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**Sandia National Laboratories
Waste Isolation Pilot Plant**

Test Plan TP 17-03

**Experimental Investigation of WIPP Salt Samples
and Clay Seams**

Task 4.4.2.6.1

Revision 1

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1 ABBREVIATIONS, ACRONYMS AND INITIALISMS

ES&H	Environment, Safety & Health
NIST	National Institute of Standards and Technology
NP	Nuclear Waste Management Procedure
RESPEC	RESPEC Inc. (Rapid City, SD)
WEIMOS	Verbundprojekt: <u>We</u> iterentwicklung und Qualifizierung der gebirgsmechanischen <u>M</u> odellierung für die HAW-Endlagerung im <u>S</u> teinsalz (Collaborative project: Further development and qualification of rock mechanical modeling for final storage of high-activity waste (HAW) in rock salt).
WIPP	Waste Isolation Pilot Plant

2 REVISION HISTORY

The initial issuance of this Test Plan was effective as of July 27, 2017. That initial version described shear tests on salt samples containing either interfaces with other materials, or seams of clay within the salt sample.

The current revision of this Test Plan, Revision 1, was completed in May 2019. Under Revision 0, all the original tests that were planned were completed and documented, and the corresponding technical notebook was reviewed and approved. After the completion of those tests, the principal investigator and the testing organization agreed that a second series of tests is desired. This second series of tests will utilize samples comprised of leftover salt from cores obtained from the original tests, and manufactured clay seams made of bentonite obtained from a commercial mine in Belle Fourche, South Dakota. The purpose of these tests is to provide test data from clay seams that are more visually analogous to the seams observed near the WIPP horizon than the clay/salt interfaces obtained for the original set of tests. In addition, it is possible that new core samples containing clay seams may be obtained from the WIPP site during the timeframe of these tests; if so, it is intended to create test samples from these cores and perform similar tests on them as the original cores.

3 PURPOSE AND SCOPE

This Test Plan describes laboratory experiments designed to measure, evaluate and quantify the effects of shear displacement along a bedding interface or clay seam on shear and fracture strength of the interface and accompanying salt. The purpose of these lab tests is to provide data that will be used to develop a constitutive model for shear strength and fracturing along bedding interfaces and seams. This model is intended to improve understanding of shear stresses and strains on bedding interfaces that can be translated to current geomechanical and WIPP performance assessment models.

In addition to applications directly related to WIPP, the data from these tests will be used to support US-German collaborative model development efforts for the Joint Project WEIMOS (2016 – 2019; “Further Development and Qualification of the Rock Mechanical Modeling for the Final HAW Disposal in Rock Salt”). Extensive collaborations with German salt repository researchers have identified four key research areas to better understand the behavior of salt for radioactive waste repositories (Hansen et al., 2016b and 2017). One subject area includes the influence of nonhomogeneities. No characterization testing has been done and nonhomogeneities have first-order effects.

There are essentially no in situ measurement data characterizing shear strength of an interface in salt and resulting effects of interface displacement and permeability. Munson & Matalucci (1983) proposed an in situ test with direct shear across a clay seam. A 1-by-1-m block in a wall containing a representative clay seam would be isolated by cutting around it. Flatjacks were proposed to be installed in slots cut around the block to apply shear and normal stresses. Displacements along and across the seam would be measured as a function of applied stress. This proposed test never occurred.

Some laboratory and computational investigations have evaluated the slip along interfaces under several different stress environments. Minkley & Mühlbauer (2007) performed direct shear laboratory tests on carnallite and salt blocks under varying normal and shear loads and shear velocities, as shown in Figure 1. With these data, they developed a shear model for interfaces that accounts for both velocity-dependent and displacement-dependent shear softening mechanisms. The plots in Figure 2, taken from Minkley &

Mühlbauer (2007), show the evolution of shear stress as a function of shear displacement for two different shear velocities.

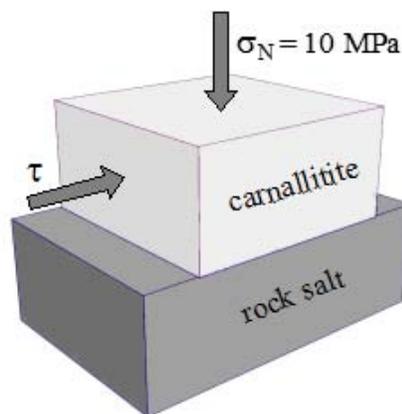
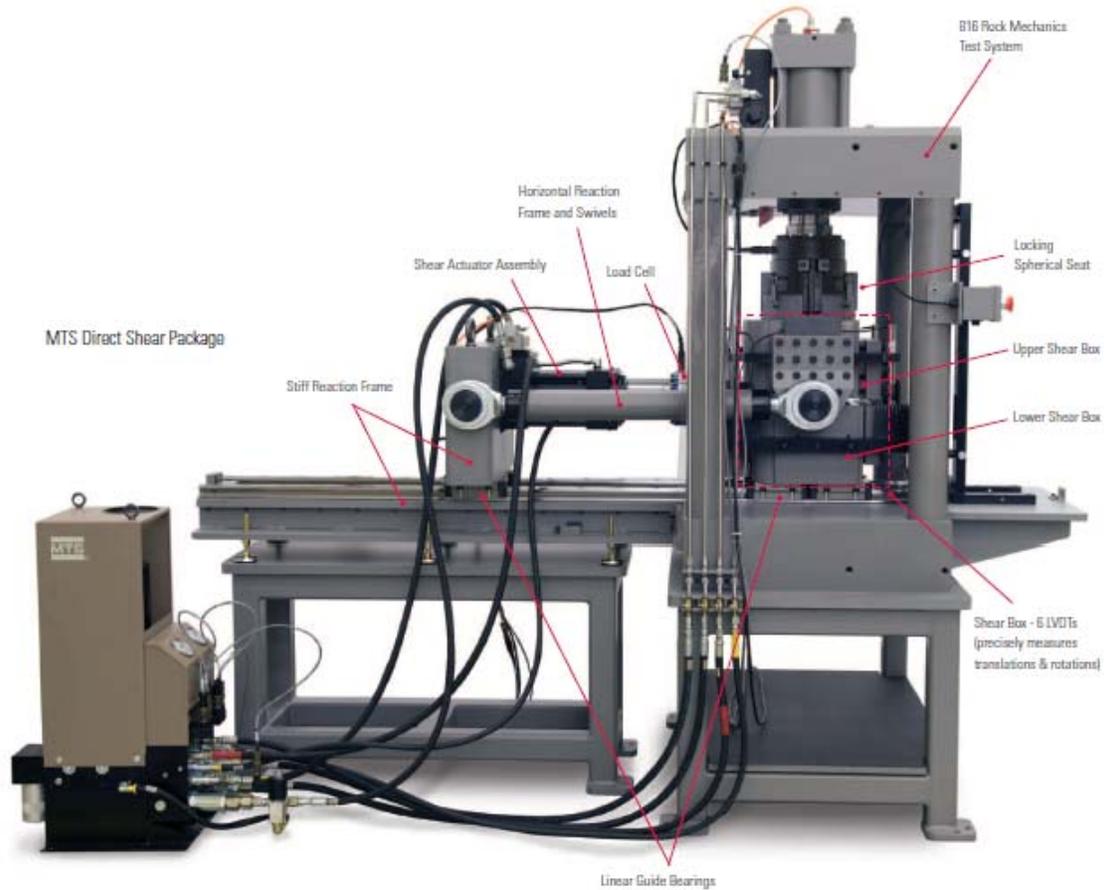


Figure 1. Direct shear test setup (Photograph of MTS direct shear machine from MTS website; bottom figure shows test setup employed by Minkley & Mühlbauer (2007)).

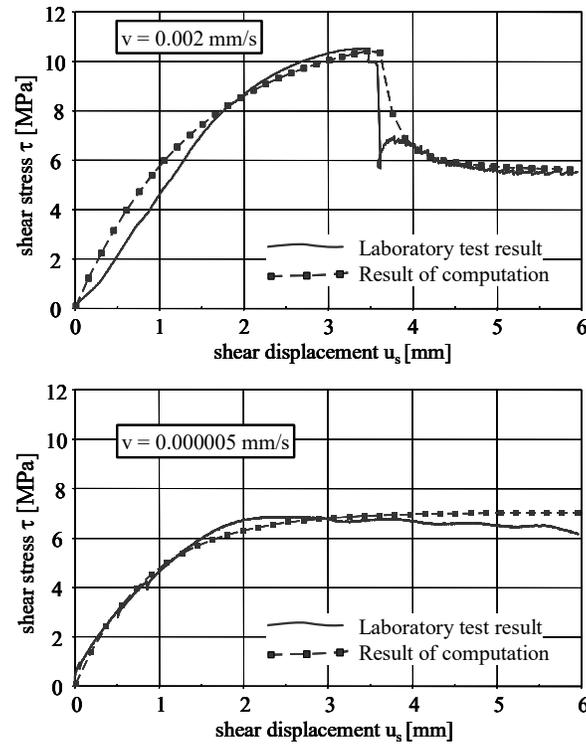


Figure 2. Shear stress vs. displacement for different shear velocities

4 EXPERIMENTAL PROCESS DESCRIPTION

4.1 Overall Strategy and Process

The series of laboratory direct shear tests will be performed on several samples of materials typical of WIPP emplacement drifts. Some test samples shall be control samples – machined blocks of halite and an appropriate other material such as anhydrite, clay, or polyhalite. These tests will be conducted at several normal and shear loads up to expected in situ pre-mining stress conditions, and at multiple shear velocities to scope for potential velocity-dependence of shear stress evolution. Tests will be performed on samples with a clay seam, or a halite/anhydrite or halite/polyhalite interface in a similar manner as that in Minkley & Mühlbauer (2007). For the completion of the laboratory phase, several test samples gathered from a field location containing a clay seam or otherwise distinct bedding discontinuity will be tested in the laboratory. A separate analysis plan is being written that will govern pre- and post-test model predictions and calibration.

4.2 Equipment

Measuring and test equipment, including Data Acquisition Systems (DAS) and instrumentation, shall follow procedure NP 12-1, “Control of Measuring and Test Equipment”. All calibrations performed on instrumentation shall be documented in the scientific notebook using standards that are traceable to NIST. The Contractor shall provide copies of the NIST traceable calibration records as part of the final records package for this work.

Note: Reference to specific commercial equipment does not constitute or imply endorsement. There are many types of instruments with similar capabilities that might be used depending on availability to the investigator.

Direct Shear Machine

For these laboratory tests to approximate in situ overburden stress conditions, normal stresses of 1000-3000 psi are required. To perform tests at these stress levels, it is recommended that laboratory tests be performed using the RESPEC direct shear machine. This machine is shown in Figure 3. It has an axial and shear load capacity of 30,000 lb each. Samples may be as large as 6" cubes, although for these tests 4" cubes or 4"-diameter cylinders will typically be used. The shear velocities range from a minimum of 0.010-0.015 in/min (0.004-0.006 mm/sec) to a maximum of 0.2 in/min (0.083 mm/sec). Potentiometric linear displacement sensors will be mounted on the shear boxes to measure displacement, from which shear will be derived. Load cells on the machine will measure horizontal loads which will be used to derive shear stress across the seam or interface. Like the system shown in Figure 1, the shear displacement is applied to the top block. Tests will be performed on intact salt samples with no interface to evaluate the test setup for any possible bending bias. Afterward, intact salt samples that include a distinct interface, such as either a clay seam or a halite/anhydrite or polyhalite interface, will be tested.

The RESPEC machine is required for these tests for several reasons. The primary reason is that the direct shear machine owned by SNL has only a 2,000 lb normal (vertical) load capacity, which will not produce the magnitude of vertical stress comparable to the in situ conditions required for the tests. Additionally, RESPEC currently has a contract with SNL and has previous experience working under the WIPP Quality Assurance program; however, their personnel may be required to undergo updated WIPP QA training. Finally, RESPEC has a relationship with Intrepid Potash, Inc., who operates a potash mine east of Carlsbad, New Mexico in similar formations as the WIPP site; through these relationships, they will be able to go into the mine and collect the required samples for the tests.

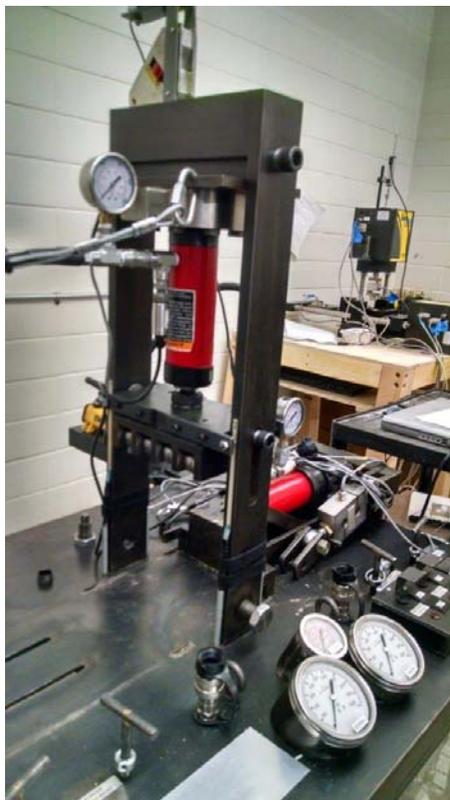


Figure 3. RESPEC's direct shear machine.

Concrete Coring Rig

The salt samples that will be used for these tests will be either from cores obtained under Revision 0 drilled from the rib of a drift, or from cores that will be obtained in the same manner from the WIPP site. The samples will be obtained using a concrete coring rig with a diamond bit core barrel having dimensions of 12-inch diameter by 22 inches long.

Hydrostatic Compression Chamber for Sample Consolidation

For the tests to be conducted under Revision 1, test samples that include a manufactured clay seam will be created. Cylindrical samples of salt will be used to sandwich a clay seam. The seam will be made of a mixture of bentonite and brine, mixed to form a paste. A seam of between ¼” to ½” thickness will be applied to the top of one cylinder of salt, after which the other cylinder will be placed on top. The entire sample will then be held under compression in a hydrostatic compression chamber for a period of approximately two weeks. The samples will be held under a consolidation pressure of 3000 psi, which is the standard pressure used for consolidation of salt. Multiple samples can be simultaneously placed in the compression chamber.

4.3 Types of Samples

Below is a description of the samples that are planned to be collected. These samples correspond to a planned test matrix. The exact number and composition of the samples may vary from the list described below due to availability of suitable samples in the Intrepid mine. All sample characteristics will be recorded in the Scientific Notebooks created for these tests.

The planned test suite consists of 28 tests, and 33 samples, described as follows:

- 4 samples of salt (halite) with no discernable seam or interface, to be considered controlled sample tests: 4 tests at 4 different normal stresses (nominally, 1000, 1500, 2000, 2400 psi), one shearing rate (probably a low rate such as 5×10^{-3} mm/s, the minimum rate for the machine)
- 4 samples with a clay seam from one region with approximately similar characteristic for all samples, “Clay Seam 1”: 4 tests at 4 different normal stresses, one shearing rate
- 4 samples with a clay seam from one region with approximately similar characteristic for all samples, “Clay Seam 2”: 4 tests at 4 different normal stresses, one shearing rate
- 4 samples with Salt/Anhydrite contact: 4 tests at 4 different normal stresses, one shearing rate
- 4 samples with Salt/Polyhalite contact: 4 tests at 4 different normal stresses, one shearing rate
- 8 samples for rate dependence tests: repeat at a single normal stress using the four interface scenarios at an additional 2 different shearing rates (if sample availability is limited, these tests may be performed with samples used in the previous tests; however, it is preferred to use new specimens for determining rate dependence, to avoid potential significant error)
- 5 samples for contingency tests; probably will be clay seam samples; these tests would allow repeat tests for any data that is questionable, or to test at additional shear rates and/or different normal stresses

For the second planned test suite, two types of samples may be used. The first type of samples will use salt from the same cores used for the first test suite, or from similar core samples available at the RESPEC facility. For these samples, a manufactured clay seam will be created between two cylinders of salt. The clay seam will have an initial thickness between ¼” to 1-1/2” (although initial tests will use ¼” and ½”

seams). The clay will be made from bentonite mixed with brine to form a paste. The samples will then be held under pressure in a hydrostatic consolidation chamber at 3000 psi for a period of about two weeks. The following minimum suite will be performed: a total of six shear tests, with two different pre-consolidation seam thicknesses of ¼” and ½”, and three different normal pressures of 500, 1000, and 1500 psi. The second type of sample that may be used, if available, will be taken from core obtained from the WIPP site. These core samples will be of similar dimensions to those obtained for the first tests (12” diameter, 22” in length). The cores will include clay seams from any of the Clay Seams D through K (and preferably G), and also from Marker Bed 139. All the shear tests for the second set will be performed using the identical method for the intact tests performed for the original set of shear tests performed earlier.

4.4 Sample Preparation

Collection and Packaging

The plan for sample collection includes a 2-week coring schedule to retrieve as many samples as possible within the timeframe. This will allow flexibility when selecting the best samples to test. Many times the condition of the sample will not be known until specimen preparation begins in the lab. The samples will be drilled from the floor of an inclined drift. The cores would be drilled using a concrete coring rig with a diamond bit core barrel having dimensions of 12-inch diameter by 22 inches long.

Sample characteristics are expected to be variable, but there are certain specifications that can be defined in this test plan. The test samples will be either 4-inch diameter cylinders (preferred) or 4”-square blocks. It is expected that up to 4 test specimens may be obtained from each of the whole 12” × 22” cores pulled. This would allow a maximum normal pressure of 2,388 psi ($= 30,000 \text{ lbs} / (\pi * r^2)$). The length of each sample will depend in part on the quality of the cores, but a 5” length will be the preferred length. Each sample will need about 2 inches of core in the upper and lower box to get the grout to set properly. This will mean that the clay seams will be less than 1”. Because of the design of the direct shear test machine, there are minimal concerns about bending or moment applied to the test sample, so sample length will be completely dependent upon core quality during sample preparation. For the anhydrite and polyhalite contact samples, there might be no seam thickness to consider.

For these tests, it will be preferable to find and recover samples with fairly uniform seam thicknesses with minimal undulations. Additionally, the condition of the seam will be inspected after coring and again after sample preparation to determine if any slip has already occurred, either in situ, after coring, or after preparation. There may be an accept/reject condition of samples based on the width of the seam undulations; this will be determined at the time of sample collection.

The 12”-diameter cored samples will require special procedures for prepping samples for shipment. They will be wrapped in plastic wrap and bubble wrap to maintain moisture. Wax coating around the samples may also be implemented to preserve the moisture content of the specimens; this method has been used for previous shipments of WIPP salt. The specific wrapping technique will be documented in the Scientific Notebook. The core will also be adequately immobilized and held under some normal pressure to ensure the seam does not separate. It is believed that this immobilization will be achieved by using hose clamps; other methods will be considered and, if used, documented. An assessment of the condition of the seam on the samples will be made when they are prepared in the lab. It should be obvious if the core has been damaged, dried out, etc.

For the tests to be conducted under Revision 1, test samples that include a manufactured clay seam will be created. Cylindrical samples of salt will be used to sandwich a clay seam. The seam will be made of a mixture of bentonite and brine, mixed to form a paste. A seam of between ¼” to ½” thickness will be applied to the top of one cylinder of salt, after which the other cylinder will be placed on top. The entire sample will then be held under hydrostatic compression in a consolidation chamber for a period of approximately two weeks. Before being put in the consolidation chamber, the sample will be wrapped in cloth to wick out any moisture squeezed out of the seam. The sample will be wrapped with a thin synthetic filter fabric and then with a thin flexible plastic sheet. The wrapped sample will be inserted into a thick Viton jacket, which will protect the sample from the pressurized oil used in the consolidation vessels. Platens vented to atmosphere will be placed on the ends of the sample, and the assembly will be placed into the consolidation vessel. Each sample will be consolidated under nominally 3000 psi hydrostatic compression for 2 weeks. Any excess pore fluid will drain through the atmospheric vents in the platens.

One of the variables that will be measured is the moisture content of the bentonite in the manufactured seam. A sample of the bentonite/brine mixture will be measured for moisture content at the time of sample fabrication. In order to minimize the amount of sample handling, a post-test measurement of seam moisture content will be done with chips from the middle and the edge of the clay seam after the shear tests have been completed.

4.5 Permeability

At the time of writing this Test Plan, we do not know if we will be able to prepare appropriate samples for gas flow measurements. If permeability determinations are made it will be performed using standard laboratory techniques that have been widely used and published. This test plan will be modified and reviewed if flow tests are to be performed. Results will be recorded in the Scientific Notebook.

4.6 Sample Control

Samples will be shipped directly to the Lead Tester for RESPEC, Stuart Buchholz, at the RESPEC laboratory where the direct shear test machine is located. The Lead Tester will retain possession during sample preparation, testing, or observational work. It is not intended to release subsamples to anyone else, but if the occasion arises, a Chain of Custody according to procedure SP 13-1, will be created by the Lead Tester in collaboration with the Principal Investigator (Steven Sobolik).

4.7 Data Quality Control

During the performance of this contract, the contractor will generate QA Records. (QA Records are identified in the “Records” section of each implementing procedure and plan.) The contractor shall adhere to the provisions of NP 17-1, “Records” and the contractor shall forward those QA Records upon their completion to the Sandia Delegated Representative (SDR) and the Principal Investigator (PI).

4.7.1 Measuring and Test Equipment

Measurements are described in each subsection of the text. LVDTs and load cells on the RESPEC direct shear machine will be calibrated in accordance with NP 12-1, “Control of Measuring and Test Equipment”; in addition, measuring devices will be identified in the Scientific Notebook in a manner that allows for

easy retrieval and reproduction. Professional laboratory practice is sufficient and calibration to NIST is unnecessary. Chain of Custody procedures are not anticipated, as there is only one custodian.

4.7.2 Data Acquisition

Data collection will be recorded in the Scientific Notebook. Quantitative data will include sample geometric and compositional characteristics (including overall dimensions, seam thickness, thickness of seam/interface undulations, sample and seam component identification, etc.), vertical and horizontal loads, shear velocities, displacement, post-test measurements of seam changes, and volume of gas flow (if flow tests are performed), which will be recorded in the Scientific Notebook. Accompanying sample photographs will be accumulated in a Scientific Notebook Supplement. Some data will be acquired by typical electronic data acquisition systems. Scientific Notebooks shall conform to the requirements of NP 20-2, "Scientific Notebooks". All Scientific notebooks shall be submitted to Sandia National Laboratories at the completion of the work. The Contractor shall address all comments developed during the final technical and quality assurance review of these Scientific Notebooks.

4.7.3 Additional Quality Assurance Requirements for Data Acquisition

All conditions adverse to quality shall be documented following NP 16-1, "Corrective Action". These corrective action requests shall be routed immediately to Sandia for review and approval by the Sandia PI, Management and WIPP Quality Assurance team.

The review of documents and reports shall require a review by WIPP qualified reviewers following NP 6-1, "Document Review Process".

Any software used in performance of this work scope, including those that operate the data collection systems, shall conform to the requirements of NP 19-1, "Software Requirements". All records developed to qualify software shall be submitted to Sandia at the completion of the contract.

5 TRAINING

Personnel involved in sample collection, preparation, testing and measurements described in this Test Plan have the requisite training and experience by virtue of hands-on experience within their respective research projects at SNL and RESPEC. The Principal Investigator has completed NP 20-1 and NP 20-2, and has performed all of activities described in this Test Plan in previous programs. Test personnel from RESPEC will be trained to WIPP requirements identified in NP 2-1, "Qualification and Training".

6 HEALTH AND SAFETY

Sample collection for the original tests will be performed at Intrepid potash/salt mines in southeastern New Mexico. Collection will occur underground, and thus will be subject to the ES&H requirements of that company's operations. Both SNL and RESPEC personnel have been trained in their own ES&H procedures, and will rely on those on the slight possibility where they deem that Intrepid's procedures are not sufficient. Drilling activities for sample collection will be performed under supervision of RESPEC in collaboration with the mine operators.

Sample collection for cores from the WIPP site will be performed by WIPP personnel operating under WIPP ES&H and QA requirements.

Sample sawing, preparation, testing, and storage will be performed by RESPEC and personnel under their testing and ES&H procedures. These procedures have been or will be confirmed to comply with SNL WIPP Quality Assurance Procedures and SNL ES&H procedures.

7 PERMITTING/LICENSING

There is no special license or permit requirement for the activities described in this Test Plan.

8 REFERENCES

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9 ACKNOWLEDGEMENT

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