Test Results and Comparison of Triaxial Strength Testing of Waste Isolation Pilot Plant Clean Salt

Spent Fuel and Waste Disposition

Prepared for US Department of Energy Spent Fuel and Waste Science and Technology

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December 5, 2016 SFWD-SFWST-2017-000101

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The attached 28-page letter report (Technical Letter Memorandum RSI/TLM-189) by RESPEC titled "Test Results and Comparison of Triaxial Strength Testing of Waste Isolation Pilot Plant Clean Salt" fulfils the FY2017 SFWST milestone M4SF-17SN010303015 (Report on Confirmatory Geomechanical Testing of Bedded Salt) in the Sandia National Laboratories Salt R&D work package.

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Technical Letter Memorandum RSI/TLM-189

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cc: Project Central File 2540 — Category A

From: Mr. Stuart A. Buchholz Manager, Materials Testing RESPEC P.O. Box 725 Rapid City, SD 57709

Stuart Bichhole

Date: August 23, 2016

Subject: Test Results and Comparison of Triaxial Strength Testing of Waste Isolation Pilot Plant Clean Salt

INTRODUCTION

This memorandum documents laboratory thermomechanical triaxial strength testing of Waste Isolation Pilot Plant (WIPP) clean salt. The limited study completed independent, adjunct laboratory tests in the United States to assist in validating similar testing results being provided by the German facilities. The testing protocol consisted of completing confined triaxial, constant strain rate strength tests of intact WIPP clean salt at temperatures of 25°C and 100°C and at multiple confining pressures. The stratigraphy at WIPP also includes salt that has been labeled "argillaceous." The much larger test matrix conducted in Germany included both the so-called clean and argillaceous salts. When combined, the total database of laboratory results will be used to develop input parameters for models, assess adequacy of existing models, and predict material behavior. These laboratory studies are also consistent with the goals of the international salt repository research program.

The goal of this study was to complete a subset of a test matrix on clean salt from the WIPP undertaken by German research groups. The work was performed at RESPEC in Rapid City, South Dakota. A rigorous Quality Assurance protocol was applied, such that corroboration provides the potential of qualifying all of the test data gathered by German research groups.

SPECIMEN ACQUISITION AND PREPARATION

The salt core tested in this program was recovered from the East Rib (Borehole E140 Core N1100) of the underground workings at the WIPP site near Carlsbad, New Mexico. Additional core sample information is provided in the Chain-of-Custody document in Attachment A. The core was shipped to RESPEC in Rapid City, South Dakota, by Sandia National Laboratories (SNL) personnel to ensure that the core was not subject to any freight damage, temperature extremes, or mishandling. The core arrived at the RESPEC laboratory on December 15, 2014, and showed no signs of damage. Photographs of the received salt core

are provided in Figure 1. The entire usable core was consumed during the testing, and any remnants were disposed of at the conclusion of the project.



Figure 1. Photographs of the Received Salt Core.

To prepare a testable specimen, a piece of salt core was cut to an approximate length-to-diameter ratio (L:D) of 2:1. The walls and ends of the cylinder were then machined in a horizontal lathe to produce a finished right-circular cylinder with ends that were flat, parallel to each other, and perpendicular to the specimen sides. A typical machining setup is shown in Figure 2 where the carbide tooling is visible next to the specimen surface. The finished specimens were then measured to determine their length and diameter. The specimens were also weighed, and a bulk-density was calculated by using the specimen dimensions to determine specimen volume. A summary of the testable specimens that were prepared is presented in Table 1. The bulk-density values are uniform and near the typical value for halite (2.15 grams per cubic centimeter [g/cc]).



Figure 2. Typical Horizontal Lathe Machining Setup for Preparing Cylindrical Specimens.

Specimen I.D.	Length (mm)	Diameter (mm)	Mass (g)	Density (g/cc)
WIPP/SNLCH106-4/4.90/2	203.26	98.72	3,343.1	2.15
WIPP/SNLCH106-4/4.90/3	203.56	98.71	3,350.4	2.15
WIPP/SNLCH106-4/5.67/2	203.79	98.75	3,350.8	2.15
WIPP/SNLCH106-4/5.67/3	202.85	98.76	3,339.1	2.15
WIPP/SNLCH106-4/5.67/4	202.07	98.78	3,327.0	2.15
WIPP/SNLCH106-5/7.15/1	202.65	98.77	3,344.7	2.15
WIPP/SNLCH106-5/7.15/3	202.85	98.76	3.350.8	2.16
WIPP/SNLCH106-5/7.93/1	203.51	98.76	3,372.8	2.16

Table 1. Summa	y of Salt S	pecimens Pre	pared for Testing
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mm = millimeters

g = grams

g/cc = grams per cubic centimeter.

All of the specimens in Table 1 have unique identification numbers for tracking within the RESPEC laboratory. A typical specimen identification number is:

WIPP/SNLCH106-4/4.90/2

where:

WIPP	=	Waste Isolation Pilot Plant
SNLCH106-4	=	SNL sample/subsample identifier
4.90	=	depth (feet)
1	=	specimen piece number.

TEST EQUIPMENT

The testing was completed by using a universal test system with two reaction columns referred to as the UTS2 system. The UTS2 is a computer-controlled, servohydraulic system manufactured by MTS Systems of Eden Prairie, Minnesota. The computer controls allow for controlling the loading in either of two modes: a stress-rate mode that uses the load-cell output as a feedback signal or a strain rate control mode that uses a Linear Variable Differential Transformer (LVDT) output to control loading. Three independently controlled heaters were mounted at the top, middle, and bottom of the pressure vessel to provide uniform heating at temperatures up to 100°C. An insulating jacket was wrapped around the outside of the pressure vessel to control heat loss.

A photograph of the test system is provided in Figure 3, which illustrates four thermocouples placed at the top, middle, and bottom of the test specimen. The fourth thermocouple was placed midheight of the specimen on the opposite side. Before testing, the thermocouples were calibrated, and the readings were used to independently adjust the temperature settings on the heaters to achieve a consistent temperature profile around the specimen.



Figure 3. Jacketed Test Specimen in the UTS2 Load Frame With Thermocouples and Acoustic Transducers.

The ultrasonic velocity setup requires that the standard steel platens in the load frame are replaced with acoustics platens. The acoustics platens shown in Figure 3 have two piezoelectric transducers imbedded in each: one for compressional (*P*) waves and another for shear (*S*) waves. The *P* and *S* waves are actively fired at 1 megahertz (MHz) from the bottom platen and received in the top platen after traveling through the salt specimen. The data-acquisition system and pulsar shown in Figure 4 beams the ultrasonic waves; listens for the wave's arrival; displays the waveform; and ultimately, outputs a raw-data file. The acoustics system was calibrated by using known aluminum standards.

The specimen volume was measured by the position of the piston pushing the confining oil into or out of the pressure vessel. A Temposonics® transducer was used for the piston position, which was calibrated by pushing oil into a burette and reading the output at ten steps throughout the range. A load cell mounted above the vessel monitors the axial loading force, and an LVDT mounted inside the hydraulic actuator at the base of the system is used to monitor the axial displacement of the loading piston and specimen length. All of the data are combined to continuously calculate the stress and strain on the specimen during the test.



Figure 4. View of the UTS2 Computer and Oscilloscope Showing Acoustic Wave-Speed Plots.

All of the instrumentation was calibrated against in-house standards that are certified traceable to National Institute of Standards and Technology (NIST) references. Calibration records indicate that the load-cell force readings and the LVDT displacement measurements are accurate to within \pm 1 percent of the reading, the volumetric measurements are accurate to within \pm 1.5 percent volumetric strain, and the specimen temperature is accurate to within \pm 2°C. Because the LVDT measures total axial displacement, including some nonspecimen contributions, a "machine softness" factor was determined that allowed the LVDT measurements to be corrected. Using a steel specimen for which accurate elastic parameters are known, the "machine softness" correction coefficient was determined to be 0.0004 millimeter per kilonewton (mm/kN). This coefficient can be multiplied by the load-cell reading with the product subtracted from the accompanying LVDT measurement to estimate specimen displacement at that point.

TEST PROCEDURE

Three shakedown tests were performed at 100°C to simulate actual test conditions and to verify that all test equipment and controls were working properly and datalogging was at an adequate rate. Once the shakedown tests were completed, the triaxial strength tests began according to the test matrix identified in the contract between RESPEC and SNL.

The test procedure included the following steps:

- 1. The test specimen setup included placing a specimen in a viton jacket and sealing the ends of the jacket over each platen using lock wire. The platens contain the acoustic transducers for measuring ultrasonic wave speeds. Honey was spread on the ends of the specimen to facilitate transmitting the sound energy between the transducer and the test specimen.
- 2. Four thermocouples were used to measure temperature within the pressure vessel. Thermocouples were placed at the top, middle, and bottom of the test specimen, and the fourth thermocouple was placed at the midheight on the opposite side of the specimen.
- 3. A small preload was applied (about 6 kN) to the specimen to close any interfaces and to ensure that pressure was applied to the seals. The acoustic signals were checked to ensure a signal was being received through the specimen.
- 4. The pressure vessel was lowered and filled with oil. The computer-controlled actuators that maintain axial force and the confining pressure increased pressure hydrostatically to the setpoint determined for the test and held the pressure constant. Once the pressure had been applied, the heaters were turned on, and the temperature was increased at a rate of 0.5°C per minute until the preferred temperature was reached. Three heaters located on the outside of the pressure vessel were independently controlled. Each heater had its own setpoint determined from previous testing. The test specimen was left at hydrostatic conditions overnight to ensure temperature equilibration throughout the test system. Insulating jackets were on the outside of the pressure vessel to reduce heat loss.
- 5. With the test specimen at the predetermined temperature and confining pressure, the test was initiated. Datalogging on the MTS computer and oscilloscope were turned on simultaneously with acoustic data (both *P* and *S* waves) set to 60-second intervals, and the MTS controller was programmed to accept data in time and strain increments. The axial stroke was increased at a rate of 0.002 millimeters per second (mm/s), which corresponds to a strain rate of 10^{-5} s⁻¹. During this time, the confining pressure was held constant. The test continued until the specimen could no longer support the applied load or the limits of the test equipment were reached.

TEST RESULTS

Eight confined triaxial, constant strain rate strength tests were performed on intact WIPP clean salt specimens. A summary of the stress, temperature, and strain rate conditions for the tests is provided in Table 2. These test conditions are a subset of the tests performed at the German laboratory facilities [Salzer et al., 2015].¹

Differential stress versus axial-strain plots for the tests ran at 25.0°C and 100.0°C are provided in Figures 5 and 6, respectively. Data obtained from tests performed under the same conditions at the German laboratories are also illustrated in these figures. Axial strains agree well between the two different datasets. At higher confining pressures, the RESPEC data do not reach the same maximum differential stress or peak axial-strain limits as the German data. This is a limitation of the axial force capacity of the RESPEC test frame. Two of the tests (3 MPa confining pressure and 25°C, 20 MPa confining pressure and 100°C) failed prematurely during the test.

¹ Salzer, K. D. Naumann, R.-M. Günther, and T. Popp, 2014. "Status of Laboratory Tests on WIPP-Salt," *Workshop 'Joint Project III: Comparison of Constitutive Models,'* Karlsruhe Institute of Technology, Karlsruhe, Germany, January 16–17.

Confining Pressure	Strain Rate (1/s)	Temperature (°C)
0.2	1×10^{-5}	25.0
1.0	1×10^{-5}	25.0
3.0	1×10^{-5}	25.0
20.0	1×10^{-5}	25.0
0.2	1×10^{-5}	100.0
1.0	1×10^{-5}	100.0
3.0	1 × 10 ⁻⁵	100.0
20.0	1×10^{-5}	100.0

Table 2.	Summary	of	Triaxial	Strength	Test	Conditions
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The volumetric strain versus axial strain for tests ran at 25.0°C and 100.0°C are shown in Figure 7 and 8, respectively. In some cases, jacket leaks occurred and volumetric measurements were truncated. As shown, the trends are similar at lower confining pressures; however, the RESPEC data show significantly more specimen compaction.

Dilation characteristics of the salt were determined where dilation implies that the specimen is being damaged through microfracturing (creation of voids). Dilation limits were determined by examining the volumetric strain and ultrasonic velocity responses during testing. The volumetric strain response indicates a small level of compaction at low-to-moderate stress differences, which is consistent with a material that is not dilating. At some elevated stress difference, the slope of the volumetric strain curve changes as the volumetric strain starts becoming more positive, which indicates volumetric expansion. Similarly, ultrasonic *P*- and *S*-wave velocity measurements were examined to determine the point at which the specimens transitioned from the compaction phase (increasing velocity) to creating void space (decreasing velocity) caused by damage. Ultrasonic velocity measurements are more sensitive to salt dilation than traditional volumetric strain measurements and indicate dilation limits at lower differential stresses. In some instances, the volumetric strain measurements did not show dilation during the test; whereas, dilation was indicated from the ultrasonic measurements. In general, dilation is indicated first by the S-wave velocity, then by the P-wave velocity, and last by the volumetric strain. Ultrasonic velocity measurements were not performed by the German laboratories for this study. Pre- and posttest photographs as well as plots of stress difference versus strain and acoustic velocities are provided in Attachment B for all of the RESPEC tests.

A plot of the dilation strengths for the tests ran at 25.0°C is provided in Figure 9. As previously discussed, dilation was indicated first by the *S*-wave velocity, then by the *P*-wave velocity, and last by the volumetric strain. Dilation was not indicated by the RESPEC volumetric strain data for the two tests performed at confining pressures of 3 MPa and 20 MPa. Also shown in Figure 9 is the German dilation strength determined by using only the volumetric strain measurement. With the exception of a single point at a confining pressure of 20 MPa, the differential stress at dilation is consistently higher for all three methods of determining dilation by RESPEC. The dilation strength should increase with increasing confining pressure. This is true for the RESPEC data; whereas, the German data do not show this relation for the tests performed at confining pressures of 1 MPa and 3 MPa.



Figure 5. Differential Stress Versus Axial Strain for Triaxial Strength Tests at 25°C.

August 23, 2016



Figure 6. Differential Stress Versus Axial Strain for Triaxial Strength Tests at 100°C.



Figure 7. Volumetric Strain Versus Axial Strain for Triaxial Strength Tests at 25°C.



Figure 8. Volumetric Strain Versus Axial Strain for Triaxial Strength Tests at 100°C.



August 23, 2016





A plot of the dilation strengths for the tests ran at 100°C is illustrated in Figure 10. Similar to the tests performed at 25°C, dilation was not indicated by the RESPEC volumetric strain data for the test performed at a confining pressure of 3 MPa. The test performed at 20 MPa prematurely ruptured before dilation was indicated by any of the three methods. The dilation strength data lie within the scatter of the German data and do not show consistently higher values as the tests performed at 25°C.

Peak strength is a measure of the maximum differential stress that the specimen can withstand before ultimately failing. The peak stress for the tests performed at 25°C and 100°C is illustrated in Figures 11 and 12, respectively. The two RESPEC tests depicted in Figure 11 are not considered valid for comparison to the German data. The test performed at 3 MPa confining pressure ruptured prematurely, and the test performed at 20 MPa did not fail before reaching the maximum axial force capacity of the test system. Additionally, the test performed at 100°C and 20 MPa confining pressure (shown in Figure 12) ruptured prematurely; thus, the peak stress is not valid for comparison. All of the remaining peak-stress data show good correlation between the RESPEC and German laboratories.

An X-Ray Diffraction (XRD) mineralogical analysis was performed on two sets of the tested material. The results indicate that the WIPP clean salt comprises more than 99 percent halite with varying amounts of polyhalite.

CONCLUSIONS

The intent of this study was to perform a limited number of triaxial strength tests to corroborate results of a much larger test matrix performed by the German laboratories. The test matrix performed by RESPEC included confined triaxial, constant strain rate strength tests of intact WIPP clean salt at temperatures of 25°C and 100°C, at multiple confining pressures, and at a strain rate of 1×10^{-5} per second. The following two main conclusions were presented by Salzer et al. [2015]:

- Strength decreases as temperature increases
- Material behavior changes from brittle to more ductile as temperature increases.

These statements were confirmed by the testing performed by RESPEC. For individual tests, axial-strain histories and peak differential stresses correlate well. Deviations were observed where the specimens prematurely ruptured or when the test equipment reached the maximum axial force capacity before reaching failure. The volumetric strain histories can be correlated; however, the RESPEC results show considerably more compaction.

One of the main objectives for triaxial strength testing is determining the dilation and peak strength at different confining pressures. The peak-stress comparisons are similar between the German and RESPEC datasets. Peak strength is determined from external axial-strain measurements, which are generally easy to perform and interpret.

Determining dilation strength is more challenging to measure and interpret, which accounts for scatter or discrepancies between the two datasets. RESPEC performed volumetric measurements to provide a direct comparison to the volumetric measurements performed by the Germans to determine the dilation strength of the salt. The volumetric measurements are performed by measuring the volume of oil that is displaced from the pressure vessel during testing. Small oil leaks in the system and expansion/contraction of the oil caused by temperature fluctuations can have a significant impact on the volumetric measurements. Because of the uncertainty in the volumetric measurements, RESPEC also











Figure 12. Comparison of the Peak Strength for the Tests Performed at 100°C.

evaluated ultrasonic velocity measurements to provide an independent method of determining dilation strength. The ultrasonic measurements were more sensitive to apparent damage and indicated dilation at lower values of differential stress. However, interpreting data for the ultrasonic and volumetric measurements can be highly subjective. Data interpretation can be improved by additional testing and comparing the different methods of determining the onset of dilation.

The limited test matrix performed by RESPEC correlated reasonably well with the German data overall. Axial stress-strain behavior at 25°C was essentially identical over comparable ranges. Axial stress-strain behavior at 100°C was replicated in three tests, with one test that failed abruptly.

Volumetric strain measurements agreed well at 25°C and low confining pressures of 0.2 and 1.0 MPa and diverged significantly at higher confining pressures. Volumetric strain measurements at 100°C were consistently lower in our experiments than in the equivalent German experiments. Where strength could be unequivocally compared, they matched well. The main discrepancy observed in the datasets was determining the dilation strength and the volumetric strain measurements. RESPEC tests exhibited less volumetric strain under most test conditions. Perhaps this is a result of a systematic testing process. RESPEC test procedure established temperature and hydrostatic pressure conditions overnight before differential pressure was applied. Our tests exhibited less volumetric strain overall and the deviation with German data increased as confining pressure and temperature increased. This observation suggests our procedure promoted more consolidation in the specimen that perhaps was induced in the German procedures. Further investigations should include an improved volumetric strain measurement system and further development and understanding of the ultrasonic velocity measurements to determine dilation strength. We will continue to explore improvements in experimental techniques as our collaborations with German salt repository researchers continues.

SAB:krl

ATTACHMENT A CHAIN-OF-CUSTODY FORM

				A	ppendix A			
ACTIVITY/ PROJECT SPECIFIC Sandia PROCEDURE National Laboratories			Form Number: SP 13-1-1 Page of1 Attach more forms as needed					
1. Initial Sample Custodia	an Terry N Printed N	lacDonald _{lame}			Organization:6	5212	Date: 12/08/2014	
2. Sample Collection or C	2. Sample Collection or Creation Information Scientific Notebook ID: NA Sample Team					n Members/Organization.		
Test Plan ID: Sandia MC	DU No.11-S-5	585		Field Log ID): NA		Tei	ry MacDonald
Sample Location: E140 /	/ N1100 (Eas	t Rib)						Phil Finley
i.e. bore	hole/core no./lal	bldg. no./etc.					e	nter n/a if none
3. Sample Identification	Date	Cont	ainer	Preser-	Analysis Request		Samp	le
Sample/Sub-Sample #	Collected	Туре	Volume	vative			Descrip	
SNLCH106-3	01/14/13	Tube	1.5 cu ft	N/A	TBD	12 in. Dia.	Halite/Poly. Core Sam	pic. SNLCH106 subsample
SNLCH106-4	01/14/13	Tube	1.5 cu ft	N/A	TBD Test	12 in. Dia.	ple. SNLCH 106 subsample.	
SNLCH106-5	01/14/13	Tube	1.5 cu ft	N/A	N/A TBD-9-11 12 in. Dia. Halite/Poly. Core Sample. S			ple. SNLCH 106 subsample.
SNLCH106-6	01/14/13	Tube	1.5 cu ft	<u>N/A</u>	TBD	-12 in. Dia.	Halite/Poly. Core Sam	ple. SNLOH100 subsample.
	01/14/13	Tube	1.5 cu ft	<u>N/A</u>	IBD IBD	12 in. Dia.	Halite/Poly. Core San	ple. SNLOH106 subsample.
SNLCH106-8	01/14/13	/14/13 Tube 1.5 cu ft N/A TBD 12 in. Dia. Halite/				Halite/Poly. Core San	pie. SNLOH 100 Subsample.	
Last Item								
4 Sample Paguiroments	Oshional	to Rosa	en Real	Ct SO	Con Color Dests	: Ham		
4. Sample Requirements	Lice Wax Im	rognated C	ardboard Tu	bes and For	am Padding	, orm		
Storage & Preservation:	Dacked in W	av Impregna	ated Cardboa	ard Tubes to	Preserve Moisture (Content: Stor	e in a Temperature C	ontrolled Environment.
Storage & Leservation.	Padded Car	board Tube	s Inside Wo	od Crate or	on covered pallet.			
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5 Custody Transfer Printed Name Signature Organization/Company					y Date-Time	Sample Condition		
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For samples that are potentially hazardous & require packaging and shipping, contact Center 6200 ES&H Coordinator of see SNE ES&H Mandal, Onper 12.								

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ATTACHMENT B CONFINED TRIAXIAL AND COMPRESSION STRENGTH TEST PLOTS AND PRE- AND POSTTEST PHOTOGRAPHS



Figure B-1. Confined Triaxial Compression Test Plot and Pre- and Posttest Photographs for Specimen WIPP/SNLCH106-4/4.90/2.



Figure B-2. Confined Triaxial Compression Test Plot and Pre- and Posttest Photographs for Specimen WIPP/SNLCH106-4/4.90/3.



Figure B-3. Confined Triaxial Compression Test Plot and Pre- and Posttest Photographs for Specimen WIPP/SNLCH106-4/5.67/2.



Figure B-4. Confined Triaxial Compression Test Plot and Pre- and Posttest Photographs for Specimen WIPP/SNLCH106-4/5.67/3.



Figure B-5. Confined Triaxial Compression Test Plot and Pre- and Posttest Photographs for Specimen WIPP/SNLCH106-4/5.67/4.



Figure B-6. Confined Triaxial Compression Test Plot and Pre- and Posttest Photographs for Specimen WIPP/SNLCH106-5/7.15/1.



Figure B-7. Confined Triaxial Compression Test Plot and Pre- and Posttest Photographs for Specimen WIPP/SNLCH106-5/7.15/3.



Figure B-8. Confined Triaxial Compression Test Plot and Pre- and Posttest Photographs for Specimen WIPP/SNLCH106-5/7.93/1.