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Grain Density Measurements of Ash Flow Tuffs: An Experimental Comparison of Water Immersion and Gas Intrusion Pycnometer Techniques

Barry M. Schwartz

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GRAIN DENSITY MEASUREMENTS OF ASH FLOW TUFFS: AN EXPERIMENTAL COMPARISON OF WATER IMMERSION AND GAS INTRUSION PYCNOMETER TECHNIQUES

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ABSTRACT

The Nevada Nuclear Waste Storage Investigations (NNWSI) Project is currently investigating tuff formations at the Nevada Test Site for feasibility as a repository site. Grain density measurements are routinely made on tuff samples obtained from cored exploratory holes and are used in conjunction with mineralogical data, downhole density logs, and lithologies to define a thermal/mechanical stratigraphy. Grain densities are used directly in the calculation of porosity and in the interpretation of the variation seen in thermal and mechanical properties. Standardized grain density procedures such as ANSI/ASTM or API do not address the problems of testing hygroscopic minerals such as clays and zeolites that commonly occur in silicic tuffs. This report compares two widely used techniques for measuring grain density: water immersion and gas intrusion. It also describes sample-handling and operating procedures necessary for repeatable grain density measurements of zeolitized and clay-bearing tuffaceous rocks. Laboratory tests included in this report show the importance of careful sample-handling on the acquisition of accurate and repeatable data. Without consistent thermal pretreatment of hygroscopic tuff samples, grain densities determined by either method can vary by as much as 10 percent due to the loss or gain of adsorbed water. Repeatable data are obtained only when pretest sample-handling procedures are both defined and rigorously followed. These data indicate that both techniques are probably sufficiently accurate and precise for most project needs. However, water pycnometer data have a higher level of precision for both zeolitized and non-zeolitized tuff samples than do gas pycnometer data.

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INTRODUCTION

The work described in this report was performed in support of the characterization of potential repository sites being performed by the Nevada Nuclear Waste Storage Investigations Project (NNWSI).

The purpose of the report is to describe and compare two widely used testing methods that determine grain densities of geologic materials. These are the water immersion and gas intrusion methods.

Grain density data are used in conjunction with mineralogy data to help define and model thermal conductivity behavior of tuffs in the correlation of mechanical properties with porosity and mineralogy, downhole density logs, and other property measurements. The results are used to help define a breakdown of the rocks into units according to their physical properties.

The porosity of tuff is usually inversely proportional to the degree of welding; i.e., the greater the degree of welding, the lower the porosity. In addition, many nonwelded tuffs contain over 50 volume percent zeolites, which, because of their hygroscopic nature, make repeatable physical property measurements, including grain density, difficult.

Grain density test procedures for geologic materials utilizing water immersion and gas intrusion methods can be found in the American Petroleum Institute Procedures,[1] and the American Society for Testing and Materials.[2] However, these procedures do not address testing of hygroscopic

materials. B. V. Zaleski[3] discusses the importance of drying geologic specimens to 110°C prior to water immersion grain density testing but does not discuss gas intrusion techniques. D. D. Dickey and E. F. Monk[4] discuss the effect that the degree of saturation of zeolitized tuffs has on grain density values using the water immersion technique; but grain density results utilizing gas intrusion are not addressed.

This report attempts to fill a gap in referenceable standardized procedures by describing sample-handling and operating procedures that will yield repeatable grain density measurements of zeolitized and clay-bearing tuffaceous rocks. The accuracies and precision of data that can be expected from water immersion and gas intrusion pycnometer techniques are also discussed.

SAMPLE DESCRIPTION

Sample numbers correspond to a depth in feet from drill hole USW G-1 as determined by the United States Geological Survey (USGS).[5] A USGS stratigraphic and lithologic log of drill hole USW G-1 is provided in Table 1. Bulk x-ray diffraction analysis is provided in Table 2.[6]

The three samples studied are the following:

- Depth interval USW G-1 2192.9 feet (Sample A) is a nonwelded tuff containing 50 to 70 percent of the zeolite clinoptilolite. There is 5 to 10 percent smectite clay content in the sample.

- Depth interval USW G-1 2246.1 feet (Sample B) is a nonwelded to partially welded tuff containing 20 to 40 percent of the zeolite mordenite.

- Depth interval USW G-1 2485.0 feet (Sample C) is a moderately to densely welded nonzeolitized tuff containing 60 to 70 percent feldspar and 25 to 35 percent quartz, with only trace smectite clay content.

Samples A and B were selected because they are both heavily zeolitized and representative of samples that readily absorb water, and they each contain a different type of zeolite. Sample C was selected because of its lack of zeolitization.

EXPERIMENTAL PROCEDURE

All samples were pulverized; in each case the entire sample was then passed through a 100-mesh sieve. Each sample was then divided into four equal parts using the coning and quartering method. One part from each sample was reserved for water pycnometer tests with the remaining three parts used for gas pycnometer tests. Approximately 15-g samples were used for each water pycnometer test and 45-g samples for each gas pycnometer test.

One-half of the powders designated for each test method were heated in air at 110°C for 16 hr with the other half heated at 205°C for 16 hr. A temperature of 110°C was chosen because it is a standard temperature used in the drying of geologic materials.[2] A bakeout temperature of 205°C was chosen on the basis of published data on zeolites that concluded that 85 percent of the water present in the zeolites was removed at 200°C.[7] Temperatures substantially greater than 200°C were not used because of possible changes in the skeletal framework of the zeolites.

All of the samples were placed in a vacuum chamber immediately after their respective heating regimes. The samples were not allowed to cool prior to placement into the vacuum chamber. The samples were actively vacuum-pumped at 13.3kPa (100 millitorr) for 30 min to remove any water that may have been absorbed during the removal of the powders from the drying ovens. The powders were stored under passive vacuum conditions for up to several days before testing. The passive vacuum was achieved by closing the main valve between the vacuum pump and the vacuum chamber. A periodic check of the vacuum gage

indicated there was no appreciable leakage. Passive vacuum conditions were used to maintain sample dryness. This method was chosen instead of using a commercial desiccant at ambient pressure because the hygroscopic nature of the powdered tuff would compete with the desiccant for moisture.

Powders tested within 2 min of removal from vacuum storage are referred to throughout this report as "dry." Samples exposed to atmospheric conditions for between 15 and 17 min prior to testing are referred to as "exposed to air." The typical environmental conditions during exposure were 24°C, 833 mbar barometric pressure, and 35 percent relative humidity. Grain density measurements on all samples were run in triplicate; i.e., three different tests using a previously untested sample were run with tests performed on "dry" powders that were heated to 110° or 205°C.

One series of tests was performed on samples "exposed to air" for approximately 15 min after being heated to 110°C. Another series of tests was performed on samples "exposed to air" for approximately 15 min after being heated to 205°C. This aspect of testing addressed the possible effect of rehydration of the hygroscopic minerals in the tuffs that can occur during sample preparation and testing. If the relative humidity is substantially higher than 35 percent, the effects of rehydration will probably be more pronounced than what occurred under the conditions in which this study was performed.

APPARATUS

Gas Intrusion Method

The gas pycnometer used was a Micromeritics Model 1303 (Figure 1). Operating procedures and data sheet are found in Appendix I. The unit consists of a sample-holding chamber capable of holding up to 40 cm^3; a cylinder fitted with a movable piston, the relative position of which is indicated on the front panel dial to five significant figures (in units of cm^3); a four-position valve; and a pressure detector.

The gas pycnometer works on the following principle: The volume of the empty sample chamber is measured by careful metering of the quantity of a nonadsorbing gas (helium) necessary to fill the chamber to a pressure level preset at the factory. Initially, the sample chamber is flushed with air and filled with powdered sample, then the sample chamber is evacuated slowly so that the powdered sample does not fluidize. The sample chamber is again filled with helium to the same preset pressure level. The decrease in the volume of helium required to fill the chamber is equal to the volume of the powdered sample in the chamber, called the grain volume. The grain density (g/cm^3) is obtained by dividing the grain weight by the grain volume. The grain weight was measured on an analytical balance.

The test is run by first weighing a dry, empty gas pycnometer sample cup and then placing the empty sample cup into the sample chamber to determine the volume of helium required to fill the sample chamber to a selected level. The powdered sample was then placed into the sample cup and weighed. The sample

chamber was then placed into the sample chamber and slowly evacuated using a vacuum pump to remove air and other contaminants. The sample chamber is again flooded with helium and the volume of helium necessary to fill the sample chamber to the same selected level is determined. The grain volume of the powder is determined by the difference of the volumes of helium gas necessary to fill the empty and partially filled sample cup.

Water Immersion Method

Operating procedures and data sheet can be found in Appendix II. The water pycnometer apparatus was assembled at Sandia National Laboratories (SNL). The system consists primarily of 100-ml glass volumetric flasks calibrated to ± 0.10 ml, a calibrated glass thermometer, double-distilled water, an evacuation chamber, and a mechanical vacuum pump. The principle of operation is that a grain volume of powder is determined from the weight of distilled water (of known density) displaced in the pycnometer by the powdered sample. The grain density (g/cm^3) is obtained by dividing the sample weight by the grain volume. The grain weight is measured on an analytical balance.

The test is run by first weighing a dry 100-ml pycnometer. The powdered sample is then placed into the pycnometer and the pycnometer reweighed. Then 40 ml of deaerated distilled water is poured into the pycnometer that is then placed in a vacuum chamber and evacuated and swirled to remove trapped air from the powder-water slurry. The contents of the pycnometer are returned to ambient pressure and temperature. The pycnometer is then filled with previously deaerated, distilled water to the scribe line and the pycnometer

reweighed. The temperature of the pycnometer contents is measured using the calibrated thermometer. The weight of the water is divided by the density of water at the measured temperature, which yields the volume of water in the pycnometer. The volume of the empty pycnometer minus the volume of water equals the grain volume of the powdered sample.

CALIBRATION TECHNIQUES

Helium Pycnometer

Before each sample was tested, a calibration run was made using steel balls of known volume. If the correct volume was measured to within ±1.5 percent, the conditions were acceptable to continue and a measurement on the tuff powder was executed. Periodically, an additional calibration check was run using 50-mesh α-quartz powder. This calibration also provided a check on procedures involving powders. The theoretical density of quartz is 2.647 g/cm^3.[2] Testing proceeded only if the measured α-quartz density was within ±1.5 percent of the theoretical value. Balance calibrations were performed in accordance with SNL Calibration Requirements.

The α-quartz was chosen as a standard calibration material because its theoretical density closely matches the average grain density for welded devitrified tuffs (2.61 g/cm^3). The α-quartz also has good chemical and physical stability in water and air and is readily available in pure form.

Due to the anhydrous nature of the α-quartz powder, any inaccuracies in the calibration runs are probably due to operator and/or instrument error, not sample-handling procedures. Therefore, the data become valuable as a check on the optimum accuracy and precision of the system, excluding sample-handling problems due to heating and exposure to the atmosphere.

Water Pycnometer

A calibrated volume for each pycnometer was determined prior to usage, employing the procedures in Appendix II. The procedure entails cleaning and drying a 100-ml volumetric flask (pycnometer), then weighing the empty pycnometer. The pycnometer is then filled with distilled water to the scribe line and the pycnometer is reweighed. The temperature of the water is then measured using a calibrated thermometer. The weight of the water divided by the density of the water at the measured temperature is equal to the volume of the pycnometer when filled to the scribe line. The pycnometer is then dried and the calibrated volume obtained is used as the true volume of the pycnometer during the next run. Balance calibrations were performed in accordance with SNL Calibration Requirements.

The water immersion method was also calibrated with the α-quartz powder. Testing proceeded if the α-quartz grain density value was within ±1.5 percent of the theoretical value.

RESULTS

Calibration Runs

Calibration run data using α-quartz powder as a reference material are shown in Table 3. Ten tests were run for each of the two test methods. The water pycnometer results show a mean grain density of 2.643 g/cm^3 with a standard deviation of 0.005 g/cm^3. The helium pycnometer results show a mean grain density value of 2.663 g/cm^3 with a standard deviation of 0.014 g/cm^3 (Table 3). Therefore, the accuracy of the water pycnometer results is higher than the helium pycnometer results, with mean errors of 0.15 and 0.60 percent, respectively.

The precision of the water pycnometer results is also greater than the helium pycnometer results with standard deviations of 0.005 and 0.014 g/cm^3, respectively.

It should be noted that all 10 helium pycnometer calibration densities were greater than or equal to the value assumed for α-quartz. However, the mean error was 0.6 percent, an excellent level of conformity in physical property testing.

Test Results

Test results and statistics are shown in Tables 4 and 5 and in Figures 2, 3, and 4.

Sample A (Zeolitized with Clinoptilolite)

For Sample A powders heated to 110°C and tested in a "dry" condition, the gas and water pycnometer mean grain density values were 2.390 g/cm^3 and 2.397 g/cm^3, respectively. Samples heated to 205°C and tested in a "dry" condition had gas and water pycnometer grain density values of 2.500 g/cm^3 and 2.447 g/cm^3, respectively.

For samples heated to 110°C and exposed to air, the gas and water pycnometer mean grain density values were 2.380 g/cm^3 and 2.403 g/cm^3, respectively. Samples heated to 205°C and exposed to air had gas and water pycnometer mean grain density values of 2.430 g/cm^3 and 2.463 g/cm^3, respectively.

The mean grain density values for samples heated to 205°C and tested in a "dry" condition were greater than those of samples heated to 110°C and tested in a "dry" condition for both the helium and water pycnometer methods. This same trend is found for the samples exposed to air.

The standard deviations of the helium pycnometer grain density values were greater than those of the water pycnometer in three out of four cases. The exception was in the 110°C test, where in the exposed-to-air samples, the standard deviation for the helium pycnometer was 0.010 g/cm^3, compared to a standard deviation of 0.015 g/cm^3 for the water pycnometer test. However, a difference of 0.005 g/cm^3 in the standard deviation is probably insignificant.

Sample B (Zeolitized with mordenite)

For Sample B powders heated to 110°C and tested in a "dry" condition, the helium and water pycnometer mean grain density values were 2.300 g/cm^3 and 2.327 g/cm^3, respectively. Samples heated to 205°C and tested in a "dry" condition had helium and water pycnometer mean grain values of 2.440 g/cm^3 and 2.397 g/cm^3, respectively.

For samples heated to 110°C and exposed to air, the helium and water pycnometer mean grain density values were 2.280 g/cm^3 and 2.347 g/cm^3, respectively. Samples heated to 205°C and exposed to air had helium and water pycnometer mean grain density values of 2.473 g/cm^3 and 2.373 g/cm^3, respectively.

As observed in Sample A, the mean grain density values for samples heated to 205°C and tested in a "dry" condition were greater than those of samples heated to 110°C and tested in a "dry" condition for both the helium and water pycnometer methods. This same trend is found in the samples exposed to air.

The standard deviations of the helium pycnometer grain density values were greater than those of the water pycnometer in three out of four cases. The exception to this sample is the same as that found in Sample A in that the standard deviation for the 110°C, exposed-to-air, helium pycnometer test was 0.010 g/cm^3, compared to a standard deviation of 0.021 g/cm^3 for the 110°C, exposed-to-air, water pycnometer test.

Sample C (Nonzeolitized)

Helium and water pycnometer mean grain density values were 2.630 g/cm^3 and 2.580 g/cm^3, respectively, for samples heated to 110°C and tested in a "dry" condition. Samples heated to 205°C and tested in a "dry" condition had helium and water pycnometer mean grain density values of 2.643 g/cm^3 and 2.600 g/cm^3, respectively.

For samples heated to 110°C and exposed to air, the helium and water pycnometer mean grain density values were 2.653 g/cm^3 and 2.617 g/cm^3, respectively. Samples heated to 205°C and exposed to air had helium and water pycnometer mean grain density values of 2.697 g/cm^3 and 2.590 g/cm^3, respectively.

The mean grain density values for samples heated to 205°C and tested in a "dry" condition were slightly greater than those of the samples heated to 110°C and tested in a "dry" condition for both the helium and water pycnometer tests. The increase, however, was the smallest of the three samples because Sample C has a lower affinity for water than the other two samples and is, therefore, less sensitive to changes in sample heating and drying regimes.

It should be noted that for Sample C, the mean grain density values for the helium pycnometer were greater than those of the water pycnometer for each temperature and drying regime tested.

The standard deviations of both the helium and water pycnometer grain density values are lower in Sample C than in Samples A and B. This is due to the decreased sensitivity of Sample C to variations in temperature and drying regimes due to the lack of zeolitization and clays in this sample.

Although the standard deviations are low compared to Samples A and B, the standard deviations of the helium pycnometer grain density values in Sample C are greater than those of the water pycnometer for each temperature and drying regime tested.

DISCUSSION

These data show that grain density values of rocks containing hygroscopic materials, such as zeolites and expandable clays, are dependent on drying temperatures and exposure times to even relatively dry air and, hence, to the level of sample hydration.

For all samples tested in a "dry" condition, the grain density values for samples heated to 205°C were greater than for samples heated to 110°C. This holds true for both the water immersion and gas intrusion techniques because more water has been removed from the rock after heating to 205°C than after heating to 110°C, thereby increasing the grain density. The effect of water removal on grain density values is reduced in Sample C, which, due to its nonzeolitic and nonclay-bearing makeup, showed the smallest increase in grain density when heated to 205°C compared to 110°C. This holds true for samples tested in a "dry" condition for both the water immersion and gas intrusion methods.

The results also indicate that the dissimilarity in measurement technique between the water and helium pycnometer tests can result in differences in grain density values even when sample preparation is well controlled.

The helium pycnometer employs a vacuum pumpdown that removes some adsorbed water bound to the powdered sample while the water pycnometer does not. This difference becomes more significant when testing hygroscopic materials such as those containing zeolites and expandable clays.

The effects of temperature changes during testing can have greater ramifications on the volume of a gas such as helium than on changing the volume of a liquid. The gas pycnometer owner's manual warns against contact with the instrument because body heat can affect the volume of the testing system.[8]

A major source of error found in the water immersion technique is the possibility of incomplete removal of trapped air from the pycnometer. Incomplete removal of trapped air will yield falsely high grain volumes and low grain densities. The meniscus is marked on the glass pycnometer at a point where the inside diameter is 3/8 in. The reading of the meniscus becomes more difficult with the increased turbidity and the effect on surface tension (between the glass and the water) that occur when testing zeolitic and clay-bearing samples.

As with any complex material, the absolute grain density of tuffaceous rock samples cannot be determined. However, a method which can be verified to have the highest accuracy using calibration standards and to have the greatest precision through repetitive testing is more desirable. Calibration data of the α-quartz (Table 3) show that the accuracy and precision of the water pycnometer are higher than those of the helium pycnometer. Data from the tuffaceous samples tested show that the precision of the water pycnometer was greater than that of the helium pycnometer in 10 out of 12 cases.

The water immersion method is a faster technique than the gas intrusion method because multiple samples can be run, side by side, in one batch. Gas

intrusion tests must be run to completion before the next sample is loaded. The calibration of the gas intrusion instrumentation is more time-consuming and more difficult than that of the water immersion apparatus.

CONCLUSION

The accuracy and precision of both water immersion and gas intrusion pycnometry are sufficient to meet most laboratory requirements. The accuracy and precision of the water immersion method are superior to those of the gas intrusion method when a known standard is analyzed. The precision of the data can be seen from an analysis of the standard deviations of tests run in triplicate of the zeolitized and nonzeolitized tuffaceous samples. The data show that the mean standard deviation was 0.013 for the water pycnometer compared to 0.025 for the helium pycnometer. In using either method, it is imperative that the level of hydration remain consistent from sample to sample. Without consistent thermal pretreatment of hygroscopic tuff samples, grain densities determined by either method can vary by as much as 10 percent due to the loss or gain of adsorbed water. In addition, the test procedures used should be documented for meaningful correlation with other properties.

Therefore, the water pycnometer technique is judged to be more suitable for both zeolitized and nonzeolitized tuffaceous samples than is the helium pycnometer technique because it requires less time to run and produces more precise data.

TABLE 1

Lithologic Log of Samples Tested From Hole USW G-1 in the Bullfrog Member of the Crater Flat Tuff[5]

Stratigraphic and Lithologic Description	Depth Interval (ft)
Tuff, ash-flow, grayish-orange-pink, light brown, and grayish-orange, nonwelded, devitrified and argillic; pumice, grayish-orange-pink, grayish-yellow, and light brown, devitrified and argillic, 2-30 mm; 10-15 percent quartz, sanidine, plagioclase, hornblende, and biotite phenocrysts; sparse dark gray and brownish-gray volcanic lithic fragments (base marked by 7 cm of reworked tuff)	2,179.0-2,209.5 (Sample 2192)
Tuff, ash-flow, light brown and grayish-orange, nonwelded to partially welded, devitrified; pumice, grayish-orange-pink, light brown, grayish-orange devitrified and vapor phase crystallization; 15-20 percent phenocrysts (quartz, sanidine, plagioclase, hornblende, biotite); rare dark gray and brownish-gray volcanic lithic fragments, commonly less than 5 mm, as large as 3 cm; slightly argillic from 2,209.5 to 2,227.0 ft and 2,306.7 to 2,307.0 ft; partially silicified from 2,244.4 to 2,258 ft (gradational); base of unit dips 35° relative to core axis	2,209.6-2,317.4 (Sample 2246)
Tuff, ash-flow, light brown to moderate-brown, moderately to densely welded, devitrified; pumice, pale yellowish-brown to pale brown, devitrified, size ranges from 2 to 30 mm, commonly 1-3 cm; 10-15 percent phenocrysts (quartz, sanidine, plagioclase, hornblende, biotite); sparse pale brown volcanic lithic fragments and moderate reddish-brown mudstone lithic fragments	2,467.0-2,547.1 (Sample 2485)

TABLE 2

Bulk X-Ray Diffraction Analysis of Samples Tested, Hole USW G-1[6]

Sample Number (Equivalent to Depth in ft)	Clinoptilolite-Heulandite	Mordenite	Smectite	Mica/Illite	Quartz	Feldspar	Cristobalite
2192.9	50-70	---	5-10	0-2	0-5	10-20	10-20
2246.1	---	20-40	---	0-5	0-5	40-60	5-15
2485.0	---	---	trace?	0-5	25-35	60-70	0-5

TABLE 3

Measured Grain Densities for α Quartz

Test No.	Gas Pycnometer Results (g/cm^3)	Water Pycnometer Results (g/cm^3)
1	2.668	2.652
2	2.663	2.641
3	2.655	2.638
4	2.688	2.642
5	2.649	2.643
6	2.657	2.636
7	2.686	2.643
8	2.665	2.651
9	2.647	2.644
10	2.656	2.642
Mean Value	2.663 g/cm^3	2.643 g/cm^3
Mean Error From Accepted Value	(+) 0.016 g/cm^3	(-) 0.004 g/cm^3
Standard Deviation	0.014 g/cm^3	0.005 g/cm^3
Relative Mean Error Percent	0.604	0.151

Accepted density of quartz is 2.647 g/cm^3 (see Reference 2).

TABLE 4

Helium Pycnometer Grain Density Results

Sample ID	Pretest Sample Treatment	Grain Density Results (g/cm^3) in Triplicate			$\bar{x}$ (g/cm^3)	Standard Deviation (g/cm^3)
		Test # 1	Test # 2	Test # 3		
A (2192')	110°C-Dry	2.37	2.37	2.43	2.390	0.035
	205°C-Dry	2.46	2.53	2.51	2.500	0.036
	110°C-Exposed to air	2.39	2.38	2.37	2.380	0.010
	205°C-Exposed to air	2.40	2.49	2.45	2.447	0.026
B (2246')	110°C-Dry	2.26	2.31	2.33	2.300	0.036
	205°C-Dry	2.46	2.44	2.42	2.440	0.020
	110°C-Exposed to air	2.28	2.27	2.29	2.280	0.010
	205°C-Exposed to air	2.45	2.43	2.54	2.473	0.059
C (2485')	110°C-Dry	2.66	2.61	2.62	2.630	0.026
	205°C-Dry	2.66	2.63	2.64	2.643	0.015
	110°C-Exposed to air	2.67	2.63	2.66	2.653	0.021
	205°C-Exposed to air	2.71	2.70	2.68	2.697	0.015

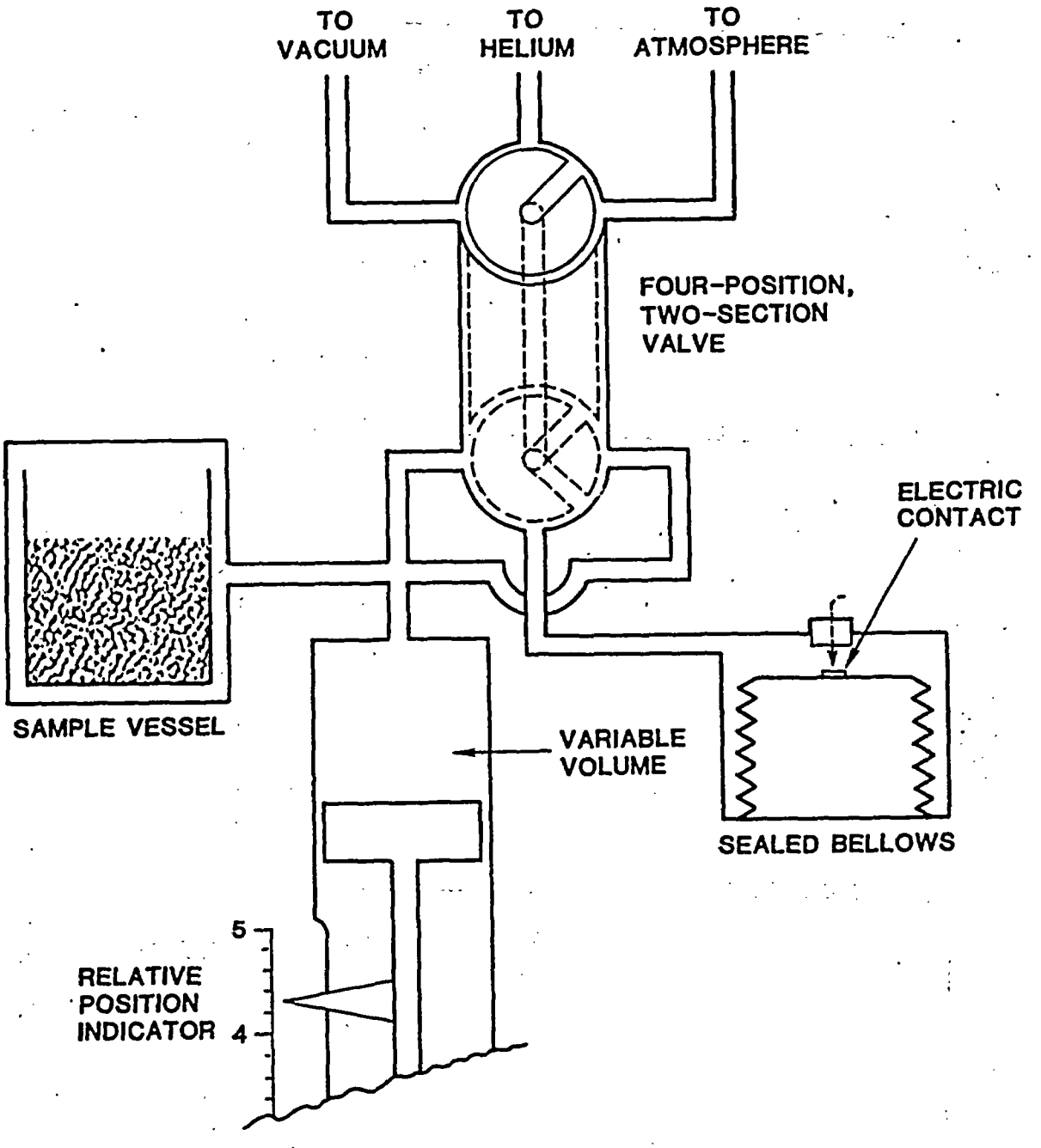
Note: "exposed to air" means exposed to ambient conditions for 15 to 17 min.

TABLE 5

Water Pycnometer Grain Density Results

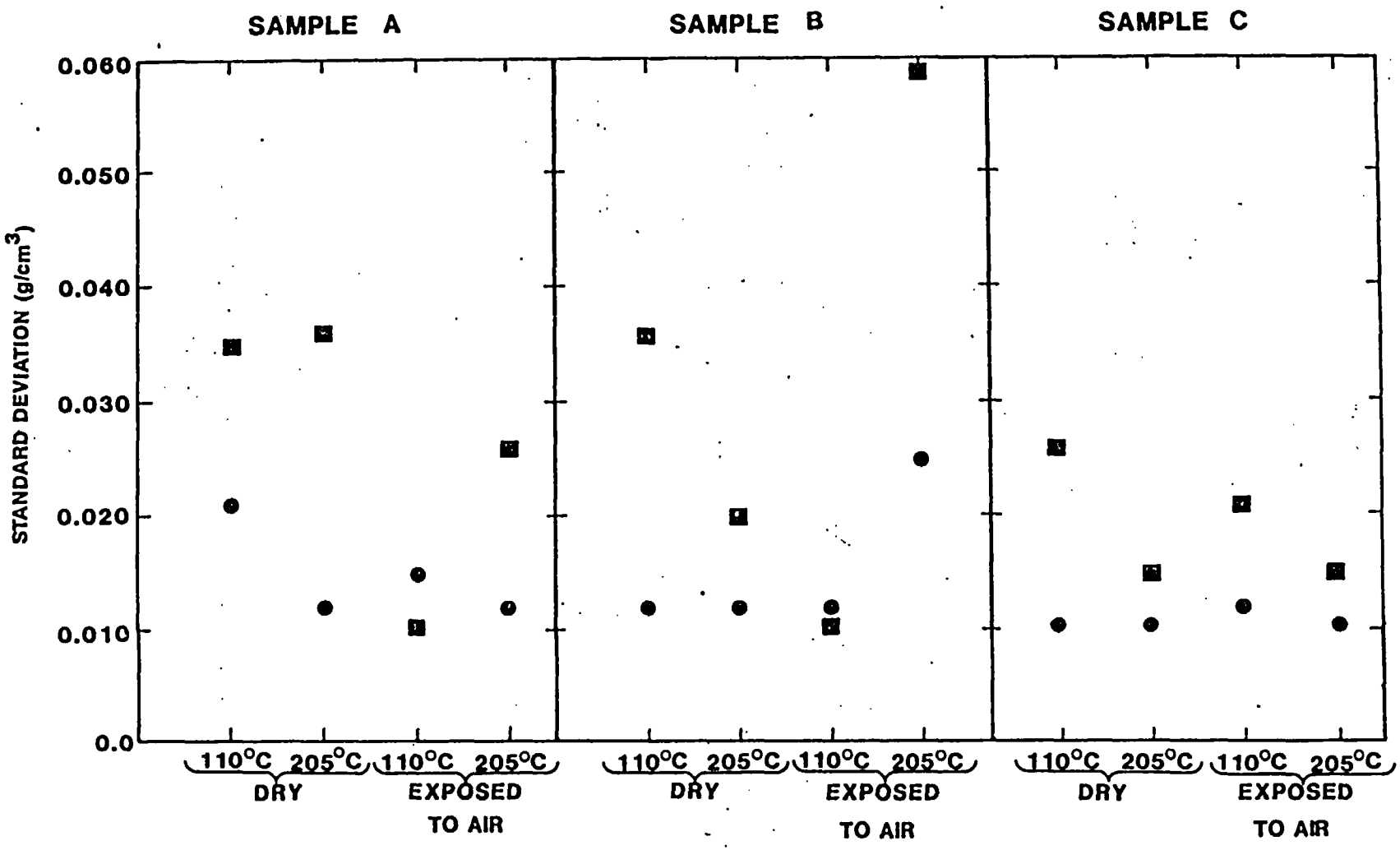
Sample ID	Pretest Sample Treatment	Grain Density Results (g/cm^3) in Triplicate			$\bar{x}$ (g/cm^3)	Standard Deviation (g/cm^3)
		Test # 1	Test # 2	Test # 3		
A (2192')	110°C-Dry	2.42	2.39	2.38	2.397	0.021
	205°C-Dry	2.46	2.44	2.44	2.447	0.012
	110°C-Exposed to air	2.40	2.42	2.39	2.403	0.015
	205°C-Exposed to air	2.45	2.47	2.47	2.463	0.012
B (2246')	110°C-Dry	2.32	2.32	2.34	2.327	0.012
	205°C-Dry	2.39	2.39	2.41	2.397	0.012
	110°C-Exposed to air	2.37	2.34	2.33	2.347	0.012
	205°C-Exposed to air	2.35	2.32	2.30	2.373	0.025
C (2485')	110°C-Dry	2.57	2.59	2.58	2.580	0.010
	205°C-Dry	2.60	2.61	2.59	2.600	0.010
	110°C-Exposed to air	2.61	2.63	2.61	2.617	0.012
	205°C-Exposed to air	2.60	2.59	2.58	2.590	0.010

Note: "exposed to air" means exposed to ambient conditions for 15 to 17 min.



Schematic of Micromeritics Model 1303 Gas Pycnometer

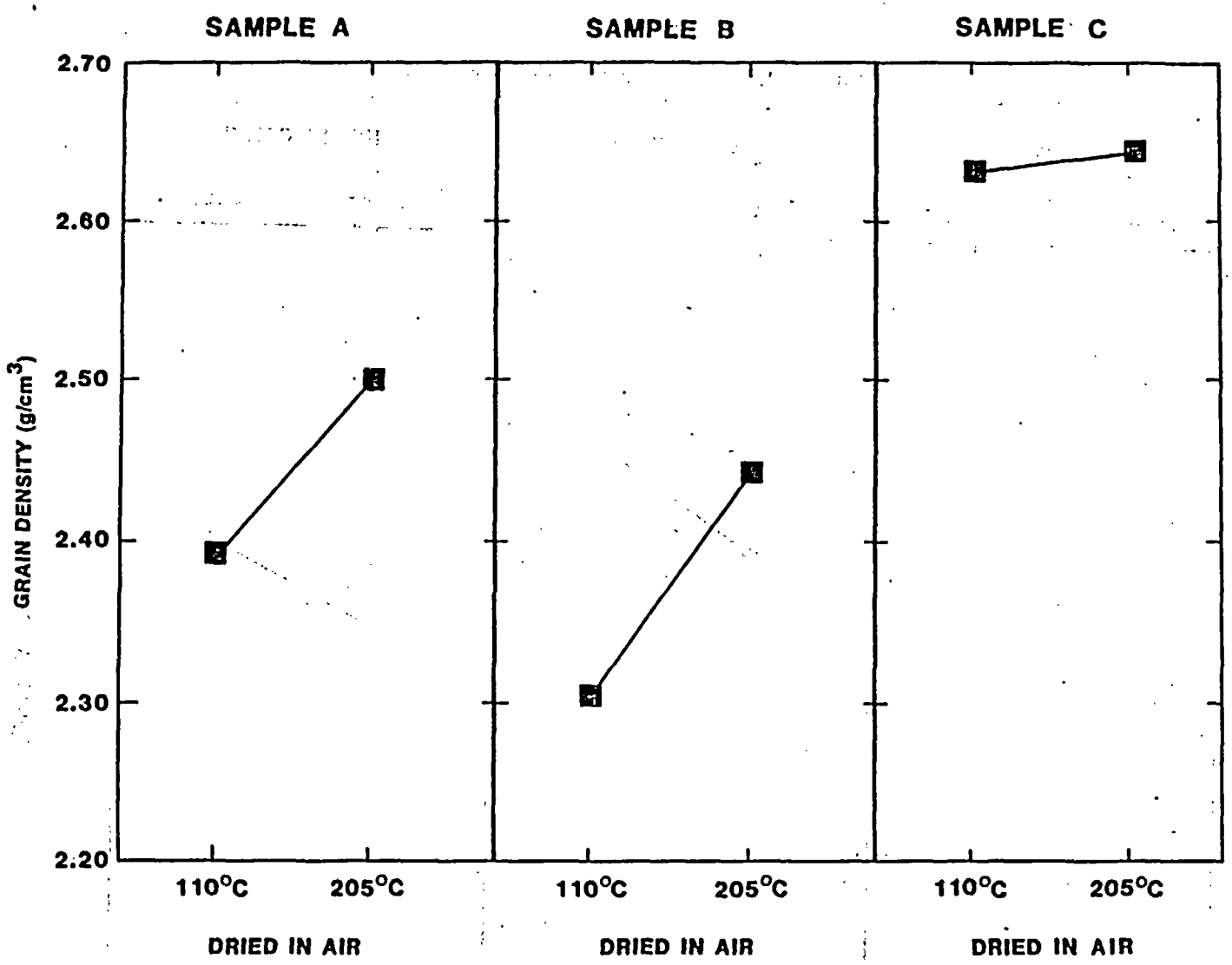
Figure 1



● WATER PYCNOMETER
■ HELIUM PYCNOMETER

Standard Deviations of Grain Density Test Data

Figure 2



Helium Pycnometer Grain Density Values

Figure 3

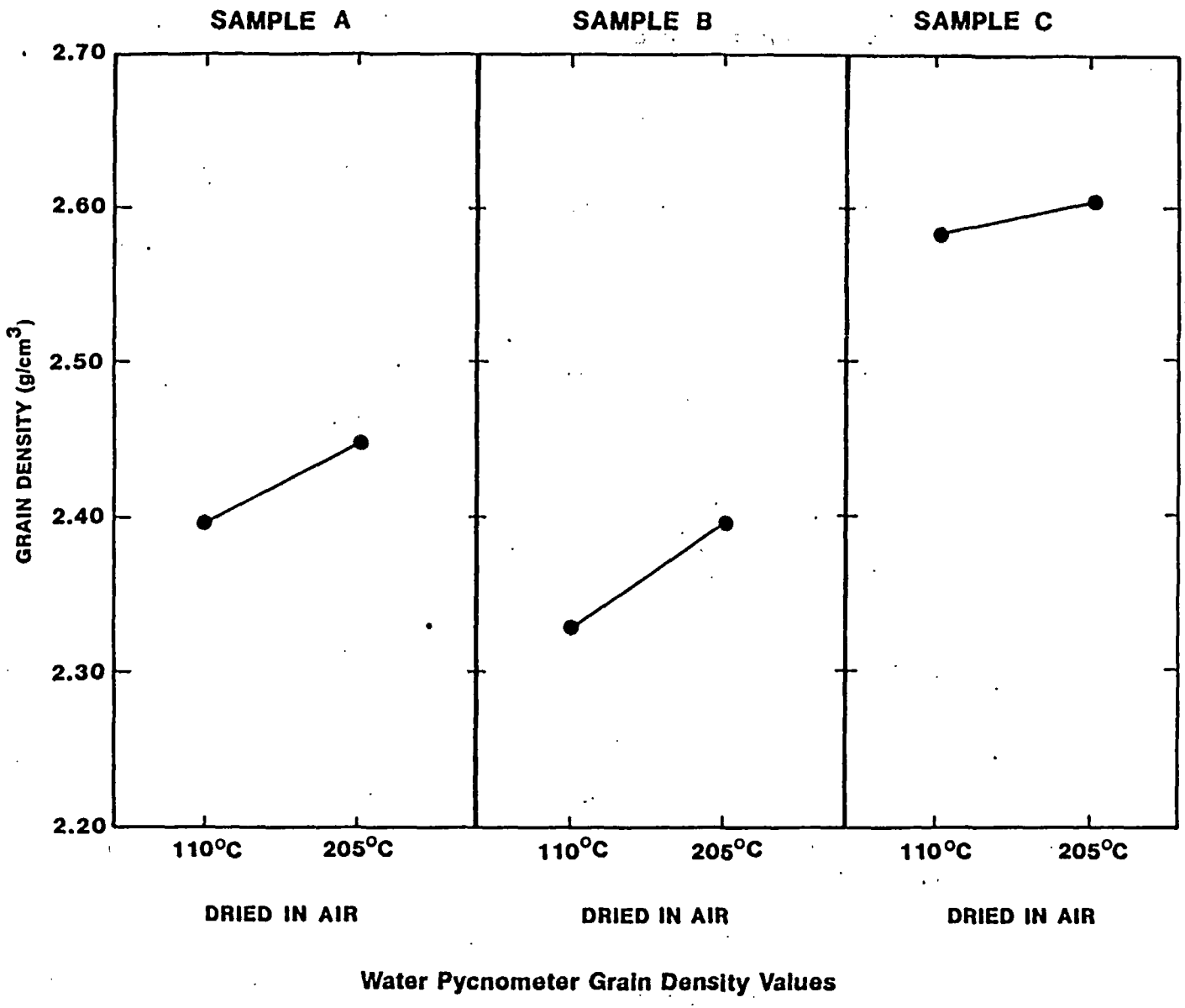

SAMPLE A
SAMPLE B
SAMPLE C
GRAIN DENSITY (g/cm3)
2.70
2.60
2.50
2.40
2.30
2.20
110°C
205°C
110°C
205°C
110°C
205°C
DRIED IN AIR
DRIED IN AIR
DRIED IN AIR
Water Pycnometer Grain Density Values

Figure 4

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Appendix I

This appendix contains an NNWSI data sheet and standard operating procedures for helium pycnometer grain density measurements.[9]

Appendix I

NNWSI Grain Density Data Sheet Using a Helium Pycnometer

Sample ID	_____	Hole ID	_____	Depth Interval (ft)	_____
Date	_____	Time	_____	Operator	_____
Temperature	_____	Barometric Pressure	_____	Relative Humidity	_____
Comments					

1. Posttest Sample Cup and Sample Weight (W) _____ grams
2. Pretest Sample Cup Weight (W_c) _____ grams
3. Posttest Sample Weight (W_e) (line 1 minus line 2) _____ grams

Nomenclature:

- a Calibration constant
- V_{LB1} Known volume of large steel ball used as standard (28.96 cm^3)
- V_{SB1} Known volume of small steel ball used as standard (16.76 cm^3)
- R_e Instrument readout when measuring volume of empty sample cup
- R_{LB1} Instrument readout when measuring volume of large steel ball
- R_{SB1} Instrument readout when measuring volume of small steel ball
- R_x Instrument readout when measuring volume of test sample
- V_e Absolute volume of test sample (cm^3)
- W_e Posttest sample weight (gram)

The order of the tests is as follows:

1. Determine the volume of the empty sample cup
2. Determine the volume of the large steel ball
3. Determine the volume of the small steel ball
4. Determine the volume of the test sample

$a = V_{LB1}/R_e$ = _____ cm^3

$V_{SB1} = a(R_e - R_{SB1})$ = _____ cm^3 (Note 1)

$V_e = a(R_e - R_x)$ = _____ cm^3

Grain Density = W_e/V_e = _____ $gram/cm^3$ (Note 2)

Quality Control [1]For ±1.5% accuracy V_{SB1} limits are 16.51 cm^3 - 17.01 cm^3
[2]For ±1.5% accuracy the quartz powder grain density limits are 2.61 gm/cm^3 - 2.69 gm/cm^3

Standard Operating Procedure for NNWSI Helium Pycnometer Grain Density Measurements

1. Turn the valve on the pycnometer to the intermediate OFF position between the gage and vacuum positions.
2. Place the sample of interest into the 40-cm^3 polyethyulene cup using clean, protective gloves and then lower the cup into the chamber.
3. Screw the black cap on the chamber until it is tight and the white dots align.
4. Check that helium is being supplied to the pycnometer at a pressure between 5 and 8 psig and that the helium bleed valve is closed (clockwise).
5. Close vacuum valve #2 (clockwise). Close vacuum valve #1 (handle pushed in).
6. Turn the vacuum system ON. When the reading on the vacuum gauge is 50 mtorr, open vacuum valve #1 (handle pushed in).
7. Turn the handle on the front of the pycnometer 10 digits below zero and then return it to exactly 0000.
8. Turn the valve on the pycnometer counterclockwise to the VACUUM position.
9. Very slowly open vacuum valve #2 (counterclockwise) watching the vacuum gage, making sure there is no sudden increase in gauge pressure. This precaution is to assure that the powders do not fluidize. Maintain vacuum pumping until the vacuum gage reads between 5 and 10 mtorr.

NOTE: If it is apparent that the powder fluidized at any time during the run, abort the run and disregard the results.

10. Turn the valve on the pycnometer counterclockwise to the Helium position for 15 s.

11. Close vacuum valve #2 (clockwise).

12. Turn the valve on the pycnometer clockwise to the vacuum position.

13. Very slowly open vacuum valve #2 (counterclockwise), watching the vacuum gage, making sure there is no sudden increase in gage pressure. This precaution is to ensure that the powders do not fluidize. Maintain vacuum pumping for 5 min after the vacuum gage reads 10 mtorr.

14. Turn the valve on the pycnometer counterclockwise to the Helium position for 15 s.

15. Turn the valve on the pycnometer counterclockwise to the AIR position for 20 s.

16. Turn the valve on the pycnometer counterclockwise to the gage position.

17. Close vacuum valve #2 (clockwise).

18. Turn the handle on the pycnometer to its upper limit (clockwise) to force gas into the sample, then back (counterclockwise) to the point where the light just comes on again.

19. Wait 90 s after the pycnometer light originally went off.

20. Determine R (dial setting) at this point, turning the dial until the light just goes OFF.

21. Turn the handle on the pycnometer 10 digits below 0000 and then return it to exactly 0000.

22. Turn the valve on the pycnometer counterclockwise to the intermediate OFF position between gage and vacuum.

23. The black cap on the pycnometer can be removed.

- END OF PROCEDURE-

Appendix II

This appendix contains an NNWSI data sheet and standard operating procedures for water pycnometer grain density measurements.[10]

NNWSI
GRAIN DENSITY MEASUREMENTS DATA SHEET
USING A WATER PYCNOMETER

Sample ID _____ Hole ID _____ Depth Interval (ft) _____

Date _____ Time _____ Operator _____

Length of Excavation _____ Drying Temp/Time _____

Flask # _____ Balance Used _____

Comments _____

1.	Weight of Pycnometer	_____	g
2.	Weight of Pycnometer plus Powder	_____	g
3.	Line #2 minus Line #1 (Grain Weight of Powder)	_____	g
4.	Temperature of Water	_____	°C
5.	Theoretical Density of Water	_____	g/cm^3
6.	Calibrated Volume of 100-ml Pycnometer	_____	ml
7.	Line #5 x Line #6 (Theoretical Weight of Water in the Calibrated Pycnometer)	_____	g
8.	Weight of Pycnometer and Powder Filled with Water to the Calibrated Fill Line	_____	g
9.	Line #2 plus Line #7	_____	g
10.	Line #9 minus Line #8 (Weight of Displaced Water)	_____	g
11.	Line #10 divided by Line #5 (Grain Volume of Powder)	_____	cm^3
12.	Line #3 divided by Line #11 = (Grain Density of Powder)	_____	g/cm^3

The operating procedure descriptions are keyed to the line numbers that appear in the Grain Density Data Sheet

Line 1 Weigh a calibrated, numbered 100-ml pycnometer, including the stopper, to the nearest 0.01 g, making sure that the pycnometer is dry and free of contamination.

Line 2 Transfer between 15 and 20 g of the powder into a preweighed and numbered 100-ml pycnometer. Weigh the pycnometer.

Dry the pycnometer and contents at 110°C for at least 16 hr at ambient pressure. The stopper should not be fitted to the pycnometer at this time.

Remove the pycnometer from the oven and evacuate between 50 and 100 mtorr vacuum until the powder is at ambient temperature. Do not allow the powder to fluidize because of excessive vacuum pumping rates.

Reduce the vacuum to ambient pressure. Fit the stopper on the pycnometer as soon as possible.

Weigh the pycnometer and stopper loaded with powder to the nearest 0.01 g.

Line 3 The grain weight of the powder is determined by subtracting the weight of the pycnometer from the weight of the pycnometer loaded with the dry powder.

Line 4 Add deaerated distilled water to a level 1/4 in. to 1/2 in. above the powder line.

Swirl the pycnometer gently to mix the sample and the water.

Place the contents of the pycnometers under vacuum and pump on the samples gently, making sure not to boil the water.

Swirl the bottle every 15 min for 2 hr to help release trapped air.

Continue vacuum pumping for a total pumpdown time of 24 hr.

Remove the pycnometer from under vacuum and cover the pycnometer with the numbered stopper.

Allow contents to warm to ambient temperature (at least 2 hr), since the temperature of the solution has been cooled because of the evaporation of water during the vacuum pumpdown.

Using a separatory funnel, fill the pycnometer to just below the calibrated scribe mark with deaerated, distilled water. The final addition of water that fills the pycnometer to the scribe mark should be made using a dropper bottle. The scribe mark should be at eye level when the final height of the meniscus is established. The lower part of the meniscus should be equal in height to the calibrated scribe mark. Step #4 should be performed within 1 hr of the completion of Step #3. Remove any water from the inside neck of the pycnometer above the meniscus, using a cotton bud. Make sure the outside of the pycnometer is dry and free of contamination. Weigh the pycnometer to the nearest 0.01 g.

Line 5 Record the posttest temperature of the water in the pycnometer to the nearest 0.5°C, using a calibrated thermometer.

Line 6 The density of the water is determined using four significant figures.

Line 7 The theoretical volume of the pycnometer.

Line 8 The theoretical weight of the water when the pycnometer is filled to the calibrated scribe mark.

-END OF PROCEDURE-

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