# Technical Letter Memorandum RSI/TLM-185

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**cc:** Dr. Gary D. Callahan, RESPEC
Project Central File 2075 — Category C

**From:** Mr. Kirby D. Mellegard
Manager, Materials Testing
RESPEC
P.O. Box 725
Rapid City, SD 57709

**Date:** September 20, 2012

**Subject:** Initial Laboratory Unconfined Compression Uniaxial Stress Test Results on Salt Specimens at Elevated Temperatures From the Waste Isolation Pilot Plant

## INTRODUCTION

This memorandum documents our test results for the initial unconfined, uniaxial stress shakedown tests on cylindrical specimens of Waste Isolation Pilot Plant (WIPP) salt performed under Task 2 of Sandia National Laboratories (SNL) Purchase Order No. 1178964, Rev. 3. In general, Task 2 calls for nine tests comprising three replicates at each of three temperatures; namely, 200°C, 250°C, and 300°C. This memorandum covers the first stage of the laboratory effort which was to complete one test at each proposed temperature to obtain preliminary information about the behavior of the salt to guide possible test matrix modifications for the remaining six tests.

## SPECIMEN ACQUISITION AND PREPARATION

The salt core tested in this program were provided by SNL and were recovered from the WIPP site near Carlsbad, New Mexico. The core recovery locations are the horizontal boreholes shown in the borehole location map in Figure 1 that was prepared for the DRZ Characterization Test Plan at the WIPP. The sources of core are the three boreholes highlighted in yellow and are identified as QGU36, QGU37, and QGU38.

A limited petrographic analysis was performed by SNL on the salt core recovered from the boreholes. The analyses were completed by Dr. D. W. Powers, Consulting Geologist, and a summary of that work is attached to this memorandum. The result of primary interest for the current uniaxial testing is the conclusion that the core from the three boreholes identified as QGU36, QGU37, and QGU38 were considered to have few impurities and the geology was similar among all three boreholes.

RSI-2075-12-016

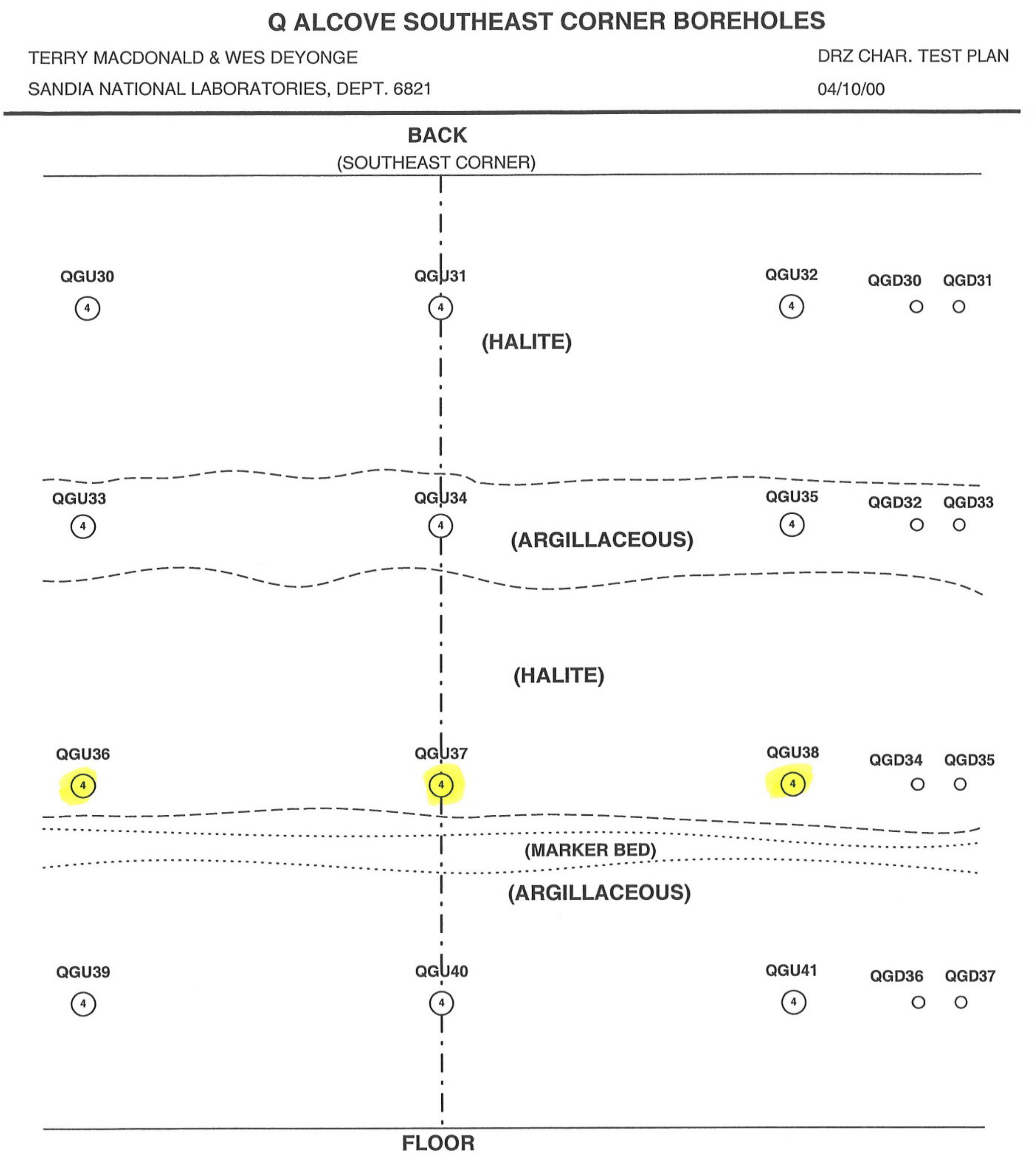


**Figure 1.** Core Recovery Borehole Locations at the Waste Isolation Pilot Plant.

The core were recovered in 2001 and were placed in environmentally secure storage at the WIPP site. The core was shipped to RESPEC in Rapid City, South Dakota, by personal courier to ensure that the core was not subjected to any freight damage, temperature extremes, or mishandling. The shipping occurred in July 2012 and a record of the shipment is shown in Figure 2. The core is in secure storage at RESPEC and will be disposed of at SNL directions at the conclusion of the project.

RSI-2075-12-017

Sandia National Laboratories
4100 National Parks Hwy.
Carlsbad NM 88221

**SHIPPER 143980**
Commercial Invoice
**Status:** Waiting for Approval

**Ship to:**
RESPEC Engineering
3824 Jet Dr.

Rapid City SD 57703
USA

| | |
|---|---|
| **RMA# or RGA#** | N/A |
| **Deliver to:** | Kirby Mellegard |
| Phone: | 605-394-6507 |
| Email: | N/A@SANDIA.GOV |
| Building: N/A | Room: N/A |
| Mail Stop: | N/A |
| Company: | RESPEC Engineering |
| Department: | N/A |
| **Address Type:** | Unclassified |
| **Date Due at Destination:** | 07/26/2012 |
| Production Related: | No |

| | |
|---|---|
| **Origination Site:** | Sandia New Mexico |
| **Ship From Site:** | Other |
| **Form filled out by:** | MACDONALD,TERRY L. |
| **Phone:** | 505/845-0408 |
| **Date Prepared:** | 2012-07-16 |
| **Requester:** | DEYONGE,WESLEY F |
| **Phone:** | 505/284-5358 |
| **Org. #:** | 06212 |

**For Shipment Processing Use**

| | |
|---|---|
| **Date Shipped:** | |
| **Carrier:** | None Selected |
| **Mode:** | None Selected |
| **Bill of Lading No.:** | |
| **Total # of Pkgs:** | 0 |
| **Total Weight:** | 0.0 lbs |
| **Total Cubic Dim:** | 0.0 |
| **Advance Notification** | **Contacted** Yes No |
| **Name and Phone:** | |
| **741 Number:** | |
| **ATS:** | |
| **TID Numbers:** | |
| **RCT Initial/Dates** | |

Arrived and unloaded at Rapid City, SD RESPEC office on 7/26/12. Inspected upon arrival - found 3 labeling errors on FCT forms SP forms were correct, RDA 7/26/12

**Reason/Authority:** To be Consumed in Testing / Incorporate into End Product
**Return Date:** NONE
**Authority Number:**
**Freight Charge Payment:** No Freight Charges
**Project:**
**Task:**
**Carrier:** None Selected
**Mode:** None Selected
**Account:**

**If shipping controlled property to a new Sandia location**
**Destination Bldg:** **Room:**

**If shipping to international destination:**
**Foreign Customs import duties and fees will be paid by:** N/A
**Export Authorization:**

**No freight charge reason:** Government Truck
**Is material being shipped from the Shipping Department building or the 6000 Igloo?** No
**Shipment Comments:** Handcarry.
**Transportation Pickup Requested:** No pickup requested
Questions about pickup call Dispatcher **844-1448** non-hazardous materials, **844-2556** hazardous materials.

**Figure 2.** Core Shipping Record for Waste Isolation Pilot Plant Salt.

An inventory of the core pieces shipped to RESPEC is listed in Figure 3. The 12 pieces that were transferred to RESPEC are highlighted in yellow. The individual core identification labels were later used when creating unique labels for individual test specimens that were fabricated from this core.

RSI-2075-12-018

**WIPP CORE WITH POTENTIAL FOR LABORATORY TESTS**

**LOCATED IN BLDG. NPHB, SHOP**

**June 18, 2012**

| Hole # | Box # | Section # | Length (ft.) | Length (in.) |
|---|---|---|---|---|
| QGU14 | 3 | QGU14-21 | 1.35 | ~16 |
| QGU14 | 4 | QGU14-25 | 0.85 | ~10 |
| QGU14 | 7 | QGU14-45 | 1.27 | ~15 |
| QGU14 | 8 | QGU14-48 | 1.05 | ~12.5 |
| QGU36 | 2 | QGU36-2-1-2 | 0.9 | ~11 |
| QGU36 | 2 | QGU36-2-2 | 1.21 | ~14.5 |
| QGU36 | 4 | QGU36-11 | 1.37 | ~16 |
| QGU36 | 5 | QGU36-13-1 | 1.1 | ~13 |
| QGU36 | 5 | QGU36-13-2-2 | 0.85 | ~10 |
| QGU36 | 6 | QGU36-14 | 1.51 | ~18 |
| QGU36 | 7 | QGU36-17 | 1.32 | ~16 |
| QGU36 | 7 | QGU36-18 | 0.76 | ~9 |
| QGU36 | 9 | QGU36-27-2 | 0.8 | ~9.5 |
| QGU36 | 9 | QGU36-28 | 1.14 | ~13.5 |
| QGU37 | 2 | QGU37-15 | 0.99 | ~12 |
| QGU37 | 3 | QGU37-20 | 1.86 | ~22 |
| QGU37 | 7 | QGU37-45 | 1.01 | ~12 |
| QGU37 | 8 | QGU37-48 | 1.66 | ~20 |
| QGU38 | 2 | QGU38-6 | 1.07 | ~13 |
| QGU38 | 2 | QGU38-7 | 0.85 | ~10 |
| QGU38 | 6 | QGU38-43-1 | 1.09 | ~13 |
| QGU38 | 6 | QGU38-43-2 | 1.33 | ~16 |
| QGU38 | 9 | QGU38-70 | 0.97 | ~11.5 |
| QGU38 | 9 | QGU38-71 | 0.87 | ~10 |

**Figure 3.** Inventory of Core Pieces Shipped to RESPEC (highlighted in yellow).

The Chain-of-Custody record for the core shipped to RESPEC is shown in Figures 4a and 4b. This record will be completed when the core is disposed of after the project is completed.

Chain of Custody

SP 13-1
Revision 5
Page 5 of 5

Appendix A

| ACTIVITY/ PROJECT SPECIFIC PROCEDURE Sandia National Laboratories | Chain of Custody | Form Number: SP 13-1-1 Page 1 of 2 Attach more forms as needed |
|---|---|---|

**1. Initial Sample Custodian** Terry L. MacDonald (Printed Name) Organization: 6212 Date: 07/16/2012

**2. Sample Collection or Creation Information** Scientific Notebook ID: _____
Test Plan ID: SNL-FCT-TP-11-0001 Field Log ID: _____
Sample Location: WIPP Underground, Q Room Southeast Corner, S-130/W-905 (i.e. borehole/core no./lab bldg. no./etc.)

Sample Team Members/Organization.
Terry L. MacDonald/SNL/6212
Wesley F. DeYonge/RESPEC/6212
enter n/a if none

| 3. Sample Identification Sample/Sub-Sample # | Date Collected | Container Type | Container Volume | Preser-vative | Analysis Request | Sample Description |
|---|---|---|---|---|---|---|
| QGU36-17 | 09-06-00 | Poly Bag | 0.11 cu ft | N/A | Heat Comp. Test | 4 in. Dia. Halite/Polyhalite Core Sample. QGU36 subsample. |
| QGU36-18 | 09-06-00 | Poly Bag | 0.07 cu ft | N/A | Heat Comp. Test | 4 in. Dia. Halite/Polyhalite Core Sample. QGU36 subsample. |
| QGU36-27-2 | 09-06-00 | Poly Bag | 0.07 cu ft | N/A | Heat Comp. Test | 4 in. Dia. Halite/Polyhalite Core Sample. QGU36 subsample. |
| QGU36-28 | 09-06-00 | Poly Bag | 0.10 cu ft | N/A | Heat Comp. Test | 4 in. Dia. Halite/Polyhalite Core Sample. QGU36 subsample. |
| QGU37-15 | 09-07-00 | Poly Bag | 0.09 cu ft | N/A | Heat Comp. Test | 4 in. Dia. Halite/Polyhalite Core Sample. QGU37 subsample. |
| QGU37-20 | 09-08-00 | Poly Bag | 0.16 cu ft | N/A | Heat Comp. Test | 4 in. Dia. Halite/Polyhalite Core Sample. QGU37 subsample. |
| QGU37-45 | 09-08-00 | Poly Bag | 0.09 cu ft | N/A | Heat Comp. Test | 4 in. Dia. Halite/Polyhalite Core Sample. QGU37 subsample. |
| QGU37-48 | 09-08-00 | Poly Bag | 0.14 cu ft | N/A | Heat Comp. Test | 4 in. Dia. Halite/Polyhalite Core Sample. QGU37 subsample. |

enter n/a if none

**4. Sample Requirements**
Handling: Use Bubble Wrap and Core Boxes.
Storage & Preservation: Packed in Poly Bag to Preserve Moisture Content; Store in a Temperature Controlled Environment.
Shipping: Wood Crate and/or PVC Pipe.
Archive: N/A
Disposition: Observation and Destructive Testing; Dispose of in Regular Trash After Testing.
Expiration Date: N/A

| 5. Custody Transfer | Printed Name | Signature | Organization/Company | Date-Time | Sample Condition |
|---|---|---|---|---|---|
| a. Relinquished by: | Terry L. MacDonald | [signature] | SNL/6212 | 07-20-12/0800 | Intact |
| a. Received by: | Wesley F. DeYonge | [signature] | RESPEC/6212 | 07-20-12/0800 | Intact |
| b. Relinquished by: | Wesley F. DeYonge | [signature] | RESPEC/6212 | 7/26/12/ 1605 | Intact |
| b. Received by: | Rodger Arnold | [signature] | RESPEC | 7/26/12/1609 | Intact |
| c. Relinquished by: | | | | | |
| c. Received by: | | | | | |

Upon sample receipt, note condition. This form (copy for your records) shall follow samples through its life, until final disposition, then send original to WIPP Records Center. For samples that are potentially hazardous & require packaging and shipping, contact Center 6200 ES&H Coordinator or see SNL ES&H Manual, Chpt. 12.

**Figure 4a.** Chain-of-Custody Documentation for Waste Isolation Pilot Plant Salt Core (Page 1 of 2).

To prepare a testable specimen, a piece of salt core was sawn to an approximate length-to-diameter ratio of L:D = 2. The walls and ends of the cylinder were then machined in a horizontal lathe to produce a finished right-circular cylinder whose ends were flat, parallel, and perpendicular to the specimen sides. A typical machining setup is shown in Figure 5 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 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 very uniform and very near the typical value for halite (2.15 grams per cubic centimeter [g/cc]), which supports the previously referenced geological assessment that the specimens are relatively free of impurities.

Chain of Custody

SP 13-1
Revision 5
Page 5 of 5

Appendix A

| ACTIVITY/ PROJECT SPECIFIC PROCEDURE Sandia National Laboratories | **Chain of Custody** | **Form Number: SP 13-1-1** Page 2 of 2 Attach more forms as needed |
|---|---|---|

**1. Initial Sample Custodian** Terry L. MacDonald (Printed Name) Organization: 6212 Date: 07/16/2012

**2. Sample Collection or Creation Information** Scientific Notebook ID: ________
Test Plan ID: SNL-FCT-TP-11-0001 Field Log ID: ________
Sample Location: WIPP Underground, Q Room Southeast Corner, S-130/W-905 (i.e. borehole/core no./lab bldg. no./etc.)

Sample Team Members/Organization.
Terry L. MacDonald/SNL/6212
Wesley F. DeYonge/RESPEC/6212
enter n/a if none

| 3. Sample Identification Sample/Sub-Sample # | Date Collected | Container Type | Container Volume | Preser-vative | Analysis Request | Sample Description |
|---|---|---|---|---|---|---|
| QGU38-43-1 | 09-12-00 | Poly Bag | 0.09 cu ft | N/A | Heat Comp. Test | 4 in. Dia. Halite/Polyhalite Core Sample. QGU38 subsample. |
| QGU38-43-2 | 09-12-00 | Poly Bag | 0.12 cu ft | N/A | Heat Comp. Test | 4 in. Dia. Halite/Polyhalite Core Sample. QGU38 subsample. |
| QGU38-70 | 09-12-00 | Poly Bag | 0.08 cu ft | N/A | Heat Comp. Test | 4 in. Dia. Halite/Polyhalite Core Sample. QGU38 subsample. |
| QGU38-71 | 09-12-00 | Poly Bag | 0.08 cu ft | N/A | Heat Comp. Test | 4 in. Dia. Halite/Polyhalite Core Sample. QGU38 subsample. |
| -----------Last Item----------- | | | | | | |
| | | | | | | |
| | | | | | | |
| | | | | | | |
| | | enter n/a if none | | | | |

**4. Sample Requirements**

| | |
|---|---|
| Handling: | Use Bubble Wrap and Core Boxes. |
| Storage & Preservation: | Packed in Poly Bag to Preserve Moisture Content; Store in a Temperature Controlled Environment. |
| Shipping: | Wood Crate and/or PVC Pipe. |
| Archive: | N/A |
| Disposition: | Observation and Destructive Testing; Dispose of in Regular Trash After Testing. |
| Expiration Date: | N/A |

| 5. Custody Transfer | Printed Name | Signature | Organization/Company | Date-Time | Sample Condition |
|---|---|---|---|---|---|
| a. Relinquished by: | Terry L. MacDonald | [signature] | SNL/6212 | 07-20-12/0800 | Intact |
| a. Received by: | Wesley F. DeYonge | [signature] | RESPEC/6212 | 07-20-12/0800 | Intact |
| b. Relinquished by: | Wesley F. DeYonge | [signature] | RESPEC/6212 | 7/26/12/1605 | Intact |
| b. Received by: | Rodger Arnold | [signature] | RESPEC | 7/26/12 1609 | Intact |
| c. Relinquished by: | | | | | |
| c. Received by: | | | | | |

Upon sample receipt, note condition. This form (copy for your records) shall follow samples through its life, until final disposition, then send original to WIPP Records Center. For samples that are potentially hazardous & require packaging and shipping, contact Center 6200 ES&H Coordinator or see SNL ES&H Manual, Chpt. 12.

**Figure 4b.** Chain-of-Custody Documentation for Waste Isolation Pilot Plant Salt Core (Page 2 of 2).

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

**WIPP/QGU38-71/1**

where:

| | | |
|---|---|---|
| WIPP | = | Waste Isolation Pilot Plant |
| QGU38 | = | borehole identifier |
| 71 | = | core piece identifier |
| 1 | = | specimen piece number. |

**Figure 5.** Typical Horizontal Lathe Machining Setup for Preparing Cylindrical Specimens.

**Table 1. Summary of Salt Specimens Prepared for Testing**

| Specimen I.D. | Length (mm) | Diameter (mm) | Mass (g) | Density (g/cc) |
|---|---|---|---|---|
| WIPP/QGU37-20/1 | 204.13 | 90.97 | 2,859.60 | 2.16 |
| WIPP/QGU37-20/2 | 207.32 | 90.96 | 2,908.50 | 2.16 |
| WIPP/QGU37-15/1 | 205.34 | 89.74 | 2,809.40 | 2.16 |
| WIPP/QGU37-45/1 | 206.59 | 91.03 | 2,903.70 | 2.16 |
| WIPP/QGU36-17/1 | 204.04 | 89.53 | 2,776.60 | 2.16 |
| WIPP/QGU36-18/1 | 204.87 | 89.59 | 2,786.35 | 2.16 |
| WIPP/QGU36-27-2/1 | 206.76 | 89.39 | 2,791.95 | 2.15 |
| WIPP/QGU36-28/1 | 206.12 | 89.51 | 2,792.55 | 2.15 |
| WIPP/QGU37-48/1 | 206.54 | 89.53 | 2,798.40 | 2.15 |
| WIPP/QGU37-48/2 | 207.88 | 89.56 | 2,820.40 | 2.15 |
| WIPP/QGU38-43/1 | 206.98 | 89.59 | 2,816.85 | 2.16 |
| WIPP/QGU38-43-2/1 | 207.71 | 89.43 | 2,820.55 | 2.16 |
| WIPP/QGU38-71/1 | 207.23 | 89.54 | 2,820.80 | 2.16 |

During the machining process where the core was finished in a horizontal lathe, the operator observed that the cutting tool frequently encountered pockets of moisture (presumably brine) as the walls of the specimen were being trimmed to final dimension. Even though the core appeared to be dry on the surface when initially mounted in the lathe, as the cutting tool proceeded to make repeated small machining passes (reducing the radius by only about 0.3 millimeter [mm] per pass), wet spots began to appear, which were evidence of noninterconnected brine inclusions. The number of inclusions increased as the cutting depth increased with each machining pass, but they were observed even during the first pass. This indicates that some isolated brine inclusions existed within about 0.3 mm of the specimen surface. A photograph of a newly machined specimen is shown in Figure 6 and the wet brine spots are visible as dark round circles scattered on the surface of the specimen.

RSI-2075-12-022

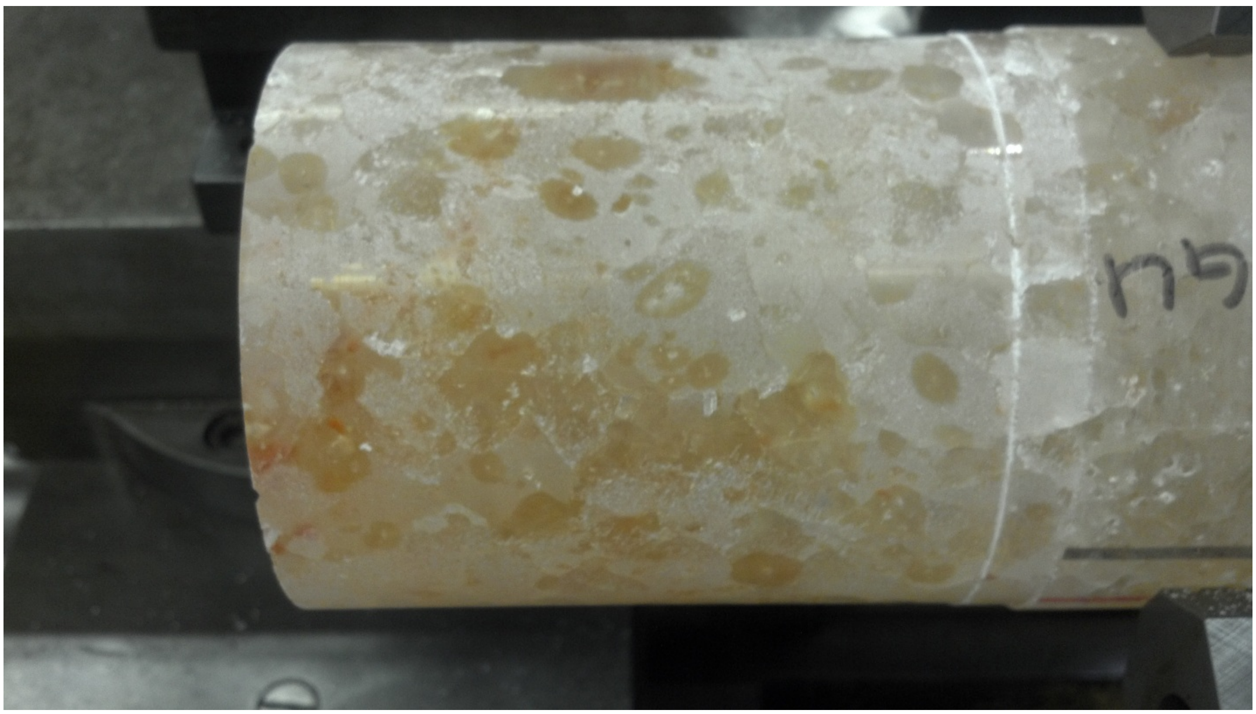

**Figure 6.** Waste Isolation Pilot Plant Salt Specimen Brine Inclusions (darker ovoid areas are wet).

## TEST EQUIPMENT

The testing was completed using a universal test system with four reaction columns referred to as the UTS4 system. The UTS4 is a computer-controlled, servohydraulic system manufactured by MTS Systems of Eden Prairie, Minnesota. The computer control allows for controlling the loading in either of two modes, a stress rate mode using the load cell output as a feedback signal or strain rate mode that uses a Linear Variable Differential Transformer (LVDT) output to control loading. An environmental chamber is mounted in the test system to provide the high-temperature environment required to perform unconfined tests at temperatures up to 300°C.

A picture of the test system is provided in Figure 7. The photograph illustrates the environmental chamber mounted in the test frame with the chamber door open for easy viewing of the interior of the chamber. Two salt specimens are inside the chamber. The specimen on the left is an Avery Island dome salt specimen instrumented with thermocouples to monitor salt specimen temperatures. There are some other thermocouples suspended in air that monitor the temperature of the air inside the chamber. Located in the load train in the middle of the chamber is a tested salt specimen (somewhat barrel shaped). Above and below the specimen are steel loading platens attached to long insulating rods that provide insulation between the hot specimen inside the chamber and the loading actuators outside the chamber. Just in view at the top of the photograph is the load cell that monitors axial loading force. An LVDT that monitors axial displacement is mounted inside the hydraulic actuator at the base of the system (not in view).

RSI-2075-12-023

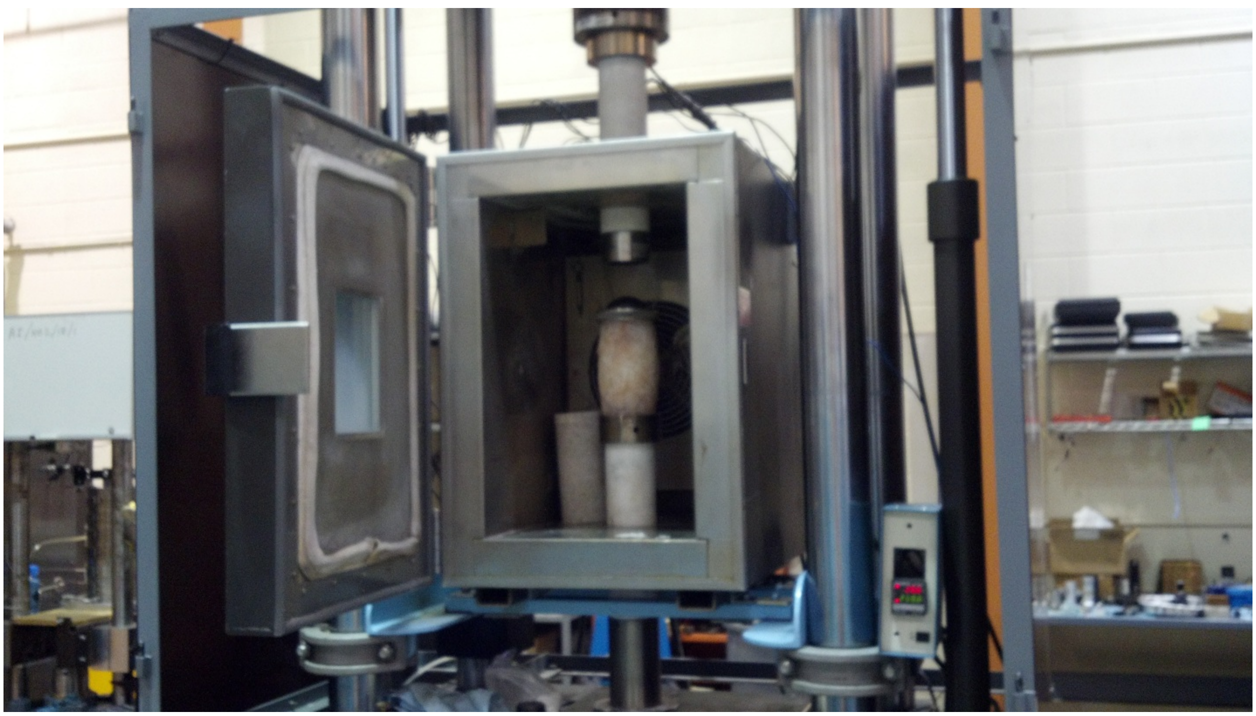

**Figure 7.** Uniaxial Test System Equipped With High-Temperature Environmental Chamber.

All three sets of instrumentation including the load cell, the LVDT, and the thermocouples were 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 ± 1 percent of reading, and the thermocouple temperatures are accurate to within ± 2°C. Because the LVDT measures total axial displacement including some nonspecimen contributions, a "machine softness" factor was determined that allowed correction of the LVDT measurements. Using a steel specimen for which accurate elastic parameters are known, the "machine softness" correction coefficient was determined to be 0.004 millimeter per kilonewton (mm/kN). This coefficient can be multiplied by the load cell reading and the product subtracted from the accompanying LVDT measurement to estimate the displacement of the specimen at that point.

## TEST PROCEDURE

Our proposed approach was to begin by performing three shakedown tests, one at each temperature. This is original research so the precise behavior of the salt specimen at these high temperatures was unknown; thus, it was assumed that the protocol described here could be adjusted after the first three tests were completed.

The test procedure was based on some underlying assumptions about the behavior of the salt. First, it was assumed that the salt would not decrepitate at these temperatures if a heating rate of about 1°C/minute was used. Second, it was assumed that the internal “structure” of a specimen is directly correlated in some fashion to the measured axial strain. This assumption implies that strain-controlled load paths would be beneficial because changes in load levels could be performed at the same strain levels in each test (i.e., the specimens would all be at the same “state” when the loads are changed) that would aid in comparison of test results to assess the effect of temperature.

The planned test procedure was defined by the following steps:

1. Bring the specimen to temperature at a rate of 1°C/minute. Thermal stabilization at the target test temperature would occur overnight. Multiple thermocouples were used to verify the air in the environmental chamber was well mixed by the chamber fan to eliminate thermal gradients surrounding the specimen.
2. Apply a small preload to the specimen (say about 0.2 MPa) to establish a reliable position to zero the LVDT (mounted in the hydraulic cylinder) used to measure axial displacement. The preload was based on a load measurement provided by a load cell located outside the environmental chamber.
3. Using the LVDT output, calculate axial strain in real time and apply deformation (load) at a strain rate of $10^{-4}$ $s^{-1}$ until reaching an axial strain level of 5 percent. This portion of the load path required 500 seconds (< 10 minutes).
4. Perform an unload/reload cycle. This step provides data for estimating a value for Young’s modulus. The unload/reload cycle is performed in load control and is completed quickly so the measured strain will be dominated by elastic deformation. At the end of the reload, resume loading at the original strain rate of $10^{-4}$ $s^{-1}$ until reaching a strain level of 10 percent. This entire step will require approximately another 10 minutes.
5. Perform another unload/reload cycle at the 10 percent strain level to obtain data for another estimate of Young’s modulus. When the reloading is completed, resume loading at the original strain rate of $10^{-4}$ $s^{-1}$ until reaching a strain level of 12 percent. This entire step will require less than 5 minutes.
6. At a strain level of 12 percent, reduce the controlled strain rate from $10^{-4}$ $s^{-1}$ to zero. This initiates a stress-relaxation test that will provide data for assessing the time-dependent deformation of the salt. The stress should display an exponential decay, and it is

assumed that the stress will be asymptotically approaching zero within a few hours. Nonetheless, the stress-relaxation phase will be allowed to continue overnight, so the next (and final) loading step will be initiated the following day.

7. The controlled strain rate is increased from zero to $10^{-4}$ $s^{-1}$, and loading continues until one of the following test termination criteria are met: (1) the specimen fails, (2) the specimen exhibits a flat stress-strain response (perfectly plastic), or (3) the specimen becomes malformed to an extent that cylindrical geometry assumptions become grossly inadequate. It is anticipated that criterion (3) could become evident at axial strains above 20 percent.

8. Test termination simply entails removal of all load and heating. When the specimen has cooled, it is preserved for possible posttest analyses that are undetermined at this point.

The above test steps were planned on three different specimens each at a different temperature; namely, 200°C, 250°C, and 300°C. These three tests were to provide valuable information for possible changes in completing the remaining six tests in the planned test matrix. In addition, these tests could provide useful data for evaluating the inelastic high temperature dependency of salt through the constant strain rate and relaxation portions of the tests and the elastic high-temperature dependency of salt through Young's modulus estimates obtained from the unload/reload portions of the tests. All specimens were preserved for possible posttest analyses that are undetermined at this time.

## TEST RESULTS

A total of four uniaxial tests were attempted—two at 300°C and one each at 200°C and 250°C. The two tests at 300°C were unsuccessful because the specimen decrepitated at temperatures less than 300°C. The tests at 200°C and 250°C were completed successfully. The test matrix is summarized in Table 2.

**Table 2. Test Matrix Summary**

| Specimen I.D. | Temperature (°C) | Status/Comment |
|---|---|---|
| WIPP/QGU37-20/1 | 200 | Successful using proposed test protocol |
| WIPP/QGU37-15/1 | 250 | Successful using proposed test protocol |
| WIPP/QGU37-20/2 | 300 | Violent decrepitation at about 280°C before loading |
| WIPP/QGU37-45/1 | 300 | Violent decrepitation at 285°C before loading |

The two tests attempted at 300°C were similar in that both of them exhibited a violent decrepitation as the specimen temperature (as estimated by thermocouples located along the central axis of the Avery Island salt specimen collocated in the test chamber) reached about 280°C and no mechanical loading could be applied. Thus there is no stress or strain data available for these tests. However, some observations can be made about how the decrepitation apparently did not depend on the rate of heating. For example, specimen WIPP/QGU37-20/2 was placed in the environmental chamber and the chamber temperature was ramped to 300°C at a rate of 1°C/minute. The decrepitation occurred at about 280°C with a violent explosion that reduced the specimen to rubble. Anticipating that the heating rate might have been too fast, the next specimen, WIPP/QGU37-45/1, was heated to just 250°C at a rate of 1°C/minute and then allowed to remain at 250°C for 24 hours. After 24 hours, the test chamber temperature was ramped to 300°C at a rate of 1°C/minute, and when the specimen temperature reached 285°C (as indicated by the thermocouples in the Avery Island specimen), violent decrepitation caused the top third of the specimen to explode into rubble. At that point, the heat was turned off, but about 5 minutes later the remaining two-thirds of the specimen also exploded, effectively reducing the entire specimen to rubble.

The pretest photographs of both specimens are shown in Figures 8 and 9. A posttest photograph shown in Figure 10 is typical of the rubble that remains after the violent decrepitation occurs. Note in Figure 10 that the Avery Island domal salt specimen still remained intact throughout both attempts at 300°C tests on the WIPP salt, which indicates that there is obviously some difference between the two salt types. The difference may be the presence of the brine inclusions noted during specimen preparation, but no extensive petrologic studies were attempted during this stage of the project.

RSI-2075-12-024


**Figure 8.** Pretest Photograph of Specimen WIPP/QGU37-20/2.

RSI-2075-12-025


**Figure 9.** Pretest Photograph of Specimen WIPP/QGU37-45/1.

RSI-2075-12-026

**Figure 10.** Posttest Photograph of Rubble of Specimen WIPP/QGU37-20/2.

The violent decrepitation observed in the two tests at 300°C is not unique to WIPP salt. Similar observations were made during the Project Salt Vault investigation in Lyons, Kansas, and reported by Bradshaw and McClain[1]. They reported observations characterized as “trapped moisture effects” that were very similar to our observations. Salt recovered from Hutchinson, Kansas, was heated and found to exhibit violent fracture at about 280°C, and the decrepitation temperature did not appear to depend on heating rate. They reported that the explosion was violent enough to lift an oven door and rupture a wire basket that was used to contain the salt sample being heated. Bradshaw and McClain considered three explanations for the decrepitation including differential thermal expansion, chemical reactions, and pressure effects of brine inclusions. They concluded that the prime cause was likely the increase in pressure resulting from the heating of the brine inclusions in the salt. Bradshaw and McClain also reported on decrepitation studies performed on salt from several other locations and found that bedded salts tended to exhibit decrepitation at about 250°C to 380°C, but no domal salts exhibited any decrepitation even at temperatures up to about 400°C. This finding is generally consistent with our observation that the Avery Island domal salt in our testing did not decrepitate even though it was exposed to the same elevated temperatures as the WIPP bedded salt specimens.

The uniaxial tests at 200°C and 250°C were successful, and the results of those tests are presented using graphs of stress and strain measurements made during the mechanical loading after the specimens had established equilibrium at their specified temperature. For all test results, axial strain is derived by first correcting the LVDT measurement for machine softness and then using the corrected LVDT displacement value to determine the current specimen length. Axial strain is then calculated as the natural logarithm of the ratio of current specimen length to original specimen length (using a sign convention of compression positive). Isochoric deformation is assumed so the lateral strain is estimated as the opposite sign value of one half the axial strain, and an updated value for the cross-sectional area of the specimen can be obtained. The current axial stress is then calculated as the ratio of the load cell measurement to the current specimen area.

The test results for the uniaxial test at 200°C are presented in Figures 11, 12, and 13 that are plots of the overall test history, the unload/reload cycles at the beginning of the test, and the final loading to ultimate strength performed at the end of the test, respectively.

The plots in Figure 11 are dominated by the stress-relaxation stage of the test where the axial strain was held constant at 12 percent overnight after the initial loading. As expected, the axial stress decreased substantially and began to stabilize at near 4 MPa before initiating the final loading to determine ultimate strength. The stress relaxation did not proceed smoothly, but rather, appeared to proceed in what might be described as a stick-slip fashion. This may be an accurate reflection of how the specimen substructure is changing, or it might indicate some inconsistency in the real-time calculation of stress and strain during the interval; however, no specific cause for the stick-slip behavior is offered yet.

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[1] **Bradshaw, R. L. and W. C. McClain, 1971.** *Project Salt Vault: A Demonstration of the Disposal of High Activity Solidified Wastes in Underground Salt Mines*, ORNL-4555 prepared by Oak Ridge National Laboratory for the U.S. Atomic Energy Commission.

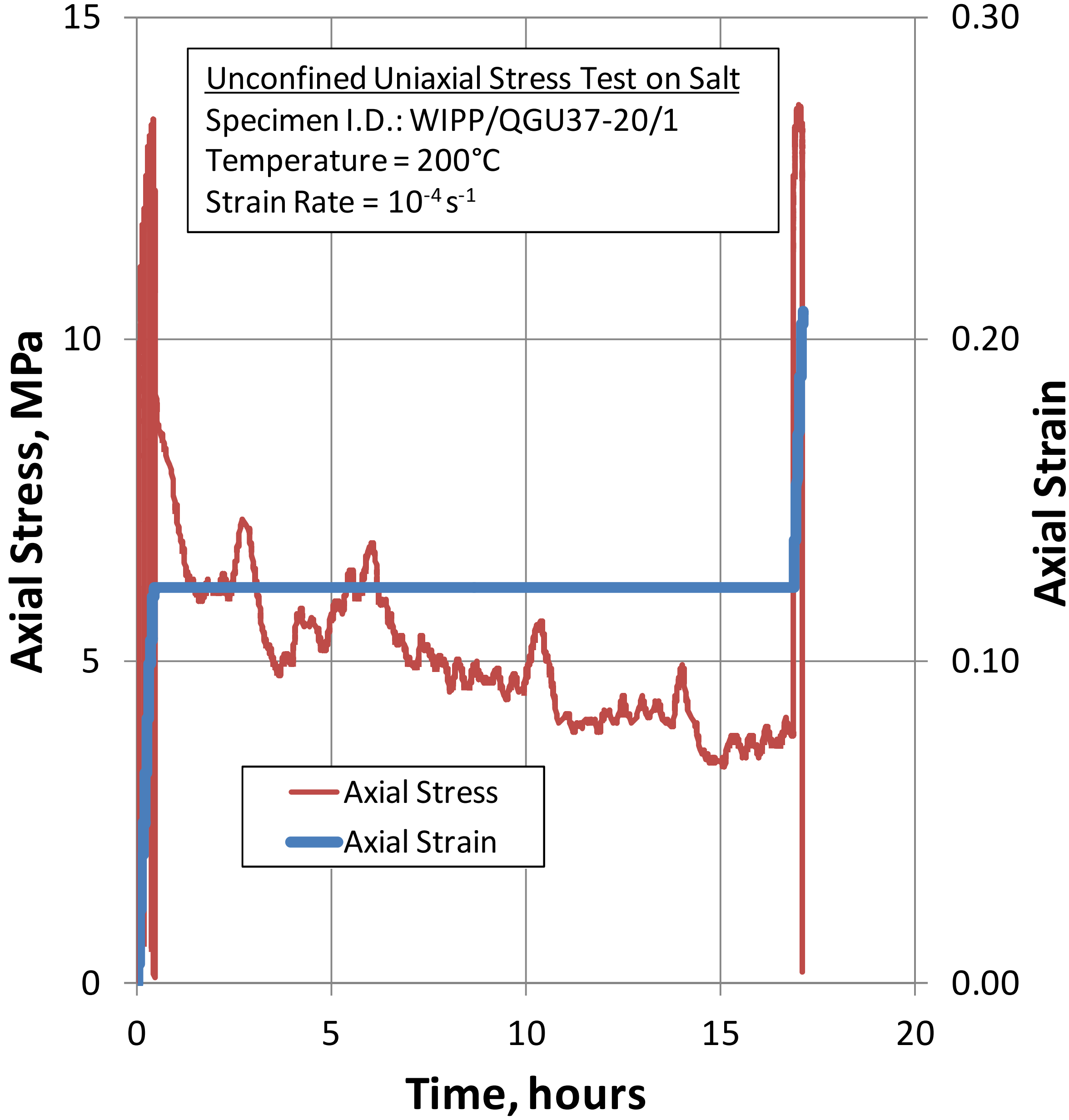


**Figure 11.** Complete Test History for Uniaxial Test on Specimen WIPP/QGU37-20/1 at 200°C.

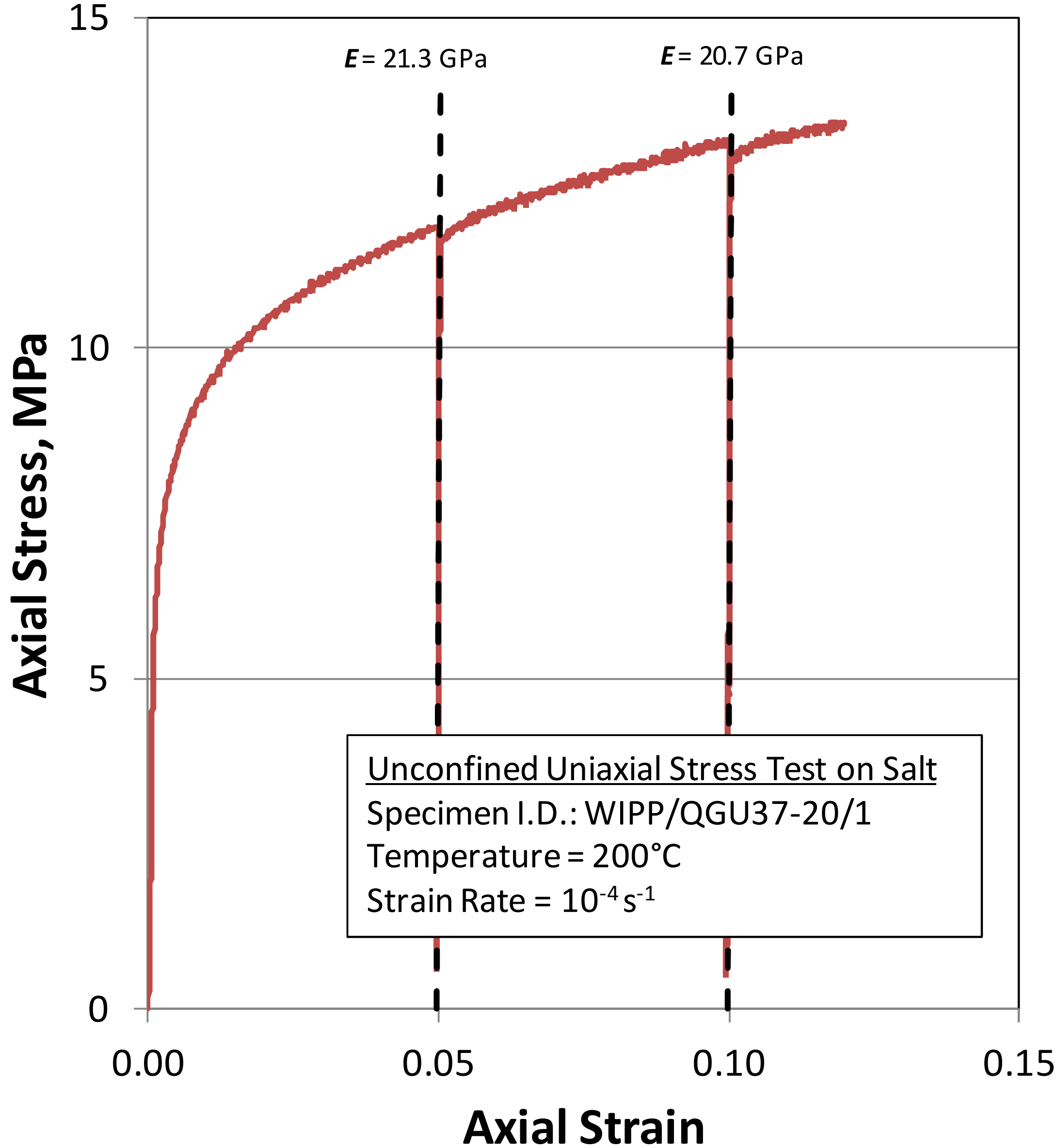


**Figure 12.** Unload/Reload Cycles on Specimen WIPP/QGU37-20/1 at 200°C.

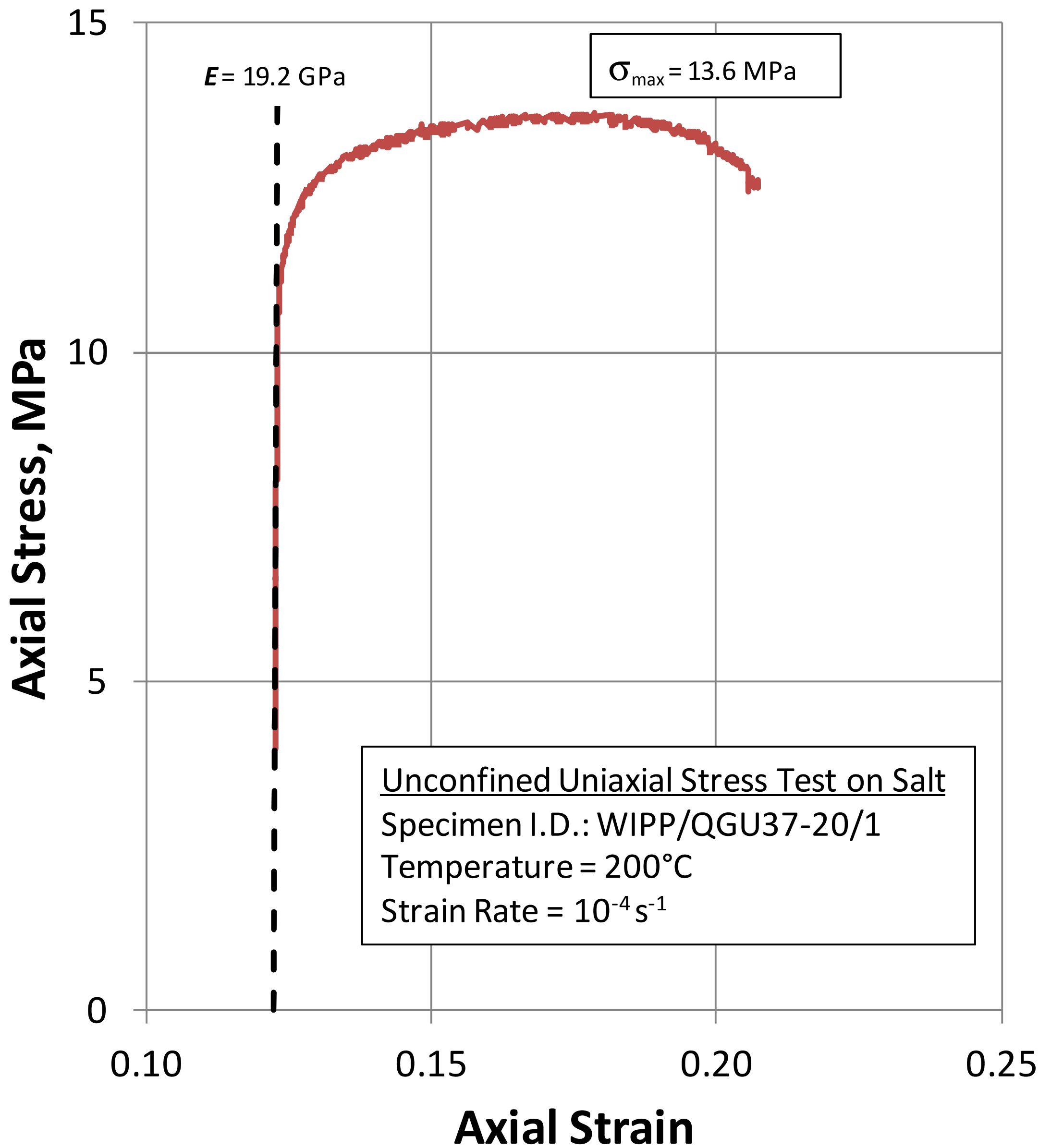


**Figure 13.** Final Loading to Ultimate Strength for Specimen WIPP/QGU37-20/1 at 200°C.

The unload/reload cycles provided in Figure 12 are typical for salt in that they exhibit very linear (elastic) behavior when the stress is less than whatever maximum stress had preceded the onset of the unloading. Fits to the linear interval of the reload portion of the cycles provides estimates of Young's modulus and those values are shown on the plot. The plot ends at an axial strain of 12 percent because that is the strain level where the stress-relaxation portion of the test began. The Young's modulus fits indicate that there might be some small effect of the strain level on the elastic constants because the fitted value at a strain of 10 percent is slightly smaller than the value estimated at a strain of 5 percent. However, the effect (if real) appears to be very small. This observation would indicate the deformation is dominated by ductile processes rather than brittle or dilatant behavior.

The final loading to determine ultimate strength was performed after the stress-relaxation stage was complete, so the axial strain was 12 percent when loading began, as illustrated in Figure 13. An ultimate axial stress of 13.6 MPa was clearly defined because loading was continued until the specimen began to lose its ability to sustain further increases in axial stress and actually began to exhibit some postpeak weakening. Another estimate of Young's modulus was obtained from the initial loading, and it indicated that the elastic constants had decreased somewhat during the stress-relaxation stage but only by about 5 percent.

The test results for the uniaxial test at 250°C are presented in Figures 14, 15, and 16 that are plots of the overall test history, the unload/reload cycles at the beginning of the test, and the final loading to ultimate strength performed at the end of the test, respectively.

The plots in Figure 14 are dominated by the stress-relaxation stage of the test where the axial strain was held constant at 12 percent overnight after the initial loading. As expected, the axial stress decreased substantially and began to stabilize at near 2 MPa before initiating the final loading to determine ultimate strength. The stress relaxation did not proceed smoothly, but rather, appeared to proceed in what might be described as a stick-slip fashion. This is the same observation that was made for the test performed at 200°C.

The unload/reload cycles provided in Figure 15 are typical for salt in that they exhibit very linear (elastic) behavior when the stress is less than whatever maximum stress had preceded the onset of the unloading. Fits to the linear interval of the reload portion of the cycles provide estimates of Young's modulus, and those values are shown on the plot. The plot ends at an axial strain of 12 percent because that is the strain level where the stress-relaxation portion of the test began. The Young's modulus fits indicate that there might be some small effect of the strain level on the elastic constants because the fitted value at a strain of 10 percent is slightly smaller than the value estimated at a strain of 5 percent. However, the effect (if real) appears to be very small. This observation would indicate the deformation is dominated by ductile processes rather than brittle or dilatant behavior.

The final loading to determine ultimate strength was performed after the stress-relaxation stage was complete, so the axial strain was 12 percent when loading began, as illustrated in Figure 16. An ultimate axial stress of 12.5 MPa was clearly defined because loading was continued until the specimen began to lose its ability to sustain in further increases in axial stress and actually began to exhibit some postpeak weakening. Additional estimates of Young's modulus were obtained from the initial loading and a second loading required to reset the

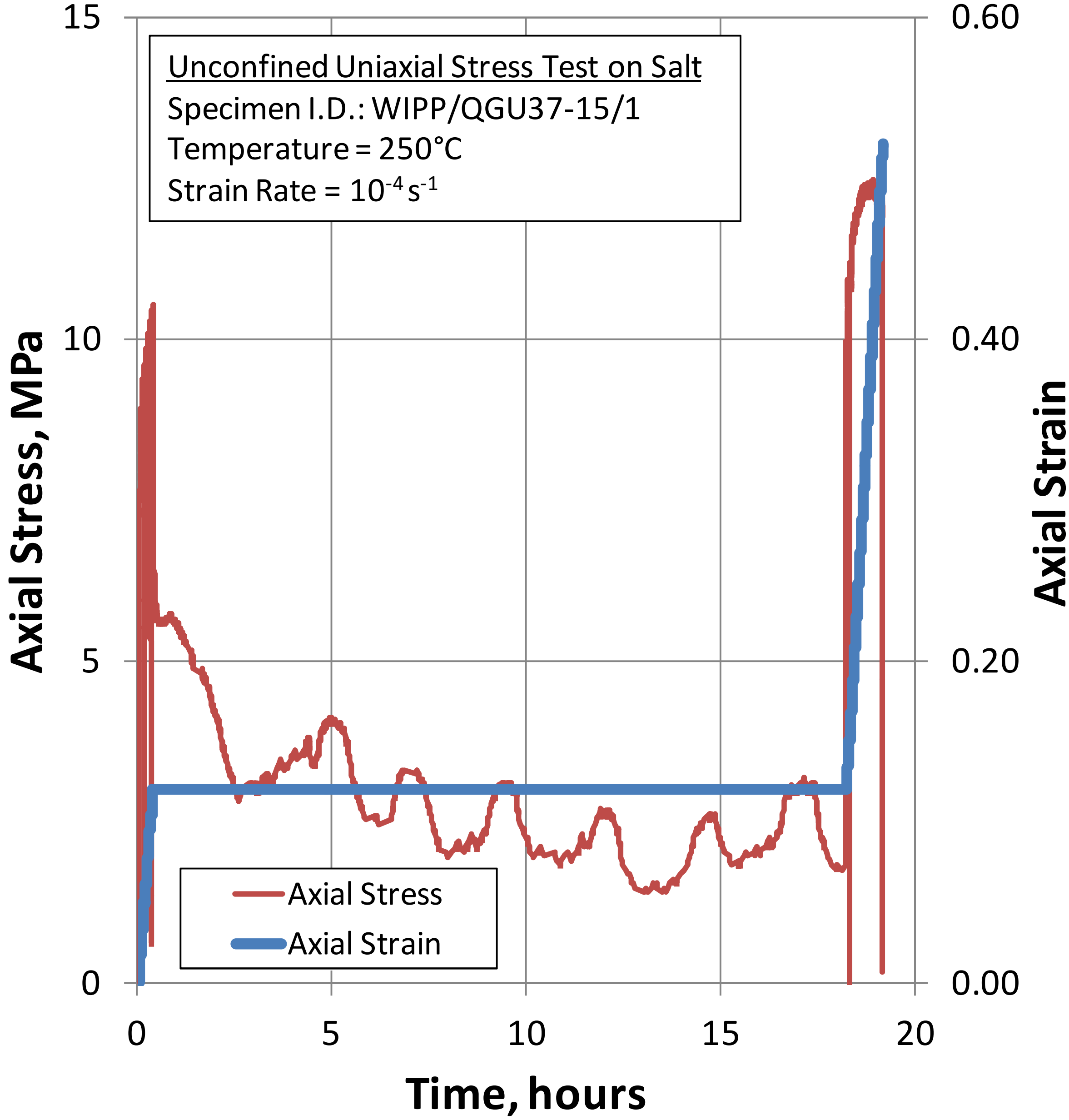


**Figure 14.** Complete Test History for Uniaxial Test on Specimen WIPP/QGU37-15/1 at 250°C.

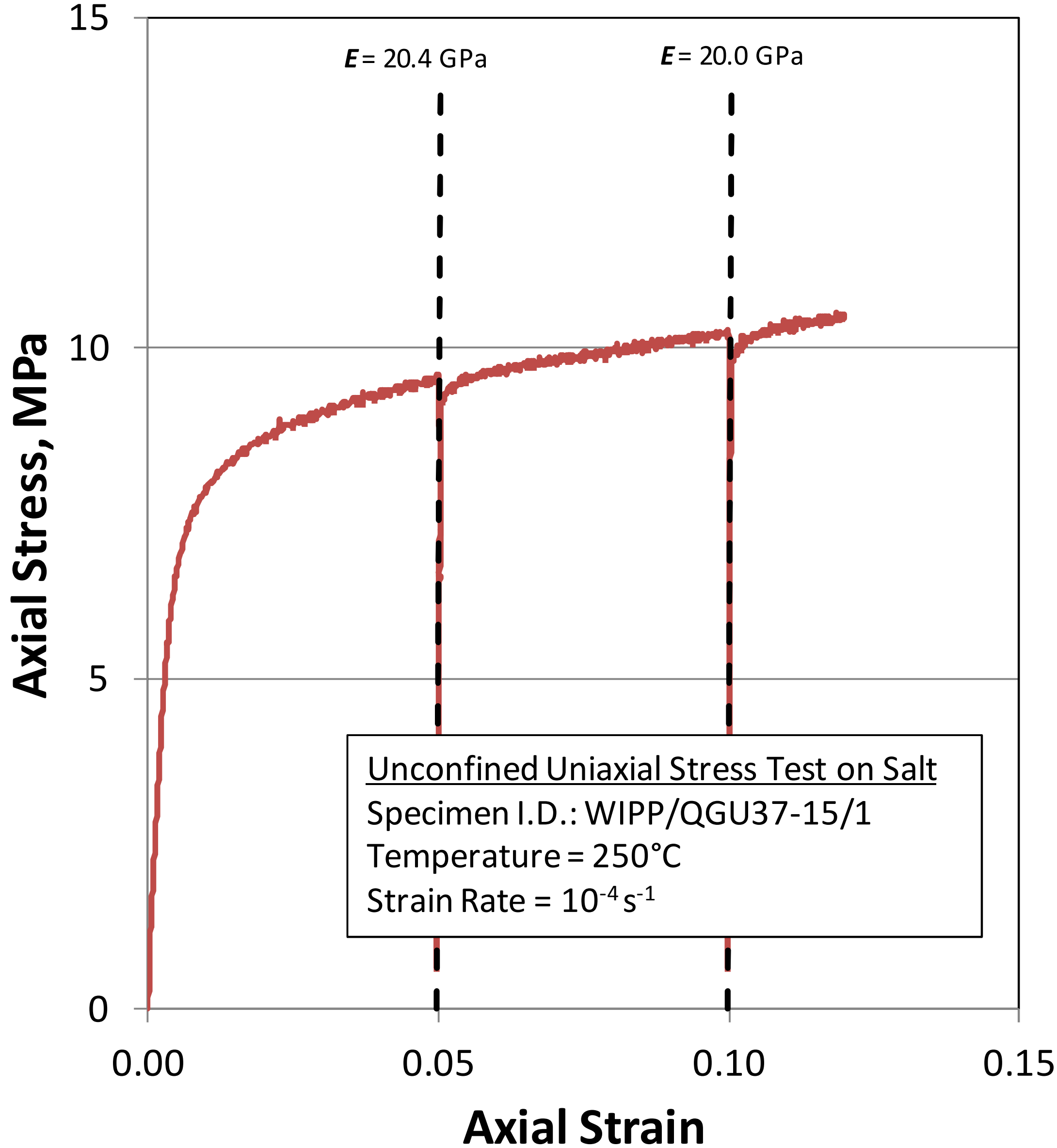


**Figure 15.** Unload/Reload Cycles on Specimen WIPP/QGU37-15/1 at 250°C.

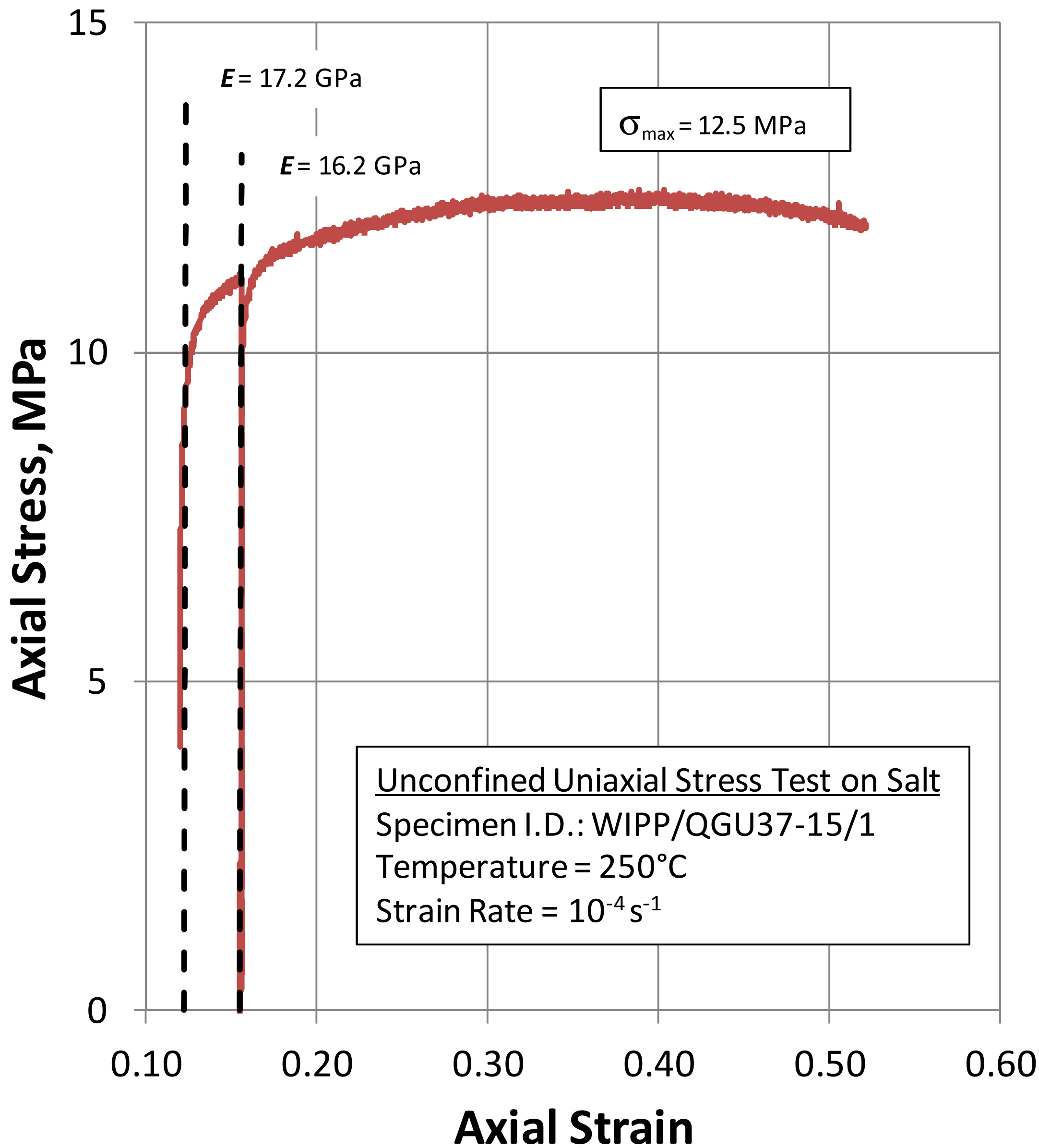


**Figure 16.** Final Loading to Ultimate Strength for Specimen WIPP/QGU37-15/1 at 250°C.

LVDT. These unload/reload cycles indicated that the elastic constants had decreased by perhaps as much as 15 percent or 20 percent during the stress-relaxation stage. This decrease might indicate some softening of the salt is being enhanced by the higher temperature in this test.

The general test results for the tests at 200°C and 250°C are summarized in Table 3. Some general comments are that Young's modulus tends to decrease with an increase in strain or temperature, at higher temperature less stress is required to induce specific strain levels, ultimate strength decreases with an increase in temperature, and the strain at ultimate strength increases with an increase in temperature. In both tests, the specimens sustained extremely high deformations while still maintaining some significant residual strength, although macroscopic vertical cracks were becoming evident by the time the tests were terminated. The posttest appearance of the specimens is displayed in the photographs provided in Figures 17 and 18.

**Table 3. Summary of Test Results**

| Specimen I.D. (Temperature) | Strain Level (%) | Stress (MPa) | Young's Modulus (GPa) | Comment |
|---|---|---|---|---|
| WIPP/QGU37-20/1 (200°C) | 5 | 11.8 | 21.3 | First unload/reload |
| | 10 | 13.1 | 20.7 | Second unload/reload |
| | 12 | 13.4 | — | Start of stress relaxation |
| | 12 | ~4 | 19.2 | End of stress relaxation |
| | ~18 | 13.6 | — | Ultimate strength |
| | 21 | 12.6 | — | Test termination |
| WIPP/QGU37-15/1 (250°C) | 5 | 9.6 | 20.4 | First unload/reload |
| | 10 | 10.2 | 20.0 | Second unload/reload |
| | 12 | 10.5 | — | Start of stress relaxation |
| | 12 | ~2 | 17.2 | End of stress relaxation |
| | 15.5 | 11.1 | 16.2 | Unload/reload for LVDT reset |
| | ~40 | 12.5 | — | Ultimate strength |
| | 52 | 11.9 | — | Test termination |

**Figure 17.** Posttest Photograph of Specimen WIPP/QGU37-20/1 Tested at 200°C.

**Figure 18.** Posttest Photograph of Specimen WIPP/QGU37-15/1 Tested at 250°C.

## RECOMMENDATIONS

The intent of performing this initial suite of uniaxial tests was to obtain a general sense of the salt deformation phenomena that could be expected of WIPP salt. A conceptual test matrix of nine tests was envisioned to investigate characteristics at temperatures up to 300°C, and these first three tests had to be attempted to evaluate the need for modifying that original vision of a high-temperature test matrix.

The initial test results presented in this memorandum clearly demonstrate that the WIPP salt will likely decrepitate (probably violently) at temperatures of about 280°C when the specimens are unconfined. It is also likely that this behavior cannot be affected by changing heating rates because the decrepitation is probably caused by a thermally driven pressure increase in entrapped brine inclusions.

The testing is scheduled to be performed at unconfined conditions, so we would recommend that future testing be limited to temperatures no greater than 275°C. Under confined conditions, the pressurized brine inclusions probably could not cause decrepitation, but confined tests are currently outside the scope of testing and would very likely require significant investments in sealing technology and development of expertise in untried test procedures. These limitations would be costly to overcome.

The test protocols for the mechanical testing worked very well for the two specimens that were tested at lower temperatures where decrepitation was not a problem. Thus we would recommend that the test protocol as documented in this memorandum be used for future testing in this project.

When the next six tests are completed, the reasonably large database with replicated tests can provide some sense of the degree of uncertainty one might expect in the high-temperature regime. The ultimate goal would be to have the ability to model that high-temperature behavior using tools that are already available. We recommend that the test results be modeled *a priori* using WIPP salt parameters already defined in the literature and accepted for use in modeling field studies at the WIPP. These salt parameters are based on data generated at lower temperatures, but the predictions will simply extrapolate outside that existing database and generate results that can be compared to the unconfined laboratory results that will be obtained from the proposed testing.

All of the work produced in the next round of tests (along with the test results in this memorandum) will be documented in a formal report. That report will also contain the results of the numerical simulation comparisons to provide some indication of how robust current modeling approaches are when dealing with temperature well in excess of the previously investigated limit of about 200°C.

KDM:krl

# ATTACHMENT A

# GEOLOGIC CORE DESCRIPTION

Dennis W. Powers, Ph. D.
*Consulting Geologist*

March 12, 2001

**Terry MacDonald and Frank Hansen**
*Sandia National Laboratories-Carlsbad*
4100 National Parks Highway
Carlsbad, NM 88220

Dear Terry and Frank:
Here is an interim summary of features and proposed activities regarding the 5 cores from the Disturbed Rock Zone I re-examined last week. These descriptions are subject to revision in the final report now in preparation.

**QGU 12**

Core was taken at about the contact between MU 4 & 5. It shows extensive subhorizontal clay laminae that developed in "salt saucers" at the depositional surface, and the core samples what would commonly be called "clay f".

Fractures were only observed within 0.3 ft of the rib face, but they have the largest aperture of any fractures. There may have been salt plucking or clay erosion by air flow during drilling.

At about 0.4 ft depth, brown clay displays slickensides @ an angle of about 45° from vertical, with the lower end of the slickensides closer to the rib face and upper end downhole. I suggest these slickensides are more likely depositional (pedogenic) than structural, but there is no way to be certain.

There is no observed strain beyond a depth of about 0.3 ft. It seems likely that the clay is absorbing strain with no visible effects, and the halite is not subject to sufficient stress within this regime to develop features observed in halite rock without significant clay.

*Recommended program*: photograph unwrapped core from 0-4.5 ft at close range from a side view; slab from 0-4.3 ft along vertical axis to see fractures and depositional features; make one cut ⊥ to the core axis at about 4.4 ft to display clay textures; re-examine, re-photograph, and re-describe slab as appropriate.

**QGU 36**

Core was taken in the lower part of MU 3. It shows coarse clear halite with few impurities.

Halite grains appear to be elongate (l:w is ~ 2:1) along a plane parallel to the rib wall due to fracturing across crystals. Strain planes due to these fractures are parallel to the rib wall near the wall, with the angle to the long core axis increasing beyond about 2.8 ft. At about 4.8 ft, strain planes are ⊥ to the long core axis, and they become more abundant beyond about 6.5 ft. Fractures in this core have no macroscopically measurable aperture.

Core breaks are parallel to the rib face near the wall; beyond about 4.8 ft, breaks tend to be ⊥ to long core axis. Break spacing is ~ 1 ft to depth of 9 ft, then is shorter to TD.

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The breaks on this core, especially near the rib face, show cleavage control as follows:

- coarse crystals (> ~ 1 inch) tend to break along the cleavage face closest to the plane of the strain fabric;
- smaller crystals tend to break along a series of close-stepped cleavages along the 2 cleavage planes where the average of these 2 planes is close to the plane of the strain fabric. The close-stepped cleavages generally appear macroscopically to have cleavage face widths in ratios between about 1:1 and 2:1.
- There are only a few instances of conchoidal to hackly fracture on these crystals.

*Recommended program:* Close-range photographs with scale of top, bottom, and one side of the core to show fabrics; slab along horizontal plane full length of core; re-examine and re-describe as appropriate; photograph one side of slab at close range.

**QGU 37**

This core has similar geology to QGU 36 and 38. It was drilled in the alcove corner.

Strain planes in this core are all ⊥ to the long axis of the core. This core is much more disked than 36 or 38 to a depth of about 14.5 ft. Beyond that depth, the core generally is in lengths of 6 inches to 1 ft. In addition, core breaks near the rib wall show a convex face on the core end nearest the rib and a fitted concave face downhole.

The crystal relief on the core breaks of QGU 37 appears to be less than that on 36. In addition, the crystals appear to have more hackly or conchoidal surfaces, few large cleavages, and very common stepped cleavage with curvature.

I suggest that the greater disking in the first 15 ft, strain planes ⊥ to the long core axis, and convex/concave breaks near the rib face are all a consequence of the location at the corner of the Q Room Alcove. It seems to me that the stress is likely to be concentrated more tightly near the corner than along the planar face and may account for all these features. I wonder if this may also account for the apparent greater fracturing rather than cleavage control on core break surfaces.

*Recommended program:* none at this time. May be useful to slab the first 2 ft along horizontal plane later to compare dislocation densities.

**QGU 38**

The geology of this core is similar to 36 and 37.

There are many similarities between 36 and 38 in fabric, as the crystals again show strain planes parallel to the rib face near the rib face. These strain planes are no longer observable beyond 4.5 ft. Strain planes ⊥ to the core axis are apparent from about 3.6 ft, co-existing with the angled strain planes.

Disking becomes much more intense from about 4.5 to 11 ft, and strain planes ⊥ to the core axis are faint or not observable beyond about 10 ft.

*Recommended program:* Close-range photographs with scale of the core from the top and one side from 0 to TD where core pieces are larger than about 4 inches; slabb from 0 - 4.5 ft along horizontal plane (?after results of QGU 36 are available).

**QGU 39**

QGU 39 was cored in the upper middle part of MU 0, at the point where clay content is beginning to increase vertically. Much of the first 7 ft of the core was taken along the edge of a dissolution pipe and shows the lateral transition between finer opaque halite to coarse clear halite, sometimes with clay concentrations along this boundary.

The core shows some of the same fabric parallel to the rib face that is observed in QGU 36 and 38. The "foliation" angle to the long axis of the core begins to increase at about 3 ft and is not discernible beyond about 3.3 ft depth.

Several fractures with discernible aperture are located at 0.75, 0.80, and 1.25 ft, and these fractures tend to parallel the rib face. Core breaks are ⊥ to the long core axis, and the rock quality is generally good in this core. There are short intervals of disking.

*Recommended program:* close-range photos with scale of top and one side of total core prior to slabbing; slab from rib face to about 7 ft along horizontal plane; examine slabs; redescribe and rephotograph as appropriate; make vertical cut ⊥ to long core axis at 7.4 ft to show clay/halite relationships and possible clay illuviation.

**Priorities**

From the recommendations above, the first priority is QGU 36, mainly because it is the core that shows the most interesting features and is well-preserved. From it, better ideas can be derived of the significant features to check on other cores.

I think the second and third priorities are the more limited items on QGU 39 and 38, probably in that order. Additional slabbing, to greater depths, may be considered if the dislocation densities begin to show patterns of interest or if other features of interest develop.

The last priority right now is probably QGU 12. Although it is interesting sedimentologically, there is very limited macroscopic evidence of strain in the DRZ (~ 0 - 0.3 ft). It will certainly be useful to compare a core like this from high on the rib face to those in the middle (QGU 36, 38) and lower part (QGU 39).

Please circulate these preliminary results as you see appropriate to develop consensus on the next steps in relating strain features to the DRZ and its properties. I am working on a report that will provide more detail of these observations.

Sincerely,

electonic file without signature

Dennis W. Powers