitable meniscus formation, then remove some water to obtain the correct height.

9. Using a cotton swab, dry the inside of the neck of the pycnometer.

10. Using a lint-free wipe, clean and dry the exterior of the pycnometer.

11. Weigh the pycnometer and contents to ± 0.0001g.

12. Using a liquid-in-glass thermometer, measure the temperature of the water in the pycnometer, and record it to the nearest 0.2°C.

13. Using the " Water Pycnometer Grain Density Measurement " data sheet, with all of the preceding information recorded, calculate the grain density of the sample. Note: The calibrated volume is interpolated, for the temperature determined in step 12, from the latest calibration data.

Bulk Properties
Yucca Mountain Project
WATER PYCNOMETER GRAIN DENSITY MEASUREMENT
per TP-_____

SAMPLE ID:_____

WATER PYCNOMETER ID:_____

Date of latest calibration:_____Nominal Pycnometer Volume:_____

DATA:

Dry Pyc. Wt.:_____g (A) Dry Pyc. + Dry Sample Wt.:_____g (B)

Dry Sample Wt.:_____g (C) = B-A

Wt. Pyc. + Sample + Water:_____g (D)

Water Temperature:_____°C (E) Water Density at (E) (See Below):_____g/cc (F)

Wt. Water Only:_____g (G) = D-B

Volume Water:_____cc (H) = G/F

Volume Pyc. at (E) from Calibration:_____cc (I)

Volume Sample:_____cc (J) = I-H

Sample Grain Density:_____g/cc (K) = C/J

Absolute Density of Water

(From Lange's Handbook of Chemistry, edited by J. Dean, 11th Edition, Sect. 10-127)

Temp °C	Density	Temp °C	Density	Temp °C	Density	Temp °C	Density
18.0	0.998595	20.8	0.998035	23.6	0.997394	26.4	0.996676
18.2	0.998553	21.0	0.997992	23.8	0.997345	26.6	0.996621
18.4	0.998520	21.2	0.997948	24.0	0.997296	26.8	0.996567
18.6	0.998482	21.4	0.997904	24.2	0.997246	27.0	0.996512
18.8	0.998444	21.6	0.997850	24.4	0.997196	27.2	0.996457
19.0	0.998405	21.8	0.997815	24.6	0.997146	27.4	0.996401
19.2	0.998365	22.0	0.997770	24.8	0.997095	27.6	0.996345
19.4	0.998325	22.2	0.997724	25.0	0.997044	27.8	0.996289
19.6	0.998285	22.4	0.997678	25.2	0.996992	28.0	0.996232
19.8	0.998244	22.6	0.997632	25.4	0.996941	28.2	0.996175
20.0	0.998203	22.8	0.997585	25.6	0.996888	28.4	0.996118
20.2	0.998162	23.0	0.997535	25.8	0.996835	28.6	0.996060
20.4	0.998120	23.2	0.997490	26.0	0.996783	28.8	0.996002
20.6	0.998078	23.4	0.997442	26.2	0.996729	29.0	0.995944

Operator: Print/Sign:_____Date/Time:_____

Appendix II
Gas Pycnometry Procedure

PROCEDURE TO DETERMINE GRAIN DENSITIES WITH A GAS PYCNOMETER

All pertinent data must be recorded on the " Grain Volume by Gas Pycnometer" data sheet.

1. Record the volumes of the sample chamber volume and expansion volume from the latest calibration onto the data sheet.

2. Weigh the empty sample cup to ± 0.001g.

3. Fill the sample cup to a minimum of three quarters full with a sample that has been prepared to the prescribed grain size and dryness.

4. Weigh the sample cup with sample to ± 0.001g, and calculate the weight of the sample only.

5. Make certain the Prep/Test valve is in the Prep position.

6. Place the sample cup with sample into the sample chamber. The vent valve should be open for this step. It is important that the sample be transferred to the sample chamber as quickly as possible after being removed from its dry atmosphere to minimize rehydration of the sample.

7. Turn the flow rate controllers on the vent and fill lines clockwise until the flow of gas would be at a minimum. This will ensure that the sample does not fluidize when the valves are opened. The rates can be higher for empty chamber and calibration volume runs. Note: For this study, samples were both purged prior to recording data, and were run without purging. When running calibration checks with and without the sample volumes, the system was always purged with the gas prior to recording data. Therefore, follow Step 8 if purging is called for, or skip to Step 9 if purging is not to be carried out.

8. Close the vent valve. Open the fill valve, allowing gas regulated to 22 ± 1 psig to enter the sample chamber. Allow the pressure in the chamber to rise to 18.5 ± 1 psig. Increase the flow rate slightly (1 to 2 turns) as the pressure rises, and its rate of increase slows (typically at approximately 10 psig). Close the fill valve and open the vent valve. Increase the flow rate progressively as the pressure drops. Allow the pressure to drop to 0 to 1 psig. Repeat this step for the prescribed number of purging cycles, always being cautious to open the fill and vent valves only after lowering the flow rates to the minimum. Proceed to Step 9.

9. With the pressure in the sample chamber near zero, and the vent valve open, open the flow rate valve on the vent line fully so that the pressure will more easily reach ambient (0.000 psig). Turn the Prep/Test valve from Prep to Test repeatedly until the pressure is

stable. With the valve in the Test position, adjust the pressure indicator to read 0.000 psig. Turn the valve to the Prep position and make certain the indicated pressure remains 0.000 psig. If it does not, then continue to alternate between Prep and Test until it does.

10. When the indicated pressure is stable at 0.000 psig in both the Prep and Test positions place the Prep/Test valve into the Prep position. Make certain the flow rate valves are turned down to a minimum flow rate.

11. Open the fill valve, allowing gas regulated to 22 ± 1 psig to enter the sample chamber. Allow the pressure in the chamber to rise to 19.500 ± 0.200 psig. Increase the flow rate slightly (1 to 2 turns) as the pressure rises, and its rate of increase slows (typically at approximately 10 psig). Close the fill valve (this will have to be done prior to reaching 19.500 psig because there is a slight increase in pressure of typically 0.750 to 1.500 psig, depending on sample volume, after the valve is closed).

12. Allow the pressure to stabilize (Note: Typically less then 15 seconds, but may need to use some judgement where more difficult, particularly zeolitic, samples are concerned), and record this volume as P_1 on the data sheet.

13. Immediately turn the Prep/Test valve to the Test position.

14. Allow the pressure to stabilize at the lower pressure (see note for Step 12), and record this value as P_2 on the data sheet.

15. Open the vent valve, and increase the venting flow rate slowly (2-3 turns initially). Increase the flow rate to maximum as the pressure approaches 0.000 psig.

16. Calculate the sample volume per the formula on the data sheet.

17. Repeat steps 9-15 until the calculated sample volumes converge on a stable value, or for a prescribed number of cycles (both methods were utilized in this study, however volume stability became the preferable condition to determine when the values were most accurate for this procedure).

18. Choose the calculated volumes from the later cycles that are within a small range of values (typically less than ± 0.015 cc, but will vary somewhat with sample behavior), and average them to arrive at the sample volume.

19. Divide the dry sample weight by the sample volume to determine its grain density.

Bulk Properties
Yucca Mountain Project
GRAIN VOLUME by GAS PYCNOMETER
per TP-_____

SAMPLE ID:_____

Pycnometer Make/Model: Micromeritics/Multivolume Pycnometer 1305
Serial No.: 429 Measurement gas: Helium
Date of latest calibration:_____Due date:_____

Operator (Print/Sign):_____Date/Time:_____

Gross Weight:_____g **Full Scale Range:_____cc**
Cup Weight:_____g **Number of Purging Cycles:_____**
Net Sample Weight:_____g

V_{cell}:_____cc **V_{exp}:_____cc**

$$V_{sample} = V_{cell} - V_{exp} / [(P_1/P_2) - 1]$$

	P_1(psi)	P_2(psi)	V_{sample}(cc)
1.	19.		
2.	19.		
3.	19.		
4.	19.		
5.	19.		
6.	19.		
7.	19.		
8.	19.		
9.	19.		
10.	19.		

V_{sample} Sum: _____cc

V_{sample} Average: _____cc

Appendix III

Information from the Reference Information Base Used in this Report

This report contains no information from the Reference Information Base.

Candidate Information for the Reference Information Base

This report contains no candidate information for the Reference Information Base.

Candidate Information for the Geographic Nodal Information Study and Evaluation System

This report contains no candidate information for the Geographic Nodal Information Study and Evaluation System.

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