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Yucca Mountain Site Characterization Project

**Calculation of Experiment Uncertainty
in Laboratory Determination of Several
Geoengineering Properties of Tuffs
From Yucca Mountain, Nevada**

F. B. Nimick, B. M. Schwartz, R. H. Price

Prepared by

Sandia National Laboratories

Albuquerque, New Mexico 87185 and Livermore, California 94550

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Calculation of Experiment Uncertainty
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of Tuffs From Yucca Mountain, Nevada

F. B. Nimick, B. M. Schwartz, and R. H. Price
Geotechnical Projects Division
Sandia National Laboratories
Albuquerque, New Mexico

ABSTRACT

A method for estimating the precision and accuracy of measured parameters is described. Examples of application of the estimating method are presented for density, porosity, compressive strength, Young's modulus, Poisson's ratio, and thermal expansion. With the exception of the coefficient of linear thermal expansion, all laboratory data for these properties for tuff samples appear to have reasonable experiment uncertainties. Uncertainties in the thermal-expansion coefficients may be as high as 39% of the values, although most uncertainties for the coefficients are probably $\leq 2\%$.

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1.0 INTRODUCTION

Yucca Mountain, located in and near the southwest corner of the Nevada Test Site (NTS) in southern Nye County, Nevada, has been identified by the U.S. Department of Energy (DOE) as a potential site for the underground disposal of radioactive waste. Responsibility for studying the suitability of Yucca Mountain as a disposal site rests with the Yucca Mountain Project (YMP), administered by the DOE offices in Las Vegas, Nevada. Sandia National Laboratories (SNL) is one of the primary YMP participants and has responsibilities for performance assessment, repository design, and the measurement of the thermal and mechanical properties of some of the tuff units at Yucca Mountain.

Preliminary data for the thermal and mechanical properties of the tuff units have permitted definition of a thermal/mechanical stratigraphy, in which units are distinguishable based on differences in one or more of the thermal and mechanical properties. A comparison of these units with the formal stratigraphic units for Yucca Mountain tuffs is provided in Figure 1.

The reason for obtaining data for the thermal and mechanical properties is to provide input for calculations that contribute to repository design and performance assessment. Implicit in the calculations is that the uncertainty in the results is dependent on the uncertainty associated with the data used as input to the calculations. Thus, quantifying the uncertainty associated with the input data is important, and reducing the uncertainty is a desirable goal.

The purposes of this report are to demonstrate a method of estimating uncertainty and to use the method to estimate the uncertainty associated with the existing laboratory determinations of several thermal and mechanical properties of samples of tuff from Yucca Mountain. Section 2 provides a general discussion of uncertainty, including accuracy and precision, and outlines the method used to obtain quantitative estimates of accuracy, precision, and uncertainty from experiment data. The remaining

Note 1: The figure shows the correlation between the thermal/mechanical stratigraphy and the geologic stratigraphy.

Note 2: The thicknesses of the units are averages for five deep core holes at Yucca Mountain.

Figure 1. Stratigraphy of Tuffaceous Units at Yucca Mountain (Source: Nimick, 1989)

sections of the paper then provide descriptions of the application of the method to existing data for the specific parameters listed below:

- saturated bulk density,
- natural-state bulk density,
- dry bulk density,
- grain density,
- matrix porosity,
- compressive strength,
- Young's modulus,
- Poisson's ratio,
- coefficient of thermal expansion, and
- thermal conductivity.

With two exceptions, each description includes a numerical example of application of the method using data for Unit TSw2, and, for each parameter, the applicability to other units is discussed. The two exceptions are grain density measured with a helium pycnometer, and thermal conductivity. For both of these parameters, information for the accuracy and precision of measured quantities is lacking, so that estimates of parameter accuracy and precision cannot be made.

2.0 DEFINITIONS AND DESCRIPTION OF METHOD

2.1 Accuracy and Precision

The uncertainty of determinations of properties is best specified using two quantities: precision and accuracy (also known as bias) (Eisenhart, 1963). Accuracy is defined as the deviation of a determination of a property from the "true" value for that property. In theory, the accuracy is unknowable because the "true" value cannot be determined. However, for the purposes of this work, the common practice of using the mean of measured values of a property as a reliable estimate of the "true" value has been followed.

Accuracy can be evaluated relatively easily for properties that are obtained from nondestructive tests on homogeneous materials. In such cases, equating the "true" value with the mean of many measured values is straightforward and reasonable. For inhomogeneous materials (e.g., rocks) replicate nondestructive tests on an individual sample can give a reliable estimate of accuracy for that sample, but the estimate may or may not apply to other samples. For destructive tests on inhomogeneous materials, experimental determination of accuracy is not likely to be feasible.

In addition to accuracy, precision must be determined. Precision is the reproducibility of successive determinations of a property. Eisenhart (1963) suggests that the calculated value of either the standard deviation or of the variance is a useful index of the precision of a series of measurements. The standard deviation as calculated for $(n-1)$ degrees of freedom (where n is the number of measurements or samples) is used for precisions discussed in this report.

The definition of precision as the reproducibility of replicate determinations of a property is not quite complete. The replicate measurements might be made successively by a single operator in a short span of time using exactly the same equipment under the same environmental conditions. In contrast, replications might be performed by different people with slightly different equipment over extended periods of time. In

presenting an analysis of precision, such modifiers as "single-operator" or "multilaboratory" are useful additions to the description.

The uncertainty in a measured parameter (e.g., where does the real value of the parameter occur relative to the measured value?) is a combination of accuracy and precision. The method of combination is discussed in the following section.

2.2 Uncertainty Estimated from Measured Parameters

An alternative to using standard reference materials to determine experiment uncertainty is to combine information on the precision (S) and accuracy (A) of measured parameters (e.g., mass, volume) to estimate values of S, A, and uncertainty (U) for the property of interest. The procedure for doing this is outlined in Abernethy et al. (1985) and is summarized below.

If an expression for a property, R, is a function of m measured parameters, P_i ($i=1,2,3,\dots,m$),

$$R = f(P_1, P_2, P_3, \dots, P_m) \quad , \quad (1)$$

then the sensitivity factors θ_i are given by

$$\theta_i = \frac{\partial R}{\partial P_i} \quad . \quad (2)$$

These factors are then used to estimate the precision and accuracy of R according to

$$S_R = \left[\sum_{i=1}^m \left(\theta_i S_{P_i} \right)^2 \right]^{1/2} \quad , \quad (3)$$

and

$$A_R = \left[\sum_{i=1}^m \left(\theta_i A_{P_i} \right)^2 \right]^{1/2} \quad . \quad (4)$$

The total uncertainty (U_R) for the mean value for R can be expressed as

$$U_R = A_R + t_{\alpha/2, n-1} S_R \quad (5)$$

where U_R is the half-width of a band in which the mean value of R is expected to occur with 99% confidence and $t_{\alpha/2, n-1}$ is the $(1 - \alpha/2)$ quantile from a Student's t distribution with $(n-1)$ degrees of freedom. For convenience, we have assumed 10 measurements for each P_i , so that the Student's t value in Equation 5 is $t_{0.995, 9} = 3.2498$.

Three points should be kept in mind when reading about the specific parameters. First, the data for these parameters often were obtained without collecting ancillary data necessary to estimate accuracy or precision. Thus, in some cases, the data necessary to estimate experiment uncertainty are not available. Second, the values of accuracy, precision, and total uncertainty discussed here do not include potential errors resulting from violations of the assumptions for a given method (e.g., that a sample used for saturated bulk density was not completely saturated before the density was measured). Instead, this paper focuses on experiment uncertainty resulting from the inherent inability to obtain perfectly accurate and precise values of measured parameters (e.g., voltage, time, length). Finally, the values estimated for accuracy and precision using the method outlined above should be the maximum values resulting from inaccuracy or imprecision in the measured parameters (assuming that all potential sources of error have been included). The assumption that the calculated values will be maximums is based on the assumed independence of the error sources. Actual values of accuracy and precision, as determined by replicate measurements of a thermal or mechanical property, should be less than the values estimated by this method.

Despite the fact that accuracy and precision for a given property often can be determined directly by measurements on materials with known properties, the estimation method is valuable for two reasons. First, if a constraint is placed on the allowable uncertainty for a given property before testing, the method can be used to estimate required accuracies and

precisions for each measured parameter. For example, if a coefficient of thermal expansion must be known to $\pm 5\%$, a displacement transducer that measures length changes with an accuracy of $\pm 7\%$ of the expected change would be unacceptable. For this case, allowable errors must be allocated between measurements of length, displacement, and temperature so that the combined error is $\leq 5\%$ of the calculated value for the coefficient of thermal expansion.

The second reason that the estimation method is useful is that some properties are determined by destructive testing (e.g., compressive or tensile strength testing). Obviously, replicate tests on a single sample are not possible for such tests, so precision is difficult to evaluate. In addition, a "true" value of strength would be very difficult to define, making a direct assessment of experiment accuracy nearly impossible. However, both precision and accuracy can be estimated for such destructive tests using the method described in this report.

3.0 APPLICATION TO GEOENGINEERING PROPERTIES

In this section, the method described in Section 2 is used to calculate the precision and accuracy of several geoengineering properties of tuff. Whenever possible the applications use existing data obtained during tests on tuff samples from Yucca Mountain.

3.1 Saturated Bulk Density

3.1.1 Density Using Small, Irregular Samples

Saturated bulk density (ρ_{sb}) is calculated from

$$\rho_{sb} = \frac{M_{s,air}}{V} \quad , \quad (6)$$

where $M_{s,air}$ is the mass of the saturated sample as determined in air, and V is the volume of the sample. Because of the usually irregular shape of the sample, V is obtained by determining the mass of the sample when submerged ($M_{s,water}$), subtracting this value from $M_{s,air}$, and dividing the result by the density of water at the test temperature, T ($\rho_{w,T}$). $M_{s,water}$ is obtained by subtracting the mass of the suspension device, usually a wire basket (M_{wb}), from the combined mass of the sample and device (M_{wb+s}). The resulting expression is

$$V = \frac{M_{s,air} - (M_{wb+s} - M_{wb})}{\rho_{w,T}} \quad . \quad (7)$$

Combining Equations 6 and 7, taking partial derivatives, and applying Equations 3 and 4 with the data listed in Table 1, we obtain $S_{\rho_{sb}} = 0.004$ g/cm³ and $A_{\rho_{sb}} = 0.003$ g/cm³. Combining these values according to Equation 5, the uncertainty in saturated bulk density is estimated to be 0.016 g/cm³.

For this example, calculations are based on data from a single sample of Unit TSw2, for which $\rho_{sb} = 2.243$ g/cm³. Thus, the "true" value for this sample is expected to lie within the band 2.243 ± 0.016 g/cm³ with 99% confidence.

TABLE 1

DATA FOR MEASURED PARAMETERS--SATURATED BULK DENSITY

<u>Property</u>	<u>Units</u>	<u>Precision</u>	<u>Accuracy</u>
Mass of Sample in Air	g	0.015 ^a	0.007 ^b
Mass of Sample Plus Basket	g	0.007 ^c	0.004 ^d
Mass of Basket	g	0.007 ^e	0.007 ^f
Density of Water	g/cm ³	2.3 x 10 ^{-5g}	2.3 x 10 ^{-4g}

a. Determined by three replicate determinations of the mass of Sample GU3-760.9/4A-B, including removing from water and blotting with moist cloth before each determination.

b. Determined by comparing the mean value of three replicate determinations of the mass of a 20-g standard, traceable to National Institute of Standards and Technology (NIST), to "true" mass.

c. Determined by 10 replicate determinations of mass of Sample GU3-760.9/4A-B suspended in wire basket in container of distilled water.

d. Taken as average of accuracies determined by three determinations (each) of the masses of 10-g and 20-g standards traceable to NIST. Accuracy is 0.000 g for 10-g standard and 0.007 g for 20-g standard.

e. Determined by 10 replicate determinations of mass of wire basket suspended in container of distilled water.

f. Determined by comparing the mean value of three replicate determinations of mass of 5-g standard, traceable to NIST, to "true" mass.

g. Calculated using a linear equation relating the density of water to temperature and assuming temperature is measured with a precision of 0.1°C and an accuracy of 1°C. Compositional corrections are unnecessary because distilled water was used for all measurements.

The values of precision, accuracy, and uncertainty estimated in this example are assumed to be representative for all of Unit TSw2. For the other thermal/mechanical units, samples with the same volumes should have better values (i.e., precisions, accuracies, and uncertainties that are of smaller absolute magnitude) because the mass of a saturated sample is highest for Unit TSw2. This conclusion assumes that the precisions and accuracies for samples from units other than Unit TSw2 are the same as

those listed in Table 1. Data are not presently available to test this assumption.

3.1.2 Density Using Large, Regularly Shaped Samples

For samples with a right-circular cylindrical shape, Equation 7 is replaced by

$$V = \pi r^2 h , \quad (8)$$

for determining sample volume, where h and r are the height and radius, respectively. The relevant values of precision and accuracy of h and r are given in Table 2. Taking partial derivatives of Equation 8 and applying Equations 3 and 4, we obtain the values of S_V , A_V , and U_V listed in Table 3.

Repeating the process for Equation 6 and using the data for sample mass listed in Table 4, values of $S_{\rho_{sb}}$ and $A_{\rho_{sb}}$ are obtained, as listed in Table 5. Table 5 also contains values of $U_{\rho_{sb}}$ as calculated from the data for $S_{\rho_{sb}}$ and $A_{\rho_{sb}}$.

All of the values in Tables 2, 3, 4, and 5 pertain to samples of Unit TSw2 except for those for the largest sample, which is from Unit TSw1. Using the same reasoning presented in the last paragraph of Section 3.1.1, the values of $U_{\rho_{sb}}$ given in Table 5 for all but the largest sample should be conservative estimates (in the sense that $U_{\rho_{sb}}$ values should be equal to or less than the values in the tables) for all other thermal/mechanical units. For samples as large as the largest one listed in Table 5, $U_{\rho_{sb}}$ for Units TCw and TSw2 could be slightly larger than the value in Table 4; no data are available with which to calculate actual values of $U_{\rho_{sb}}$ for these two units.

TABLE 2

PRECISIONS AND ACCURACIES FOR DIMENSIONS
OF CYLINDRICAL SAMPLES

<u>Sample Size (cm)</u>		<u>Precision^a (cm)</u>		<u>Accuracy^b (cm)</u>	
<u>Height</u>	<u>Radius</u>	<u>Height</u>	<u>Radius</u>	<u>Height</u>	<u>Radius</u>
5.08	1.27	0.0025	0.0025	0.0127	0.0064
10.16	2.54	0.0025	0.0025	0.0127	0.0064
16.51	4.13	0.0025	0.0025	0.0254	0.0064
25.40	6.35	0.0025	0.0025	0.0254	0.0064
45.72	11.43	0.0025	0.0025	0.0254	0.0064
53.34	13.33	0.0025	0.0025	0.1270	0.0127

a. Assumed values.

b. Taken to be equivalent to the tolerances required by the machining process.

TABLE 3

PRECISION, ACCURACY, AND UNCERTAINTY
FOR VOLUME OF CYLINDRICAL SAMPLES

<u>Sample Size (cm)</u>		<u>Volume (cm³)</u>			
<u>Height</u>	<u>Radius</u>	<u>Value</u>	<u>Precision</u>	<u>Accuracy</u>	<u>Uncertainty</u>
5.08	1.27	25.74	0.102	0.267	0.598
10.16	2.54	205.93	0.409	1.069	2.398
16.51	4.13	884.70	1.079	3.061	6.568
25.40	6.35	3217.59	2.553	7.240	15.537
45.72	11.43	18765.00	8.273	23.458	50.344
53.34	13.33	29798.12	11.260	90.858	127.451

TABLE 4

DATA FOR MASSES OF CYLINDRICAL SAMPLES

<u>Sample ID</u>	<u>Mass (g)</u>		
	<u>Value</u>	<u>Precision^a</u>	<u>Accuracy</u>
BB 12A-1	58.73	0.020 ^b	0.010 ^c
28-2-A	481.4	0.005 ^d	0.010 ^e
BB 11D-1	2116.6	22.7 ^d	45.4 ^f
BB 10A-1	7598.3	249	1919
#22-2	44910.2	64 ^h	1224 ⁱ
BB 1B-1	59045.1	91 ^j	1588 ^k

- a. All data for precision taken on dry masses. Precision of values from saturated, surface-dry rock samples will be somewhat worse than precision of values for dry masses.
- b. Determined by three replicate determinations of mass of 50-g standard traceable to NIST.
- c. Determined by comparison of mean value of three replicate determinations of mass of 50-g standard, traceable to NIST, to "true" mass.
- d. Assumed to be 50% of accuracy value.
- e. Assumed to be the same as the value obtained for 50-g standard; three replicate determinations of mass of 500-g standard gave accuracy and precision of 0.000.
- f. Obtained by comparing single determinations of mass of 5-lb (2,268-g) standard, traceable to NIST, to "true" mass.
- g. Obtained by interpolation for next higher and lower values.
- h. Two replicate determinations of mass of 100-lb (45,359-g) standard traceable to NIST.
- i. Determined by comparing mean value of two replicate determinations of mass of 100-lb (45,359-g) standard, traceable to NIST, to "true" mass.
- j. Assumed value.
- k. Obtained by comparing single determination of mass of 115-lb (52,163-g) standard, traceable to NIST, to "true" mass.

TABLE 5

PRECISION, ACCURACY, AND UNCERTAINTY FOR SATURATED BULK
DENSITY OF CYLINDRICAL SAMPLES

Sample ID	Size (cm)		Saturated Bulk Density (g/cm ³)			
	Height	Radius	Value	Precision	Accuracy	Uncertainty
BB 12A-1	5.08	1.27	2.282	0.002	0.010	0.016
28-2-A	10.16	2.53	2.356	0.005	0.012	0.028
BB 11D-1	16.51	4.13	2.392	0.026	0.052	0.136
BB 10A-1	25.40	6.35	2.361	0.008	0.060	0.086
#22-2	45.72	11.43	2.393	0.004	0.065	0.078
BB 1B-1	53.34	13.33	1.982	0.003	0.054	0.064

3.2 Natural-State Bulk Density

Natural-state bulk densities (ρ_{nsb}) were determined only for the small samples discussed in Section 3.1.1. These densities are calculated from

$$\rho_{nsb} = \frac{M_{ns,air}}{V} \quad , \quad (9)$$

where $M_{ns,air}$ is the mass of the natural-state sample (i.e., the sample with a saturation state that is assumed to have remained unchanged since the sample was removed from the ground) as determined in air. Because of the usually irregular shape of the sample and the need to cover the sample with beeswax to preserve the saturation state, V is determined using the following equation:

$$V = \frac{M_{ws,air} - (M_{wb+ws} - M_{wb})}{\rho_{w,T}} - \frac{M_{ws,air} - M_{ns,air}}{\rho_{wax}} \quad , \quad (10)$$

where $M_{ws,air}$ is the mass of the waxed sample as determined in air, $(M_{wb+ws} - M_{wb})$ is the quantity equivalent to $(M_{wb+s} - M_{wb})$ in Equation 7, and ρ_{wax} is the density of the beeswax. Unfortunately, no data were recorded for masses including the wire basket; thus, precision and accuracy

values have been estimated directly for the mass of the waxed sample submerged in water.

The values of precision and accuracy listed in Table 6 apply for the measured parameters. Taking partial derivatives of Equation 10 and applying Equations 3 and 4, we obtain $S_V = 0.159 \text{ cm}^3$ and $A_V = 0.450 \text{ cm}^3$ for a sample from Unit TSw2 (GU3-753.0) having a volume of 65.20 cm^3 .

TABLE 6
PRECISIONS AND ACCURACIES FOR
SAMPLE MASS AND DENSITIES OF WATER AND WAX

<u>Property</u>	<u>Units</u>	<u>Precision</u>	<u>Accuracy</u>
Natural-State Mass in Air	g	0.015 ^a	0.003 ^a
Mass of Waxed Sample in Air	g	0.015 ^a	0.003 ^a
Mass of Waxed Sample in Water	g	0.008 ^a	0.003 ^a
Density of Water	g/cm ³	2.3×10^{-5a}	2.3×10^{-4a}
Density of Wax	g/cm ³	0.0088 ^b	0.025 ^c

a. See footnotes for Table 1.

b. Determined by replicate measurements during wax-density calibrations.

c. Assumed; the true value of wax density is unknown.

Repeating the process for Equation 9, values are obtained for $S_{\rho_{nsb}}$ and $A_{\rho_{nsb}}$ of 0.006 g/cm^3 and 0.016 g/cm^3 , respectively. Combining these values in Equation 5 yields a value for $U_{\rho_{nsb}}$ of 0.035 g/cm^3 , relative to a natural-state bulk density of 2.321 g/cm^3 .

As was true for saturated bulk density, the volumes of samples from all thermal/mechanical units are roughly equivalent. Assuming similar precisions and accuracies for $M_{ns,air}$ and V for all units, we conclude that the values of $S_{\rho_{nsb}}$, $A_{\rho_{nsb}}$, and $U_{\rho_{nsb}}$ given in the preceding paragraph all should be conservative values, in the sense that correlative values for thermal/mechanical units other than TSw2 would be less than or equal to the values stated above.

3.3 Dry Bulk Density

Dry bulk density (ρ_{db}) is calculated from

$$\rho_{db} = \frac{M_{d,air}}{V} , \quad (11)$$

where $M_{d,air}$ is the mass of the dry sample as determined in air, and V is the volume determined during saturated bulk density measurements (Section 3.1.1). For the same small, irregular sample discussed in Section 3.1.1 (GU3-760.9/4A-B), the precision and accuracy of $M_{d,air}$ are 0.015 and 0.007 g respectively, relative to $M_{d,air} = 19.271$ g. Using the same values of S_V and A_V given in Section 3.1.1, we obtain $S_{\rho_{db}} = 0.006$ g/cm³ and $A_{\rho_{db}} = 0.003$ g/cm³. Combination of these values yields $U_{\rho_{db}} = 0.022$ g/cm³, relative to a dry bulk density of approximately 2.36 g/cm³. As with the saturated and natural-state bulk densities, the uncertainty value estimated for Unit TSw2 should be a good estimate of an upper bound on uncertainties for thermal/mechanical units other than Unit TSw2.

3.4 Grain Density

Grain densities were determined using two different methods. The methods are discussed separately in the following sections.

3.4.1 Water-Immersion Pycnometer

Grain density (ρ_g) is calculated from

$$\rho_g = \frac{M_g}{V_g} , \quad (12)$$

where M_g and V_g are the mass and volume of a powdered sample, respectively. The mass is obtained by the difference in masses of an empty, 100-ml pycnometer (M_p) and of the pycnometer with the sample inside (M_{p+g}):

$$M_g = M_{p+g} - M_p . \quad (13)$$

The volume of the sample is obtained by calculating the volume of water displaced by the sample:

$$V_g = V_p - V_w , \quad (14)$$

where V_p is the calibrated volume of the 100-ml pycnometer, and V_w is the volume of water that can be added to the pycnometer containing the powdered sample to bring the water level to the 100-ml scribe line. This water volume is obtained from

$$V_w = \frac{M_w}{\rho_{w,T}} = \frac{M_{p+g+w} - M_{p+g}}{\rho_{w,T}} , \quad (15)$$

where M_{p+g+w} is the mass of the pycnometer containing the sample and filled with water to the scribe line. The calibrated volume of the pycnometer is obtained from

$$V_p = \frac{M_{p+w} - M_p}{\rho_{w,T}} , \quad (16)$$

where M_{p+w} is the mass of the pycnometer filled with water to the scribe line. Combining Equations 14, 15, and 16, we get

$$V_g = \frac{M_{p+w} - M_p - M_{p+g+w} + M_{p+g}}{\rho_{w,T}} . \quad (17)$$

The values of precision and accuracy listed in Table 7 apply for the measured parameters. Taking partial derivatives of Equation 17 and applying Equations 3 and 4, we get $S_V = 0.004 \text{ cm}^3$ and $A_V = 0.037 \text{ cm}^3$, relative to a volume of 9.86 cm^3 . The correlative values for M_g are $S_{M_g} = 0.002 \text{ g}$ and $A_{M_g} = 0.017 \text{ g}$, relative to a grain mass of 24.794 g . Combining this information for grain density, we get $S_{\rho_g} = 0.001 \text{ g/cm}^3$ and $A_{\rho_g} = 0.010 \text{ g/cm}^3$, relative to a measured value of 2.515 g/cm^3 . The calculated

TABLE 7

PRECISIONS AND ACCURACIES USED IN CALCULATIONS OF GRAIN DENSITY^a

Property	Units	Value ^a	Precision	Accuracy
Mass of pycnometer + 100 ml H ₂ O	g	168.982	0.002 ^b	0.023 ^c
Mass of pycnometer	g	69.434	0.001 ^d	0.010 ^e
Mass of pycnometer + powder + H ₂ O	g	183.942	0.002 ^b	0.023 ^c
Mass of pycnometer + powder	g	94.228	0.002 ^d	0.014 ^e
Density of Water	g/cm ³	0.997363	2.3x10 ^{-5f}	2.3x10 ^{-4f}

a. Data for Sample GU3-753.0.

b. Determined by three replicate determinations of mass of a 150-g standard traceable to NIST.

c. Determined by comparing the mean value from three replicate determinations of mass of a 150-g standard, traceable to NIST, to the "true" mass.

d. Obtained by linear interpolation between precision indicated by footnote b and precision of 0.001 g obtained by three replicate determinations of mass of NIST-traceable 30-g standard.

e. Obtained by linear interpolation between accuracy indicated by footnote c and accuracy of 0.003 g obtained by comparing the mean value from three replicate determinations of mass of NIST-traceable 30-g standard with "true" mass.

f. See footnotes for Table 1.

uncertainty is $U_{\rho g} = 0.013 \text{ g/cm}^3$. As with the bulk densities, an uncertainty value of 0.013 g/cm^3 should be a good estimate of an upper bound on uncertainties for thermal/mechanical units other than Unit TSw2.

3.4.2 Gas-Intrusion Pycnometer

Equation 12 applies for calculation of grain density for this method as well, but the method of determining V_g is different. With a gas pycnometer, V_g is found directly using

$$V_g = V_{c+g} - V_c, \quad (18)$$

where V_{C+G} is the volume of a sample cup containing the powdered sample, and V_C is the volume of the empty sample cup.

Unfortunately, no data are available for the precision or accuracy for either V_{C+G} or V_C , so that no estimates of the precision and accuracy of V_G , and thus of ρ_g , can be made.

3.5 Matrix Porosity

Matrix porosity (ϕ) as a volume fraction usually is calculated using

$$\phi = 1 - \frac{\rho_{db}}{\rho_g} \quad . \quad (19)$$

Using the estimated precisions and accuracies for ρ_{db} (Section 3.3) and ρ_g (3.4.1), the following values can be calculated for Unit TSw2:

$$\begin{aligned} S\phi &= 0.002 \quad , \\ A\phi &= 0.004 \quad , \text{ and} \\ U\phi &= 0.010 \quad , \end{aligned}$$

relative to the calculated value for ϕ of 0.054.

Because ρ_{db} and ρ_g vary between thermal/mechanical units, the values of $S\phi$, $A\phi$, and $U\phi$ listed above for Unit TSw2 cannot be extrapolated to other units. Assuming that the values for precision and accuracy of ρ_{db} and ρ_g used in this example apply for all of the thermal/mechanical units and using average values for ρ_g and ρ_{db} , the values of $U\phi$ listed in Table 8 are obtained.

For the welded units, and especially for Unit TSw3, the total estimated uncertainty is a substantial percentage of the actual porosity. However, it should be kept in mind that the assumed values for precision and accuracy are maximum values (see Section 3.1.1 for discussion), so that the uncertainty estimates in Table 8 also are likely to be overestimates.

TABLE 8

UNCERTAINTY FOR MATRIX POROSITY

Unit	$U\phi$	ϕ	$\frac{U}{\phi} (\%)$
TCw	0.014	0.107	13.1
PTn	0.013	0.420	3.1
TSw1	0.010	0.144	6.9
TSw2	0.010	0.122	8.2
TSw3	0.014	0.032	43.8
CHn1v	0.013	0.345	3.8
CHn2v	0.009	0.281	3.2
CHn1z	0.013	0.324	4.0
CHn2z	0.014	0.216	6.5

3.6 Elastic Properties3.6.1 Young's Modulus

Young's modulus (E) is calculated using

$$E = \frac{\sigma_2 - \sigma_1}{(\epsilon_a)_2 - (\epsilon_a)_1} \quad , \quad (20)$$

where $(\epsilon_a)_2$ and $(\epsilon_a)_1$ are the axial strains induced by the stresses σ_2 and σ_1 . In turn, $(\epsilon_a)_2$ and $(\epsilon_a)_1$ are calculated using

$$(\epsilon_a)_2 = \frac{(\Delta L)_2}{L_0} = \frac{L_2 - L_0}{L_0} \quad (21a)$$

and

$$(\epsilon_a)_1 = \frac{(\Delta L)_1}{L_0} = \frac{L_1 - L_0}{L_0} \quad , \quad (21b)$$

where L_0 is the original sample length (or the original length over which displacements are measured) and $(\Delta L)_2$ and $(\Delta L)_1$ are the changes in L_0

caused by changing the stress level from the reference level (usually zero) to σ_2 and σ_1 . Alternatively, if the span of the length-measuring device (L) includes endcaps or other components of a testing device, an axial strain ϵ_a is obtained by

$$\epsilon_a = \frac{\Delta L}{L} = \frac{(\Delta L)_T - \Sigma(\Delta L)_i}{L}, \quad (22)$$

where $(\Delta L)_T$ is the change in length of all of the different materials spanned by the device, and $\Sigma(\Delta L)_i$ is the sum of the length changes of all materials other than the sample.

3.6.1.1 Length Change of Sample Only

Combining Equations 20 and 21, and expressing σ_2 and σ_1 as the ratios of applied force (F_2 and F_1) to cross-sectional area (A^*), we get

$$E = \frac{L_0(\sigma_2 - \sigma_1)}{(\Delta L)_2 - (\Delta L)_1} = \frac{L_0(F_2 - F_1)}{A^*[(\Delta L)_2 - (\Delta L)_1]}. \quad (23)$$

The precisions and accuracies of force and area are discussed in Section 3.7. The precision and accuracy of L_0 (the sample height) are presented in Table 2.

Specification of precision and accuracy for values of ΔL presents a similar problem as is encountered for F ; the answer depends on the specific gauge used to measure ΔL , the range of the gauge, and where in the range the values of ΔL occur.

As an example, suppose a sample has a Young's modulus of 35 GPa as determined between 10 and 50% of a failure stress of 165 MPa. The values of precision and accuracy listed in Table 9 apply.

Using the information above, calculated values of S_E and A_E are 0.09 and 0.34 GPa, respectively. The value of U_E calculated from these two values is 0.63 GPa.

TABLE 9

PRECISIONS AND ACCURACIES USED IN CALCULATIONS OF YOUNG'S MODULUS

Parameter	Value	Precision	Accuracy	Uncertainty
L_G	$1.02 \times 10^{-1} \text{ m}$	$2.5 \times 10^{-5} \text{ m}^a$	$1.27 \times 10^{-4} \text{ m}^a$	$2.08 \times 10^{-4} \text{ m}$
F_2	$1.67 \times 10^5 \text{ N}$	$1.67 \times 10^2 \text{ N}^b$	$3.34 \times 10^2 \text{ N}^b$	$7.60 \times 10^2 \text{ N}$
F_1	$3.34 \times 10^4 \text{ N}$	$3.34 \times 10^1 \text{ N}^b$	$6.69 \times 10^1 \text{ N}^b$	$1.75 \times 10^2 \text{ N}$
A^*	$2.03 \times 10^{-3} \text{ m}^2$	$3.99 \times 10^{-6} \text{ m}^c$	$1.02 \times 10^{-5} \text{ m}^{2c}$	$2.32 \times 10^{-5} \text{ m}^2$
$(\Delta L)_2$	$2.54 \times 10^{-4} \text{ m}$	$1.25 \times 10^{-7} \text{ m}^d$	$1.52 \times 10^{-6} \text{ m}^d$	$1.93 \times 10^{-6} \text{ m}$
$(\Delta L)_1$	$6.24 \times 10^{-5} \text{ m}$	$1.25 \times 10^{-7} \text{ m}^d$	$1.25 \times 10^{-7} \text{ m}^d$	$5.31 \times 10^{-7} \text{ m}$

a. Taken from Table 2.

b. Calculated by linear interpolation of load-cell calibration data for precision and accuracy. Load-cell capacity is $500 \times 10^3 \text{ lb}$ ($2.2 \times 10^6 \text{ N}$); relevant range is 0 to $100 \times 10^3 \text{ lb}$ (0 to $4.4 \times 10^5 \text{ N}$).

c. Taken from Table 11.

d. Calculated using data obtained during calibration of axial LVDTs.

The results in the preceding example for Unit TSw2 cannot be extrapolated directly to other units. The values of F_2 , F_1 , $(\Delta L)_2$, and $(\Delta L)_1$ all depend on both the failure stress and the Young's modulus of the material as well as on calibration data for the load-cell and linear variable differential transformers (LVDT) used in the relevant experiments. Thus, values of accuracy, precision, and uncertainty for the Young's moduli of other thermal/mechanical units will need to be calculated using test-specific data and the approach outlined in this section.

3.6.1.2 Length Changes in Testing Equipment and Sample

Calculation of the precision and accuracy of Young's modulus using Equations 20 and 22 is unrealistic for this report. Too many quantities can vary, depending on the materials used for equipment components and the testing configuration. Inherent in the method, however, is the likelihood that a value of Young's modulus obtained using Equations 20 and 22 will be less accurate and less precise than a value calculated using Equation 23

because of the increase in the number of measured parameters, each with associated inaccuracy and imprecision.

3.6.2 Poisson's Ratio

Poisson's ratio (ν) is calculated using

$$\nu = - \frac{\left(\epsilon_t\right)_2 - \left(\epsilon_t\right)_1}{\left(\epsilon_a\right)_2 - \left(\epsilon_a\right)_1} \quad (24)$$

where ϵ_t is the transverse, or radial, strain, defined as

$$\epsilon_t = \frac{\Delta r}{r_0} = \frac{r - r_0}{r_0} \quad (25)$$

and where Δr is the change in the radius (r_0) of a cylindrical sample resulting from a change in the stress applied to the sample.

Combining Equations 21, 24, and 25, we have

$$\nu = \frac{L_0}{r_0} \left[\frac{(\Delta r)_1 - (\Delta r)_2}{(\Delta L)_2 - (\Delta L)_1} \right] \quad (26)$$

The data in Table 10 apply for estimation of the precision and accuracy of Poisson's ratio. The calculated values of S_ν , A_ν , and U_ν are 0.002, 0.006, and 0.012, respectively, compared to a value of 0.30 for ν .

The preceding estimation of S_ν and A_ν assumes direct linear measurement of the change in transverse dimension. Other techniques with which the change in radius can be determined are available, including measurement of changes in sample circumference or volume. These techniques also can provide reliable determinations of radial strain and, thus, of Poisson's ratio.

As discussed for Young's modulus (Section 3.6.1.1), the numerical results of this example, obtained using data from Unit TSw2, cannot be

TABLE 10

PRECISIONS AND ACCURACIES USED IN CALCULATIONS OF POISSON'S RATIO

Parameter	Value	Precision	Accuracy	Uncertainty
L_0	$1.02 \times 10^{-1} \text{ m}$	$2.5 \times 10^{-5} \text{ m}^a$	$1.27 \times 10^{-4} \text{ m}^a$	$2.08 \times 10^{-4} \text{ m}$
$(\Delta L)_2$	$2.54 \times 10^{-4} \text{ m}$	$1.25 \times 10^{-7} \text{ m}^b$	$1.52 \times 10^{-6} \text{ m}^b$	$1.93 \times 10^{-6} \text{ m}$
$(\Delta L)_1$	$6.24 \times 10^{-5} \text{ m}$	$1.25 \times 10^{-7} \text{ m}^b$	$1.25 \times 10^{-7} \text{ m}^b$	$5.31 \times 10^{-7} \text{ m}$
r_0	$2.54 \times 10^{-2} \text{ m}$	$2.5 \times 10^{-5} \text{ m}^a$	$6.4 \times 10^{-5} \text{ m}^a$	$1.45 \times 10^{-4} \text{ m}$
$(\Delta r)_1$	$8.71 \times 10^{-7} \text{ m}$	$6.53 \times 10^{-9} \text{ m}^c$	$1.31 \times 10^{-8} \text{ m}^c$	$3.43 \times 10^{-8} \text{ m}$
$(\Delta r)_2$	$1.52 \times 10^{-5} \text{ m}$	$1.14 \times 10^{-7} \text{ m}^c$	$2.28 \times 10^{-7} \text{ m}^c$	$5.98 \times 10^{-7} \text{ m}$

a. Taken from Table 2.

b. Taken from Table 10.

c. Calculated using data obtained during calibration of radial LVDT.

extrapolated directly to other units. Estimation of precision, accuracy, and uncertainty for the Poisson's ratios of other thermal/mechanical units should be performed using test-specific data and the approach outlined in this section.

3.7 Compressive Strength

Compressive strength (σ) is calculated using

$$\sigma = \frac{F}{A^*}, \quad (27)$$

where F is the force applied to the cross-sectional area (A^*) of a sample. In turn, A^* is calculated by

$$A^* = \pi r^2, \quad (28)$$

where r is the radius of the cylindrical sample. Values of precision and accuracy for r are listed in Table 2. Calculated values for A^* are given in Table 11.

TABLE 11

PRECISIONS AND ACCURACIES FOR CROSS-SECTIONAL
AREAS OF CYLINDRICAL SAMPLES

<u>Sample Radius (cm)</u>	<u>Sample Area (cm²)</u>		
	<u>Value</u>	<u>Precision*</u>	<u>Accuracy*</u>
1.27	5.07	0.0199	0.0511
2.54	20.27	0.0399	0.1021
4.13	53.59	0.0649	0.1661
6.35	126.68	0.0997	0.2553
11.43	410.43	0.1795	0.4596
13.33	558.23	0.2094	1.0637

*Calculated using precision and accuracy data for radii given in Table 2.

The precision and accuracy for the load depend on the capacity of the load cell used to measure the load, the range used for the load cell, and where in the range the strength of the sample lies. For example, if a sample with a radius of 2.54 cm is tested using the SNL load cell with a capacity of 1000×10^3 lb described in Price et al. (1985) and the load necessary to achieve sample failure is 3.34×10^5 N, the values of S_f and A_f are both 111.2 N (as determined from load-cell calibrations). If the same sample is tested using the SNL load cell with a capacity of 500×10^3 lb, both precision and accuracy would be 100.1 N (as determined from load-cell calibrations). The values of A_o , S_o , and U_o for the strength of 165 MPa (corresponding to the load given above) are 0.83, 0.33, and 1.90 MPa, respectively, for the larger of the two load cells.

As discussed for the two elastic properties (Sections 3.6.1 and 3.6.2), the numerical results of this example, obtained using data for Unit TSw2, cannot be extrapolated directly to other units. Estimation of the precision, accuracy, and uncertainty for the compressive strengths of other thermal/mechanical units should be performed using test-specific data and the approach outlined in this section.

3.8 Thermal Expansion

The coefficient of linear thermal expansion (α) can be expressed in two ways:

$$\text{average: } \alpha = \frac{\Delta L}{L_0(\Delta T)} = \frac{L - L_0}{L_0(\Delta T)} , \quad (29a)$$

and

$$\text{instantaneous: } \alpha = \lim_{T_2 \rightarrow T_1} \frac{L_2 - L_1}{L_0(T_2 - T_1)} , \quad (29b)$$

where ΔL is the length change (from the original length L_0) caused by the temperature change ΔT , and L_2 and L_1 are the sample lengths at temperatures T_2 and T_1 . (Note that neither of these equations is strictly true as ΔL becomes a significant fraction of L_0 .) The length change can be measured directly (e.g., in a single-pushrod dilatometer) or by comparison with the length change of a standard material for the same temperature interval (e.g., dual-pushrod dilatometer). The example discussed below applies to data gathered at SNL using a dual-pushrod dilatometer. Although an SNL contractor has also obtained thermal-expansion data that were determined with a single-pushrod dilatometer, supporting information (in the form of precisions and accuracies for measured parameters) is insufficient for estimating the precision and accuracy of thermal-expansion coefficients.

The data in Table 12 apply for estimating the precision and accuracy of α .

Taking partial derivatives of Equation 29a and applying Equations 3 and 4 using the data in Table 12, the calculated values of S_α , A_α , and U_α are $0.80 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$, $1.66 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$, and $4.26 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$, respectively, relative to a value of $10.8 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$ for α for the temperature interval of 25° to 50°C .

TABLE 12

PRECISIONS AND ACCURACIES USED IN CALCULATIONS OF THE
COEFFICIENT OF LINEAR THERMAL EXPANSION

<u>Parameter</u>	<u>Value</u>	<u>Precision</u>	<u>Accuracy</u>	<u>Uncertainty</u>
ΔL	$6.858 \times 10^{-6} \text{ m}$	$5.08 \times 10^{-7} \text{ m}^a$	$1.016 \times 10^{-6} \text{ m}^b$	$2.67 \times 10^{-6} \text{ m}$
L_0	$2.54 \times 10^{-2} \text{ m}$	$1.27 \times 10^{-5} \text{ m}^c$	$2.54 \times 10^{-5} \text{ m}^b$	$6.67 \times 10^{-5} \text{ m}$
ΔT	25°C	0.1°C^c	1°C^d	1.32°C

- a. Estimated repeatability of reading displacement from plot of displacement as a function of temperature.
- b. Assumed to be twice the value for precision.
- c. Assumed value.
- d. Criterion applied for calibration of thermocouple.

This example used data for welded, devitrified tuff for a 25°C temperature interval. If the temperature interval is larger but the same value of α applies, then S_α and A_α would be smaller by the factor $25/\Delta T$. (Note that, to date, most coefficients of thermal expansion used on the YMP have been calculated for $\Delta T = 50^\circ\text{C}$.) For other tuffs, all of which have smaller values of α for the 25° -to- 50°C interval, values of S_α and A_α will be similar, assuming that precisions and accuracies are the same as listed in Table 12. The similarity results because $S_{\Delta L}$ and $A_{\Delta L}$ are the dominant factors in the calculation of the precision and accuracy of α for this example.

Ten determinations of α for platinum (determined as a calibration check by SNL) give the values of precision and accuracy shown in Table 13 as a function of temperature interval. For these data, the largest values of precision and accuracy are approximately 11% of the relevant value of α . Most of the precisions and accuracies are better than 5% of values of α . Thus, the values of precision and accuracy estimated for tuff samples based on the data in Table 12 appear to be conservative in the sense that the estimated precision and accuracy values for tuff are higher than the correlative (measured) values for platinum.

TABLE 13

THERMAL-EXPANSION DATA FOR PLATINUM*

<u>Temperature Interval (°C)</u>	<u>Coefficient of Linear Thermal Expansion (10⁻⁶°C⁻¹)</u>		
	<u>Value</u>	<u>Precision</u>	<u>Accuracy</u>
25-50	8.95	0.95	-1.02
50-100	9.08	0.29	0.06
100-150	9.26	0.33	-0.11
150-200	9.38	0.44	0.01
200-250	9.52	0.27	0.17
250-300	9.62	0.28	0.25

*Data obtained for 10 individual determinations of expansion as a function of temperature.

3.9 Thermal Conductivity

The expression relating thermal conductivity to measured parameters depends on the technique used for measurement. The only technique used to gather thermal-conductivity data for SNL on YMP samples has been the transient-line-source technique. The relevant equation for calculation of thermal conductivity (K) is

$$K = \frac{P}{4\pi\Delta T} \ln \frac{t_2}{t_1} , \quad (30)$$

where P is the power input to the line heater, and ΔT is the temperature difference between times t_2 and t_1 at a single measurement location (usually the midpoint of the heater). As discussed in Nimick (1989), information on the precision and accuracy of P, ΔT , and t are not available, so S_K and A_K cannot be calculated.

4.0 CONCLUSIONS

A summary of the precisions, accuracies, and uncertainties estimated in the examples in Section 3 is provided in Table 14. Most of the estimated uncertainties are $\leq 2\%$ of the values for the relevant parameters. A notable exception is for the coefficient of linear thermal expansion, for which the uncertainty is 39% of the value. An uncertainty of this relative magnitude would be unacceptable in future testing; the estimate suggests that plans for determination of thermal-expansion behavior should include careful consideration of estimated error and methods of achieving substantial improvements in the precisions and accuracies of measured parameters. [For example, having the precision and accuracy of the measurement of length change improve each by an order of magnitude over those listed in Section 3.8 would reduce U_α by almost an order of magnitude (i.e., the relative error would be close to 5%).]

The other parameter in Table 14 for which the uncertainty is a large percentage of the measured value is matrix porosity. Examination of Table 8 shows that an uncertainty of 0.01 is to be expected, regardless of the measured value for porosity. Thus, for samples with low porosity, the uncertainty reasonably can be expected to be a large percentage of the measured value.

As pointed out in Section 2.2, the method used in this report does not include potential inaccuracies or imprecisions resulting from violations of assumptions for a test method. Thus, any discussion of experimental uncertainty should touch on the correctness of application of a test method in addition to any calculations of accuracy or precision based on the estimation method.

As discussed throughout the report, all numerical results obtained in the examples used data from Unit TSw2. For density and thermal-expansion, the results can also be used as estimates of precision, accuracy, and

TABLE 14

EXAMPLES OF ESTIMATED PRECISION, ACCURACY, AND UNCERTAINTY^a

Property	Units	Value	Precision	Accuracy	Uncertainty	Uncertainty Meas. Value (%)
Saturated Bulk Density	g/cm ³	2.243	0.006	0.003	0.016	0.71
Natural-State Bulk Density	g/cm ³	2.321	0.006	0.016	0.035	1.51
Dry Bulk Density	g/cm ³	2.38	0.006	0.003	0.022	0.92
Grain Density	g/cm ³	2.515	0.001	0.010	0.013	0.52
Porosity	--	0.054	0.002	0.004	0.010	18.52
Compressive Strength	MPa	165	0.33	0.83	1.90	1.15
Young's Modulus	GPa	35	0.09	0.34	0.63	1.80
Poisson's Ratio	--	0.30	0.002	0.006	0.012	4.00
Coefficient of Linear Thermal Expansion (25°-to-50°C interval)	10 ⁻⁶ °C ⁻¹	10.8	0.80 ^b	1.66 ^b	4.26 ^b	39.44

a. All values in the table are for welded, devitrified material from the Topopah Spring Member (thermal/mechanical Units TSw1 and TSw2).

b. Values for 50°C intervals for ΔT would be half of the listed value.

uncertainty for other thermal/mechanical units. For matrix porosity, Young's modulus, Poisson's ratio, and compressive strength, estimates of precision, accuracy, and uncertainty need to be obtained using test-specific data.

Table 15 summarizes the measured parameters for which data on precision and accuracy must be available before the precision and accuracy of the geoenengineering properties discussed in this report can be estimated. The information in Table 15 should be considered during planning and performance of a testing program.

TABLE 15

**TEST PARAMETERS RELEVANT TO ESTIMATION OF PRECISION
AND ACCURACY OF GEOENGINEERING PROPERTIES**

<u>Geoengineering Property</u>	<u>Parameters^a</u>	<u>Section</u>
Saturated Bulk Density (Irreg. Samples)	$M_{s,air}, M_{wb+s}, M_{wb}, \rho_{w,T}$	3.1.1
Saturated Bulk Density (Reg. Samples)	$M_{s,air}, r, h$	3.1.2
Natural-State Bulk Density	$M_{ns,air}, M_{ws,air}, M_{wb+ws}, M_{wb}, \rho_{w,T}, \rho_{wax}$	3.2
Dry Bulk Density	$M_{d,air}, M_{wb+s}, M_{wb}, \rho_{w,T}$	3.3
Grain Density (Water Pyc.)	$M_{p+w}, M_p, M_{p+g+w}, M_{p+g}, \rho_{w,T}$	3.4.1
Grain Density (Gas Pyc.)	$M_{p+g}, M_p, V_{c+g}, V_c$	3.4.2
Matrix Porosity	None; calculated using ρ_{db} and ρ_g	3.5
Young's Modulus	$L_o, F_2, F_1 A^*, (\Delta L)_2, (\Delta L)_1$	3.6.1
Poisson's Ratio	$L_o, (\Delta L)_1, (\Delta L)_2, r_o, (\Delta r)_2, (\Delta r)_1$	3.6.2
Compressive Strength	F, A^*	3.7
Coefficient of Thermal Expansion	L_2, L_1, L_o, T_2, T_1	3.8
Thermal Conductivity ^b	$P, \Delta T, t_2, t_1$	3.9

a. See the relevant section for definition of parameter symbols.

b. Parameters listed for thermal conductivity are for the transient-line-source technique; other techniques will have different parameters.

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APPENDIX A

INFORMATION FROM, AND CANDIDATE INFORMATION FOR, THE SITE AND ENGINEERING PROPERTY DATA BASE AND THE REFERENCE INFORMATION BASE

No information in this report has been taken from, nor is intended for entry into, either the Site and Engineering Property Data Base or the Reference Information Base.

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<p>1 J. W. Bartlett, Director (RW-1)
Office of Civilian Radioactive
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U.S. Department of Energy
1000 Independence Avenue, S.W.
Washington, D.C. 20585</p> <p>1 T. H. Isaacs (RW-4)
Office of Strategic Planning
and International Programs
Office of Civilian Radioactive
Waste Management
U.S. Department of Energy
1000 Independence Avenue, S.W.
Washington, D.C. 20585</p> <p>1 J. D. Saltzman (RW-5)
Office of External Relations
Office of Civilian Radioactive
Waste Management
U.S. Department of Energy
1000 Independence Avenue, S.W.
Washington, D.C. 20585</p> <p>1 Samuel Rousso (RW-10)
Office of Program and Resources
Management
Office of Civilian Radioactive
Waste Management
U.S. Department of Energy
1000 Independence Avenue, S.W.
Washington, D.C. 20585</p> <p>1 J. C. Bresee (RW-10)
Office of Civilian Radioactive
Waste Management
U.S. Department of Energy
1000 Independence Avenue, S.W.
Washington, D.C. 20585</p> <p>1 C. P. Gertz (RW-20)
Office of Geologic Disposal
Office of Civilian Radioactive
Waste Management
U.S. Department of Energy
1000 Independence Avenue, S.W.
Washington, D.C. 20585</p> | <p>1 S. J. Brocoun (RW-22)
Analysis and Verification Division
Office of Civilian Radioactive
Waste Management
U.S. Department of Energy
1000 Independence Avenue, S.W.
Washington, D.C. 20585</p> <p>1 D. D. Shelor (RW-30)
Office of Systems and Compliance
Office of Civilian Radioactive
Waste Management
U.S. Department of Energy
1000 Independence Avenue, S.W.
Washington, D.C. 20585</p> <p>1 J. Roberts (RW-33)
Office of Civilian Radioactive
Waste Management
U.S. Department of Energy
1000 Independence Avenue, S.W.
Washington, D.C. 20585</p> <p>1 G. J. Parker (RW-332)
Office of Civilian Radioactive
Waste Management
U.S. Department of Energy
1000 Independence Avenue, S.W.
Washington, D.C. 20585</p> <p>1 Associate Director (RW-40)
Office of Storage and Transportation
Office of Civilian Radioactive
Waste Management
U.S. Department of Energy
1000 Independence Avenue, S.W.
Washington, D.C. 20585</p> <p>1 Associate Director (RW-50)
Office of Contract Business
Management
Office of Civilian Radioactive
Waste Management
U.S. Department of Energy
1000 Independence Avenue, S.W.
Washington, D.C. 20585</p> <p>1 C. G. Russomanno (RW-52)
Office of Civilian Radioactive
Waste Management
U.S. Department of Energy
1000 Independence Avenue, S.W.
Washington, D.C. 20585</p> |
|--|---|

- 1 D. U. Deere, Chairman
Nuclear Waste Technical
Review Board
1100 Wilson Blvd. #910
Arlington, VA 22209-2297
- 1 Dr. Clarence R. Allen
Nuclear Waste Technical Review Board
1000 E. California Blvd.
Pasadena, CA 91106
- 1 Dr. John E. Cantlon
Nuclear Waste Technical Review Board
1795 Bramble Dr.
East Lansing, MI 48823
- 1 Dr. Melvin W. Carter
Nuclear Waste Technical Review Board
4621 Ellisbury Dr., N.E.
Atlanta, GA 30332
- 1 Dr. Donald Langmuir
Nuclear Waste Technical Review Board
109 So. Lookout Mountain Cr.
Golden, CO 80401
- 1 Dr. D. Warner North
Nuclear Waste Technical Review Board
Decision Focus, Inc.
4984 El Camino Real
Los Altos, CA 94062
- 1 Dr. Dennis L. Price
Nuclear Waste Technical Review Board
1011 Evergreen Way
Blacksburg, VA 24060
- 1 Dr. Ellis D. Verink
Nuclear Waste Technical Review Board
4401 N.W. 18th Place
Gainesville, FL 32605
- 5 C. P. Gertz, Project Manager
Yucca Mountain Project Office
U.S. Department of Energy
P.O. Box 98608--MS 523
Las Vegas, NV 89193-8608
- 1 C. L. West, Director
Office of External Affairs
DOE Field Office, Nevada
U.S. Department of Energy
P.O. Box 98518
Las Vegas, NV 89193-8518
- 12 Technical Information Officer
DOE Field Office, Nevada
U.S. Department of Energy
P.O. Box 98518
Las Vegas, NV 89193-8518
- 1 P. K. Fitzsimmons, Director
Health Physics & Environmental
Division
DOE Field Office, Nevada
U.S. Department of Energy
P.O. Box 98518
Las Vegas, NV 89193 8518
- 1 D. R. Elle, Director
Environmental Protection Division
DOE Field Office, Nevada
U.S. Department of Energy
P.O. Box 98518
Las Vegas, Nv 89193-8518
- 1 Repository Licensing & Quality
Assurance Project Directorate
Division of Waste Management
U.S. Nuclear Regulatory Commission
Washington, D.C. 20555
- 1 Senior Project Manager for Yucca
Mountain Repository Project Branch
Division of Waste Management
U.S. Nuclear Regulatory Commission
Washington, D.C. 20555
- 1 NRC Document Control Desk
Division of Waste Management
U.S. Nuclear Regulatory Commission
Washington, D.C. 20555
- 1 P. T. Prestholt
NRC Site Representative
301 E. Stewart Ave.
Las Vegas, NV 89101
- 1 E. P. Binnall
Field Systems Group Leader
Building 50B/4235
Lawrence Berkeley Laboratory
Berkeley, CA 94720
- 1 Center for Nuclear Waste
Regulatory Analyses
6220 Gulebra Road
Drawer 28510
San Antonio, TX 78284

- 3 L. J. Jardine
Technical Project Officer for YMP
Mail Stop L-204
Lawrence Livermore National
Laboratory
P.O. Box 808
Livermore, CA 94550
- 4 R. J. Herbst
Technical Project Officer for YMP
N-5, Mail Stop J521
Los Alamos National Laboratory
P.O. Box 1663
Los Alamos, NM 87545
- 1 H. N. Kalia
Exploratory Shaft Test Manager
Los Alamos National Laboratory
Mail Stop 527
101 Convention Center Dr.
Suite 820
Las Vegas, NV 89109
- 1 J. F. Divine
Assistant Director for
Engineering Geology
U.S. Geological Survey
106 National Center
12201 Sunrise Valley Dr.
Reston, VA 22092
- 6 L. R. Hayes
Technical Project Officer
Yucca Mountain Project Branch--MS 425
U.S. Geological Survey
P.O. Box 25046
Denver, CO 80225
- 1 V. R. Schneider
Asst. Chief Hydrologist--MS 414
Office of Program Coordination
& Technical Support
U.S. Geological Survey
12201 Sunrise Valley Drive
Reston, VA 22092
- 1 R. B. Raup, Jr.
Geological Division Coordinator
MS 913
Yucca Mountain Project
U.S. Geological Survey
P.O. Box 25046
Denver, CO 80225
- 1 D. H. Appel, Chief
Hydrologic Investigations Program
MS 421
U.S. Geological Survey
P.O. Box 25046
Denver, CO 80225
- 1 E. J. Helley
Branch of Western Regional Geology
MS 427
U.S. Geological Survey
345 Middlefield Road
Menlo Park, CA 94025
- 1 Chief
Nevada Operations Office
U.S. Geological Survey
101 Convention Center Drive
Suite 860, MS 509
Las Vegas, NV 89109
- 1 D. Zesiger
U.S. Geological Survey
101 Convention Center Dr.
Suite 860 - MS509
Las Vegas, NV 89109
- 1 R. V. Watkins, Chief
Project Planning and Management
U.S. Geological Survey
P.O. Box 25046
421 Federal Center
Denver, CO 80225
- 1 A. L. Flint
U.S. Geological Survey
MS 721
P.O. Box 327
Mercury, NV 89023
- 1 D. A. Beck
U.S. Geological Survey
1500 E. Tropicana, Suite 201
Las Vegas, NV 89119
- 1 P. A. Glancy
U.S. Geological Survey
Federal Building, Room 224
Carson City, NV 89701
- 1 Sherman S. C. Wu
Branch of Astrogeology
U.S. Geological Survey
2255 N. Gemini Dr.
Flagstaff, AZ 86001

- 1 J. H. Sass
Branch of Tectonophysics
U.S. Geological Survey
2255 N. Gemini Dr.
Flagstaff, AZ 86001
- 1 DeWayne A. Campbell
Technical Project Officer for YMP
Bureau of Reclamation
Code D-3790
P.O. Box 25007
Denver, CO 80225
- 1 S. M. Dash
Science Applications International
Corp.
14062 Denver West Parkway, Suite 255
Golden, CO 80401
- 1 K. W. Causseaux
NHP Reports Chief
U.S. Geological Survey
421 Federal Center
P.O. Box 25046
Denver, CO 80225
- 1 V. M. Glanzman
U.S. Geological Survey
913 Federal Center
P.O. Box 25046
Denver, CO 80225
- 1 J. H. Nelson
Technical Project Officer for YMP
Science Applications International
Corp.
101 Convention Center Dr.
Suite 407
Las Vegas, NV 89109
- 2 SAIC-T&MSS Library
Science Applications International
Corp.
101 Convention Center Dr.
Suite 407
Las Vegas, NV 89109
- 1 Elaine Ezra
YMP GIS Project Manager
EG&G Energy Measurements, Inc.
Mail Stop D-12
P.O. Box 1912
Las Vegas, NV 89125
- 1 R. E. Jackson, Program Manager
Roy F. Weston, Inc.
955 L'Enfant Plaza, Southwest
Washington, D.C. 20024
- 1 Technical Information Center
Roy F. Weston, Inc.
955 L'Enfant Plaza, Southwest
Washington, D.C. 20024
- 1 D. Hedges, Vice President,
Quality Assurance
Roy F. Weston, Inc.
4425 Spring Mountain Road, Suite 300
Las Vegas, Nevada 89102
- 1 D. L. Fraser, General Manager
Reynolds Electrical & Engineering Co.
Mail Stop 555
P.O. Box 98521
Las Vegas, NV 89193-8521
- 1 R. F. Pritchett
Technical Project Officer for YMP
Reynolds Electrical & Engineering Co.
MS 408
P.O. Box 98521
Las Vegas, NV 89193-8521
- 1 B. W. Colston
General Manager & President
Las Vegas Branch
Raytheon Services Nevada
Mail Stop 416
P.O. Box 95487
Las Vegas, NV 89193-5487
- 1 R. L. Bullock
Technical Project Officer for YMP
Raytheon Services Nevada
Suite P250, MS 403
101 Convention Center Dr.
Las Vegas, NV 89109
- 1 R. E. Lowder
Technical Project Officer for YMP
MAC Technical Services
101 Convention Center Drive
Suite 1100
Las Vegas, NV 89109

- 1 C. K. Hastings, Manager
PASS Program
Pacific Northwest Laboratories
P.O. Box 999
Richland, WA 99352
- 1 A. T. Tamura
Science and Technology Division
Office of Scientific and Technical
Information
U.S. Department of Energy
P.O. Box 62
Oak Ridge, TN 37831
- 1 Carlos G. Bell, Jr.
Professor of Civil Engineering
Civil and Mechanical Engineering
Department
University of Nevada, Las Vegas
4505 South Maryland Parkway
Las Vegas, NV 89154
- 1 C. F. Costa, Director
Nuclear Radiation Assessment
Division
U.S. Environmental Protection
Agency
Environmental Monitoring Systems
Laboratory
P.O. Box 93478
Las Vegas, NV 89193-3478
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Battelle Columbus Laboratory
Office of Nuclear Waste Isolation
505 King Avenue
Columbus, OH 43201
- 1 T. Hay, Executive Assistant
Office of the Governor
State of Nevada
Capitol Complex
Carson City, NV 89710
- 3 R. R. Loux, Jr.
Executive Director
Nuclear Waste Project Office
State of Nevada
Evergreen Center, Suite 252
1802 North Carson Street
Carson City, NV 89710
- 1 C. H. Johnson
Technical Program Manager
Nuclear Waste Project Office
State of Nevada
Evergreen Center, Suite 252
1802 North Carson Street
Carson City, NV 89710
- 1 John Fordham
Water Resources Center
Desert Research Institute
P.O. Box 60220
Reno, NV 89506
- 1 Dr. Martin Mifflin
Water Resources Center
Desert Research Institute
2505 Chandler Avenue
Suite 1
Las Vegas, NV 89120
- 1 Eric Anderson
Mountain West Research-Southwest
Inc.
2901 N. Central Ave. #1000
Phoenix, AZ 85012-2730
- 1 Department of Comprehensive Planning
Clark County
225 Bridger Avenue, 7th Floor
Las Vegas, NV 89155
- 1 Planning Department
Nye County
P.O. Box 153
Tonopah, NV 89049
- 1 Lincoln County Commission
Lincoln County
P.O. Box 90
Pioche, NV 89043
- 5 Judy Foremaster
City of Caliente
P.O. Box 158
Caliente, NV 89008
- 1 Economic Development Department
City of Las Vegas
400 East Stewart Avenue
Las Vegas, NV 89101

- 1 Community Planning & Development
City of North Las Vegas
P.O. Box 4086
North Las Vegas, NV 89030
- 1 Director of Community Planning
City of Boulder City
P.O. Box 367
Boulder City, NV 89005
- 1 Commission of the European
Communities
200 Rue de la Loi
B-1049 Brussels
BELGIUM
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YMP Research and Study Center
Reynolds Electrical & Engineering
Co., Inc.
MS 407
P.O. Box 98521
Las Vegas, NV 89193-8521
- 1 Amy Anderson
Argonne National Laboratory
Building 362
9700 So. Cass Ave.
Argonne, IL 60439
- 1 6300 T. O. Hunter
- 1 6310 T. E. Blejwas, Actg.
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