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Sandia National Laboratories
Waste Isolation Pilot Plant

Test Plan TP 16-01

Characterization of Reconsolidated Crushed Salt from the BAMBUS Site

Task 4.4.2.6.1

Revision 0

Effective Date: November 3, 2015

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1 ABBREVIATIONS, ACRONYMS AND INITIALISMS

BAMBUS	Backfilling and Sealing of Underground Repositories for Radioactive Waste in Salt
CBFO	Carlsbad Field Office
CCA	Compliance Certification Application
EDS	Energy Dispersive X-Ray Spectroscopy
IC	Ion Chromatography
IfG	Institut für Gebirgsmechanik GmbH (Leipzig, Germany)
M&TE	Measure and Test Equipment
NIST	National Institute of Standards and Technology
NP	Nuclear Waste Management Procedure
SEM	Scanning Electron Microscope
UNM	University of New Mexico
WIPP	Waste Isolation Pilot Plant

2 REVISION HISTORY

This is the initial issuance of this Test Plan.

3 PURPOSE AND SCOPE

This Test Plan describes observational petrofabrics and physical measurements of samples of reconsolidated crushed salt from the Backfilling and Sealing of Underground Repositories for Radioactive Waste in Salt (BAMBUS) field experiment (Bechthold et al. 2004). Details of the forensic examinations are explained here in sufficient detail that the research could be repeated by independent peers with equal expertise. In fact, several of the techniques applied and measurements described in this Test Plan were reported previously (Bechthold et al. 2004). The crushed salt backfill associated with the BAMBUS forensic work reported earlier has now experienced another decade of consolidation in the underground setting. Collaborators in international salt repository investigations have agreed that another series of characterization studies on the BAMBUS reconsolidated salt would help elucidate large-scale, long-term consolidation processes. In late August 2015, German research groups acquired new borehole core from the BAMBUS site. This acquisition is thoroughly described in Appendix A: BAMBUS Core Acquisition.

There is an interest in Germany to examine and measure current characteristics of the backfill, make an assessment of its properties, and describe reconsolidation processes and mechanisms. The overall work is being performed by our German colleagues at the Institut für Gebirgsmechanik GmbH Leipzig (IfG). At a site visit to IfG in connection with an independent project meeting, four relatively small cores were obtained by the Test Plan author (Hansen). Documentation is provided in 4.8 Sample Control and in Appendix A.

Crushed salt reconsolidation is an important consideration for the Waste Isolation Pilot Plant (WIPP) and any other salt repository in which crushed or run-of-mine salt plays a role in isolation systems. Salt as a geologic medium has several attributes favorable to long-term isolation of material placed in mined openings. Salt formations are largely impermeable and induced fractures heal. Permanent isolation also depends on our ability to construct geotechnical barriers that achieve high-performance characteristics attributed to the native salt formation. Crushed salt is commonly used as backfill in operating mines for structural and sealing purposes. Salt repository seal concepts have often included elements of reconstituted granular salt. A lingering uncertainty with geotechnical barriers constructed of granular salt pertains mostly to the limits involved with testing over extended time periods and at an appropriately large scale. To strengthen the technical basis for salt seal systems, natural and anthropogenic analogues can be used to illustrate cases where salt reconstitutes itself into an impermeable medium. Invoking analogue examples provides an independent line of reasoning to safety case performance arguments. Therefore, these forensic studies of the reconsolidated BAMBUS salt add to the analogue database as well as provide pertinent information for technical assessments being made by our German colleagues.

In previous cooperation with international colleagues, the Carlsbad Field Office (CBFO) advanced scientific knowledge of salt properties pertinent to WIPP through Work Packages undertaken by BAMBUS collaborators. Overall the experiment itself and the post-test forensics comprise many elements of direct relevance to WIPP operations and its performance assessment as well as salt

repository future activities. Sandia's previous investigations associated with BAMBUS focused on crushed salt reconsolidation and the excavation disturbed zone. Studies for this Test Plan characterize basic properties and characteristics of reconsolidated crushed salt backfill from the BAMBUS setting.

4 EXPERIMENTAL PROCESS DESCRIPTION

4.1 Overall Strategy and Process

The author of this Test Plan is also collaborating with the University of New Mexico (UNM) on a research project sponsored by Nuclear Engineering University Partnerships: *Improving the understanding of the coupled thermal-mechanical-hydrologic behavior of consolidating granular salt.* The UNM research is examining granular salt consolidation through an integrated program of laboratory measurements, observations, and model development. UNM researchers have been taught specimen preparation techniques, measurement methods, and received hands-on observational instruction and interpretation in pursuit of their various thesis objectives. The BAMBUS core provides a "real-life" example that can be compared to their measurements on laboratory samples. The students will apply their techniques and make measurements on BAMBUS core samples using experimental processes described here.

4.2 Equipment

Microstructural observations are made with optical and electron beam microscopes. There are many optical scopes and scanning electron microscopes (SEM) that would provide equally sufficient images. The Principal Investigator (Hansen) is collaborating in a mentoring capacity with thesis work by Melissa M. Mills and Laxmi Paneru under the supervision of Professor John C. Stormont at UNM. Characterization of the BAMBUS cores is directly related to their ongoing research interests. Interpretation of microstructures, porosity, density, permeability and other characteristics will enhance their thesis database and provide high-quality results for our international collaborators. The equipment and techniques described in this Test Plan are being employed in their respective research. Below are descriptions of equipment and techniques applied, such that any other equally trained person can replicate 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.

Optical Microscope

A Leitz Ortholux II optical microscope, Figure 1, uses combined transmitted and incident polarized light with a five objective nosepiece to examine samples. The microscope is equipped with a Leica camera and Leica Application Suite software to capture images. The optical scope will be used for point-counting porosity and petrophysical descriptions. Much of the microscopic documentation is captured by photography.

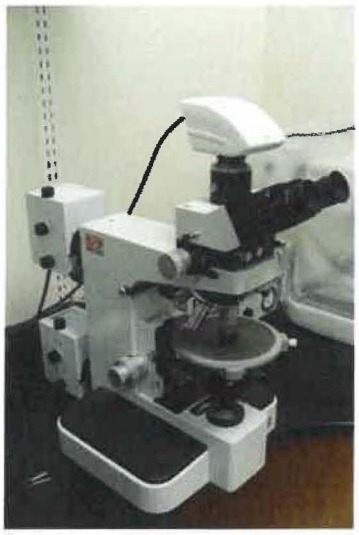

Figure 1. Leitz Ortholux II optical microscope

Stereo Inspection Microscope

A Lynx eyepiece-less stereo inspection microscope, Figure 2, with dynascope technology is used to look at larger (hand-held) samples. The optics allow for a large viewing area with a 3-dimensional, high-resolution image. The range of stereo zoom magnification is 3.5x to 120x provided by the step magnification multiplier.

Figure 2. Lynx stereo inspection microscope

Scanning Electron Microscope

The JEOL 5800LV SEM, Figure 3, uses a beam of electrons to scan a sample in order to create surface images at high magnification (up to 300,000X). The JEOL is equipped with secondary and backscattered electron imaging detectors as well as a cathodoluminscence (CL) imaging detector. The instrument uses the Oxford Isis 300 analytical system, which includes an energy dispersive spectroscopy (EDS) detector, to acquire sample images and composition.

Figure 3. JEOL 5800LV scanning electron microscope

An FEI Nova 200 Nanolab, Figure 4, is an instrument with a dual beam system of a focused ion beam column and SEM column. The SEM column is equipped with a tungsten filament for imaging in low or high vacuum levels. The detector has a resolution of 3.5 nm operating at 30 kV at high vacuum and less than 15 nm at 3 kV when operating in low vacuum. The instrument is also equipped with INCA Synergy 350 with HKL Premium EBSD System, which allows elemental analysis of samples as well as the capability of generating phase maps from elemental maps and Cameo data. A HKL Premium Electron Backscatter Detector can be used within the machine for crystallographic determination to create orientation maps of microstructures, generate combined orientation and elemental maps, and generate pole figures.

Figure 4. FEI Nova 200 Nanolab SEM

Sputter Coater

To prepare non-conducting or poorly conducting samples for observation in an SEM, a sputter coater must be used. The EmiTech KX950 sputter coater, Figure 5 allows samples to be thinly coated with carbon or gold-palladium to increase electrical conduction. This device is equipped with a turbo pump evaporator for complete automatic control during evaporation of the chamber to low pressures while having a dry gas inlet to improve coating deposition. This sputter coater is also used with a K150X film thickness monitor that measures the thickness of the coating that has been deposited on a crystal within the chamber.

Figure 5. EmiTech KX950 sputter coater with K150X film thickness monitor

Diamond Wire Saw

The BAMBUS samples need to be cut and prepared in special ways to make thin-sections, obtain aggregated samples, extract single crystals, and prepare thermal conductivity samples. A diamond wire saw, shown in Figure 6, will be used to prepare subsamples. Samples of interest are clamped in place on the platform to ensure they do not move while cutting. The saw mechanism changes rotating directions using magnetic sensors, which allows the wire to be pulled back and forth. A small amount of weight (about 5 grams) on the wire arm slowly lowers the wire through the sample. Sensors located on the sides of the arm control the stroke and shut-off.

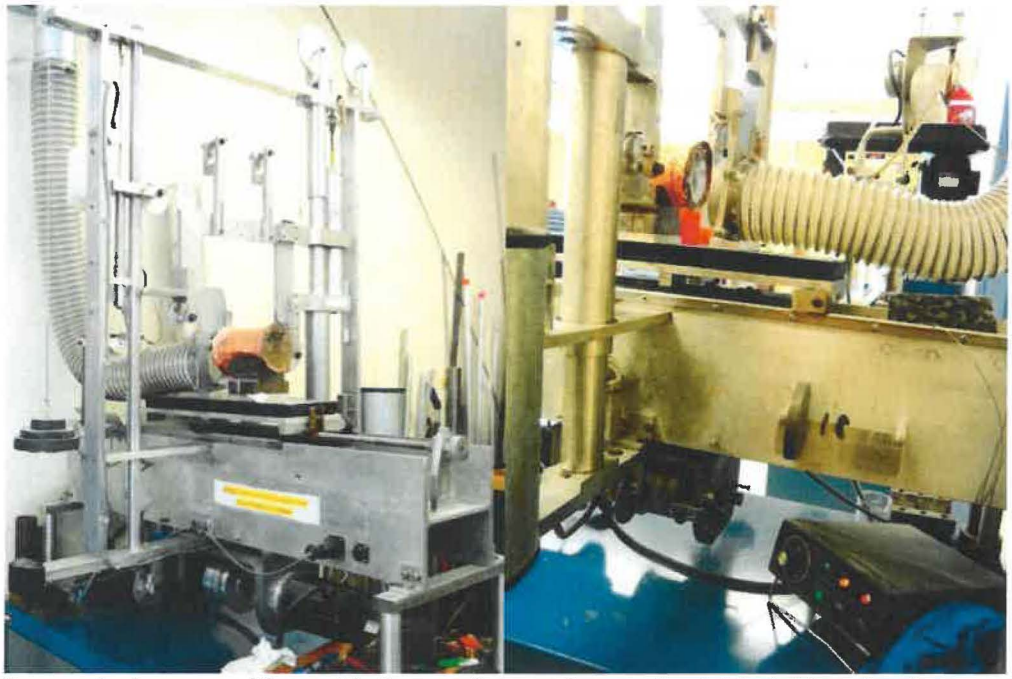

Figure 6. Diamond wire saw with ventilation, pulley system, adjustable table, wheel, pump and pump controller

Diamond Wire Saw Replacement

The diamond wire generally only lasts about 10 to 15 cuts for salt samples of this nature, which means it needs to be replaced regularly. Replacing the wire is a sensitive procedure since it is important not to kink or overlap the wire on the rotary wheel (Figure 7). Once the wire breaks, all of the old wire must be taken off. Since the wire can be tangled around the rotary drum, wire cutters are needed. Once all the wire is off, it is essential to clean residue from the grooves where the wire winds. A cup of warm water and a thick-haired brush is used to clean the wheel. The wheel is turned on slow-speed and the wet brush used to remove the residue. The wheel is dried with a cloth.

With the wheel clean, the wire can be restrung through the pulleys. At the end, the wire is strung through a hole and wrapped around a thumb-tight screw on the rotary wheel. Next, the wire must be wound around the wheel, certifying that the wire coming off the last pulley is in line with its starting location on the wheel and then rotating it manually about 25 times. After a sufficient amount of wire is wound, the opposite side of the wire is cut from the spool and attached to the wheel to complete the loop. Finally, the pump is turned on at a slow speed to make sure the wire is centered on the wheel as it is pulled back and forth. If there are less than 2 segments of wire on one side of the wheel, the wire is not centered and the magnetic sensors need to be adjusted to change the stopping point.

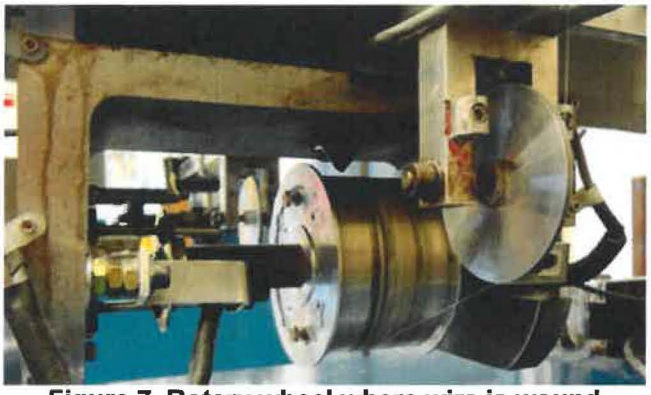

Figure 7. Rotary wheel where wire is wound

IsoMet Low Speed Saw

A Buehler IsoMet low speed saw, shown in Figure 8, is used for making precision cut thin sections with a diamond tipped saw. Once the surface of an impregnated sample is glued to a glass slide, the slide is suctioned into a vacuum chuck holder with the help of a small amount of vacuum grease to create a tight seal. The holder is then screwed onto the arm above the blade and its position adjusted by a micrometer. The micrometer allows for manual determination of thickness for each cut. Weights on the arm control the loading of the specimen, which governs the speed of the cut. For salt specimens, isopropanol is used as the cutting liquid and is poured into a reservoir below the blade, refilling regularly due to evaporation.

Figure 8. Buehler IsoMet low speed saw

Ecomet III Polisher and Grinder

Figure 9 shows a Buehler Ecomet III polisher and grinder, which is used to polish thin sections after cutting and also to create smooth, even surfaces for porosity samples. Abrasive, circular paper, ranging in grit size from 400 to 1200, is adhered to a rotating wheel. The paper is wetted with isopropanol and samples are held on the wheel with minimal pressure until a smooth surface is acquired.

Figure 9. Buehler Ecomet III polisher and grinder

4.3 Types of Samples:

Below we describe essentially all the types of sub-samples that could be used in these observational studies. We may not create all these types of sub-samples because they may not provide distinguishing features or information. However, all sub-sample preparation is described.

Cleavage Chips

Cleavage chips produced from individual crystals can be used to evaluate deformational processes in that grain. A single grain is isolated by disaggregation. A small, sharp chisel is used to cleave along the Miller Indices {100} plane. With practice, flat chips 1 to 3-mm thick can be produced. Typically the chips are etched. Observations can be made using the optical microscopy or SEM.

Thick-Thin Sections and Thin Sections

Sample material is impregnated with epoxy as described subsequently. Excess epoxy can be cut away with a knife, band saw, or other convenient method. Impregnated samples are then cut to size for a petrographic slide. Thin sections for optical petrography are challenging to make because they are extremely thin--having an optical path difference between 530 and 560 nanometers. Fortunately, much of the information of interest can be obtained from thick-thin sections (1-3 mm thick). Optical microscopy is used for observations of deformation processes, grain boundary conditions, void spaces, fabric, mineralogy and any other notable characteristics. Petrographic sections can also be etched and examined optically or by SEM.

Fragments

Fragments can be obtained several ways. Part of observational studies of reconsolidated salt involves use of a "fresh face" that has not been cut or polished. Usually fragments from cut ends can simply be broken by flexure. This exposes a clean surface, which exhibits diagnostics of sample cohesiveness, grain boundary characteristics, and other evidence of microprocesses. The SEM is better for observation because of the uneven surfaces. Fragments provide a sense of 3-D imaging, particularly useful for examining grain boundaries.

4.4 Sample Preparation

Impregnation

Typically, the solid state of the sample is locked in place by impregnation with low viscosity epoxy. Samples are shaped to a convenient size for thin- or thick-section preparation using the slow-speed, low damage wire saw described elsewhere. There are many types of epoxy that can be used. We use a two-component epoxy which is stained blue and has performed well in similar sample preparation (RF 1366 commercially available epoxy from Resin Formulators). The sub-sample is placed in a convenient disposable container (a paper cup or aluminum foil, for example). The sample is completely immersed in epoxy and placed in a bell jar, which is put under approximately 10 psi vacuum (approximately 3 psia inside the bell jar). After the system has been evacuated, the vacuum is removed slowly by venting and atmospheric pressure helps force epoxy into the evacuated voids of the specimen. This process is repeated 3-4 times to ensure that the specimen has been fully impregnated. The epoxy is then allowed to cure.

Polishing

Cutting with the diamond-wire saw creates a relatively smooth surface with small ridges. To improve imaging on the stereo-dynascopic or optical microscopes, uneven surfaces need to be smoothed by polishing, which is done using a clean, flat surface, a sheet of sandpaper, and isopropanol. For salt, 400 to 600 silicon carbide sand paper has been shown to produce an acceptable surface. Isopropanol is used as the wetting agent and a figure-eight motion seems to create a nice finish.

Etching

Individual cleavage chips are etched in a solution of methanol saturated with $PbCl_2$ and stopped with butanol. Etching requires practice. Typically, the single chip is held with a tweezers and agitated in the $PbCl_2$ solution for 3 to 4 seconds and stopped immediately by transfer to butanol. Excess liquid can be drawn off by dropping the chip on a dry Kimwipe®. Etching highlights the substructure in the crystal lattice, which can be immediately examined on an optical scope. This quick examination also allows evaluation of how well etching was done. Crisp etches with sharp contrast and resolution allow for better evaluation of the substructure. After cleavage chips are successfully etched, they are mounted with carbon tape and coated in gold-palladium for observation under the SEM where a higher magnification and resolution of the etched surface can be seen.

Coating for SEM

Using the sputter coater shown in Figure 5 above, samples could be coated with carbon or gold-palladium. A carbon rod with a 3mm sharpened spigot shaped head is ignited and evaporated producing a 25nm thick coat for a 5 second pulse. Carbon coating is generally used for unetched mass samples and gold-palladium is used to for etched surfaces to obtain high-resolution coating. It is a constant challenge to produce an optimal thickness. Too thick of a coating obscures subgrain features, while too thin will not sufficiently highlight them enough to be observable. From experience, a 9.5nm thickness produces the best results.

4.5 Observation Techniques

Point Counting

Point counting is a common technique for manually determining the porosity of a sample by counting the number of void spaces and solid spaces. A thick-thin or thin section is placed on a mechanical sliding stage, shown in Figure 10, on an optical microscope. We have found that for granular salt, racking the stage horizontally 40 mm and 20 mm vertically at nominally 1-mm intervals provides accurate coverage. The petrographic section is moved within the stage in a grid-like pattern. At each grid point a determination (one count) of solid or void under the cross hairs is made by the observer. Typically, 300 to 500 counts are made to obtain an measurement considered representative for the entire sample. A single sample can be re-measured by changing the direction of movement.

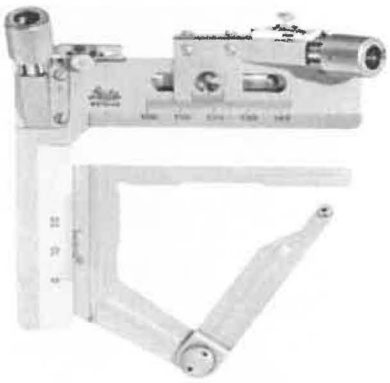

Figure 10. Leitz mechanical slide stage for optical microscope

An example from previous work is shown in

Table 1. The ratio of void to solid space was calculated and multiplied by 100 to determine the percent porosity. There are other means to estimate porosity. In this particular example, laboratory measurements of geometric change roughly approximated the porosity to be 14%. The average obtained by point counting is 13.8%. On a section by section basis, point counting porosity is quite precise, depending on the skill of the microscopist. The accuracy shown here is characteristic of the data quality objective—justifiably only one significant figure after the decimal point.

Table 1. Results of the point counting technique on a thin section sample

Trial #	Void #	Solid #	Ratio	Porosity
1	44	257	0.146	14.6%
2	40	260	0.133	13.3%
3	41	259	0.136	13.6%
			Average	13.8%

ImageJ Particle Size and Counting Analysis

ImageJ® is an image processing program that is being evaluated for its application along with other characterization techniques. ImageJ is used to determine particle size and number of particles from photomicrographs of thick-thin and thin sections. The sections are photographed in reflected light on the optical microscope. These photomicrographs are then opened with the ImageJ software where the scale bar on the image can be measured to set the scale for the analysis. Then, the image is converted to monochrome format and a threshold set to allow the program to distinguish between the background (dark) and a particle or grain (light). From the output data, an outline image of numbered particles can be shown as well as a separate window with each particle area. This analysis tool *might* be useful when determining salt grain change in size after consolidation and another means to determine porosity of the sample.

4.6 Thermal Conductivity and Porosity

Thermal conductivity is a key measurement of interest for reconsolidating granular salt. In a high-level waste salt repository, thermal activation drives deformation processes of the formation salt. Thermal conductivity varies with porosity. Therefore, it is good practice to measure porosity and thermal conductivity of the same sample, if possible.

As described in Appendix A, we were allotted a small amount of core. As outlined in Appendix A, we might be able to produce three samples for thermal conductivity and porosity measurements. Two nearly identical pieces will be cut using the diamond-wire saw. Each piece is polished on one side to get a smooth surface using fine sand paper and isopropanol. This operation cleans the salt dust and removes the asperities present on the surface. After polishing, a Kapton® thermal sensor is pressed between the two cut pieces. One of the cut sample pieces is placed on the mounting table of the sample holder and its height is adjusted so that the surface of the sample will be at the same level as the sensor. The second cut sample disc is placed on the sensor, and a mounting pressure is applied using a screw. A heat pulse of 637.7 to 760.3 mW is supplied by the Thermal Constant Analyzer to the sample pieces and thermal properties are measured in 20 seconds. Heat energy is dissipated by waiting 20 minutes between runs. Typically, three to five measurements at each temperature are made and averaged. In terms of data quality objectives, standard deviation may range between 10^{-2} to 10^{-3} W/(mK).

After thermal properties are measured, porosity measurements are obtained on the same material. The thermal conductivity sample is cored to obtain a 1-inch or 1.5-inch-diameter circular disk. The choice of diameter depends on the feasibility of coring, governed by the degree of consolidation. The cored sample is then cut on the diamond wire saw to a height of 0.5 inches and later polished to obtain a uniform geometry.

Helium gas expansion porosimeter is used to determine the effective porosity of sample cores. It is based on Boyle's law of gas expansion. The apparatus consists of a reference volume and a sample cell volume, separated on either side by a valve V_2 as shown in Figure 11. Before the actual porosity measurement is conducted, the sample cell of the porosimeter is calibrated under two different conditions: sample cell with all the billets inside and sample cell without a 0.5-inch thick billet. The cored sample disc is put in the sample cell to replace a billet of an equivalent diameter and volume. The external valve V_3 on the right is left open to the atmosphere and central valve V_2 is closed. The reference section is filled through the left valve V_1 with helium at a load pressure of P_o= 100 psi. After the reference section is shut in from main gas supply and pressure Po is recorded, the sample section is shut by closing the valve to the atmosphere. After this, the central valve V_2 is opened to allow the gas in the reference section to pass into the sample section. The pressure is allowed to equilibrate for 15 seconds, and the equilibrium pressure P is recorded for at least three measurements and averaged. This practice follows helium gas expansion porosimeter user's manual provided by Frank Jones and Associates, Inc.

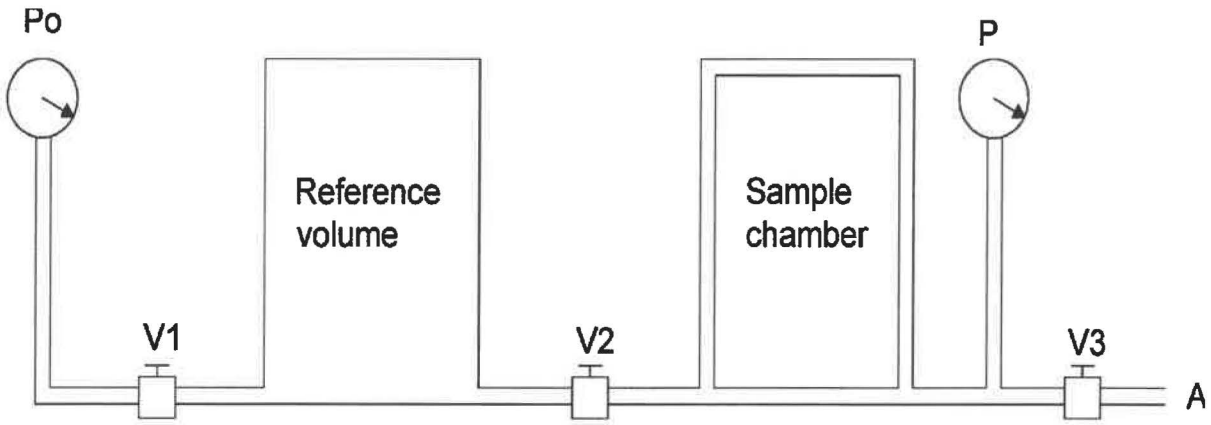

Figure 11. Schematic of porosimeter method

The grain volume is given by,

$$G=B+\frac{Pof}{Pf}R-\frac{Pos}{Ps}R$$

where, P_{os}, P_s= initial and final pressures during a porosity test, when a 1" or 0.5" billet is replaced by an equivalent volume of sample.

The reservoir volume is given by,

$$R=\frac{B}{\frac{Pob}{Pb}-\frac{Pof}{Pf}}$$

where, B=volume of the removed 1" or 0.5" billet.
P_{ob}, P_b= initial and final pressures during calibration of the cell, when a 1" or 0.5" billet is removed.
P_{of}, P_f= initial and final pressures during calibration of the cell, when all the billets are inside the cell.

Measurement of diameter and height is carried out at a number of locations on the sample using calipers. Volume is calculated from dimension averages and bulk density is calculated from mass and volume. Grain density is calculated from the grain volume and mass.

Porosity φ is calculated by:

$$\varphi = 1 - B_D/G_D$$

where, B_D= bulk density (g/cc) and G_D= grain density (g/cc)

Another method called the small volume method, determines the total porosity of the sample. The sample core used in the porosimeter method is also used in this method. Bulk density is calculated from measured mass and volume. Grain density is assumed as 2.16 g/cc.

Example Calculation (as will be documented in the Scientific Notebook or Supplemental Binder).

The porosimeter's sample cell is also referred as sample chamber or sample cup. There are two different types of sample cups: 1" diameter and 1.5" diameter. Below is the explanation of a 1" diameter cup calibration with illustrations. The 1" diameter sample cup has four billets of varying heights: 1-one-inch billet, 1-0.5-inch billet and 2-0.25-inch billets.

Calibration#1:
The sample cup is calibrated with all the above mentioned billets inside. The data obtained from this calibration may be tabulated as shown below. P_{of} and P_f are the upstream and equilibrium pressures, respectively. The "f" in the sub-script refer to "full", i.e., when the cup is full of billets.

CALIBRATION#1: All the billets inside the cell			
Initial pressure (P_{of}), Psi	**Equilibrium pressure (P_f), Psi**	**P_{of}/P_f**	**Average P_{of}/P_f**
100.28	83.94	1.195	1.199
100.21	83.45	1.201	
100.11	83.37	1.201	

Calibration#2:
The cup has all the billets inside except for the 0.5-inch billet. Sub-script "B" refers to calibration with a billet removed.

CALIBRATION#2: 0.5-inch billet removed			
Initial pressure (P_{oB}), Psi	**Equilibrium pressure (P_B), Psi**	**P_{oB}/P_B**	**Average P_{oB}/P_B**
100.09	44.99	2.225	2.225
100.12	45.02	2.224	
100.11	45.00	2.225	

The reference volume is given by:

$$R = \frac{B}{\frac{Pob}{Pb} - \frac{Pof}{Pf}}$$

Where, B= volume with 0.5-inch billet removed (provided by the manufacturer).

After these two calibrations, the 0.5-inch billet is not replaced in the sample cell. Instead, a sample core of 0.5-inch height is placed inside the sample cell and tested for porosity with the other 3 billets inside. The precision of these measurements is higher than the data quality objective for bulk porosity, which is nominally the same as the point count technique (0.1%).

4.7 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, including work on reconsolidated salt similar to the BAMBUS core (Figure 12) (Bauer, et al. 2015a; 2015b). For anticipated high flow rate conditions (high porosity, up to 900 sccm down to 0.1 sccm), gas flow is measured using a constant head (or pressure) technique with an inert gas as the permeant; this was accomplished with a flow meter panel, wherein an array of flow meters is used with specific ranges of operation. Results will be recorded in the Scientific Notebook.

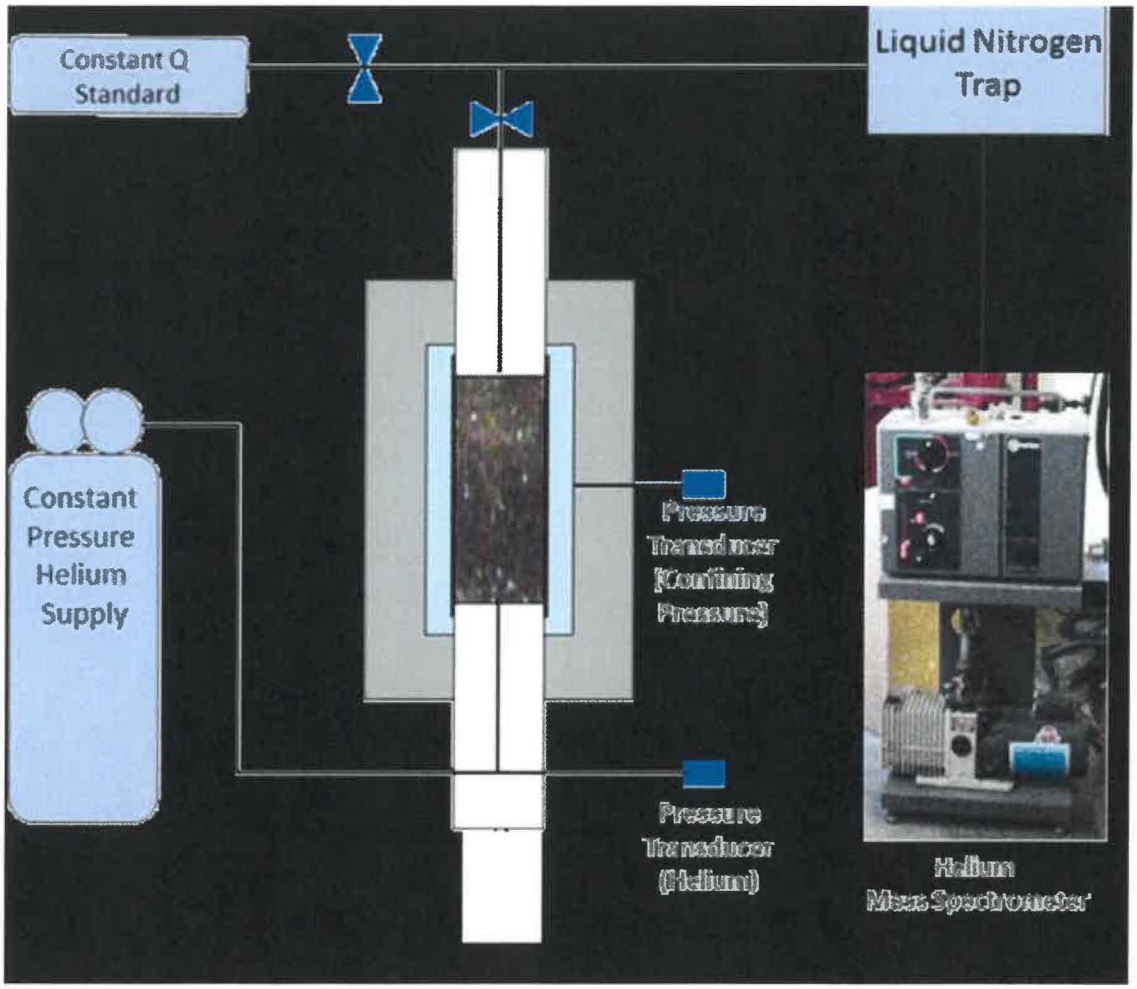

Figure 12. Schematic of the permeability measurement set-up based on the helium mass spectrometer

For permeability calculations, Darcy's law is expressed as the measured flow rate of fluid crossing a unit area and is proportional to the pressure differential measured across the ends of the specimen. The permeability of the sample, k, is calculated by the following equation:

$$k = Qx \cdot \mu \cdot L / (A \cdot \Delta P)$$

where:

k is the permeability (m^2)

Q_X is the flow rate in the axial direction of the specimen (m^3/s)
μ is the gas viscosity (Pa sec)
ΔP is the pressure differential measured across the ends of the specimen (Pa)
L is the length of the specimen (m), and

A is the cross-sectional area perpendicular to the axis of the specimen (m^2).

In terms of data quality objectives, previous measurements on similar samples having porosities greater than 0.25 yielded permeability in the 10^{-12} to 10^{-13} m^2 range; this is likely the upper limit of permeability measurement limit as restricted by the high pressure tubing used in our flow-through system. For porosities of 0.15 to 0.2, the apparent permeability is observed to decrease to 10^{-13} to 10^{-14} m^2 range.

4.8 Sample Control

Samples were given directly to the Principal Investigator by IfG scientists and hand-carried to the Geomechanics Laboratory at Sandia Laboratories. The Principal Investigator will retain possession during sample preparation, testing, or observational work. It is not intended to release subsamples to students or anyone else, but if the occasion arises, a Chain of Custody according to procedure SP 13-1 will be created by the Principal Investigator. Appendix A describes the raw material provided by IfG. Samples were not collected or created by a person working on WIPP; however, consistent practice for unique identification of subsamples will be followed.

4.9 Data Quality Control

4.9.1 Measuring and Test Equipment

Measurements are described in each subsection of the text. There are few measurements of length and weight. Ordinary calipers with accuracy of 0.01 mm and scales with accuracy of 0.05 grams will yield calculated values within 0.5 to 1.0%. In accordance with NP 12-1, Control of Measuring and Test Equipment, 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 (although standard practice in the Geomechanics Laboratory). Chain of Custody procedures are not anticipated, as there is only one custodian. Subsamples will be marked as indicated in Appendix A.

4.9.2 Data Acquisition

Data collection will be recorded in the Scientific Notebook. Few quantitative data will be recorded, such as possible permeability or porosity calculations. Most information comes by qualitative observations made on the optical or SEM microscope, which is recorded in the Scientific Notebook. Accompanying photographs will be accumulated in a Scientific Notebook Supplement. There will be no data acquired by typical electronic data acquisition systems. Procedure NP 20-2 Scientific Notebooks will be followed. As described in the text above, much of the scientific inquiry here is observational in nature.

5 TRAINING

Personnel involved in sample preparation, microscopy, 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 UNM. 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. In fact, the PI developed many of the techniques described. Graduate students will be trained to WIPP requirements identified in NP 2-1, Qualification and Training.

6 HEALTH AND SAFETY

Research described in this Test Plan will be undertaken in three locations: 1. University of New Mexico Northrup Building, 2. Building 849 Sandia Geomechanics Laboratory, and 3. Building 823 of Sandia Tech Area. Scanning electron microscopy and sample sputtering will take place in the Northrup facilities and involve operator M. Mills and observer F. Hansen. Health and safety requirements relevant to work conducted on the campus of UNM are the purview of UNM. Since the work will be done at UNM by UNM students, they will be following the ES&H requirements set out by the university. The UNM instruments will be operated by UNM students approved and trained for such operation. The Principal Investigator (Hansen) helps interpret microstructural features in an observer role. As suggested by Shane R. Page, Sandia Center 6200 ES & H Coordinator, acknowledgement of this arrangement has been confirmed by email from UNM Professor John C. Stormont, stating that they will indeed follow UNM practices and requirements. This working arrangement is set in place for the benefit of all parties in case of unlikely event of an incident.

Sample sawing, impregnation, porosimeter measurements, and perhaps permeability (if samples are sufficient) will occur in within the Geomechanics Laboratory at Sandia Building 849 and are governed by SNL NEPA ID: NM16-0004; Bldg 849 Geomechanics Laboratory Materials Characterization Testing and PHS #: SNL07A00110-010, entitled: Bldg 849 Geomechanics Laboratory. M. Mills, L. Paneru and F. Hansen will prepare samples and L. Paneru will perform porosimeter measurements in Bldg 849. Laboratory operations describe the non-radiological hazards associated with these activities and procedures to deal with those hazards, including training requirements for personnel active in the laboratory.

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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Bauer, S.J., J.Y. Lee, and W.P. Gardner. 2015b. Helium-Mass-Spectrometry-Permeameter for the Measurement of Permeability of Low Permeability Rock with Application to Triaxial Deformation Conditions. SAND2015-1406 C. 49th US Rock Mechanics/Geomechanics Symposium. San Francisco, CA.

DOE 1996. Title 40 CFR Part 191 Compliance Certification Application for the Waste Isolation Pilot Plant. DOE/CAO-1994-2184. U.S. Department of Energy, Carlsbad, NM

9 ACKNOWLEDGEMENT

Melissa Mills and Laxmi Paneru are graduate students at UNM working under supervision of Professor John C. Stormont. They have provided images and discussion of techniques described in sections of this Test Plan.

Appendix A BAMBUS Core Acquisition

The following figures and photographs were provided by IfG for identification of sample origin. Figure A1 is a plan view of the underground area from whence the new BAMBUS cores were extracted. Notably the coring occurred proximal to the surface of heater 4 as indicated in Figures A2, A3 and A4. Figures A5, A6, and A7 are photographs of the core. Table A1 summarizes the available core and planned usage.

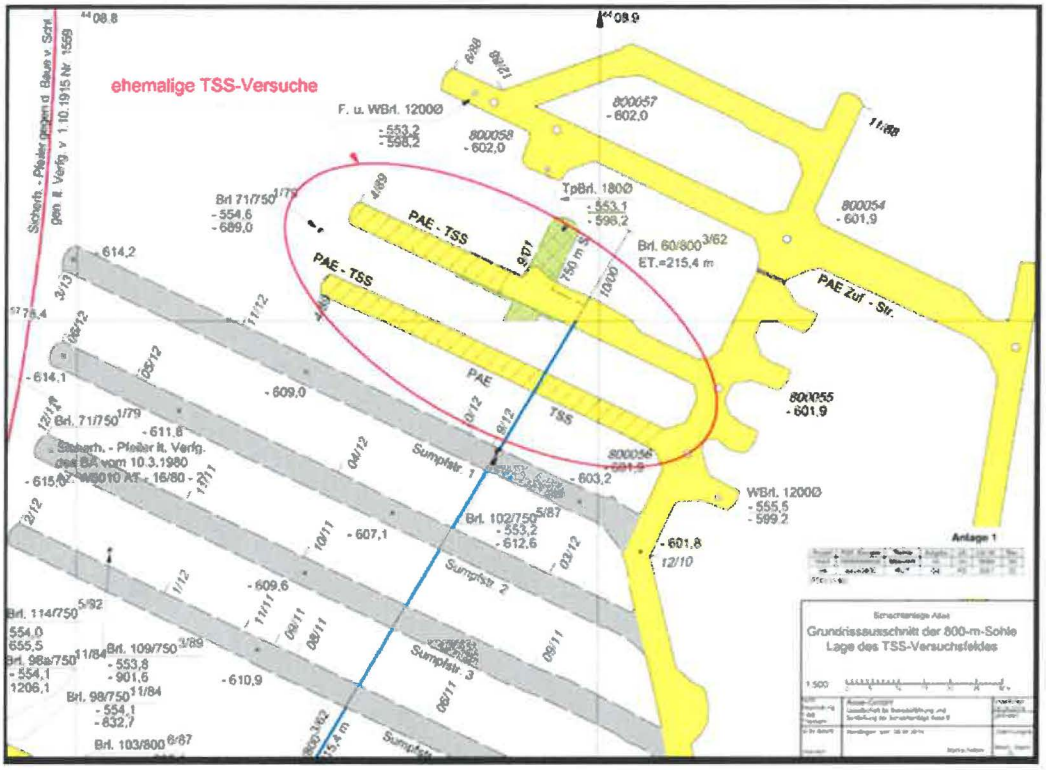

Figure A-1. Area of Thermal Structural Experiment

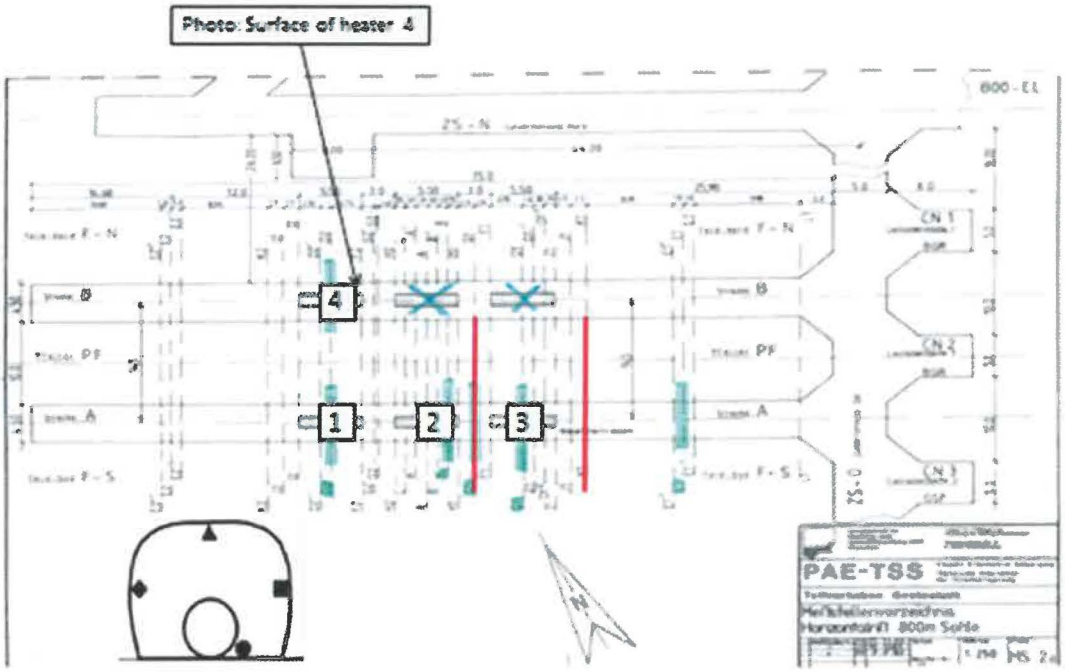

Figure A-2. Plan view of source of new core

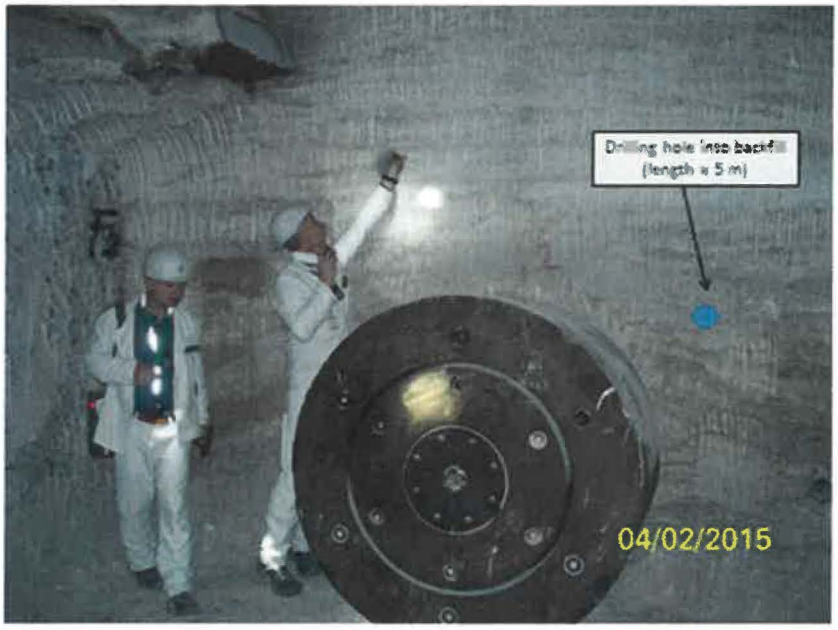

Figure A-3. Man showing Borehole Kbrg-1

Figure A-4. Photographs of Borehole KBrg-2

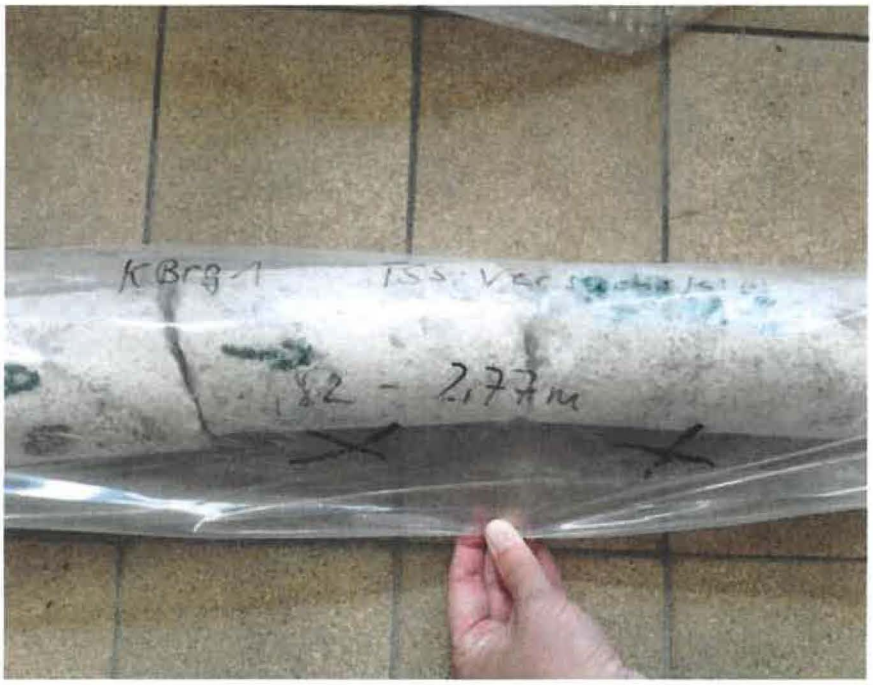

Figure A-5. Two samples from KBrg-1 (1.82-2.77m)

Figure A-6. Sample from KBrg-2 (2.5m)

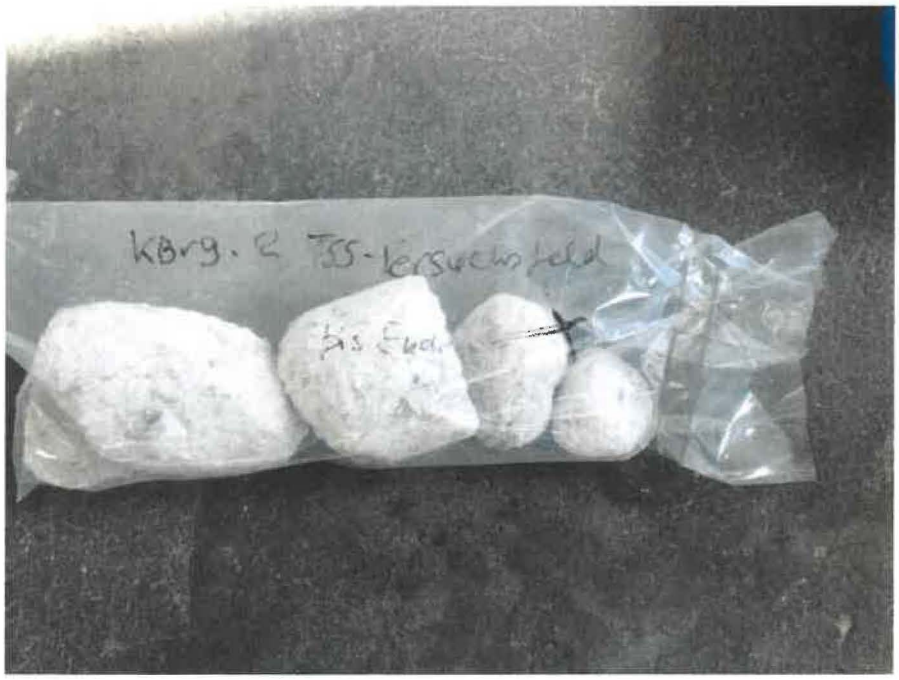

Figure A-7. Sample from KBrg-2 (bis end)

Table A-1. Original core identification and planned sub-samples

Sample Original ID	Sub-sample	Use	Disposition
KBrg-1(1.82 m)	BH1-1.82m-A	Thermal/Porosity	
	BH-1.82m-B	SEM/Optical Microscope	
	BH-1.82m-Center	Permeability	
KBrg-1(2.77 m)	BH1-2.77m-A	Thermal/Porosity	
	BH1-2.77m-B	SEM/Optical Microscope	
	BH1-2.77m-Center	Permeability	
KBrg-2(2.5 m)	BH2-2.5m-A	Thermal/Porosity	
	BH2-2.5m-B-1	SEM/Optical Microscope	
	BH2-2.5m-B-2		
	BH2-2.5m-Center	Permability	
KBrg-2 (bis end)	BH2-HD-A	Thin Sections	
	BH2-HD-B	SEM/Optical Microscope	