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Section I

Name/Title of Deliverable/Milestone: Thermal Conductivity of Crushed Salt

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Thermal Conductivity of Crushed Salt
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FIGURES

1 ABBREVIATIONS AND ACRONYMS

Cal Lab	SNL Primary Standards Calibration Laboratory
DAS	Data Acquisition System
DOE	Department of Energy
ES&H	Environmental Safety and Health
HA	Hazard Analysis
JSA	Job Safety Analysis
NQ	non-QA
PA	Performance Assessment
PHS	Primary Hazard Screening
PI	SNL Principal Investigator, or Designee
QA	Quality Assurance
QAPD	QA Program Document
SNL	Sandia National Laboratories
SP	Activity/Project Specific Procedure
US	United States
TCHM	Holometrix TCHM-LT thermal conductivity instrument
TRU	Transuranic
WIPP	Waste Isolation Pilot Plant

2 REVISION HISTORY

This is the original version of this Test Plan. Future revisions will be documented and appear in this section, as applicable. Changes to this Test Plan, other than those defined as editorial changes per Sandia National Laboratories, shall be reviewed and approved by the same organization that performed the original review and approval.

3 PURPOSE AND SCOPE

Reconsolidation of crushed salt is a very important physical phenomenon when backfilling or sealing nuclear waste repositories in salt is considered. There is a long history of testing crushed salt backfill for salt repository applications. Over the years, the mechanical properties pertaining to salt reconsolidation has been a topic of great interest to international salt repository studies as exemplified at symposia (for example: Aubertin and Hardy, 1996 and Wallner et al., 2007). A preponderance of these studies has been at room temperature, with a few tests at elevated temperatures up to 100°C. Today there is a renewed national and international interest in salt reconsolidation at elevated temperature, particularly as applied to disposal of heat-generating nuclear waste. This Test Plan puts forward the experimental procedure for a laboratory study to determine the thermal conductivity of reconsolidated crushed salt, emphasizing testing as a function of porosity.

The primary purpose of these experiments is to quantitatively evaluate the thermal conductivity of consolidated crushed salt as a function of porosity. A secondary purpose is to observe the potential temperature dependence of thermal conductivity of porous crushed salt. To successfully complete these experiments, challenging geomechanics techniques will have to be developed. The salt used in these experiments is “mine-run,” which means the aggregate was produced during normal mining operations. The planned laboratory studies are intended to provide data representing thermal conductivity behavior as a function of porosity and temperature up to 200^{o}C. The porosity and deformational processes produced during sample pressing will be determined by microscopic examination of the salt substructures.

The key objective is to collect thermal conductivity measurements of

reconsolidated salt as a function of porosity. Starting with loose salt with a porosity of about 40% we will cold press samples in a Teflon holder and/or die to successively lower porosities, and measure the thermal conductivity. Previous work, sample handling and test procedures are described below.

The work will be done to QA standards as delineated in Chapter 9 of this Test Plan. In all cases, this work will follow good scientific practice including: maintaining current instrument calibrations, following a documented procedure for sample preparation, and preservation of samples. The post-test microscopy methods are identified in Section 4.2.

4 EXPERIMENTAL PROCESS DESCRIPTION

This section describes the experimental processes required to study the thermal conductivity of crushed, natural (domal and bedded) and "salt lick" salt. Experiments are performed at temperatures and with porosities applicable to salt repository conditions.

4.1 Description of the Proposed Experiments

4.1.1 Relevance of earlier work

Considerable work has previously been done to determine the thermal conductivity of bedded and domal salt at a range of temperatures and pressures (e.g., Smith, 1976; Durham, et al., 1983; Durham, et al., 1984; Durham and Abey, 1981a; Durham and Abey, 1981b). The temperature regime of interest for these studies is generally from room temperature to as high as 500°C. Confining pressures ranging from ambient to 50 MPa are found to have little effect on thermal conductivity. Some researchers have also acknowledged effects from the composition of the salt, from pure NaCl to salt with up to 20% insoluble impurities (Durham et al., 1983; Sweet and McCreight, 1980, van den Broek, 1982). Other work has determined thermal conductivity for bedded salt, with no systematic focus on variables within the salt (e.g., Liu et al., 2011; Birch and Clark, 1940).

Little work has been done to determine the effect of variable porosity on salt thermal conductivity. Naturally occurring salt tends to have low porosity, from 0% for individual crystals to roughly 2–3% for polycrystalline samples. Limited work has been done on salt samples with higher porosity (Acton, 1977), but this work was not designed to systematically analyze the effect of porosity. It was instead focused on samples of variable grain and crystal size.

The most comprehensive approach is from Bechthold et al., (1999) and Bechthold et al., (2004), the combined data from which provide a fairly complete range of salt thermal conductivity values from 0–40% porosity. Our work intends to verify and augment these data by systematically measuring porosity effects in a single test series and at temperatures from 100–200°C. This will analyze the simultaneous dual controlling effects of porosity and temperature. This project does not currently focus on pressure effects, as salt thermal conductivity has been shown to be

independent of confining pressure (e.g., Durham, et al., 1983; Durham, Boro and Beiriger, 1984).

A generalized equation—the geometric mean of rock conductivity and pore conductivity—used to model the bulk thermal conductivity of a two-medium material is found in Ingebritsen et al. (2006, citing Sass et al., 1971):

$$K_b = K_r^{1-n} K_f^n \qquad (1)$$

where *K* is thermal conductivity, *b* is bulk material, *n* is porosity, and *r* and *f* denote rock and fluid, respectively. Inputting values for salt (rock) and air (pore fluid) at different temperatures (Table 1) creates an estimate of the range of values likely to be encountered in this work (Figure 1).

	25°C	50°C	100°C	150°C	200°C
K_{salt} (W/mk)	6.512	6.213	5.620	5.091	4.646
K_{air} (W/mk)	0.026	0.028	0.032	0.035	0.039

Table 1: Thermal conductivity values of salt and air used in the equation to model bulk two-medium thermal conductivity. Salt values come from Smith (1976); air values are calculated using the equation $k = (1.5207 \times 10^{-11} \times T^3) - (4.8574 \times 10^{-8} \times T^2) + (1.0184 \times 10^{-4} \times T) - 0.00039333$, where T is temperature in K. Equation (1) is uncorroborated but matches values published elsewhere.

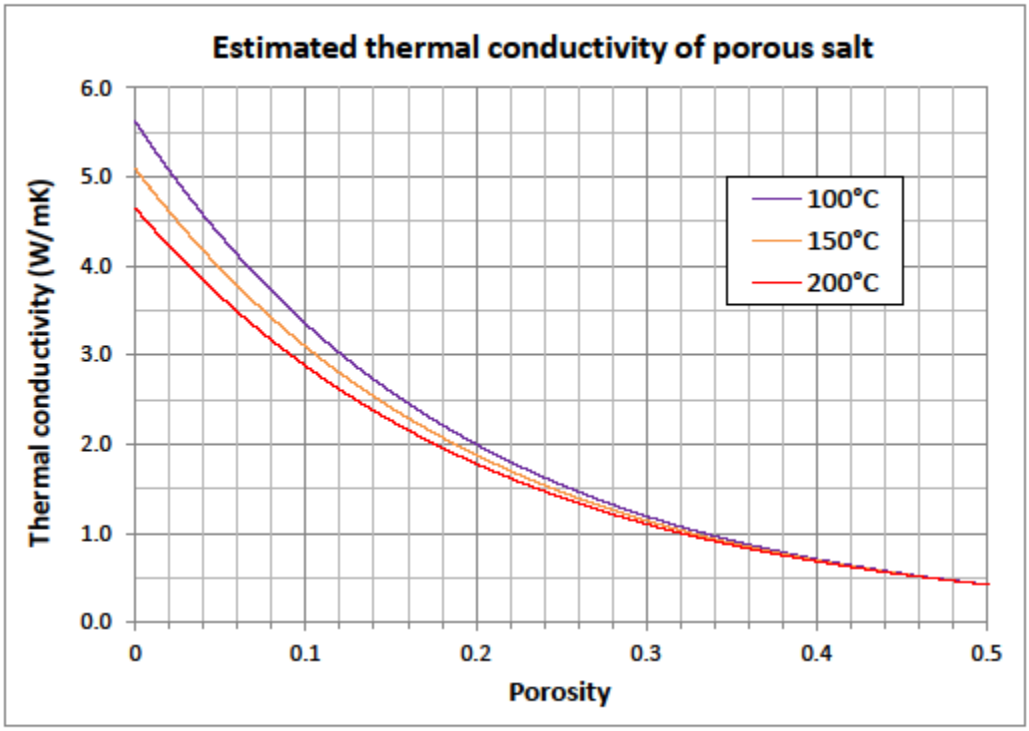

Figure 1: Estimated values of salt thermal conductivity based on a general two-medium mixing equation.

4.1.2 Overview of the Holometrix TCHM-LT thermal conductivity instrument

The Holometrix TCHM-LT thermal conductivity instrument (TCHM) is a guarded heat flow meter designed to measure thermal resistance of an unknown material. The guarded heat flow method measures and compares the temperature gradient across a sample to the flow of heat through the sample in order to calculate the thermal resistance of the sample. Knowing sample thickness, the system calculates the thermal resistivity of the sample; sample thickness is measured at test temperature. Thermal conductivity is the reciprocal of thermal resistivity.

To make these calculations, a sample is placed in the TCHM test stack and the stack is clamped shut by a load of 70 psi, provided by bottled nitrogen[b] (Figure 2). When the sample is clamped, its upper and lower surfaces contact, respectively, an upper heated plate and a heat flux transducer mounted above a lower heated plate (Figure 3). The temperature of each plate is controlled by an attached heater. The lower heater is always hotter than the upper heater, resulting in heat flow from the lower sample surface to the upper sample surface. The heat flux transducer

b Shop air is not recommended; see Appendix B for an explanation.

measures the amount of heat passing through the sample (Q).

Figure 2: From left to right: The test stack in the opened position, ready to be loaded; the test stack with sample loaded, ready to be clamped; the test stack clamped by bottled nitrogen (out of sight) at 70 psi.

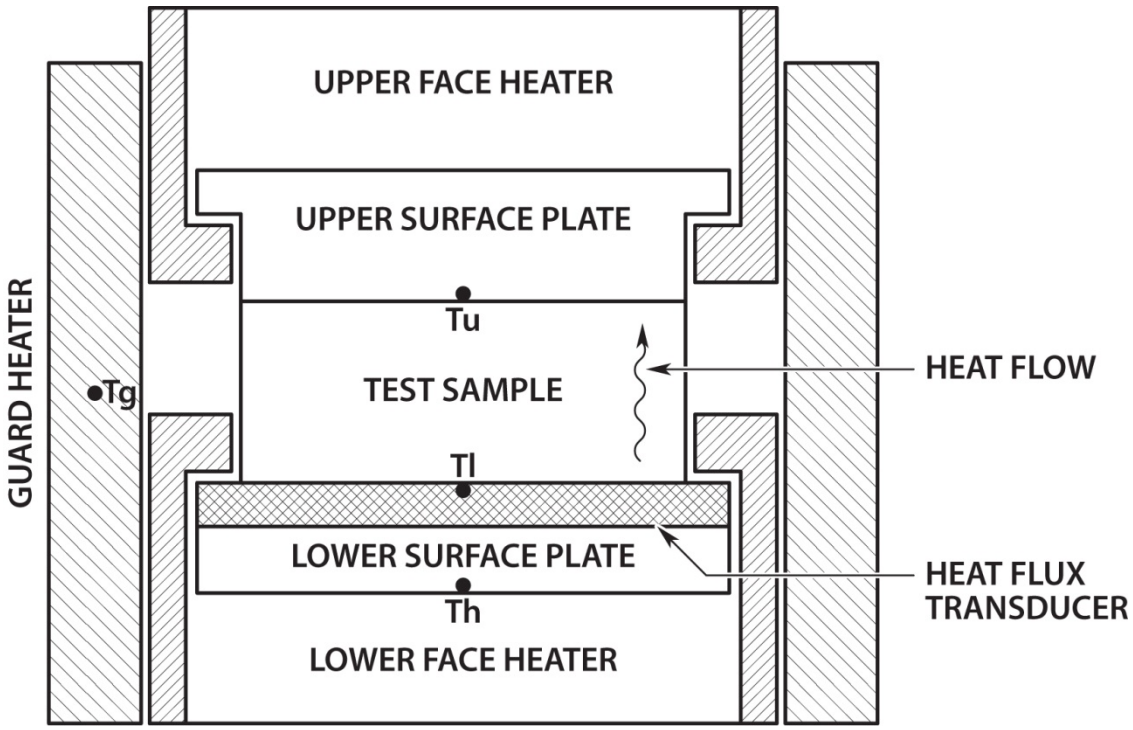

Figure 3: Schematic diagram of the TCHM test stack assembly. Note thermocouples at the upper (T_u) and lower sample surfaces (T_l), in the guard heater (T_g) and at the lower heater (T_h). The lower heater is the reference point by which the upper and guard heaters maintain a constant temperature difference.

Thermocouples record temperature at the lower heater (T_h); the lower sample

surface, or interface of the sample with the heat flux transducer (T_l); and the upper sample surface, or interface of the sample with the upper plate (T_u). A differential controller uses feedback from these thermocouples to maintain a fixed temperature difference across the test stack (T_h-T_u) of 25–30°C. As temperature in the lower heater increases or decreases, so does temperature in the upper heater.

The heater-plate-sample assembly is surrounded by a cylindrical guard heater to minimize heat loss from lateral heat transfer (Figure 3). A thermocouple (T_g) is embedded in the guard heater. A differential controller uses feedback from this thermocouple and another in the lower heater to maintain a fixed temperature difference between the lower and guard heaters (T_h-T_g) of 15–20°C. The guard temperature is maintained at roughly the mean sample temperature.

To calculate thermal conductivity, the TCHM measures thermal resistance of the sample using the equation

$$R_s = N\left(\frac{T_l - T_u}{Q}\right) - R_o = \frac{d}{K}$$

Where R_s is sample thermal resistance, *N* is a proportionality constant, *Q* is heat flow, R_o is the sum of contact thermal resistances within the system, and *d* is sample thickness. Dividing by *d* removes the thickness term and gives sample thermal resistivity, which is the reciprocal of sample thermal conductivity. The system calibration solves for *N* and R_o, which are constants for any given method of assembling the sample. During testing, the TCHM determines the ratio T_l-T_u/Q, or ΔT/Q, for every specified temperature set point.

TCA200 software (TCA) controls the system and records data. The TCA records temperature and heat flow every 60 seconds. The average ΔT/Q ratio is recorded after every 10 scans. After 2 blocks of 10 scans, the TCA checks that all ratios within the second block are within 1% of each other. If they are, then the sample is at equilibrium and the system can move on to the next temperature set point. If not, then the TCA checks that the average ratio of the second block is within 1% of the average ratio of the first block. If it is, then the sample is at equilibrium and the system can continue to the next temperature set point. If not, then additional blocks of 10 scans will be performed until the sample reaches equilibrium.

The TCHM can reach a maximum sample temperature of 200°C. A circulating water bath provides liquid-cooled heat sinks near the upper and lower heaters

(Figure 4) to protect the TCHM from heat damage as well as enabling a lower minimum test temperature. Using tap water, the minimum sample temperature is 40°C, although this can be reduced to -100°C by using other coolant liquids such as liquid nitrogen. The TCHM can measure thermal conductivities from 0.1–10 W/mK, or a thermal resistance range of 0.001–0.050 m^2K/W. The estimated accuracy is 2–5% when sample thermal resistance is greater than 0.005 m^2K/W and 5–10% when thermal resistance is less than 0.005 m^2K/W. We therefore ascribe an accuracy of ~5% to thermal resistance measurements with better accuracy at higher resistance and/or thickness.

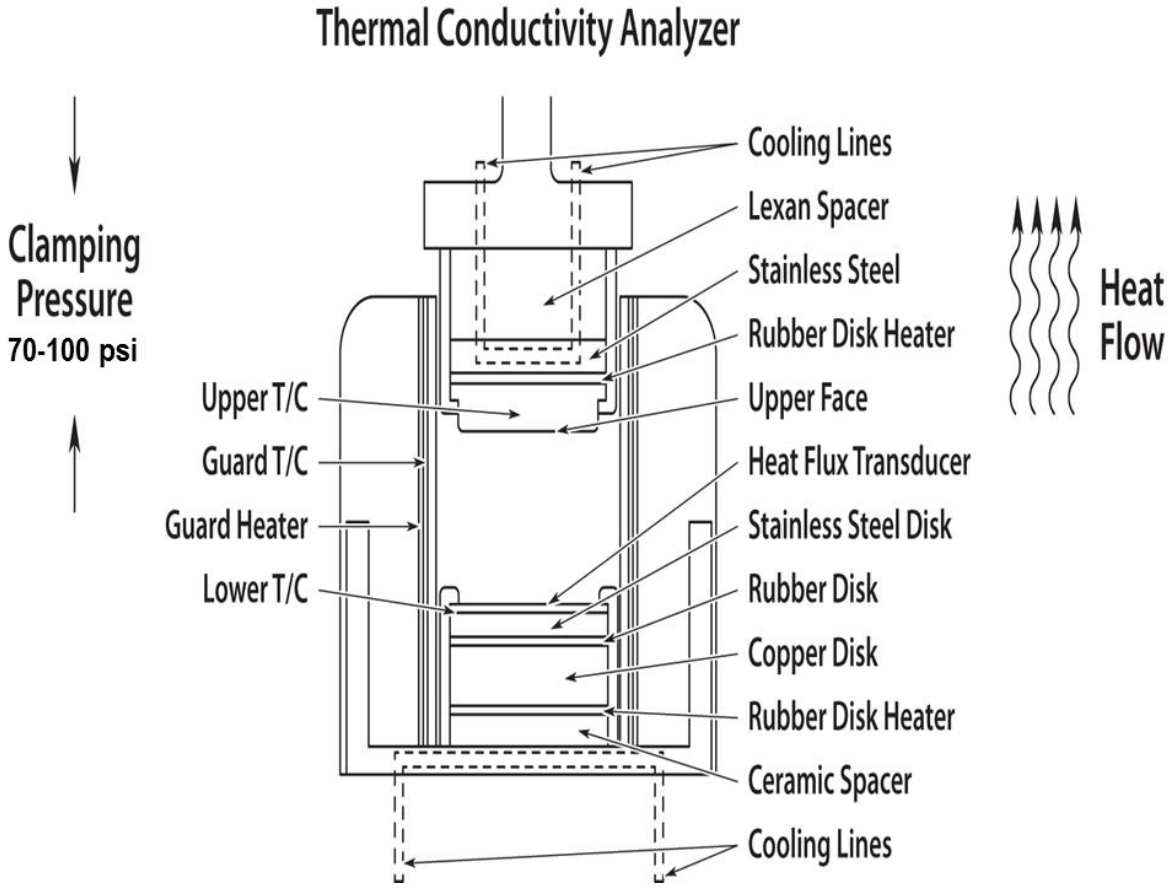

Figure 4: Detailed schematic of the TCHM test stack. Note cooling lines directed past the upper and lower heaters.

4.1.3 System calibration

Calibration of the TCHM is essential for accurate data analyses. The calibration

solves for the constants N and R_o in the equation from Section 4.1.2. This enables sample assemblies to take any form necessary by predetermining, over a range of thermal resistances, the built-in thermal resistance unique to that setup. Because the process assesses built-in factors affecting thermal resistivity over the entire system, the TCHM must be recalibrated whenever:

- there is a change in the compressive load on the test stack;
- there is a change in the temperature difference between the upper/lower or guard/lower heaters (see Section 4.1.2);
- the heat flux transducer is replaced;
- or the method of sample assembly is changed.

Calibration of the TCHM can take up to a week. The calibration process measures the thermal resistivity of a known material—either fused quartz or Pyrex—over a range of sample resistances estimated to be appropriate for the materials later to be tested as determined by a literature survey. The TCA then verifies the calibration by using the calibration to determine the thermal conductivity of an "unknown" known material, i.e. separate calibration standard—either Pyrex or fused quartz, whichever was not used at first. If the results are within acceptable error (nominally 5%) of the pre-known thermal conductivity value, then the calibration has been successful.

If samples have resistance greater or lower than the extremes of the calibration, the TCA will project from the calibration in order to estimate N and R_o at higher or lower resistivities (Figure 5). This is done by matching a linear or quadratic equation, as necessary, to the slope of calibration data. Ideally, however, the calibration will cover the full range of resistivities necessary.

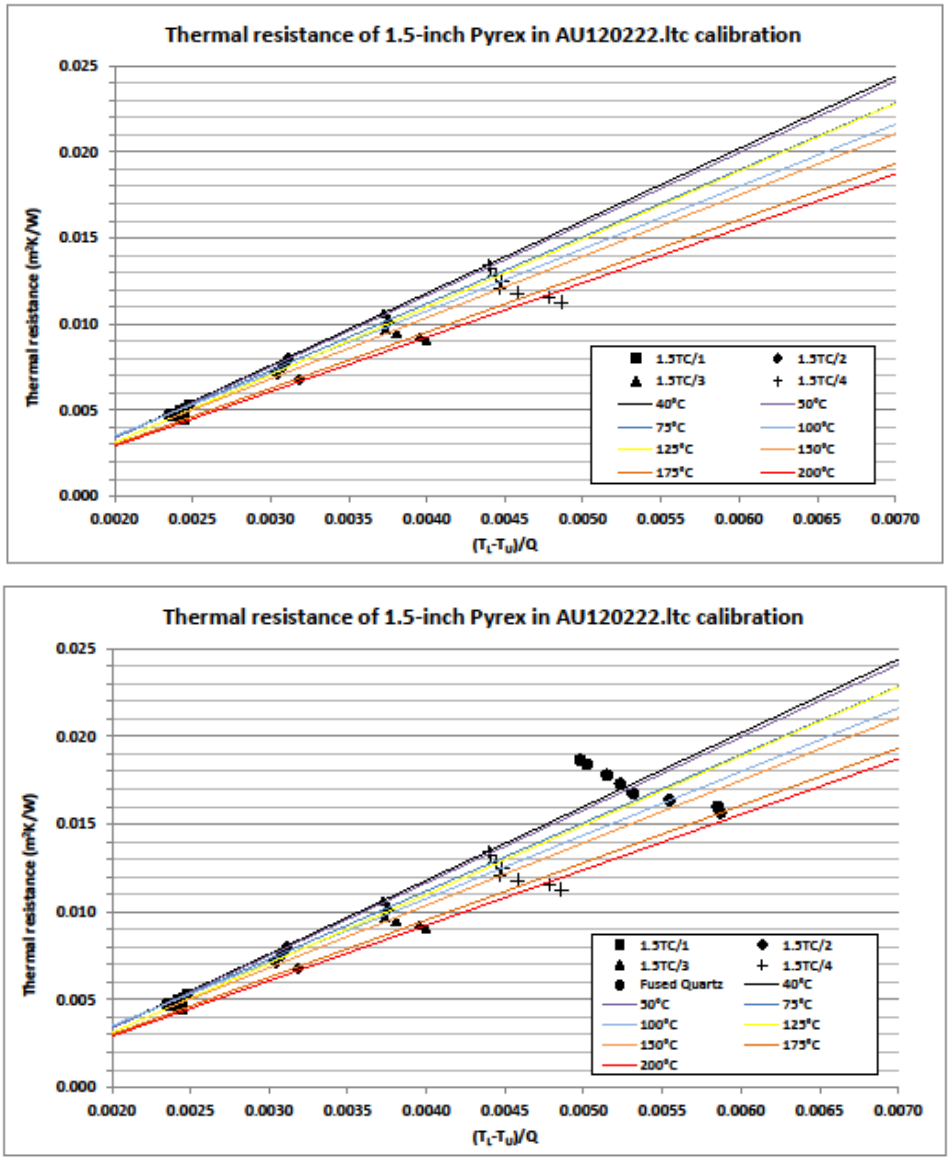

Figure 5: A) Sample resistance data from a calibration run of 4 Pyrex samples at 8 temperatures from 40°C to 200°C; B) a fused quartz verification sample has higher resistance than any of the Pyrex calibration samples, but its resistance can be estimated by projecting from the calibration data.

For an effective calibration run, at least three samples (fused quartz or Pyrex) of different thickness must each be tested over the full temperature range of interest and using the intended sample assembly. The different thicknesses will define the calibration's upper and lower resistances, so it is helpful to choose samples from the full range of acceptable thickness (~4–20 mm). The calibration temperatures

(mean sample temperature, represented by the guard temperature T_g) should be in increments of 25°C or 50°C to develop a well constrained calibration curve.

A simple calibration might run from 50°C to 200°C and include 3 fused quartz samples with thicknesses 5 mm, 10 mm and 20 mm. For each of these samples, a calibration test could be performed with temperature set points at 50°C, 100°C, 150°C, and 200°C. After performing these tests, a verification run could be performed on a 10-mm-thick Pyrex sample. The verification run will calculate the thermal conductivity of Pyrex at each temperature set point and compare it to known values at that temperature.

Calibrations are used to account for the thermal resistivity of other materials in the sample assembly (Figure 4).

The system calibration used for this work consists of seven 38.1-mm-diameter fused quartz disks assembled in the sample setup described in Section 4.1.5. These disks have thicknesses of 5 mm, 6 mm, 9 mm, 12 mm, 15 mm, 18 mm, and 20 mm. Each fused quartz sample is measured in increments of 25°C from 50°C to 200°C. The verification test is done with a 19-mm-thick, 38.1-mm-diameter Pyrex sample assembled in the same way.

4.1.4 Preparation of salt

Sample material includes loose mine-run (crushed) salt from the Waste Isolation Pilot Plant (Figure 6a); cohesive domal salt (Figure 6b); and cohesive “salt lick” (Figure 6b). The bulk of testing is performed using the mine-run salt, which can be easily compacted into samples of variable porosity, thickness, and grain size. Domal and salt lick salt have low porosity (< 3%). They are useful for completing the low-porosity region of the thermal conductivity curve, providing an additional check on low porosity measurements. The domal and salt lick salt used are relatively pure NaCl, and contain only a small fraction of the impurities seen in the mine-run salt.

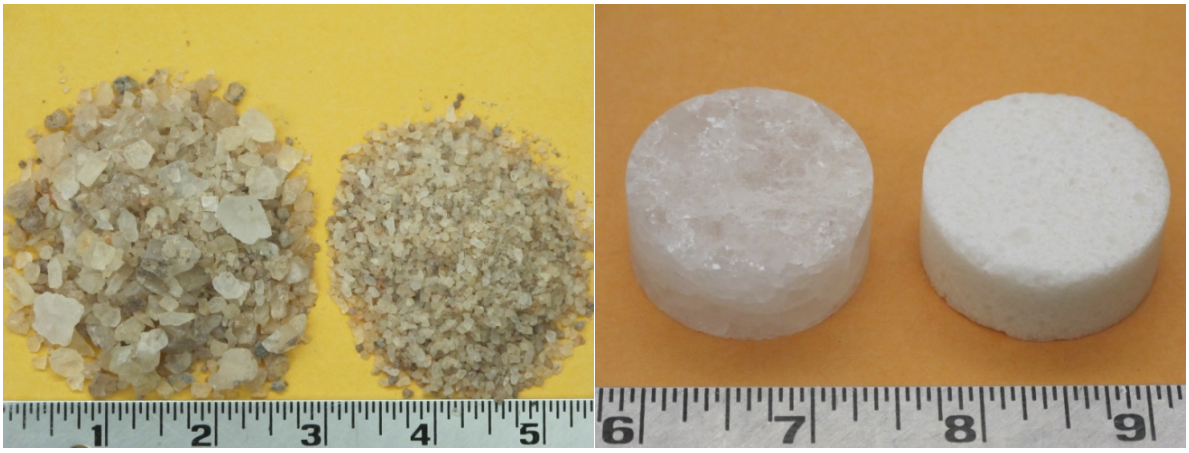

Figure 6: A) Non-sieved (left) and sieved (right) mine-run crushed salt used to make samples of variable porosity; B) domal salt (left) and salt lick salt (right) 38.1-mm cores used to obtain thermal conductivity measurements at low porosities. Scale in inches.

The salt is dried and stored at 100°C for at least three days prior to measurement. Experience developed herein has shown that this is sufficient time to dry WIPP salt of adsorbed water, and this gives this salt a similar starting material condition to salt tested mechanically. The salt loses 0.1–0.2% of its mass in the first 1–3 days, after which there is no noticeable or consistent change in mass.

As all tests will be performed at 100°C or higher and samples will be heated to test temperatures before data collection begins, moisture content is expected to remain low in samples during testing. The test series will take several months, so in order to restrict re-adsorption of moisture onto the salt, it will be stored in the oven at 80–100°C until it can be tested. Immediately before testing a sample, the salt will be removed from the oven, loaded into a Teflon ring, and placed into the thermal conductivity apparatus as described in section 4.1.5. The apparatus heaters will be turned on and brought to the test temperature immediately to prevent the sample from remaining at ambient temperatures for long. If sample material needs to be removed from the oven for extended periods of time, it will be stored in an airtight storage container with desiccant packs and it will be replaced in the oven for at least a day before testing. Plastic bags are insufficient to keep the salt dry, as most plastic bagging material is relatively permeable to atmospheric moisture.

4.1.5 Sample preparation

After salt has been sieved and dried at 100°C for several days, specimens are assembled as right circular cylinders with a diameter of 50.8 mm, a minimum

height of 5 mm, and a maximum height of 20 mm. The diameter and range of heights are limited by the maximum and minimum sample sizes for which the TCHM can effectively measure sample resistivity. Samples may be composed of either mixed or uniform grain size, provided the 10:1 ratio of sample size to grain size is maintained. Sample ends should be smooth, flat, and parallel.

Because many specimens are composed of loosely compacted mine-run salt, Teflon rings are used to contain the material. The Teflon has an inner diameter of 38.1 mm and an outer diameter of 50.8 mm, with variable heights from 5 to 20 mm. The Teflon ring is capped on the bottom by copper foil conforming to the outer diameter of the ring, and on top by a 1.25-mm-thick aluminum disk matching the inner diameter of the Teflon (Figure 7). These caps contain the salt within the Teflon, protect the TCHM from corrosion, and help create smooth and parallel ends. The aluminum disk is thick in order to allow testing of samples that are slightly shorter than the Teflon ring by bridging the gap between the sample and the upper plate, which contacts the sample from above (Figure 3). Although the domal and solar salt samples are cohesive and do not require containment, the same setup is used in order to maintain consistent sample size and preparation, and in order to use a common calibration file for all samples.

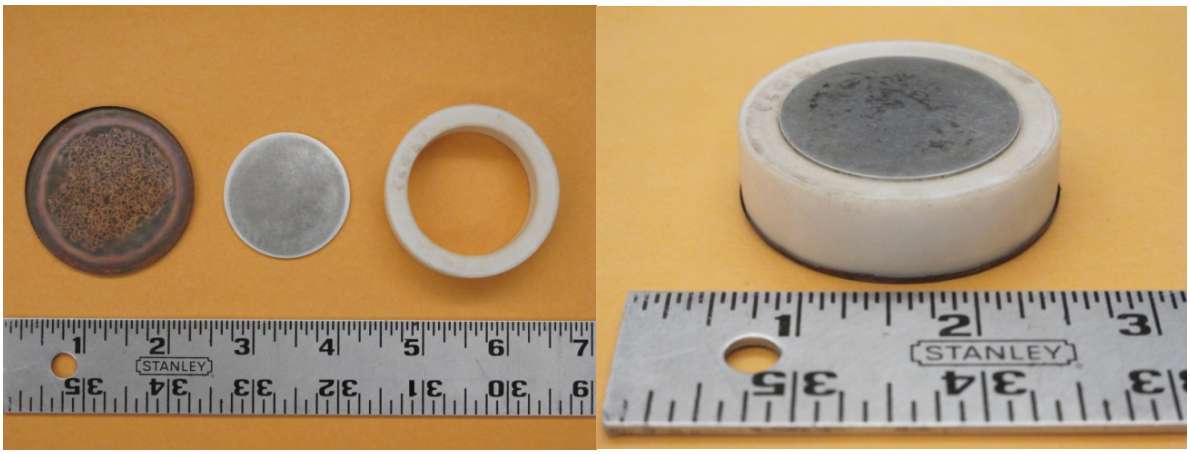

Figure 7: A) Components of the sample holder, from left to right: copper foil base, aluminum top cap, Teflon ring; B) the assembly ready for testing. Scale in inches.

The TCHM manual states that ideal sample thickness should follow the guideline $40\lambda > d > 5\lambda$, where d is sample thickness in mm, and λ is estimated sample thermal conductivity in W/mK. Because thermal conductivity is affected by porosity, and the samples used in this work have porosities from 0–40%, no one

sample thickness satisfies this guideline. Nonetheless, in order to keep sample preparation consistent, we will vary porosity and maintain a common sample thickness throughout the suite of tests. Sample thickness will begin at 16 mm, which most closely satisfies the guidelines based on the modeled values in Figure 1 while minimizing the amount of necessary calibration projection by maintaining a low maximum estimated thermal resistance.

For a sample of given thickness, the volume of the Teflon chamber is calculated and the volume of salt necessary to achieve the desired porosity is then calculated as a fraction of the chamber volume. This salt volume is used to determine the required salt mass by using a mine-run salt density of 2.140 g/cc—the average of 26 measurements of finely crushed mine-run salt displacement of isopropanol (Table 2). (We note that the salt density determined herein differs slightly from that reported [2.18 g/cc] in Hansen et al, 2003).The required salt mass is then tamped or die-compacted to the specified sample thickness. For samples with relatively high porosity (25–50%), specimens can be manually assembled by pouring grains into the Teflon disk and gently tamping them down. For samples with lower porosities (5–25%), specimens are die-packed at room temperature: Salt is poured into the die and compacted to the desired height over a period of 5 minutes. The sample is then allowed to sit for 10 minutes to allow the salt to plastically deform and prevent expansion when the load is released. Confirmation of these porosities will be obtained through optical microscopy of epoxy-impregnated samples.

Sifted WIPP mine-run salt	
Grain size (mm)	Density (g/mL)
0.25	2.131
0.25	2.066
0.25	2.076
0.59	2.204
0.59	2.160
0.59	2.157
1.00	2.159
1.00	2.118
1.00	2.122
1.41	2.131
1.41	2.144
2.00	2.148
2.00	2.143
2.36	2.135
2.36	2.138
2.79	2.155
2.79	2.130
3.35	2.146
3.35	2.155
3.99	2.144
3.99	2.128
3.99	2.155
4.75	2.145
4.75	2.149
6.35	2.153
6.35	2.148
Average	2.140

Table 2: Density of Waste Isolation Pilot Plant mine-run crushed salt, determined by displacement of isopropanol with finely crushed salt.

The TCHM instruction manual recommends using a paste (e.g., Dow Corning Heat Sink Compound 340) to reduce contact thermal resistances where the sample contacts the upper and lower plates. Our work suggests that this paste is unnecessary and because the paste is difficult to remove from the components of the TCHM, we avoid its use.

4.1.6 Test procedure

Once the sample has been assembled, it is placed it in the test chamber and the test stack is clamped. The heater temperature (T_h) is manually raised to the point at which the sample will be at test temperature (Table 3). (Sample temperature is represented by T_g and is always lower than T_h, as described in Section 4.1.2.) The heater heats and cools at about 1°C per minute, so this step may take an hour or more and drives off moisture re-adsorbed during the sample preparation phase.

Heater Temperature (°C)	Sample Temperature (°C)
121	100
144	125
171	150
196	175
221	200

Table 3: The test temperature is represented by the guard temperature (T_g), which is maintained at the average mean sample temperature. The heater temperature (T_h) is always higher than the sample temperature.

Because porosity measurements are based on sample volume, and in order to calculate accurate thermal conductivity values, it is important to know the actual sample thickness during testing. The sample will be clamped at 70 psi axial pressure, which may compact the low-porosity, loose-salt samples. It will also be heated to as much as 200°C, which will cause it to expand.

Measurable change in thickness due to heating can be seen using a micrometer to monitor movement of the TCHM stack height (Figure 8). As indicated in the figure, when sample height increases due to thermal expansion, the stack height decreases. It is therefore important to maintain a single temperature for each test, as only one sample thickness can be entered for each test. This method has shown thickness to increase by as much as 4.1% of original sample height over 154°C. In another instance, the stack height increased by 3.4% when the sample was heated from 100°C to 200°C. The coefficients of thermal expansion, α, for NaCl at 100°C and 200°C are about 1.25×10^{-4} K^{-1} and 1.35×10^{-4} K^{-1}, respectively (e.g., Kumar, 1994; Enck and Dommel, 1965). Using an average value of 1.30×10^{-4} K^{-1} (the increase in α from 100°C to 200°C is nearly linear), about a third of this increase

can be attributed to salt expansion, or a 1.3% increase in total sample height. The additional expansion is attributed to expansion in other components of the test stack. A comparison of thermal conductivity calculated from the original sample height and the at-temperature sample height shows a difference of up to 7% (Figure 9).

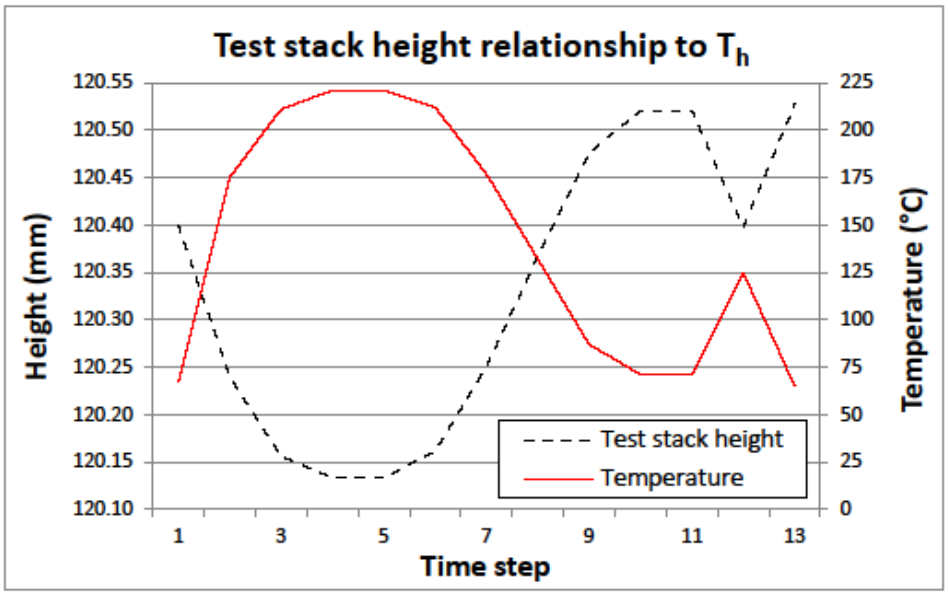

Figure 8: The height of the test stack changes noticeably with changes in sample temperature.

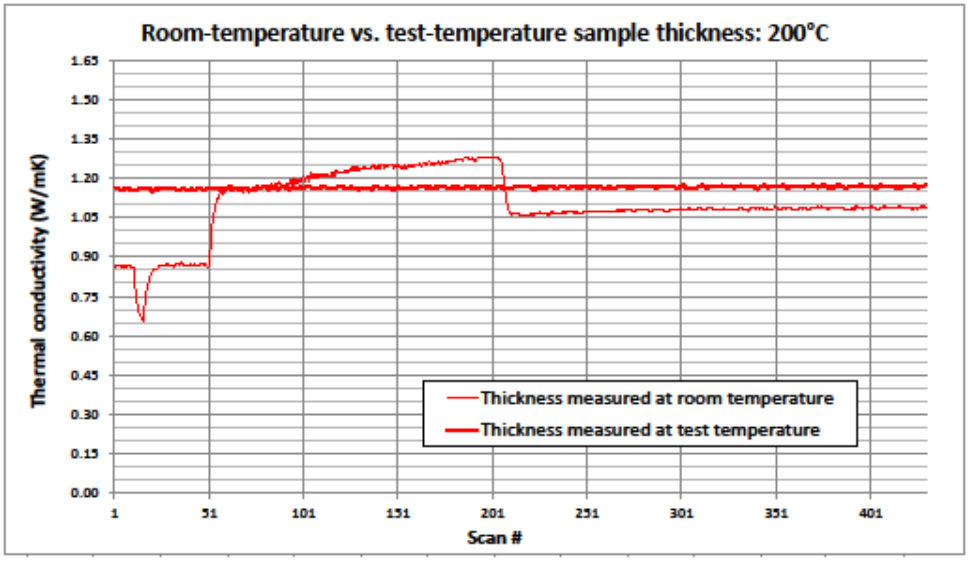

Figure 9: The difference in thermal conductivity measurements for the same sample with thickness measured at room temperature and at test temperature.

Because at-temperature thickness measurements increase the accuracy of porosity estimates and thermal conductivity calculations, the sample thickness is not measured until it reaches test temperature. This is done indirectly by measuring the height of the test stack with a caliper. Caliper height measurements of the test stack when loaded with relatively incompressible samples of known thickness follow a linear trend, with sample thickness equal to 138.91 – 1.0036 × C, where *C* is the caliper measurement in mm (Figure 10). The R^2 value of this equation is 1.00.

When the thickness is measured, it is input into the TCA control program, along with the test temperatures, and the test can begin. The temperature range of interest is 100–200°C. We will test each sample in separate test runs at 100°C, 150°C, and 200°C. The TCA allows for analyses at multiple temperatures during a single test run, but we will test only one temperature at a time due to the change in sample thickness with temperature—the TCA records only one sample thickness for each test run, so it is unable to adjust its calculations in response to changes in sample thickness.

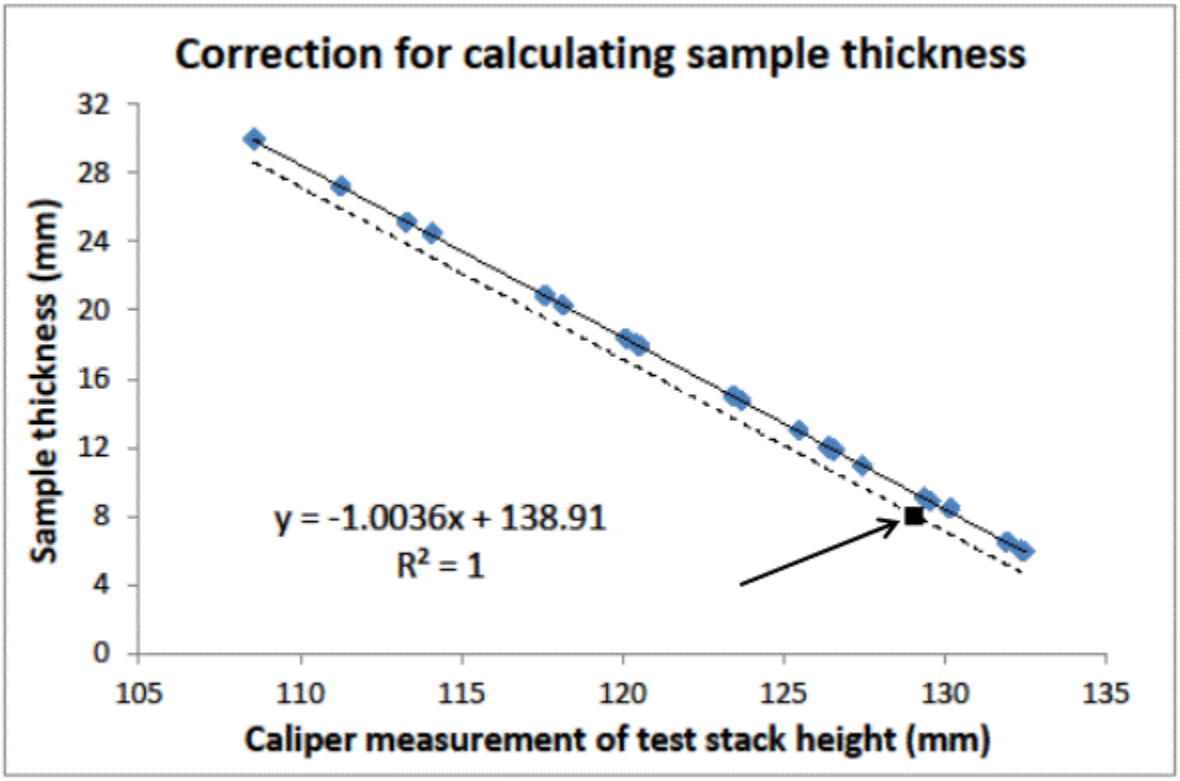

Figure 10: Test stack height has a direct relationship to sample height. The offset dashed line accounts for the thickness of the copper base plate and aluminum cap. The black square represents a stack height of 129.08 mm, or sample thickness of 8.05 mm.

Consecutive thermal conductivity calculations at the same temperature show less than ±0.5% difference. The TCA will make five measurements on each sample at each temperature in order to average these slight inconsistencies. Such a test will take 3–4 hours, but the TCHM does not need to be attended once a test has begun. At least 30 tests will be run in order to establish the thermal conductivity of salt as a function of porosity from 0% to about 40%. This is deemed adequate to achieve the data quality objective. Based on technical judgment of the PI, this number of tests should provide adequate definition via statistical averages. The samples for these tests will be of roughly equal thickness and grain size, as described in Section 4.1.5.

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4.2 Observational Microscopy

Post-test microscopy will help determine the actual porosity of salt samples used in this work. During testing, the porosity of each sample is calculated indirectly from the volume, mass and density of the salt. It is important to assess the accuracy of these porosity estimates. This will be done by point-counting thin sections made from samples impregnated with blue- or rhodamine-dyed epoxy. Scanning electron microscopy may augment optical microscopy in order to do analysis at high magnification. Energy dispersive spectroscopy could be also used to identify other mineral types entrained in the salt.

Sandia has existing activity- and project-specific procedures (SPs) that are available online at http://www.nwmp.sandia.gov/onlinedocuments. For the experiments in this Test Plan we intend to use the scanning electron microscope (SEM) and the optical microscope, which involve the following SPs:

- SP 12-11 Fischer Scientific Stereomaster Microscope
- SP 12-13 Olympus BX-60 Microscope
- SP12-17 Scanning Electron Microscope Imaging and Energy Dispersive Spectroscopy

For analysis of crushed salt samples, we will follow the procedures described in the SPs. We will document the results and process in a logbook (not an official scientific notebook). The procedures identified in the above SPs are not dependent on the specific scanning electron microscope or the brand name of the optical scope, which happen to be included in the SP title. Use of a logbook will provide results as Non-Q. This strategy will be reviewed as work progresses.

4.3 Modifications to the Experimental Process

Modifications to the test procedures outlined in Section 4.1 may be required during as testing proceeds. These modifications will be enacted at the direction of the PI, and will be documented in an official scientific notebook as part of QA records. Such modifications are not deviations and will not be reported as non-conformances requiring corrective action.

If test conditions deviate appreciably from the anticipated execution of the current version of this Test Plan, the Test Plan will be revised.

5 TEST EQUIPMENT

This Test Plan requires equipment to dry mine-run salt at 100°C; compact salt at loads on the order of 20,000 pounds; and measure the temperature and thermal resistance of a sample heated to 200°C. The equipment may consist of “off the shelf” items ordered directly from suppliers, standard equipment provided by service companies, and/or custom-built equipment designed and built for a specific task governed by the Test Plan. All equipment operators will follow the supplier’s/designer’s operation and calibration recommendations (as required). All equipment with calibration requirements and quality-affecting operations will be documented as part of the QA records and controlled by FCT QAP 12-1 (see Subsection 9.4).

6 DATA-ACQUISITION PLAN

Both manually- and electronically-collected data will be acquired during the test activities. The following types of data may be recorded:

- data from the DAS
- manually collected test data.

6.1 Scientific Notebook(s)

A scientific notebook(s) will be used in accordance with FCT QAP 20-2 (see Subsection 9.4) to document all activities and decisions during the Test Plan (except as noted in Section 4.1). Specific information that may be entered in the scientific notebook(s) consists of:

- a statement of the objectives and description of work to be performed, as well as a reference to this Test Plan;
- a written account of all activities associated with the development and implementation of the mine-by test;
- documentation of safety meetings;
- a list of equipment used during each activity, including make, model, and operating system (if applicable);
- traceable references to calibration information for instruments and/or gauges calibrated elsewhere; and
- discussions of the information and/or observations leading to decisions to initiate, terminate, or modify test activities.

All entries in the scientific notebook(s) will be signed and dated by the person making the entry. The scientific notebook(s) for this Test Plan will be reviewed by an independent, technically-qualified individual at a minimum of every six months to verify that sufficient detail has been recorded to retrace the activities and confirm the results.

Manually collected data may also be recorded on specially prepared forms rather than in the scientific notebook(s) when that process will provide a more efficient means of data collection and tracking. In particular, a standard geomechanics test data form should be prepared and completed as each test is carried out. These will be included in a supplementary binder to the scientific notebook.

6.2 Electronic Data Acquisition

The DAS will be used to record instrumentation data during the test. Electronic data file-management information will be documented in the scientific notebook(s) for these activities.

6.3 Manual Data Acquisition

Manual data collection will be carried out during the test using a scientific notebook(s) or forms designed specifically for each activity or data type. Information will be documented such that duplication of information will be minimized. The PI will determine the means of documenting manually-acquired data and will ensure that all quality-affecting information is documented.

6.4 On-Site Validation

During the test activities, the PI will evaluate the data, as they are acquired. The data will be diagnosed for any equipment failure and/or procedure-induced effect that may degrade the data quality. The PI will take immediate action (if required) to make any necessary changes to the equipment configuration or the procedures to assure the data quality is consistent with the objectives of these activities.

The PI will use real-time evaluation of the acquired data during test activity to assure that the data are usable in a detailed interpretation, the conditions can be maintained over the planned duration of the activity, and an activity will not be terminated before the minimum objectives can be achieved under the given time restraints. The PI may utilize some or all of the following procedures and analytical tools:

- real-time inspection of signal quality to assure useable data

- real-time analysis of the acquired data to assess transducer functioning and proper operation of the DAS; and

If at any time the PI determines that a test activity objective cannot be accomplished due to time constraints, problems concerning the performance of the equipment, or unsuitability of initial conditions, the PI will consult with cognizant personnel to terminate the activity, or develop a recovery plan. The PI will document all real-time evaluation of data and conditions in the scientific notebook(s).

7 SAMPLING AND SAMPLE CONTROL

Crushed salt samples will be prepared under this Test Plan using mine-run salt. Following preparation and testing, the samples may be placed in on-site inventory or transported to other locations for further characterization, e.g., optical microscopy. Sample handling and transport will be controlled following requirements in FCT QAP 13-1 *Control of Samples.*

8 TRAINING

All personnel who will perform quality-affecting activities under this Test Plan will have training in the SNL QA program and relevant procedures according to FCT QAP 2-1 *Qualification and Training.*

9 QUALITY ASSURANCE

9.1 Quality-Affecting Activities

Activities performed under this Test Plan are quality affecting, except for those specifically noted in section 4.1. The intent of the data and observations made in these studies are expected to be used in design considerations and repository performance expectations.

9.2 Quality Assurance Program Description

Activities are conducted in accordance with the requirements specified in the FCT Quality Assurance Program Document. A complete discussion of this integration is given in Appendix A.

9.3 QA Procedures

The QAPs and SPs that may apply to work performed under this Test Plan include:
FCT **QAP 2-1** Qualification and Training
FCT **QAP 5-1** Implementing Procedures
FCT **QAP 6-1** Document Review Process
FCT **QAP 6-2** Document Control
FCT **QAP 9-1** Analysis
FCT **QAP 13-1** Control of Samples and Standards
FCT **SP 13-1** Chain of Custody
FCT **QAP 20-1** Test Plans
FCT **QAP 20-2** Scientific Notebooks

Modification to these procedures may be required during testing activities. Such modifications are not deviations and will not be reported as non-conformances that require corrective action. However, the PI will document modifications in the scientific notebook(s) as they occur as part of the QA records.

9.4 Manufacturers QA Procedures

Manufacturers' QA procedures that may apply to work performed under this Test Plan:

None.

9.5 Data Integrity

Care will be taken throughout the performance of the operations for this Test Plan to ensure the integrity of all data collected including documentation on hard copy and data collected on storage media. Duplicate copies of all data will be produced as quickly as possible and the duplicate copies will be maintained at a location separate from the test site to ensure that data are not lost.

9.6 Records

Records will be maintained as described in this Test Plan and applicable FCT QA implementing procedures. These records may consist of bound scientific notebook(s), loose-leaf pages, forms, printouts, or information stored on storage media. The PI or designee will ensure that the required records are maintained and submitted to the designated storage location.

9.6.1 Required QA Records

As a minimum, QA records will include:

- Scientific notebook(s);
- Calibration records for all controlled equipment;
- Equipment-specification sheets or information (if available);
- All forms containing manually-collected data;
- Standard geomechanics test forms, fully completed
- Chain of Custody Forms

9.6.2 Miscellaneous Non-QA Records

Additional records that are useful in documenting the history of the activities, but are considered non-QA records may be maintained and submitted to the SNL FCT Quality Assurance SharePoint Site or EIMS FileNet. . These records include:

- safety briefings;

- ES&H documentation;
- as-built diagrams of equipment
- equipment manuals and specifications;
- equipment manifests; and

These records do not support regulatory compliance and, therefore, are not quality-affecting information.

9.6.3 Submittal of Records

QA records generated through the implementation of this Test Plan shall be prepared and submitted to the SNL FCT Quality Assurance SharePoint site and submitted to EIMS Filenet in accordance with the SNL Records Management Manual and IM 100.2.2 Control of Records (manage and Protect Information). EIMS Filenet in accordance with the SNL Records Management Manual and IM 100.2.2 Control of Records.

10 HEALTH AND SAFETY

The safety practices and policies will meet the requirements of the SNL ES&H Manual. Operational safety will be addressed through an ES&H Primary Hazard Screening (PHS), a Hazard Analysis (HA), and a Pressure Safety Data Package (if required) developed by SNL. Work planning and controls will be implemented and records will be maintained by and in accordance with Division 6000 corporate policy. For example a specific JSA was developed for the lead jacket handling.

11 PERMITTING/LICENSING

There are no special licenses or permitting requirements for the work described in this Test Plan.

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Appendix A - SDI Test Activities Quality Assurance

Organization

Sandia National Laboratories' organization is fully described on the Sandia Internal Website (Techweb). The activities described in this Test Plan are the responsibility of Organization 06914 (Geomechanics), within SNL's Geoscience, Climate, and Consequence Effects Center (06900). Figure A-1 also shows the QA organizational interfaces for the activity.

This R&D activity, managed as Work Package FT-12SN081801, is conducted by Sandia National Laboratories under contract to U.S. DOE as part of the DOE-NE Fuel Cycle Technologies (FCT) program Used Fuel Disposition (UFD) Campaign. As an FCT UFD R&D activity, it is conducted in accordance with the FCT QAPD[2], SNL's DOE approved QA Program Description (SNL-QAPD)[3] and SNL's UFDC Preliminary Quality Assurance Implementation Plan[4] (SNL-UFCD-QAIP). Management decided to augment basic requirements for this QRL3 activity, based on the potential utility of the results. Hence, this Appendix to the activity Test Plan is provided in compliance with the provisions of SNL-UFCD-QAIP Section 4.

Quality Assurance Program

To summarize the minimum requirements for this QRL3 activity, as described in SNL-UFCD-QAIP, the activity is to be conducted in compliance with the SNL-QAPD, with the additional requirement that deliverables receive a technical review in accordance with the FCT QAPD Appendix B.

Management decided that certain quality assurance improvements would be beneficial to the conduct of this activity. A meeting was held on January 6, 2011, involving Organization 06914 management and technical staff, and the FCT QA POC to grade the quality assurance requirements to apply the Consolidation of Crushed Salt at Temperatures up to 250^{o}C under Hydrostatic and Shear Stresses test plan activity. Management has decided to apply the same QA approach to this activity. Table A-1 reflects the outcome of the grading considerations.

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2 U.S Department of Energy, Office of Nuclear Energy, Fuel Cycle Technologies Quality Assurance Program Document, Washington, D.C., October 13, 2010.

3 Sandia National Laboratories Quality Assurance Program Description, June 25, 2010, WebFileShare ID: WFS1043674

4 Sandia National Laboratories Used Fuel Disposition Campaign Preliminary Quality Assurance Implementation Plan; Fuel Cycle Research & Development; Prepared for U.S. Department of Energy Used Fuel Disposition Campaign December 2010 FCR&D-USED-2011-000019; January 13, 2011.

Quality Assurance requirements flow down from the FCT QAPD, the SNL-QAPD and SNL-UFCD-QAIP, as illustrated in Figure A-1. Predominantly, procedures from the Sandia Corporate Policy System (CPS) apply to this activity's quality elements, consistent with the approved SNL-QAPD. In selected instances, specific procedures were developed to improve quality to approximate NQA-1 levels for certain quality elements. Table A-1 identifies the CPS procedures that are generally applicable as well as the specific procedural augmentations that apply.

Figure A-1 – General QA Requirements Flow Down for SDI Activities

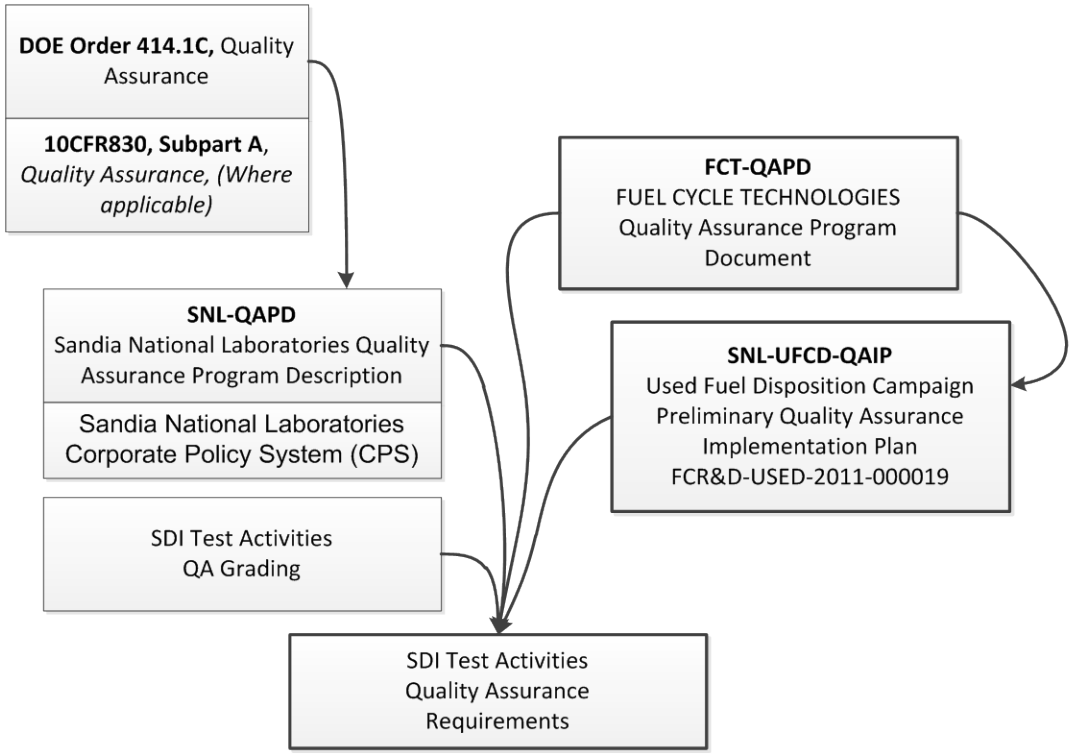

Table A-1 Graded Quality Assurance Requirements for SDI Activity

NQA-1 (2008) Requirement Excerpt5	**Summary of Grading**	**Procedures as Appropriate**
Organization - Responsibilities for the establishment and implementation of the quality assurance program shall be defined. The organizational structure, functional responsibilities, levels of authority, and lines of communications for activities affecting quality shall be documented.	A description of performing organizations placement within laboratory organization, including interfaces, is provided above. Rely on CPS procedures, including those listed in adjacent column, as appropriate.	**CG 100.1** - Establish the Decision-Making Framework **CG 100.1.1** - Create and Maintain the Mgmt. Structure **CG 100.1.2** - Create or Change a Policy - Process - or Procedure **CG 100.1.3** - Define Roles - Responsibilities - Accountabilities - and Authorities **CG100.6.8** - Identify and Manage Corporate Issues **CG100.6.16** - Conduct Management Reviews **CG100.6.17** - Conduct EO/LLT Management Reviews

5 Refer to ASME NQA-1-2008 (Revision of ASME NQA-1-2004) Quality Assurance Requirements for Nuclear Facility Applications for complete description of requirement.

Quality Assurance Program - The program shall identify the activities and items to which it applies. The program shall provide control over activities affecting quality to an extent consistent with their importance.	A description of the Quality Assurance Program, requirements flow down, relationships between FCT QA, NNSA/SSO QA and laboratory QA organizations, is provided above. Rely on CPS procedures, including those listed in adjacent column, as appropriate. Specific qualifications and training required of MOW involved in the activity are addressed by the specific FCT QAP identified.	Multiple CPS procedures in **HR100.2**. **FCT QAP 2-1** Qualification and Training
Design Control - The design shall be defined, controlled, and verified. Design inputs shall be specified on a timely basis and translated into design documents. Design interfaces shall be identified and controlled. **(Note: Includes provisions applicable to use of computer programs.)**	Rely on CPS procedures, including those listed in adjacent column, as appropriate. Related controls are addressed by the specific FCT QAP identified. Note: Requirements specifically identified for software determined to be N/A, because no software is designed as part of this activity.	**ME100.3.1 -** Perform Work **ME100.3.2 –** Manage Projects Throughout Their Lifecycle **ME100.3.3 -** Apply Configuration Management Principles to Documents and Physical Items **FCT QAP 20-1** Test Plans

Procurement Document Control - Applicable design bases and other requirements necessary to assure adequate quality shall be included or referenced in documents for procurement of items and services. To the extent necessary, procurement documents shall require Suppliers to have a quality assurance program consistent with the applicable requirements of NQA-1.	Rely on CPS procedures, including those listed in adjacent column, as appropriate.	Multiple procedures in **SCM100** – Manage Property, Material and Services through the Supply Chain
Instructions, Procedures and Drawings - Activities affecting quality and services shall be prescribed by and performed in accordance with documented instructions, procedures, or drawings that include or reference appropriate quantitative or qualitative acceptance criteria for determining that prescribed activities have been satisfactorily accomplished.	Rely on CPS procedures, including those listed in adjacent column, as appropriate. Specific controls are addressed by the FCT QAP identified.	**CG 100.1.2** - Create or Change a Policy - Process - or Procedure **FCT QAP 5-1** Implementing Procedures

Document Control - The preparation, issue, and change of documents that specify quality requirements or prescribe activities affecting quality such as instructions, procedures, and drawings shall be controlled to ensure that correct documents are being employed.	Rely on CPS procedures, including those listed in adjacent column, as appropriate. FCT QAPs listed will be modified to rely mostly on CPS, to provide explicit information for the task.	**IM100.2.1** - Control of Documents **IM100.2.2** - Control of Records **HR100.2.15** - Maintain Training Records in TEDS LMS **HR100.5.7** - Manage Corporate Human Resources Records **FCT QAP 6-1** Document Review Process **FCT QAP 6-2** Document Control
Control of Purchased Items and Services - The procurement of items and services shall be controlled to ensure conformance with specified requirements.	Rely on CPS procedures, including those listed in adjacent column, as appropriate.	**ME100.3.1** - Perform Work **SCM100.2.2** - Acquire Property (Requirements and Instructions - Inspect and Return Property section) **SCM100.3.10** - Do's and Don'ts for Requesters and SDRs During Contract Management Activities (Requirements and Instructions - step 3)

Identification and Control of Items - Controls shall be established to assure that only correct and accepted items are used or installed.	Rely on CPS procedures, including those listed in adjacent column, as appropriate.	**ME100.3.1** - Perform Work **SCM100.2.2** - Acquire Property (Requirements and Instructions - Inspect and Return Property section) **SCM100.3.3** – Manage Property **SCM100.3.10** - Do's and Don'ts for Requesters and SDRs During Contract Management Activities (Requirements and Instructions - step 3) **SCM100.3.13** – Manage Suspect or Counterfeit Items **SCM100.3.14** - Store General Materials at Sandia National Laboratories
Control of Special Processes - Special processes that control or verify quality, such as those used in welding, heat treating, and nondestructive examination, shall be performed by qualified personnel using qualified procedures in accordance with specified requirements.	Rely on CPS procedures, including those listed in adjacent column, as appropriate. Additional controls are addressed by the FCT QAP identified.	**ME100.3.1** - Perform Work **FCT QAP 9-1** Analysis

Inspection - Inspections required to verify conformance of an item or activity to specified requirements or continued acceptability of items in service shall be planned and executed.	Rely on CPS procedures, including those listed in adjacent column, as appropriate.	**ME100.3.1 -** Perform Work **SCM100.2.2** - Acquire Property (Requirements and Instructions - Inspect and Return Property section) **SCM100.3.10** - Do's and Don'ts for Requesters and SDRs During Contract Management Activities (Requirements and Instructions - step 3) **SCM100.3.13** – Manage Suspect or Counterfeit Items
Test Control - Tests required to collect data such as for siting or design input, to verify conformance of an item or computer program to specified requirements, or to demonstrate satisfactory performance for service shall be planned and executed. **(Note: Applicable to testing of computer programs, hardware and operating systems.)**.	Activity specific controls are addressed by the FCT QAPs identified. Note: Requirements specifically identified for software determined to be N/A, because of the nature and use of data recording and later processing.	**FCT QAP 20-1** Test Plans **FCT QAP 20-2** Scientific Notebooks
Control of Measuring and Test Equipment - Tools, gages, instruments, and other measuring and test equipment used for activities affecting quality shall be controlled, calibrated at specific periods, adjusted, and maintained to required accuracy limits.	Rely on CPS procedures, including those listed in adjacent column, which requires a measurement assurance plan consistent with Primary Standards Lab (PSL) practices.	**ME100.3.1 -** Perform Work

Handling, Storage and Shipping - Handling, storage, cleaning, packaging, shipping, and preservation of items shall be controlled to prevent damage or loss and to minimize deterioration.	Rely on CPS procedures, including those listed in adjacent column, as appropriate. Additional controls are addressed by the FCT QAP / FCT SPs identified.	**SCM100.3.3** – Manage Property **SCM100.3.14** - Store General Materials at Sandia National Laboratories **FCT QAP-13-1** Control of Samples **FCT SP-13-1** Chain of Custody
Inspection, Test, and Operating Status - The status of inspection and test activities shall be identified either on the items or in documents traceable to the items where it is necessary to ensure that required inspections and tests are performed and to ensure that items that have not passed the required inspections and tests are not inadvertently installed, used, or operated.	Rely on CPS procedures, including those listed in adjacent column, as appropriate.	**ME100.3.1** - Perform Work **CG100.5.5** - Control Item and Process Nonconformances **SCM100.3.13** - Manage Suspect or Counterfeit Items

Control of Nonconforming Items – Items that do not conform to specified requirements shall be controlled to prevent inadvertent installation or use.	Rely on CPS procedures, including those listed in adjacent column, as appropriate.	**SCM100.3.13** - Manage Suspect or Counterfeit Items **SCM100.3.13** - Manage Suspect or Counterfeit Items **CG100.6.6** - Perform Corrective Action **CG100.6.9** - Conduct Root Cause Analysis and Extent of Condition Reviews **CG100.5.5** - Control Item and Process Nonconformances
Corrective Action - Conditions adverse to quality shall be identified promptly and corrected as soon as practicable. In the case of a significant condition adverse to quality, the cause of the condition shall be determined and corrective action taken to preclude recurrence.	Rely on CPS procedures, including those listed in adjacent column, as appropriate.	**CG100.6.6** - Perform Corrective Action **CG100.6.9** - Conduct Root Cause Analysis and Extent of Condition Reviews

Quality Assurance Records - The control of quality assurance records shall be established consistently with the schedule for accomplishing work activities. Quality assurance records shall furnish documentary evidence that items or activities meet specified quality requirements. Quality assurance records shall be identified, generated, authenticated, and maintained, and their final disposition specified.	Rely on CPS procedures, including those listed in adjacent column, as appropriate. Transitory working information and non-record materials will be managed in the ANEP SharePoint site. The ANEP SharePoint site will be used for interim storage of records for convenience FCT deliverables and associated records will be managed in accordance with the FCT Records Management Plan upon finalization of the deliverable. (Submittal of records to the Fuel Cycle Research and Development Document Management System will maintain the associations between deliverables and supporting documentation, to the extent practicable.)	**IM100.2.1** - Control of Documents **IM100.2.2**- Control of Records **IM100.2.3** - Prepare and Release Information **IM100.2.4** - Cancelled - Manage Records Throughout their Lifecycle **IM100.2.5**- Identify and Protect Unclassified Information

Audits - Audits shall be performed to verify compliance to quality assurance program requirements, to verify that performance criteria are met, and to determine the effectiveness of the program. Audit results shall be documented and reported to and reviewed by responsible management. Follow-up action shall be taken where indicated.	Rely on CPS procedures, including those listed in adjacent column, as appropriate. Note: It was determined that auditing the activity was not considered as value added, surveillance/assessment was considered adequate.	**CG100.6.3** - Perform Assessments **CG100.6.7** - Conduct Independent Internal Audits

Appendix B: Memo regarding the use of bottled gas vs. shop air

Albuquerque, New Mexico 87185-0705

date: 12/09/97

to: Nancy Brodsky, Dept 6117

from: Glenn T. Barker, Dept. 6116

subject: Summary of the Thermal Conductivity Analyzer Modifications

After the failure of the TCA to pass the verification check at the conclusion of the WA-0329 thermal conductivity tests, a thorough examination was conducted of both the hardware and software used for the TCA. The purpose of this memo is to document the order which various theories for the poor performance were addressed, the conclusions drawn, and any improvements that were made.

The last failed verification check was made during the June/July 1997 timeframe, and the manner in which it failed showed that over time (several months) the TCA output gradually decreased. The first thought was to find a slow external influence which might explain this observation since the short term performance appeared to be acceptable. It was noted that at that time, the average humidity in the thermal properties lab was consistently 50% RH or higher; and since in the onset of thermal conductivity testing (~February 1997) laboratory humidity had steadily risen due to the change of seasons and the method of air conditioning. The first hypothesis was that some how humidity was effecting the TCA. Numerous calibration/verification runs were made after using various methods to control moisture surrounding the TCA. Some of the methods included: system shutdown in between calibrations and verifications, removal of the test cavity cover to allow damp fiberous insulation to dry, and application of mechanical rough pump vacuum to the bell jar surrounding the test cavity. All attempts appeared to have no effect on performance.

It was also noted that occasionally one of the two pneumatic clamp sole-noid valves would stick open. When this occurred, generally it was quite apparent since compressed air could be audibly heard leaking from the top of the analyzer. Typically, when this did occur, the condition would correct itself (the valve would seat) in ten minutes or less, so testing would not commence until this happened. The next hypothesis then was to suppose that something more subtle perhaps was also occurring with this marginal solenoid. A pressure guage was installed at various points in the pneumatic circuit, and all fittings were leak checked - but nothing could explain a long term degradation in TCA output. It wns noted however, that TCA ouput was very sensitive to pneumatic clamping pressure. Minor adjustments (± 5 psi) to the TCA pressure regulator translated to large changes in TCA ourput (10-20%). Since the TCA was so sensitive to changes in clamping pressure, both pneumatic solenoid valves were replaced - the TCA performance was unchanged.

During the previous investigations it was noted that the maximum pressure available from the facilities compressed air line had dropped below 100 psi (cracking pressure of the relief valve on the supply manifold). When the system was first installed, maximum pressure exceeded 100 psi which was how the relief valve operation was periodically checked. Since the pneumatic pressure was regulated down to 75-80 psi by the pressure regulator on the TCA this was not believed to be a concern. However, in light of the sensitivity to clamping pressure, a series of tests were conducted off of a compressed gas cylinder instead of the facilities compressed air port. The first set of tests appeared to be inconclusive, but the pressure regulator resolution used for the test was inadequate for use in the TCA, so a second series of tests were conducted with a more appropriately sized regulator. It was this second series of tests which showed a marked improvement in consistency of the TCA output. It was also noted from examination of the calibration output report, that the standard deviation of the data used to calculate the slope of the calibration line, Output vs. Resistance at a given temperature, provided a good relative measure of the quality (machine performance) of the data. The standard deviation reported as "Sigma N" varies as a function of temperature but in general terms this value improved

from -.27 at low temperatures with facilities compressed air, to -.09 at low temperatures with a compressed gas cylinder.

After switching to a compressed gas cylinder as a pnuematic source for the clamp, efforts were then directed to software concerns. Comparisons of the test reports generated from previous work performed at Holometrix to those generated here at Sandia, showed that there was a difference in the software codes used. Since, prior to use, the software is validated by the conduct of a calibration and verification, this difference was viewed as a secondary importance - however still a concern. The most obvious difference showed in the intermediate calculation of thermal conductivity, which appeared to be off be a factor of about 2. Manual (calculator) checks of calibration/verifications proved accurate, only the intermediate results appeared affected. By stepping through the code it was discovered that on this version of the code, the same array name which contained the heat flux data was used for two different purposes. The result of which explained the factor of two difference in the intermediate results, however unknown until now, it also caused the program to declare equilibrium prematurely. At this point it was decided to make a concentrated effort to clean up the code and incorporate several additions as well. The software "TCA200LT.EXE" version 2 included the following changes from version 1.0:

1) Renamed one of the two conflicting arrays containing heat flux data.
2) Added code to spool all "raw" data to file.
3) Added code to prevent printer problems from aborting test.
4) Added the date to printed report in several places.
5) Added code to calculate temperatures from voltages per A/D manufacturer's recommendation.
6) Added code to enable users to calibrate using either Pyrex 7740 or Quartz, then verify with the opposite material.
7) Organized code into logical sections, eliminating skipped code, consolidating similar routines and adding ample comments to source code.

Software was validated by numerous calibration/verification runs as well manual (calculator) checks and finally (attached) spread sheet calculated comparison of code generated reports.

Next, the TCA was outfitted with newly calibrated (Sandia Primary Standards lab) thermocouples for all QA affecting channels. Also installed at this time, was a new pnuematic clamp actuator (installed as a precautionary measure), and a passive R-C filter network in series with the heat flux transducer to filter out electrical noise.

Subsequent tests have shown favorable results in the long term (up to 6 weeks) stability of the TCA even after major disassembly between calibration and verification checks. Differences between calibration values and verifications appear to stem primarily from the uncertainty for the reported values of thermal conductivity for the Pyrex standards themselves. Verification errors seem to consistently run about 6% low at the low (>25°C) temperatures, and about 2% at higher temperatures (<200°C).