Report on Laboratory Tests of Drying and Rewetting of Intact Rocks from the Drift-Scale and Single-Heater Tests

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Introduction

The primary focus of this report is measurement of moisture-retention curves of tuff as a function of temperature for both drying and rewetting conditions. The report contains descriptions of experimental designs and procedures, data, observations, preliminary analyses, and future work.

Knowledge of unsaturated transport properties is critical for understanding the movement of water through the unsaturated zone. Evaluation of the performance of a potential nuclear waste repository also depends on knowledge of these properties. Moisture-retention data are important input for numerical models of moisture movement in unsaturated porous media. Also important is the effect of sample history on the moisture-retention curves and whether there is significant hysteresis between wetting and drying measurements.

This report presents the moisture-retention curves of the Single-Heater Test (SHT) and Drift-Scale Test (DST) core samples measured thus far. We have completed the measurement of the moisture-retention curves of those core samples in the wetting phase at 95°C. The moisture-retention curves at 95°C in the drying phase will be determined in the next quarter. The highest temperature at which the moisture-retention curves will be determined will be 95°C. Previous measurements of the matric potential of tuff are reported by Roberts and Lin (1995) and in the *Near-Field and Altered-Zone Environment Report* (Wilder, 1996).

Experimental Procedure

Sample Description and Preparation

Samples from the DST and the SHT from the Exploratory Studies Facility (ESF), Nevada Test Site (NTS), were prepared from cores taken from the chemistry boreholes. Tables 1 and 2 list the identification numbers, boreholes, depths, wet and dry densities, and porosities. Thirteen DST samples and eleven SHT samples were prepared. The samples were subcored to a diameter of between 4.4 cm and 6.1 cm and cut into disks approximately 2.5 to 3.0 mm thick. Samples with obvious large cavities and inhomogeneous inclusions were avoided. The porosity of each sample was determined by subtracting the dry density from the saturated density and dividing by the water density. The average porosity for the DST samples is $10.0 \pm 1.7\%$; for the SHT samples, it is $11.1 \pm 1.1\%$.

Calculation of matric potential

The matric potential is the pressure potential that arises from the interaction of water with a solid matrix (Marshall and Holmes, 1992). In tuff, we assume that the suction potential is equal to the matric potential. Matric potential is defined by Kelvin's Law as

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$$y = b R T \ln (e/e_0) / M \quad (1)$$

where y is the matric potential in MPa, b is the density of water at the temperature of interest in g/cm^3 , R is the universal gas constant ($8.314 \text{ J}/\text{Kmol}$), T is temperature in K, e/e_0 is the relative humidity RH, and M is the molecular weight of water ($18.015 \text{ g}/\text{mole}$).

To determine the moisture-retention curve, samples were placed in the humidity chamber at a specific temperature and $\sim 20\%$ the RH. Measurements began on dry samples. When the weights reached a constant value for several days (samples were weighed daily), it was assumed that equilibrium was established. When a stable weight was achieved, the RH was increased and the process repeated at the same temperature. The maximum saturations achieved at the highest RH ($\sim 98\%$) were between 30% and 40% (vide infra). A balance with a sensitivity of 0.01 mg calibrated to a traceable standard was used to weigh the samples. Saturation is calculated by comparing weights with dry weights and taking into account porosity. The process was then repeated for the drying portion of the measurement. This cycle of measurement was repeated at different temperatures.

The following procedure was used to perform the measurements. First, the samples were machined and the dimensions measured. Next, the samples were placed in a vacuum oven at 35°C to reach a completely dry state. The samples were then placed in the relative humidity chamber at 25.1°C and 20% RH. When a stable weight (corresponding to a saturation) was achieved, the RH was increased to 50%. This was repeated as the RH was increased to 80%, and then to 95%. After this, the RH was decreased according to the following steps: 80%, 50%, and 20% RH. This procedure was repeated for all samples at the next temperatures: 49.6°C and 95°C . The SHT samples at 25.1°C had additional measurements performed, during both wetting and drying, at 35% and 65% RH.

Measurement Uncertainty

The uncertainties involved in the determination of the moisture retention curves include the measurements of weights, relative humidity and sample size. The sample dimensions are used to determine sample wet and dry densities. It is estimated that the thickness of the sample can be determined to $\pm 0.01 \text{ mm}$ and diameter to $\pm 0.03 \text{ mm}$. For the samples used here, this results in an error in sample volume of $\sim \pm 0.4\%$. The uncertainty in dry weight is estimated to be $\sim 0.00002 \text{ g}$ and for wet weight $\sim 0.0001 \text{ g}$. The error in the wet weight is higher than that of the dry condition because of the difficulty in achieving and maintaining saturation levels of 100% at elevated temperature. These uncertainties result in errors in dry and wet densities of $\sim 3\%$. When propagated through to porosity, the error is $\sim 1.0\%$ porosity, or $\sim 7\text{--}11\%$ of the measurement (Bevington and Robinson, 1992).

When repetitive measurements, such as the determination of weights at a specified relative humidity, are made on samples over several days, the uncertainty in the measurement is often less than the statistical uncertainty in the mean of the measured parameter. In such cases the error is taken as one standard deviation of the mean of the repeated measurements. The errors in saturation determined at

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specific temperature and RH vary from ~0.07 to 0.4% Sw. Thus, the relative uncertainty is between ~1% and 10%, with a 1% to 2% error most common.

The uncertainty in the relative humidity is approximately $\pm 2\%$ RH. When propagated through equation 1 to matric potential, the absolute uncertainties are fairly low, but the relative uncertainties are high at the matric potentials closest to zero.

The temperatures reported are the mean temperatures recorded during the time periods of the measurements. The highest statistical uncertainty (one standard deviation) is about $\pm 0.6^\circ\text{C}$, but is generally less than $\pm 0.3^\circ\text{C}$.

Results and Discussion

DST Samples

The samples of the DST used in the moisture retention curve measurements are listed in Table 1. Moisture retention curves of the DST samples at temperatures of 25.1°C , 49.6°C , and 93.7°C are shown in Figures 1 to 3 respectively. For temperature of 93.7°C only the wetting phase of the moisture retention curves are available now. The drying phase data will be available later. Only the 'average' properties are shown for clarity. The averages are the mean saturation and matrix potential of all samples. The error bars for saturation are the standard deviation from the average saturation at a matrix potential level of all samples. There is very little hysteresis observed between the wetting and drying curves. Previous measurements on similar samples showed significant hysteresis under certain conditions (Wilder, 1996). There is also very little difference between the 25.1 and 49.6°C curves. But the data at 93.7°C show significantly lower moisture retention than the lower temperature curves. For comparison, samples from USW-G4 (NTS borehole) showed a significant temperature dependence of moisture retention on temperature (Roberts and Lin, 1995).

Table 1. Drift Scale Test samples prepared for moisture retention experiments

Sample ID	Borehole	Depth (m)	Wet Density (g/cm ³)	Dry Density (g/cm ³)	Porosity
01002163-1	CHE-1	92.3 –93.2	2.371	2.286	0.0853
01002176-1	CHE-2	128.9 –129.6	2.322	2.211	0.111
01002179-1	CHE-5	39.4 –40.1	2.386	2.303	0.0831
01002189-1	CHE-6	43.4 –44.1	2.307	2.216	0.0914
01002190-1	CHE-6	63.9 – 64.6	2.375	2.292	0.0835
01002194-1	CHE-6	99.7 –100.4	2.330	2.224	0.106
01002199-2	CHE-7	51.9 – 52.6	2.301	2.174	0.127
01002200-1	CHE-7	80.0 –80.7	2.363	2.273	0.0895
01002206-2	CHE-10	15.7 –16.3	2.329	2.227	0.102
01002207-2	CHE-10	27.3 –28.0	2.400	2.311	0.0891
01002209-2	CHE-10	75.6 –76.3	2.379	2.254	0.125
01002212-1	CHE-10	91.6 –92.3	2.368	2.243	0.124
01002215-1	CHE-10	124.0–124.7	2.361	2.282	0.0796
Mean*		13 samples	2.35 $\pm$ 0.03	2.25 $\pm$ 0.04	0.100 $\pm$ 0.017

* Statistical mean for 13 samples; errors represent one standard deviation for all samples collectively

Figure 1. Matric potential versus average saturation for 13 DST samples at 25.1°C. Solid line indicates the wetting curve, and dashed line indicates drying curve. Point at 100% saturation is inferred.

Figure 2. Matric potential versus average saturation for 13 DST samples at 49.6°C. Solid line indicates the wetting curve, and dashed line indicates drying curve. Point at 100% saturation is inferred.

Figure 3. Matric potential versus average saturation for 13 DST samples in the wetting phase at 93.7°C. Point at 100% saturation is inferred.

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SHT Samples

The samples of the SHT used in the moisture retention curve measurements are listed in Table 2. Moisture retention curves of the SHT samples at temperatures of 25.1°C, 49.6°C, and 93.7°C are shown in Figures 4 to 6 respectively. For temperature of 93.7°C only the wetting phase of the moisture retention curves are available now. The drying phase data will be available later. Only the 'average' properties are shown for clarity. The averages are the mean saturation and matric potential of all samples. The error bars for saturation are the standard deviation from the average saturation at a matric potential level of all samples. Similar to the case for the DST samples there is neither hysteresis evident nor a notable temperature dependence. Also, stronger temperature dependence of the moisture retention curves is shown between 93.7°C data and those at the lower temperatures.

Table 2. Single Heater Test samples prepared for moisture retention experiments.

Sample ID	Borehole	Depth (m)	Wet Density (g/cm ³)	Dry Density (g/cm ³)	Porosity
0047525.2	CHE-1	2.20	2.348	2.247	0.102
0047525.2A	CHE-1	2.30	2.350	2.249	0.102
0047526.2	CHE-1	4.60	2.349	2.240	0.109
0047527.2	CHE-1	8.20	2.345	2.246	0.0998
0047528.2	CHE-1	12.5	2.331	2.224	0.107
0047529.2	CHE-1	14.0	2.344	2.235	0.109
0047530.2A	CHE-2	14.8	2.294	2.167	0.127
0047531.2	CHE-2	5.00	2.332	2.222	0.111
0047533.2	CHE-2	12.7	2.290	2.156	0.135
0047534.2	CHE-2	15.2	2.331	2.229	0.103
0047535.2	CHE-2	17.6	2.314	2.195	0.119
Mean*		11 samples	2.33±0.02	2.22±0.03	0.111±0.011

* Statistical mean for 11 samples; errors represent one standard deviation for all samples collectively

Figure 4. Matric potential versus average saturation for 11 SHT samples at 25.1°C. Solid line indicates the wetting curve, and dashed line indicates drying curve. Point at 100% saturation is inferred.

Figure 5. Matric potential versus average saturation for 11 SHT samples at 49.6°C. Solid line indicates the wetting curve, and dashed line indicates drying curve. Point at 100% saturation is inferred.

Figure 6. Matric potential versus average saturation for 11 SHT samples in the wetting phase at 93.7°C. Point at 100% saturation is inferred.

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The moisture retention curves are dependent on temperature. The effect is not significant between 25°C and 50°C, but seems to be significant between 50°C and 94°C. The temperature effect on the moisture retention curves is always to decrease the level of saturation at a matric potential level. It is true for both the DST and the SHT samples. The maximum saturation can be reached in those samples depends on temperature. At 25.1°C the maximum saturation achieved is ~30% for the DST samples and ~36% for the SHT samples. At 49.6°C this increases to ~40% for the DST samples and ~39% for the SHT samples. But at 93.7°C it decreases to about 28% for the DST samples and about 29% for the SHT samples. These saturations are higher than those reported for USW-G4 and LBT samples which were ~30% saturation at the highest RH.

Future work

The measurements on both DST and SHT samples will continue for the drying phase at 95°C. Measurements at a lower temperature will be repeated in order to assess the effect of higher temperature excursions on the repeatability of the previous (low temperature) measurements. A new set of measurements of liquid sorptivity is currently planned and samples are being prepared. These measurements will consist of one dimensional sorptivity measurements into cylinders of intact tuff as functions of temperature. The one dimensional sorptivity experiment will take place in an oven for temperatures below 100°C and in a high pressurized humidity chamber for temperatures greater than 100°C.

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