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Title: Draft Test Plan for Brine Migration Experimental Studies in Run-of-Mine Salt Backfill

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Draft Test Plan for Brine Migration Experimental Studies in Run-of-Mine Salt Backfill

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EXECUTIVE SUMMARY

The primary objective of the experimental effort described here is to aid in understanding the complex nature of liquid, vapor, and solid transport occurring around heated nuclear waste in bedded salt. In order to gain confidence in the predictive capability of numerical models, experimental validation must be performed to ensure that (a) hydrological and physiochemical parameters and (b) processes are correctly simulated. The experiments proposed here are designed to study aspects of the system that have not been satisfactorily quantified in prior work. In addition to exploring the complex coupled physical processes in support of numerical model validation, lessons learned from these experiments will facilitate preparations for larger-scale experiments that may utilize similar instrumentation techniques.

DRAFT TEST PLAN FOR BRINE MIGRATION EXPERIMENTAL STUDIES IN RUN-OF-MINE SALT BACKFILL

1.   Introduction

Laboratory experiments that focus on brine migration in run-of-mine (RoM) backfill are necessary to validate parameters and models used in numerical simulations of heated nuclear waste in bedded salt, to support the planning/design of thermal tests at intermediate and field scales (Robinson et al., 2012), and elucidate key coupled physical processes that may be observed in the larger-scale tests and in an operational high-level waste (HLW) repository. Coupled thermal-hydrological-chemical (THC) modeling performed to date has demonstrated that under certain conditions a heat pipe may be sustained in crushed salt backfill. Creation of a heat pipe leads to enhanced heat transfer effects, significant porosity change, and a substantial impact on the predicted temperature and moisture state of the system (Stauffer et al., 2013). However, model verification and validation are required to accurately predict the onset of heat pipe formation and impacts of this phenomenon. This test plan describes experiments to investigate migration of brine and water vapor in RoM salt, under representative generic salt repository conditions but at a scale that can be achieved in the laboratory.

Our experiments will be designed to examine as many factors as possible in a semi-realistic, although highly down-scaled, scenario. It is not expected that the small-scale laboratory results will produce identical effects when upscaled to the repository level. Instead, these bench-scale experiments will provide validation of the numerical models, and the models will be used to design and predict results of larger intermediate- and field-scale heater experiments in bedded salt. It is necessary to know, in advance of a heated field test, the maximum temperatures expected for a given heat load, the likely redistribution of fluid and salt, where to place instruments to look for evidence of physical processes of interest, and how to use chemical tracers to track fluid and gas migration. Ultimately, all of the experiments and models, together, will be the key to understanding the processes and evolution of a system for disposal of high-temperature waste in bedded salt formations.

2.   Background

The following sections briefly describe the physical processes involved in vapor and brine migration in heated salt, modeling results from a coupled THC numerical code, and prior relevant experimental work, both in-situ and in the laboratory.

2.1   Theory
2.1.1   Brine and Vapor Migration around HLW

Our investigations focus on a generic bedded salt repository design known as the in-drift disposal concept. Canisters of HLW are emplaced on the floor after excavation of the room, and backfilled with crushed RoM salt. There are complex and strongly coupled thermal, hydrological, chemical, and mechanical processes that take place when heated waste is added to this system.

If the temperature of the waste exceeds the boiling point of brine, water will evaporate and migrate in the vapor phase before recondensing beyond the boiling front. If conditions are conducive to it, a heat pipe may be established in the crushed salt (Figure 1). In a heat pipe, free water is vaporized in the boiling region, advects and diffuses along concentration gradients to the cooler regions where it recondenses, and some of the liquid flows back toward the heat source by gravity flow and along capillary pressure gradients where it is boiled off and the cycle is repeated. The replenishing fluid is saturated brine; when boiled off, it deposits a layer of salt around the heater. This leads to the build-up of a low-porosity "rind" while the heat pipe is sustained. Models of this effect (section 2.2) and in-situ evidence for heat pipe formation have been demonstrated (section 2.3).
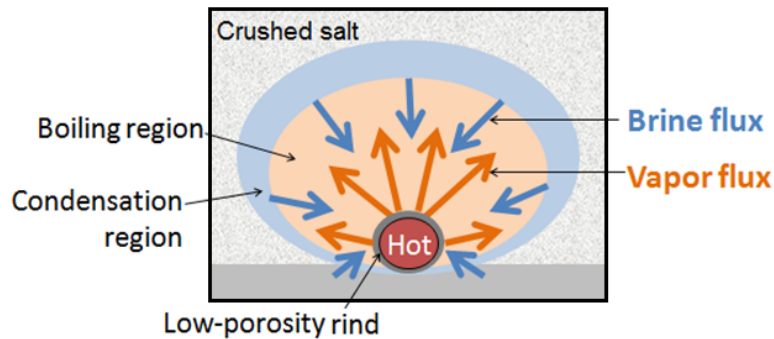
Figure 1. Heat pipe in salt.

Water availability affects the onset and vigor of heat pipe formation. Sources in the system that may feed the heat pipe include intergranular "pore" fluids in the RoM, intragranular fluid inclusions which may migrate to grain boundaries, water bound to clays and other accessory minerals in impure salt which may be released under heating, and inflow from the excavation damage zone (EDZ) into the RoM backfill. If water is available but is not readily mobile in the system, the heat pipe formation will be disrupted. Initially, permeability to air in the crushed salt backfill is high, allowing for efficient water vapor removal from the salt pile. However, porosity may be decreased significantly by redistribution of salt in a heat pipe, causing strongly coupled feedbacks in the salt pile (Olivella et al., 2011). Capillary suction and relative permeabilities are uncertain parameters that also affect fluid mobility.

Dry-out of the crushed salt backfill is influenced by vapor flow in the test drift, which may be caused by barometric pumping or pressure differentials between the sealed-off waste space and the ventilated access drifts. Evaporation from the RoM backfill pile will depend on temperature, relative humidity, saturation, airflow, and pressure in the ventilated spaces. Understanding water vapor migration is a key component of predicting moisture behavior in the RoM salt pile, which has strong feedbacks on heat transport. For example, in the current coupled models, thermal phase equilibrium is assumed between the vapor phase (air + water vapor), liquid phase (brine), and solid phase (rock salt). If thermal equilibrium between phases is not in fact happening at the flow rates seen in the heat pipe simulations, models could be over predicting the amount of vapor condensation.

2.1.2   Evaporation and water vapor diffusion

Within pores, water partitions between the liquid phase and the vapor phase in an amount determined by temperature, pressure, and ion concentration in the liquid (Stauffer et al., 2013). In FEHM, thermal equilibrium is assumed to instantaneously exist between the gas, liquid, and solid phases.

Between pores, movement of water vapor occurs by advection and diffusion between pores, greatly modified by the presence of liquid saturation and salt grains. The free-air diffusivity is modified by a tortuosity term that may depend on porosity and saturation; in the Millington-Quirk empirical model, for example, tortuosity is given by

$$\tau = \theta_a^{7/3}/\phi^2 \qquad\qquad \text{Eq. 1}$$

and the effective diffusion coefficient is

$$D_{va} = \tau\phi\theta_a D_{va}^0 \rho_v \frac{0.101325}{P}\left(\frac{T+273.15}{273.15}\right)^{1.81} \qquad\qquad \text{Eq. 2}$$

where θ_a is the air-filled porosity, ϕ is total porosity, $D_{va}^o = 2.23 \times 10^{-5}$ m^2/s, T is temperature in C, and P is pressure in MPa (Millington and Quirk, 1961; Zyvoloski et al., 1997). However, the Millington-Quirk model has been shown to be unreliable for some geologic media (Jin and Jury, 1996). It has not been studied for RoM salt.

Loss of water vapor from the salt pile occurs by evaporation and diffusion across the RoM/air boundary. Ventilated air flow in the drift may allow efficient removal of water vapor and fluctuating relative humidity, reflecting the local weather, but generally considerably drier than equilibrium moisture conditions in a closed brine-saturated system at in situ repository temperatures. Water loss from the salt pile will depend on the concentration gradient between water vapor in the "unventilated" pores (diffusion), advection from the salt pile into the air, and evaporation that depends on relative humidity, temperature, and water availability at the surface of the pile (replenishment by suction).

2.1.3   Thermal models in crushed salt and air

The thermal conductivity of salt depends on porosity and temperature. The following relationships were developed by combining experimental evidence from the Asse mine in Germany and from WIPP, and are implemented in FEHM:

$$K_{T-300}(\phi) = 1.08(-270\phi^4 + 370\phi^3 - 136\phi^2 + 1.5\phi + 5) \qquad\qquad \text{Eq. 3}$$

$$K_T(\text{T},\phi) = K_{T-300}(\phi)\left(\frac{300}{\text{T}}\right)^{1.14} \qquad\qquad \text{Eq. 4}$$

Where T is temperature in Kelvin and ϕ is porosity.

Heat transfer in the air space between the RoM salt and the drift wall or roof is controlled by a combination of conductive, convective, and radiative heat transfer. Conductive heat transfer is proportional to the temperature gradient and area of contact:

$$q_{cond} = -k_{t,a} A \frac{dT}{dx}$$
Eq. 5

where $k_{t,a}$ is the thermal conductivity of air (0.03 W/m*K; Bejan, 1995). The thermal conductivity of air is significantly lower than that of crushed or intact salt. However, heat transfer by convection in air is considerably more efficient. The Nusselt number (Nu) can be used to relate the effectiveness of heat transfer across a gap by convection as compared to conduction (Bejan, 1995):

$$k_{c,eff} = Nu * k_{t,a}$$
Eq. 6

where

$$Nu = 0.069 Ra^{1/3} Pr^{0.074}, \; 3x10^5 < Ra < 7x10^9$$
Eq. 7

and the Rayleigh (*Ra*) and Prandtl (*Pr*) numbers are given by

$$Ra = \frac{g\beta(T_1 - T_2)H^3}{\alpha v}$$
Eq. 8

and

$$Pr = \frac{v}{\alpha},$$
Eq. 9

where *g* is the acceleration due to gravity, *β* is the thermal expansion coefficient, T_1 is the temperature at the top of the RoM salt, T_2 is the temperature of the roof of the waste drift, *H* is the height of the air gap, *α* is the thermal diffusivity, and *v* is the kinematic viscosity. For high-temperature waste in a drift with a 4-foot air gap, Rayleigh numbers exceed the range given in Eq. 7, and extrapolation of the relationship is required (Stauffer et al., 2013). When using this approximate model for convective heat transfer, as opposed to modeling it directly, the appropriate rate of heat transfer for convection is then

$$q_{conv} = -k_{c,eff} A \frac{dT}{dx},$$
Eq. 10

analogous to the expression for conduction above, but using the "effective thermal conductivity" for heat transfer by convection.

Heat transfer by radiation in air is also quite efficient, with the rate of heat transfer from an object of area A_1 in an enclosure A_2 given by (Kreith and Bohn, 1997):

$$q_{rad} = A_1 F_{1\text{-}2} \sigma (T_1^4 - T_2^4),$$ Eq. 11

where $F_{1\text{-}2}$ is a shape factor representing the areas and emissivities of the materials comprising object 1 (the salt pile) and object 2 (the drift walls), $\sigma = 5.67 \times 10^{-8}$ W/m^{2}K^4, and temperatures T_1 and T_2 are in Kelvin. However, an effective coefficient of heat transfer, analogous to the thermal conductivities for conduction and convection described above, can also be calculated as:

$$k_{r,eff} = \frac{q_{rad}H}{A_1(T_1 - T_2)}$$ Eq. 12

with q_{rad} given above (Eq. 11). Combining the effective heat transfer coefficients for a total effective conductivity gives (Kreith and Bohn, 1997):

$$k_{t,eff} = k_{t,air} + k_{c,eff} + k_{r,eff}$$ Eq. 13

This is the model used in FEHM to approximate heat transfer by convection and radiation, and it requires additional experimental validation, because heat loss through the air gap must be modeled accurately in order to predict final temperature and moisture around heated nuclear waste. Although these relationships for heat transfer by convection and conduction are extremely scale-dependent, they can be computed for any particular experimental setup and compared to the numerical model of that experiment to test for validity. In particular, the experimental setup may not have a mass of salt above to re-radiate, simplifying the shape factor for radiative transfer $F_{1\text{-}2}$ (approximated for a plane parallel system to represent the relative flat top of the backfill and drift ceiling, for example) to simply the emissivity of the salt pile, ε_1. The emissivity of salt, both crushed and intact, is not well-known (Svalstad, 1983).

2.2   Prior Modeling Results

Numerical modeling has been performed by several groups of researchers, but the difficulty in handling the strongly coupled THMC processes has prevented fairly realistic simulation of the HLW temperature regime until recently. The new modeling results have highlighted areas where experimental validation is needed. Our experimental focus will be on the coupled thermal, hydrological, and chemical processes, which are included in LANL's Finite Element Heat and Mass transfer code (FEHM) (Zyvoloski et al., 1997).

2.2.1   Drift-scale models

FEHM modeling results at the drift scale suggest that a wide range of behavior (from extensive heat pipe activity to none) can occur in the RoM salt based on availability of both water and heat and the ability of water to move at low saturations (Stauffer et al., 2013). One of the primary indicators of a heat pipe is porosity change. In drift-scale simulations, porosity change from the initial 35% in the model is seen in Figure 2. In this case the only parameter varied was the heat load in the canisters. For increasing heat loads ranging from 250W to 750W in Figure 2, the porosity changes are significantly enhanced for higher heat loads and temperatures. In all cases there is little water flowing into the RoM salt from the EDZ, the initial saturation of the RoM pile is 10%, and the maximum capillary suction in the RoM salt is 1.0 MPa. One metric for total porosity change is following:

PC_{tot} = sum over all RoM nodes (abs(initial porosity – final porosity).

This metric produces a volume of porosity and sums both gains and losses. For case A, this value is 0.16 m^3, for case B it is 0.46 m^3, while for case C PC_{tot} = 1.18 m^3.
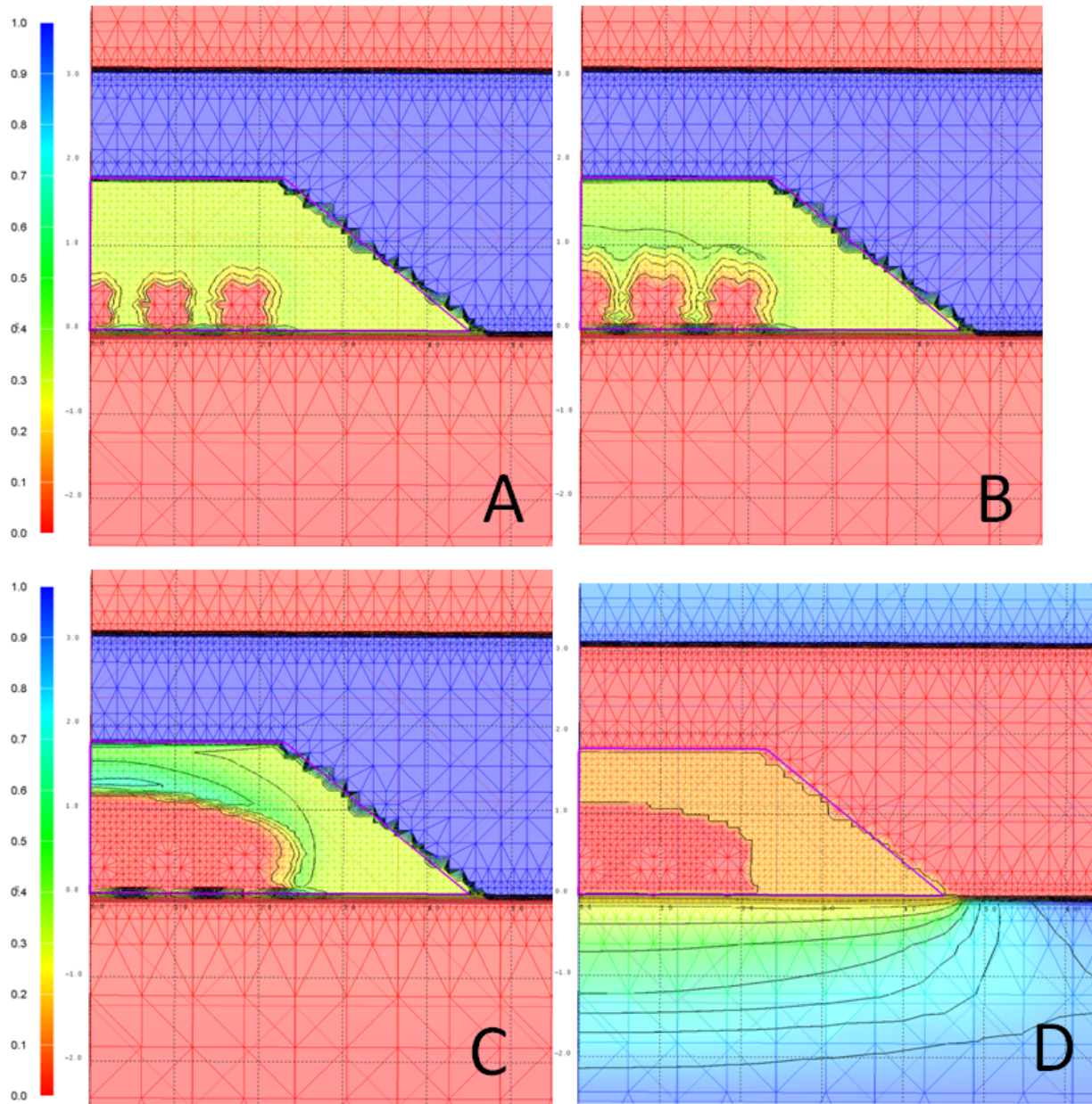

Figure 2: Porosity after 2 years of heating for A) 250W B) 500W and C) 750W per heater. D) Saturation corresponding to the porosity in C.

The large changes in porosity seen in the 750W case are caused by the heat pipe effect whereby purified water vapor is driven away from the heat source and saturated brine is pulled back

towards the heat from wetter regions to the very dry region that develops around the heaters. Note that the model does not consolidate the crushed salt when porosities become very large in the condensation halo. The dry-out region can be seen in Figure 2D.

Other sets of simulations have demonstrated the impact of initial available water and capillary suction properties on porosity redistribution (Stauffer et al., 2013). Initial saturation in the crushed salt is an uncertain parameter, depending on many factors such as how long the RoM backfill has been allowed to sit in the ventilated repository before enclosing the waste. Additionally, it is extremely difficult to quantify the amount of water that will seep into the excavated drift; past brine flow investigations have produced extremely varied results (Kuhlman and Malama, 2013). For this reason, initial saturation was varied from 0.01 to 0.10 and the effect on the heat pipe was modeled. Very low saturation cases did not produce a heat pipe, even with clay dehydration added to the code to release water at appropriate temperatures for dehydration reactions. At some intermediate saturation, depending on clay content, a heat pipe becomes possible. There are strong temperature differences between the wet initial condition that created a heat pipe and the other initial states that do not.

Capillary suction affects the available water in the RoM salt through interactions with the EDZ beneath the pile. With greater natural suction, more water can be pulled into the RoM salt, providing more drive for the heat pipe. Similarly, more suction allows the heat pipe to strengthen, as increased suction is more efficient at pulling saturated brine back towards the hot region. Varying from 0.1 MPa of maximum suction in the RoM salt to 0.5 MPa impacts the two year porosity distribution. The PC_{tot} for these cases is 0.29 m^3 and 0.38 m^3, respectively. In both cases the initial saturation is 5% with 750W canisters.

2.2.2   Laboratory-scale models

Models were also developed to test whether the heat-pipe results seen at the drift scale would likely be reproducible in the laboratory. Initial results indicate that it will be possible to see porosity change after only a few days of heating for a fairly simple setup. Modeling was performed for various designs, including a thin tank, a square tank, a rectangular tank; the heater was placed on the floor of the tank, or raised in the crushed salt pile; and the presence or absence of a saturated layer beneath the heater was considered. Models such as these will be used throughout the experimental design process to design the exact setup and predict the detectability and redistribution of tracers such as HDO, D_2O, and $H_2^{18}O$.

An example of one small-scale 3-D model is shown in Figure 3. In this case, a square tank of dimensions 0.3 by 0.3 m (1.0 by 1.0 ft) has a heating element elevated to 5 cm above the bottom of the tank. Elevating the heater provides additional insulation to heat loss from the bottom, unless the tank bottom is well-insulated. The initial porosity was 35% in the RoM salt and initial saturation was 5%. Temperature, saturation, and porosity at 1 and 5 days are shown in Figure 4. The porosity change around the heater would likely be observable in the laboratory.

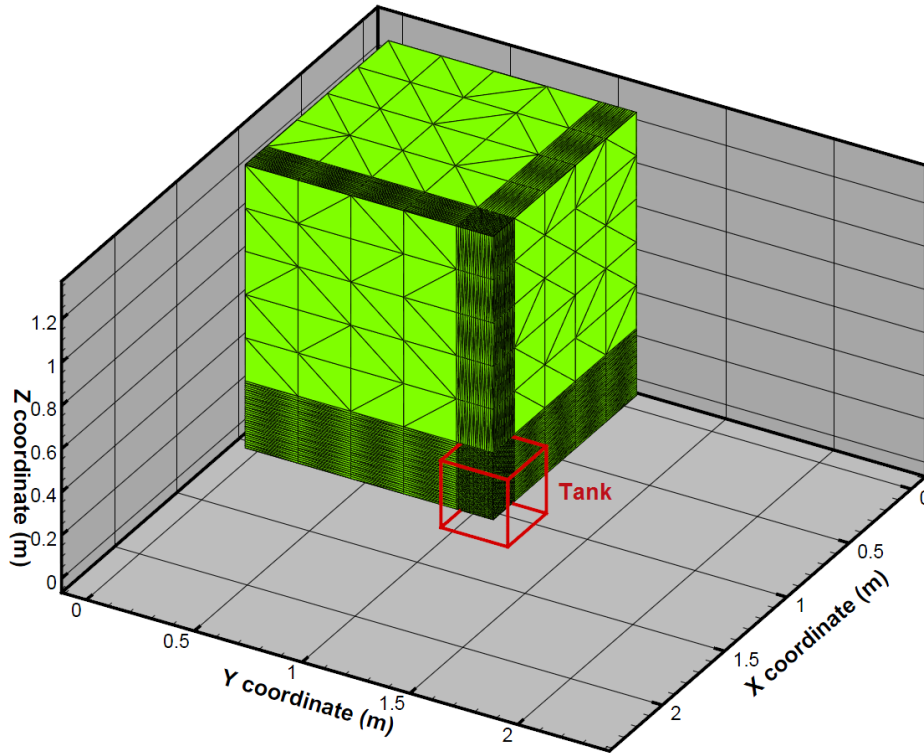Figure 3. Cut-away showing one-quarter of the computational mesh for the laboratory-scale model with a 1 ft by 1 ft by 1 ft tank. Minimum mesh size was 1 cm in the tank, and maximum mesh size was 24.5 cm in the air.
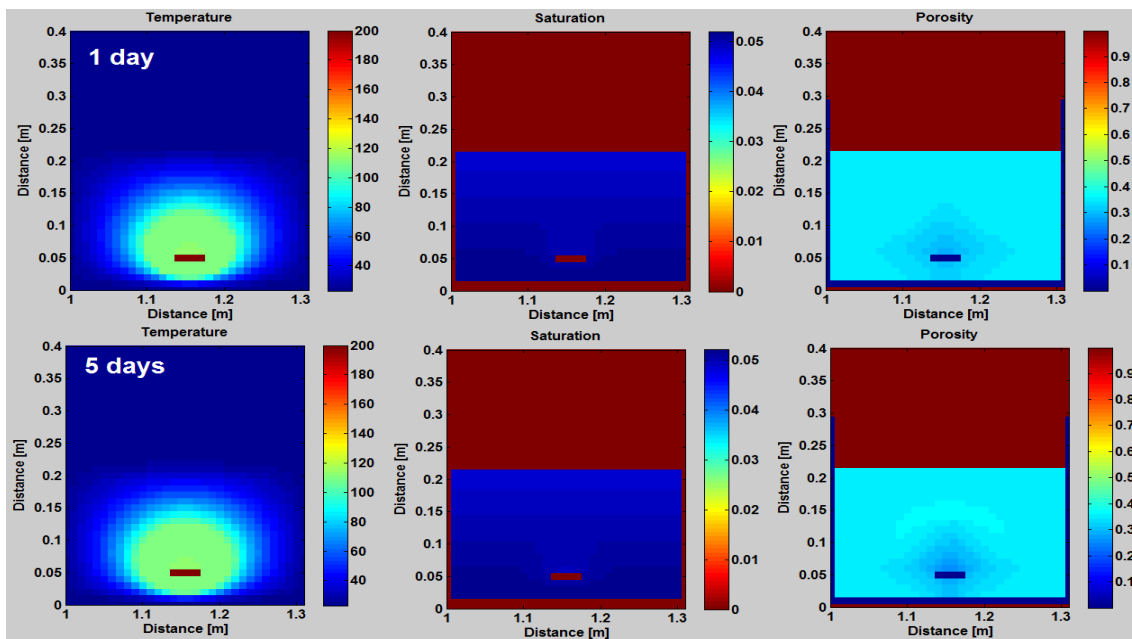
Figure 4. Model results (temperature, saturation, and porosity at 1 day and 5 days) for a 0.3 by 0.3 m (1.0 by 1.0 ft) open tank with salt to 20 cm height. The heater is elevated by 5 cm in the crushed salt.

Sensitivity studies can be quickly performed with the model to test different setups and design an experiment with high likelihood of heat pipe formation. For example, for a particular scenario, the effect of temperature, initial saturation, and maximum capillary suction are shown in Figure 5. This model is of a larger tank than the case above; dimensions were 40 cm by 80 cm by 20 cm. In this case, likely due to model assumptions and the setup chosen, initial saturation and maximum capillary suction have little effect on heat pipe vigor (extent of the 0.2 porosity contour), while temperature of the heater has a significant effect. The results below are preliminary; additional modeling will be performed in as part of the experimental design effort.

Figure 5. Effect of temperature, initial saturation, and maximum capillary suction on porosity (ϕ) after 5 days of heating. The $\phi = 0.2$ contours are shown. Initial porosity was 0.35.

2.3   Prior Experimental Results

Kuhlman and Malama (2013) provide an overview of in-situ experiments at WIPP. To date, few dedicated laboratory experiments have been performed on crushed RoM-like material; the results of these will guide and inform our proposed experiments to avoid unnecessar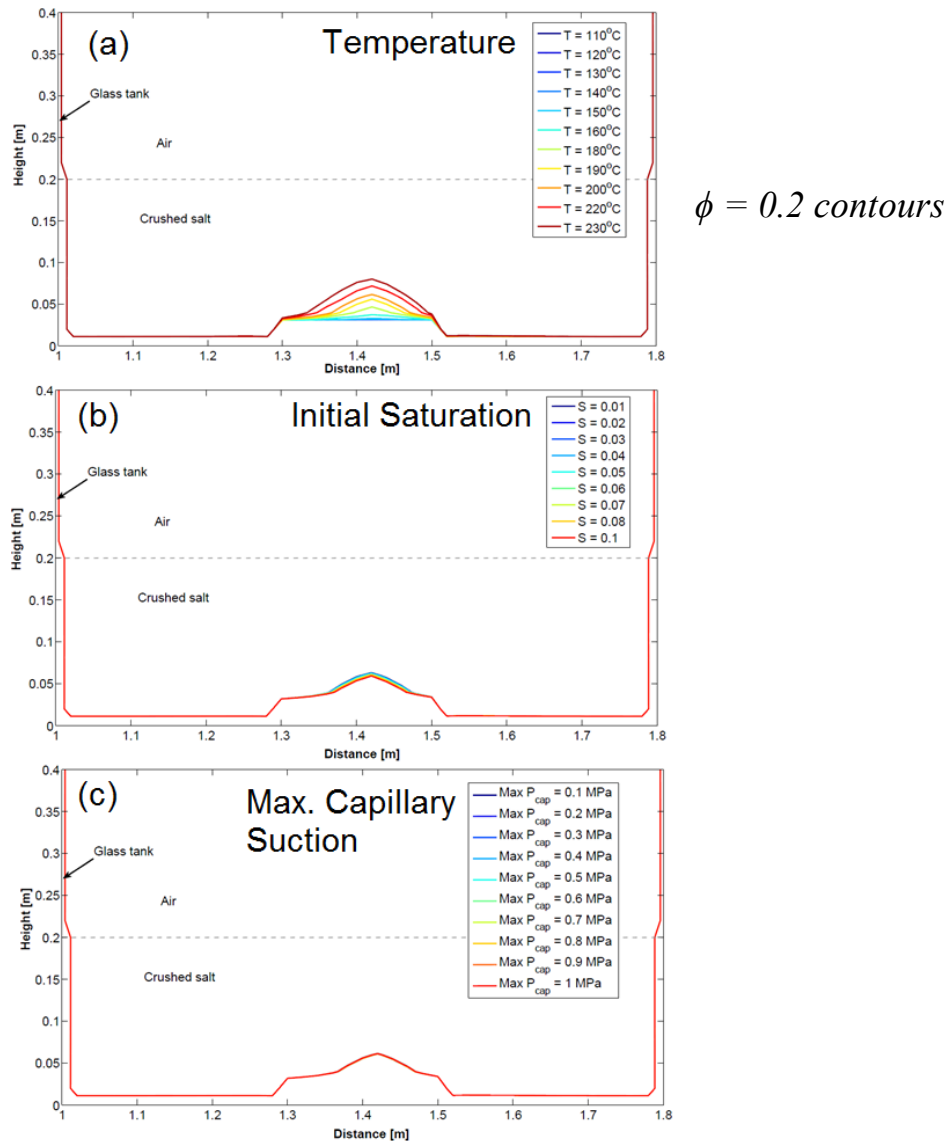y replication and show where additional confirmatory experiments are needed. Relevant historical and recent prior experiments, both laboratory and in-situ, are listed below. Additional information about these experiments may be found in the references herein.

There is experimental evidence for porosity change from a heat pipe in 1980s heater tests at WIPP in boreholes backfilled with crushed salt (Krumhansl et al., 1991). After heating to up to 130°C, heaters in both open boreholes and boreholes backfilled with crushed salt were found to be enclosed in solid rock salt with final bulk density within 99% of that of intact salt (2160 kg/m^3) (Figure 6) (Brady et al., 2013). The in-situ appearance of a rind, indicating heat pipe activity, provides the primary motivation for studying the phenomenon in much greater detail to gain an understanding of whether such features are likely to form during HLW repository operation.

Figure 6: Heater from borehole BO45 showing porosity reduction as a likely result of heat pipe activity (Krumhansl et al., 1991). From Brady et al. (2013).

A key aspect of utilizing prior experimental work is lessons learned from instrumentation tests in the harsh, high-salinity and potentially acidic environment produced by heated WIPP salt. Otto and Miller (2013) provide a review of many instrumentation techniques used by themselves and others to measure saturation, humidity, matric potential, and more. For saturation measurements, they had success with water content reflectometers (also known as time-domain reflectometry, or TDR), although minor corrosion on the probes and the need for good contact with salt grains

created complications that were solved. Levitt (unpublished report, 2014) used neutron probes for moisture measurements in salt and also had success. All methods require calibration.

Other relevant in-situ and laboratory experiments, both published and in progress, include the following; additional details about the most relevant experiments are given in section 4:

- Olivella et al. (2011) – laboratory experiment directly showing porosity redistribution in crushed salt under high thermal load as a result of heat pipe formation
- Stauffer et al. (2013), Caporuscio et al. (2013) – preliminary experiments to test/validate the FEHM thermal conductivity function of porosity and temperature in crushed salt
- Caporuscio et al. (2013) – experiments on clay and impurity properties in WIPP samples
- Cinar et al. (2006) – retention characteristics of "compacted" salt, an intermediate state between crushed and reconsolidated salt; relation between permeability and porosity
- LANL-CFO Instrumentation studies (Otto and Miller, 2013)
- LANL-CFO Air ventilation tests (Otto and Miller, 2013)
- LANL-CFO 2013 Evaporation tests (Otto, personal communication, 2014)
- LANL stable isotope experiments (Boukhalfa et al., unpublished, 2014) – fractionation factor for water vapor and liquid above spiked (D_2O) brine at various temperatures

3.   Experimental Objectives

The objectives of this experimental effort are summarized in Table 1. They are subdivided into three categories: (1) validating numerical models, (2) supporting the planning of more elaborate and expensive large-scale tests, and (3) understanding specific physical processes that can be observed under controlled conditions in the laboratory.

1. Validate physicohemical and hydrologic parameters and processes used in numerical simulations of heated nuclear waste in bedded salt, with a focus on RoM salt backfill:

The observed spatial and temporal change of porosity, temperature, and moisture will be compared to numerical predictions of the same. The comparisons will be used to challenge all sub-models and assumptions used in the numerical simulations. Currently, data gaps exist in the following physical processes as modeled: evaporation rate as a function of temperature, relative humidity, saturation in the RoM salt, and air turnover rate; water vapor diffusivity in porous RoM backfill; thermal models in RoM salt and air, with parameters such as emissivity for radiative heat transfer considered highly uncertain; and the effect of compaction during porosity reduction. It also may be possible to study the range of possible relative permeability and capillary suction functions of saturation by fitting to the experimental data, although these will not be directly measured in the tests.

2. Support the planning/design of thermal tests at intermediate and field scales:

The small-scale experiments and the validated models will be used to determine, in advance, the expected temperature and moisture distribution and whether a heat pipe is expected to occur for a given large-scale experimental scenario. This may lead to different design choices in instrumentation and tracer usage in these more extensive experiments. The coupling of modeling

and laboratory work on the small-scale experiments will also challenge the assumption that the numerical model has the ability to accurately predict the outcome of a very complex heated system.

The small-scale experiments will also help ensure that the larger-scale experiments are designed and instrumented with enough measurement capability and sensitivity to meet objectives for those experiments (e.g., is the spatial frequency of temperature sensors sufficient to support parameter estimation in the matching of model to experimental outcome? Are the isotopic tracers of high enough concentration to produce a measurable signal at the locations of interest?). Sampling for water isotopes in the vapor phase presents some unique challenges related to the volumes of air required. The small-scale experiments will give researchers a chance to test methods of sampling the vapor phase to minimize perturbation of the air in the test.

These experiments also afford the opportunity to test laboratory equipment and determine its efficacy in a high salinity environment. Evidence already suggests that some traditional measurement techniques will fail when subjected to the salt and brine. These problems should be detected and solved in a cheaper test than during the field-scale operation.

3. Understand key coupled physical processes that may be observed in the larger-scale tests and in operation of a HLW repository:

The ultimate objective of all of our work is to help understand the safety and efficacy of HLW storage in salt. A thorough understanding of the physical processes is required to meet that objective. While prior research has been done on many aspects of hydrogeological processes in porous media, little data exist for these processes in solid salt with a saturated brine. We aim to study water vapor loss across the boundary from porous RoM salt to air; water vapor diffusion in porous RoM salt; the particulars of heat pipe development, timescales of establishment and operation, and extent of porosity change; and sensitivity of heat pipe vigor to temperature of heaters, water content in the RoM salt, and air properties.

	Objective	Instrumentation needs
1	Validate physicohemical and hydrologic parameters and processes used in numerical simulations of heated nuclear waste in bedded salt, with a focus on run of mine (RoM) salt backfill	
1a	Evaporation rate as a function of temperature, relative humidity, saturation in the RoM salt, air turnover rate	Water vapor in air, relative humidity, in-situ initial and transient saturation in salt, tracers (D_2O, $H_2^{18}O$ and SF_6), temperature, heating block and temperature control
1b	Water vapor diffusivity in porous RoM backfill	Isotopic and non-partitioning tracers (D_2O, $H_2^{18}O$, SF_6)
1c	Relative permeability/capillary suction functions of saturation	Not directly measured in this test plan; inverse modeling from experimental results to simulation parameters
1d	Thermal models in RoM salt and air	Temperature spatially in salt and air, tracers (D_2O)
1e	Compare observed distributions and numerical predictions of salt, temperature, and moisture spatially and temporally. Challenge all assumptions used in the numerical simulations	Forensic state of the system: saturation, temperature, and porosity change
2	Support the planning/design of thermal tests at intermediate and field scales	
2a	Determine, in advance, whether a heat pipe is expected to occur for a given experimental scenario	Multiple trials with different conditions (e.g., saturation, sieve-sorting of salt, temperature)
2b	Ensure that the larger-scale experiments are designed and instrumented with enough measurement capability and sensitivity to meet objectives	Test all instruments that may be used in the field (D_2O, $H_2^{18}O$, SF_6 collection and analysis, thermocouples, relative humidity, moisture sensors, etc.)
2c	Test new experimental equipment and determine its efficacy in a high salinity environment	Test all instruments that may be used in the field during this experiment (see above, 2b)
3	Advance understanding of key coupled physical processes	
3a	Vapor transport: diffusion models in porous material	Isotopic and non-partitioning tracers, measured throughout salt pile (D_2O, $H_2^{18}O$, SF_6); water vapor pressure sensors
3b	Water vapor loss across the boundary from porous RoM salt to air	Isotopic tracers, measured in salt pile boundary and in air; in-situ transient saturation
3c	Heat pipe: timescales of establishment and operation, extent of porosity change	Temperature resistant dye, observation port, and forensic samples
3d	Sensitivity of heat pipe vigor to temperature of heaters, water content in the RoM salt, air properties and turnover	Perform multiple experimental trials with different conditions (see above, 2a)

Table 1. Objectives of the experimental effort and instrumentation needs.

4.   Experimental Methods

The sections that follow discuss three main types of experiments: bench-scale heat pipe tests (section 4.1), individual process experiments (section 4.2), and an intermediate-scale experiment to be performed in Carlsbad (section 4.3). The experiments will address a complimentary, overlapping set of the objectives listed above (Table 1). For each experiment, the relevant prior experimental work, proposed instrumentation, and draft plan of experimental conduct is given in the subsections that follow.

4.1   Description of Proposed Bench-Scale Heat Pipe Experiments

These small-scale experiments will be designed to produce heat pipes in a tank of dimensions 1 to 2 ft on a side. The purpose is to demonstrate the sensitivity of heat pipe formation on various parameters and controls and to test physical processes under temperatures and relative humidity similar to the RoM salt in the immediate vicinity of the high-temperature canisters. To gain a better understanding of the sensitivities, several trials of the experiment will need to be performed. The number of trials will depend on availability of salt, prior experiment success, and funding.

4.1.1   Relevance of earlier work

To our knowledge, the most relevant prior work on heat pipes in crushed salt are the in-situ heater experiments described in section 2.3 and the laboratory work of Olivella et al. (2011). In the experiment by Olivella et al., a heat pipe was established in a 5 cm by 10 cm cylindrical apparatus with fixed temperature plates on either end and a closed system for mass. Porosity change was observed along the length of the cylinder as expected for a heat pipe, with dissolution at the condensation front and buildup of a low-porosity rind. They provided destructive testing of the system at various times (7, 15, 30, and 65 days) to determine the spatial and temporal pattern of porosity change and saturation, and compared model results to the experimental. The experimental results were well-matched by the numerical model CODE-BRIGHT for the same scenario (Olivella et al., 2011). Their work also produced a surprising result, which is that the final state of the system was relatively insensitive to initial saturation, a finding that contradicts longer-term FEHM drift-scale model results (2 years) but agrees with some bench-scale modeled scenarios using FEHM. Our work is intended to confirm the results of Olivella et al. in a somewhat more realistic scaled system with controlled airflow allowed and more space around the heater for RoM backfill.

Research on instrumentation in the high-salinity WIPP environment has been performed by LANL-Carlsbad Field Office (Otto and Miller, 2013). Lessons learned from their experiments with various methods of determining porosity, saturation, and matric potential will be applied to the choice of instrumentation in all of the experiments discussed in this test plan. Neutron probe moisture measurements in salt have been tested on WIPP RoM (Levitt, unpublished report, 2014), and similar measurement techniques may be used here.

4.1.2   Preparation/Setup

Figure 7 shows a draft schematic of the proposed setup for heated experiments. As the experiment is underway, minor changes to the layout may be required based on equipment availability and cost, modeling results, and lessons learned about the proposed techniques.
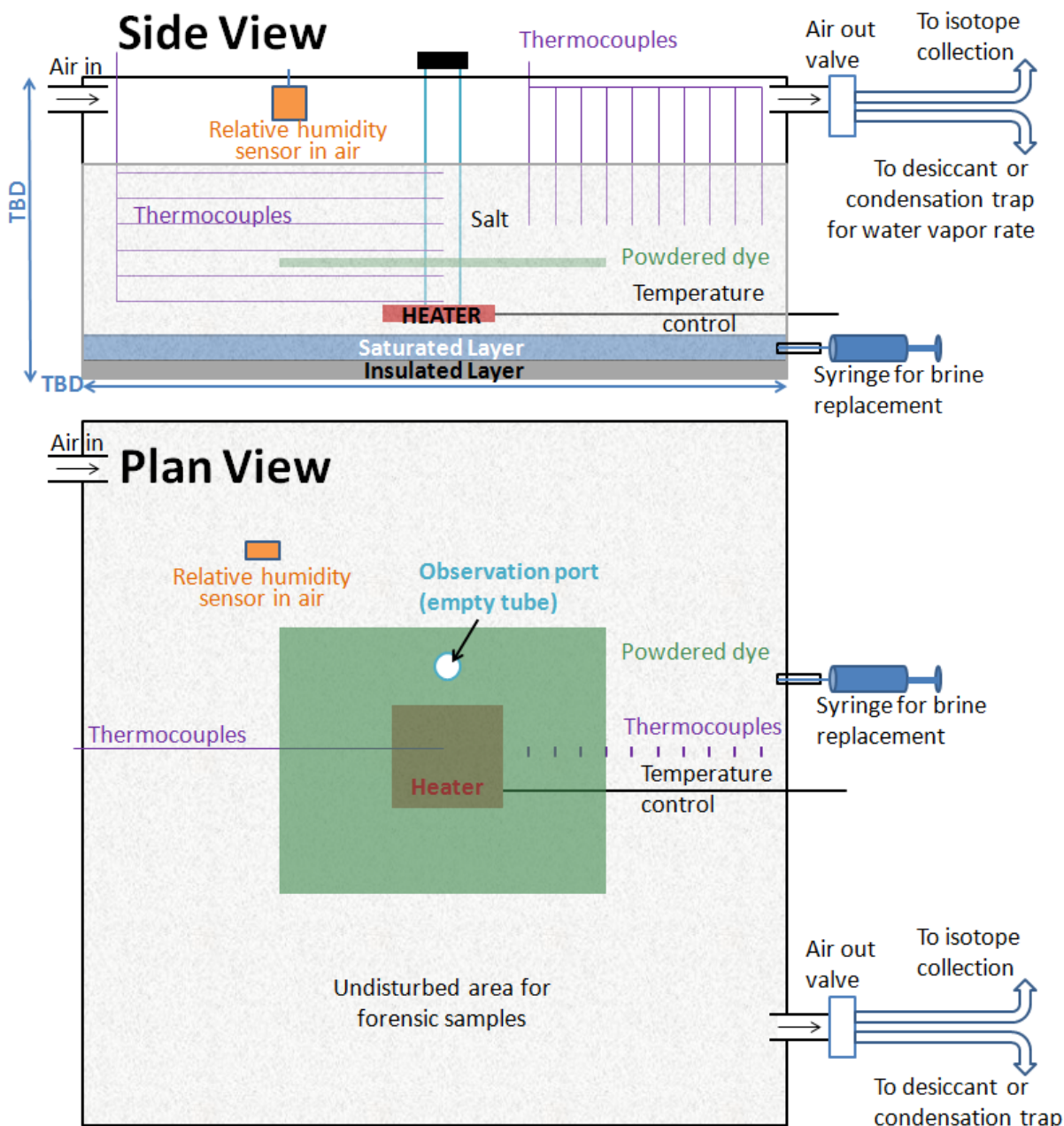
Figure 7. Draft schematic of the heated experiment.

The dimensions of the container will be 1 to 2 ft on a side (exact size to be determined), and the apparatus will be square. Instrumentation will be present in 3 of the 4 sides of the apparatus, but because the effect of the equipment on the heat pipe itself is unknown, the 4th side will be unadulterated. Forensic studies will be performed after the experiment is concluded, primarily on

the side that did not previously have equipment, although analysis of the other sides will reveal information about how the presence of the equipment changed the final state of the system. Depending on the size of the apparatus, it is possible that the off-the-shelf acrylic tank may need reinforcing (wood framing) on the outside to support the weight of the salt inside. The bottom of the tank will be insulated inside to protect the acrylic from the heater.

One difficulty in realism of the experiment is simulating the availability of moisture from the EDZ and surrounding intact salt. In practice, when a borehole is drilled into bedded salt, it seeps with an influx rate that is difficult to predict as a function of temperature and which depends on geologic heterogeneity (Kuhlman and Malama, 2013). In the drift-scale numerical models, the permeability of the EDZ is determined by comparisons with data from WIPP. In the laboratory, it will be difficult to use a material below the heater and RoM salt that realistically simulates the transfer of water from the EDZ to the crushed salt. We propose including a saturated layer below the heater as a water source, although we recognize that suction characteristics between this layer and the RoM salt may not accurately represent EDZ fluxes. The saturated material may be additional RoM salt or an inert material such as sand or glass beads. For comparing numerical models to the experiments, this material should have reasonably well-understood retention characteristics. Alternatively, a porous or ceramic plate may be used at the bottom to allow water to enter the RoM salt at a known rate. Brine will be inserted into the apparatus by a syringe through a port at the bottom of the tank. If replacement brine is desired during the course of the experiment, it may be supplied through the same port.

Mine-run salt in the experiment will be sieved to a yet-to-be specified diameter, based on calculations and experience, that will allow the material to behave as a continuum in the down-scaled experiment. Ultimately, a balance will need to be reached between overall size of the apparatus (ideally, smaller; i.e., easier to build, using less material, and cheaper) and grain size distribution (ideally, larger; i.e., more like the in-situ RoM grain diameters, but small enough to model as a continuum). It may be advantageous to use pure halite in the heat pipe experiment, although it will be less useful for testing the thermal models developed for RoM salt. The advantages to using pure, clean salt of varying size include a potential lack of availability of large quantities of WIPP RoM, as well as impurities in RoM (clays) causing interference in measurements.

Instrumentation will be set up with as few penetrations in the tank as possible. The instrumentation would instead penetrate a lid, which would be fabricated from materials to be determined and well sealed/insulated. Where the tank is penetrated for wiring or instrumentation, O-rings and seals will be used to prevent leaking.

The tank will be outfitted with inlet and outlet ports for air, to be hooked to gas flow equipment and gas collection equipment (for both isotope and water vapor mass measurement). These procedures will be tested and refined during the individual process experiments (section 4.2.2.1). The air out may be diverted to either a collection system for isotopic measurements or a collection trap (condensation or desiccant cartridge) for water vapor which would be used to quantify the flow rate of water vapor with time out of the system. A valve would be used to direct air out to either instrument. The valve system would be the same as the one described for the water loss experiment (section 4.2.2.1) and would be tested first in that setting.

Thermocouples will be emplaced in the salt by a procedure that allows accurate measurement of their locations while filling the apparatus with salt. Thermocouple location may need to be re-determined at the end of the experiment if solid redistribution has caused them to shift. The number of thermocouples will be based on size, price, etc., but ideally the resolution of the thermocouples will be as high as possible in a vertical line above the heater and a horizontal line at a chosen depth.

An observation port will consist of an empty tube, glass or acrylic, at a particular distance from the heater where porosity redistribution is expected to take place. The tube will be used for (a) camera and (b) a neutron probe if possible. The size of the observational equipment will be used to determine the minimum diameter of the observation tube (e.g., neutron probe diameter is approximately 1.5 in.).

Condensation on the observation tube may be a problem for optical observations. For this reason, we propose including a layer of powdered dye in the salt which would intersect the observation tube. Changes to the dye layer when reached by the condensation front would at least be visible, even if other observations are impossible. Lessons learned from early iterations of the experiment would help determine whether porosity change will be visible throughout the experiment in the observation tube. The tube will be capped when not in use to avoid heat conduction via the tube.

A relative humidity sensor suited for use in high-salinity (e.g., marine) environments will be placed in the air above the salt pile for continuous data collection via data logger. RoM pile saturation information is desired during the course of the experiment, in addition to forensic samples that will be destructively measured for saturation (by heating and weighing). These measurements may be made through the neutron logging in the observation port (discussed above) or an additional method such as reflectometry (Otto and Miller, 2013), which is not shown in Fig 7. Thermocouple psychrometers are proposed as an instrumentation capability in the water loss experiment (section 4.2.2.1). They could be used in this experiment as well.

Stable isotope spiking of the initial ponded water at the bottom will serve as a tracer to help isolate and model individual processes. Isotope use in the individual process experiments (section 4.2) will help clarify isotope fractionation behavior in more complex systems with phase change and diffusion. The predicted versus observed breakthrough time of isotopes in the vapor phase, plus forensic analysis of isotope distribution in the liquid, will ensure that our models of vapor transport and of isotope-fractioning processes are accurate. We propose using either a Picarro-type cavity ring-down laser spectrometer, which would in theory allow for continuous measurement of isotopic enrichment of water vapor. In practice, the Picarro measurements may be sensitive to the production of acidic vapor from heating the impure RoM salt (Boukhalfa et al., unpublished report, 2014), and would require the equipment to be set up in the same laboratory as the machine. If the Picarro is not practical, a GV Instruments Isoprime continuous flow isotope ratio mass spectrometer may be used instead. In that case, a condensation trap with liquid nitrogen or dry ice would be used.

4.1.3   Equipment Needed

Whenever possible, equipment already owned by the laboratories will be utilized. Items marked with a * are optional, but may be needed.

Apparatus
Acrylic tank (dimensions TBD)
Lid materials and insulation, sealant
*Wood/screws for framing
Glass or acrylic observation tube (dimensions TBD)
Seals, O-rings for tank penetrations
Heater
Insulating material
Test materials
Run-of-Mine salt or pure halite
Saturated brine prepared by dissolving RoM salt in DI water
Stable isotope tracers (D_2O, $H_2^{18}O$) to be determined
Laboratory equipment
Soil sieve(s)
Scales
Oven for drying samples
Saw for cutting forensic samples
*Small coring device for forensic samples
Air inlet equipment (dry air or variable relative humidity capability)
Vapor collection outlet equipment, including: 2-way valve, tubing, heat tape, clamps
Fluid syringe
Instrumentation
Thermocouples (number TBD)/recording capability for the thermocouples
Relative humidity sensor for air/recording capability for the RH sensor
Camera for visual observations in tube
*Neutron probe or TDR
*Thermocouple psychrometers (option)
Stable Isotope Analyzer (Picarro or mass spectrometer) and sample collection equipment
Desiccant cartridges or condensation trap for collection of water vapor at outlet
*Powdered dye

4.1.4   Test procedures

1) Sieve the salt to a maximum diameter, to be determined.
2) Perform pre-test analyses, will include post-sieving grain size distribution, bulk density, grain density, porosity, and initial saturation of the RoM salt. Saturation will be determined by oven-drying and weighing.
3) Conduct pre-test modeling to predict onset of heat pipe formation, likely maximum temperatures, extent of porosity redistribution, etc.
4) Construct experiment apparatus, including: tank, framing, lid, air in/air out tubing and connections.

5) Begin adding RoM salt, ponded water with isotopic tracers, heater.
6) Meanwhile, instrument the apparatus with thermocouples as RoM salt is added. Measure locations of thermocouples accurately.
7) Hook up the air inlet and outlet equipment. Prepare for vapor collection.
8) Turn on the heat.
   a. Air out – sample for isotopes, water vapor content
   b. Observation port – look for change in porosity, dye movement
   c. Temperature, relative humidity measurements throughout
9) Allow system to run for as long as useful information is being collected and for as long as the pre-test modeling suggests that the heat pipe will produce porosity change. Turn off the heat.
10) Collect forensic samples.
   a. Observational evidence of porosity change while sawing back into salt.
   b. Core saw or slab cuts for samples for bulk density, saturation spatial distribution, isotope distribution.
   c. Dye – see how far it traveled.
11) Replicate experiments as desired with different heat loads, water availability, etc.

4.1.5   Analysis

A model will be built of the final experiment as-performed. All of the time series and forensic data will be compared to model results. Discrepancies between the model and the experimental results will provide key information about model assumptions, data gaps, parameterization problems, etc. The individual process experiments (described in section 4.2 below) will be used both before and after the full heat pipe experiment, based on these results, to test emergent hypotheses and refine the model.

4.2   Description of Proposed Bench-Scale Individual Process Experiments

These bench-scale experiments will be designed to test specific physical processes under conditions similar to the RoM backfill at lower temperatures than the heat pipe experiments. The experiments will include control of the air above the crushed RoM salt, including airflow and relative humidity. Expanding the temperature range beyond ambient is desired, and external heaters will be used (e.g., heat lamp), while internal temperatures will be measured.

There are two proposed individual process experiments: (#1) measure water loss from a RoM salt pile, and (#2) quantify diffusion parameters in RoM. The objectives of the evaporation experiment are to understand water vapor migration by diffusion and evaporation in a column of RoM, particularly loss at the air boundary in the presence of airflow; and to test the fractionation of condensable isotopic tracers in the system. The objectives of the diffusion experiment are to validate the tortuosity model currently used in FEHM (Millington-Quirk) and the model for effective diffusivity as a function of temperature, pressure, and tortuosity (Eq. 2). It is expected that most of the same equipment can be used for the two experiments, and that once the equipment is tested, validated, and set up, other experiments and replicates can be performed with relative ease.

In these experiments, the use of tracers will be especially key to quantifying evaporation and fluid migration; the experiments will also test out procedures for measuring the tracers for larger-scale experiments. Both of the experiments will rely on isotopic tracers in water vapor (HDO, D_2O, $H_2^{18}O$, or HTO) to track processes in the salt pile. In the diffusion experiment, a nonreactive, noncondensible gas-phase tracer (such as SF_6) is desired as well to help separate fractionation as a result of diffusion from evaporation and mixing of water vapor isotopes throughout the column.

4.2.1   Relevance of earlier work

A passive evaporation test has been performed underground at WIPP, with evaporation of brine from a beaker over time measured by weight change, along with local temperature, relative humidity, wind speed, and barometric pressure. A control beaker of distilled water was also measured. They found that that a layer of salt built up on the brine beaker, which then began to show hygroscopic behavior and gained water from the air, even at relative humidities of around 25-30% (Otto, unpublished data, 2013-2014). These experiments were terminated after the February 2014 truck fire in WIPP and no new in-situ data will be available in the short term. Our proposed experiment will measure the variables directly relevant to evaporation in the immediate vicinity of the salt (e.g., relative humidity, air flow, temperature) and will control relative humidity and air flow in and out of the chamber. Our experiment will measure water vapor collected instead of weight change of the whole system, and will take place in a column apparatus to understand spatial characteristics of brine and water vapor migration.

A follow-on experiment to the in-situ WIPP evaporation studies is planned for the laboratory at LANL-CFO. This experiment will control and measure the same properties (temperature, relative humidity, airflow, weight change at the beaker-scale) but in a controlled environment allowing for the testing of various scenarios. In all cases, water content determination will be made by weight loss determination, some ICP-MS analyses will be done to support and characterize salt used, pre-screened mesh sizes will be used, temperature throughout the salt will be measured based on modeling needs. Overall RH will be monitored and controlled (per lab/experiment design) and flow parameters (rate and humidity content) will be controlled and adjusted. Figure 8 shows the experiment setup for the follow-up evaporation tests. Data from these experiments will be used in conjunction with the proposed experiments in this test plan to help understand water loss in RoM salt.
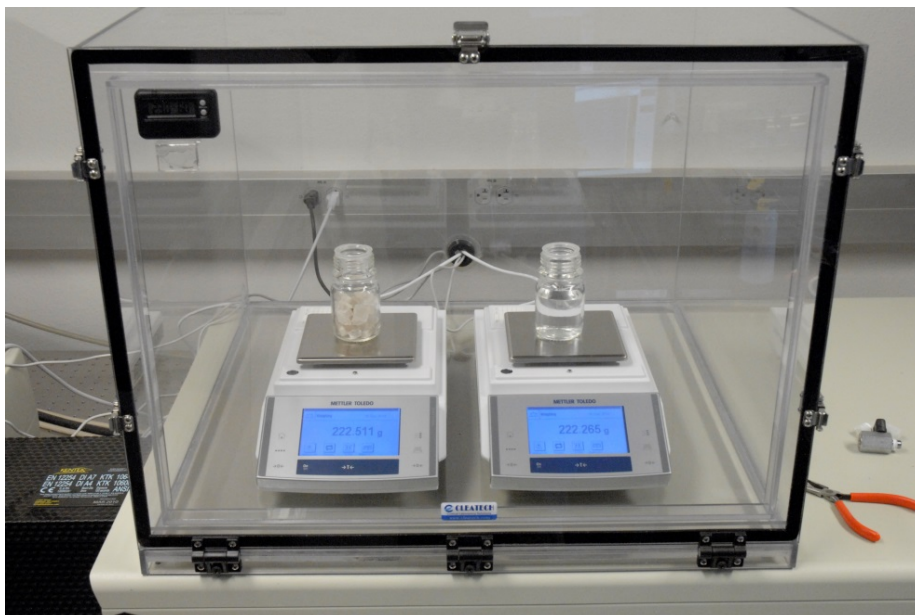
Figure 8. Relative humidity environment chamber configured with balance for weight loss/gain measurements.

Prior experiments to test water loss and evaporation in natural systems (e.g., with lysimeters) are plentiful, but typically are focused on effects such as radiation and insolation, evapotranspiration and the effects of crop cover, etc. Even for bare-soil experiments, the difference between the repository setting and an outdoor setting will be significant. Therefore, we do not see much literature of use on evaporation rates from bare porous material. Furthermore, our system includes saturated brines and hygroscopic behavior of salt and impurities that will be different from soil-water systems.

Experiments on isotope fractionation while a soil column undergoes evaporation have been performed, both outdoors and in the laboratory (e.g., Shurbaji et al., 1995; Barnes et al., 1989). None of these experiments have been performed for RoM salt-sized grains and for saturated brines. Typically, the experimental results have been compared with simplified models of vapor transport to show the deficiencies in the models. We expect our comparatively full-physics model (e.g., thermal effects on diffusive transfer through the vapor pressure as a function of temperature) should compare well with the data, but where it does not, we plan to investigate further.

Water vapor diffusion in soils has been studied by some prior researchers (e.g., Jin and Jury, 1996; Bruckler and Ball, 1989). The experiments typically measure diffusion through variably saturated materials and make comparisons with the Millington-Quirk and other models for tortuosity (Eq. 1 and 2 above). These experiments provide valuable insight into experimental design and setup, but have not been performed for material like RoM salt and with brine.

4.2.2   Preparation/Setup

The two primary experiments are described below, one to test evaporation and water loss from the salt pile with air turnover in the experimental chamber, and one to study water vapor diffusion in RoM salt. For replicate experiments on the same apparatus in either case, pre-treatment of the available RoM salt may be required to ensure that replicate experiments are starting from the same initial conditions. For example, the original RoM salt may be split into batches, heated at a specified temperature for the same amount of time, tested for saturation, and stored properly to avoid changes from varying ambient environmental conditions.

4.2.2.1   Evaporation experiment

A draft layout of the proposed evaporation experiment is shown in Figure 9. As the experiment is underway, minor changes to the layout may be required based on equipment availability and cost, pre-experiment modeling results, and lessons learned about the proposed techniques.
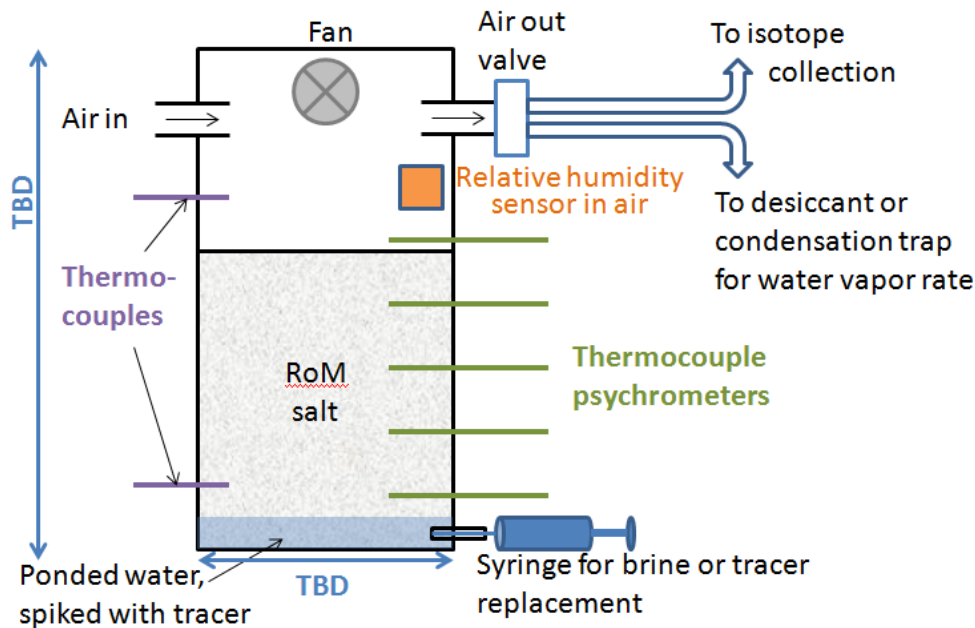

Figure 9. Draft schematic of the evaporation experiment. Thermocouple psychrometers, shown here entering the column at various heights, are an optional instrumentation capability.

The apparatus will consist of a column of dimensions to be determined by prior calculations, but approximately 6 in. by 3 in. in cross-section and approximately 2 ft high. Lateral variation through the column is not desired; this should essentially be a 1-D experiment. The apparatus may be set on a scale for backup weight loss measurements, although wires and tubing connected to the apparatus may prevent highly accurate weight measurements. For that reason, the primary method for determining water loss rate will be through desiccant cartridges or a condensation trap. A layer of ponded water approximately 1 in. high will be spiked with an isotopic tracer (HDO, D_2O, $H_2{}^{18}O$, or HTO).

The flow rate and relative humidity of the air in will be controlled by an air tank with a regulator, flow meter, and desiccant trap to dry the air before entering the apparatus. A fan will gently mix the air in the void space above the RoM salt, with care taken not to force significant air down into the salt pile (the fan will be oriented so airflow is parallel to the salt surface). Air out will be routed by a valve to either to a desiccant (or condensation trap) to measure the quantity of water vapor produced, or to a collection system for measurement of the isotope ratios of the vapor. The gaps in water vapor produced by the system while the air/water vapor mixture is directed to isotope collection will be interpolated from the times that the vapor out is directed to measure quantity of water vapor. To avoid condensation on the outlet lines, heat tape may be used to keep the temperatures slightly hotter than ambient. The same system will be used in the heat pipe experiment, and its efficacy will be tested here.

Initially, air in will be very dry air, to avoid varying isotopic ratios of the inlet air affecting the measured concentrations of isotopic tracers. However, depending on the quantity of spiking of the ponded water, this effect could turn out to be minimal. There may be equipment available at LANL to allow different relative humidities of the air in.

Calculations will be made in advance to determine the ideal depth of ponded water. The first experiments will have no replenishment of the ponded water, but later experiments may involve an inlet at the bottom to keep the ponded layer present, with a syringe to add brine or a new tracer to the system. The case where the ponded water dries out will provide transient information about water loss and dry-out. The case with ponded water replenishment will reach a steady-state water loss and the tracer in the replenished water could change after equilibrium is established. The breakthrough of the second tracer would provide additional information about vapor migration.

The isotope tracer behavior will be modeled to test assumptions about fractionation as a condensable species (e.g., HDO, D_2O) moves through the system. The enrichment of the tracer in water vapor as the system approaches dehydration (as the ponded water dries out) may provide a useful diagnostic for dry-out in a field-scale or intermediate-scale heater test. The concentration of tracers in the ponded water will be determined based on equipment specifications.

One extremely useful continuous measurement would be saturation in the salt pile, ideally at several depths. Within a small column experiment, it is extremely difficult to determine transient saturations, although post-test, destructive forensic sampling can be performed. The techniques discussed in section 4.1.2 (neutron probe, TDR) may be used here if possible in the smaller container. On the other hand, the entire experiment could be performed in the larger heat pipe apparatus if necessary. We also propose using thermocouple psychrometers to measure water vapor pressure in the porous RoM salt, with very small batch experiments performed on salt samples to calibrate the psychrometers to known saturations.

Temperature of the laboratory will be measured, and thermocouples inside the experiment will verify the internal temperatures, near the ponded water and in the air. Thermocouple psychrometers will be inserted into the apparatus at several heights to measure water vapor pressure with position in the RoM pile and air. Furthermore, a relative humidity sensor in the air will provide backup measurements and allow for calibration of the thermocouple psychrometer in air.

4.2.2.2   Diffusion experiment

A draft layout of the proposed diffusion experiment is shown in Figure 10. As the experiment is underway, minor changes to the layout may be required based on equipment availability and cost, pre-experiment modeling results, and lessons learned about the proposed techniques.
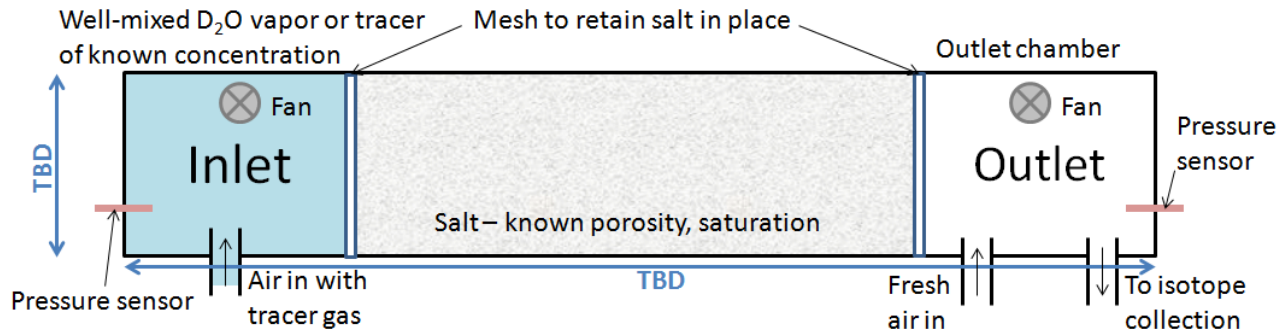

Figure 10. Draft schematic of the diffusion experiment.

Temperature will be measured in the laboratory immediately surrounding the experiment. If replicates are desired at other temperatures to test temperature dependencies of the diffusion coefficient, a heat lamp maybe employed and if temperature gradients are established, multiple thermocouples will be used alongside the apparatus to measure differences from end to end.

The key difficulty with the diffusion experiment is avoiding any advective air flux that would override the diffusive process. Air in and air out within the outlet chamber will be at very low rates and equally matched to avoid producing a pressure gradient across the system. High-resolution pressure sensors or differential pressure sensors will be placed in the inlet and outlet gas chambers to measure the pressure differential. If one is generated, the flux induced by pressure gradients may be removable from the results by modeling of the advection-diffusion equation. Examples exist in the literature of similar experiments on soils with water vapor (e.g., Jin and Jury, 1996).

For this experiment it would be particularly valuable to include a noncondensible tracer along with the heavier water isotopes. For example, SF_6 is a relatively conservative gaseous tracer (low concentrations in water by Henry's law partitioning) that is frequently used and detectable at low concentrations. The breakthrough of the noncondensible tracer will provide primary information about the tortuosity and effective diffusion coefficient of the RoM salt to help separate out effects that are specific to condensable tracers such as enhanced vapor diffusion (Webb and Ho, 1998).

The air inlet will have a known concentration of vapor tracer. The procedure for fixing isotopic concentrations in the vapor in is to be determined. It may be advantageous to try a pulse concentration change instead of a step concentration change, to avoid having to maintain a constant concentration of isotopic tracer in the air inlet chamber. In that case, the air inlet would begin with fresh air, switch to the vapor tracer, and quickly return to fresh air. However, it may be the case that change in concentration of the vapor tracer is insignificant.

The air outlet chamber would be well-mixed with an air turnover rate sufficient that the system has a zero-concentration boundary condition (in contrast to a fixed-flux boundary condition).

4.2.3   Equipment Needed

Much of the instrumentation needed for these experiments will overlap with heat pipe experiment (e.g., inlet and outlet air equipment, thermocouples, thermocouple psychrometers, relative humidity sensor, etc). Only additional materials required for these experiments, some of which may be optional (marked with an asterisk), are listed below. If the materials are necessary for only one of the two experiments, the number is indicated in parentheses.

Additional equipment beyond the heat pipe experiment (section 4.1.3):

2 tanks (acrylic columns), size TBD
2 fans
High-resolution differential pressure sensors (experiment #2 only)
*SF_6 and SF_6 sensor (experiment #2 only)

4.2.4   Test procedures

4.2.4.1   Evaporation experiment

1) Sieve the salt to a maximum diameter, to be determined.
2) Pre-test analysis will include post-sieving grain size distribution, bulk density, grain density, porosity, and initial saturation of the RoM salt. Saturation will be determined by oven-drying and weighing.
3) Conduct pre-test modeling to predict time of dry-out for a given depth of ponded water and air flow rate, plus likely isotopic tracer breakthrough times.
4) Construct experiment apparatus, including: tank, air in/air out tubing and connections.
5) Begin adding RoM salt and ponded water with isotopic tracers.
6) Meanwhile, instrument the apparatus with thermocouples and thermocouple psychrometers as RoM salt is added. Measure locations of all instruments accurately.
7) Hook up the air inlet and outlet equipment. Prepare for vapor collection.
8) Begin dry air flow in at a specified rate. Collect data:
    a. Air out – sample for isotopes, water vapor content
    b. Continuous temperature, relative humidity data
9) Allow system to run until ponded water has dried out and water vapor in air out equals water vapor in air in (i.e., extremely dry).
10) Replicate experiments with different conditions of ponded water (e.g., replenishment including different tracers), different relative humidity of air in, different ambient temperatures (e.g., using an external heat lamp).

4.2.4.2   Diffusion experiment

1) Sieve the salt to a maximum diameter, to be determined.
2) Pre-test analysis will include post-sieving grain size distribution, bulk density, grain density, porosity, and initial saturation of the RoM salt. Saturation will be determined by oven-drying and weighing.

3) Conduct pre-test modeling to predict time of isotopic tracer breakthrough. Use this to determine the length scale of experiment.
4) Construct experiment apparatus, including: tank, air in/air out tubing and connections, mesh partitions to hold up the salt.
5) Add RoM salt between mesh partitions.
6) Instrument the apparatus with pressure sensors and external thermocouples.
7) Hook up the air inlet and outlet equipment. Prepare for vapor collection.
8) Add gaseous isotopic tracer of known concentration to the inlet chamber. Collect data:
    a. Air out – sample for isotopes, water vapor content
    b. External temperature
9) Allow system to run until equilibrium breakthrough is observed.
10) Replicate experiments with different ambient temperatures (e.g., using an external heat lamp), saturation of the RoM salt, and other tracers (particularly a noncondensible tracer).

4.2.5   Analysis

4.2.5.1   Evaporation experiment

We will study the transient rate of water loss as compared to models of the same, and analyze any differences. Is loss from the pile limited by water availability to the top of the pile (suction, retention characteristics) or energy? Different temperature settings will be required to determine this. Water loss rate as a function of spatially varying saturation in the pile may be converted to an empirical formulation for use in the FEHM model.

We will study the effect of dry-out of the ponded water on isotopic enrichment. Additionally, isotopic tracers measured as fraction of total water vapor, with time, will yield information about fractionation from diffusion and evaporation across boundary. This breakthrough curve will be modeled and the results will provide valuable information about isotope fractionation from various processes in the system.

4.2.5.2   Diffusion experiment

We will fit an effective, measured D_{va} to modeled breakthrough curves (analytical and numerical). We will compare effective D_{va} to D^0_{va} (free air diffusion coefficient) for same tracer. These will be used to determine effective tortuosity (Eq. 1 and 2) as a function of measured properties. We will use replicate experiments to isolate factors (particularly breakthrough with a noncondensible tracer), and to test emergent hypotheses.

4.3   Description of Proposed Intermediate-Scale Experiments

The need for intermediate-scale experiments is well discussed in prior sections. These, for all environmentally-relevant studies, are needed to experimentally integrate and address the coupling of subsurface processes. For the specific case of generic RoM salt experiments, where heat flow and water movement issues predominate, a number of physical and geochemical processes will contribute to the observed heat flow and these are highly coupled in a way than can only be properly simulated in the laboratory by performing intermediate scale experiments. It is the key goal of this task to perform intermediate-scale (defined as meter-scale) experiments

in a way that can challenge and test modeling predictions. These data will be used to validate model predictions and establish that the right processes are identified in the models. This work will also meet the objective of providing feedback on the utility of equipment used for monitoring key parameters. Some development work will be identified that will support the real-system in-mine studies that are eventually planned.

The key elements of this task are:

1. Put in place the needed infrastructure to perform intermediate-scale experiments. This includes a laboratory facility designed to support this work, but also the needed instrumentation and experimental protocols within the current QA structure that are needed.
2. Conduct a series of intermediate-scale experiments that will be technically organized to address multiple issues. This is expected to be a multi-investigator effort and will directly challenge the ability of current modeling approaches to predict heat and water transport under conditions that approach the actual/expected in-mine demonstration.

4.3.1   Relevance of earlier work

There is much in the literature about the key and important role that scaled-up experiments can provide. This is a necessary step in the development of models and is essential to confirm that we have a sufficient understanding of the important subsurface processes and how they influence and interact with each other (e.g., extent and nature of their coupling). This has already been reviewed in prior sections of this report.

Although some in-mine experiments and laboratory studies were already performed (see discussions in Section 4.1.1 and 4.2.1), there has not been a significant study performed at the intermediate scale (approximately 1 m^3) – so this is a key and new contribution to this overall scientific activity and is needed to transition from the smaller bench-top experiments to the field application. The advantage of intermediate-scale experiments is that even though they will likely have and include a significant degree of process coupling, we still have many ways to control the conditions of the experiments in a way that can more easily challenge the model and accelerate (i.e. condense) the time needed. In this way, these tests will function as a final check on the predictive capability of the modeling and provide needed/critical data in a more timely fashion.

4.3.2   Preparation/Setup

Preparation for the intermediate-scale experiment may require additional benchtop experiments to ensure that the intermediate-scale experiment goes smoothly. In particular, further instrument durability tests will be performed in a benchtop apparatus similar to those discussed above (sections 4.1 and 4.2) prior to beginning planning and procurement for the intermediate-scale test (Figure 11). Equipment will be recycled from previous experiments whenever possible.
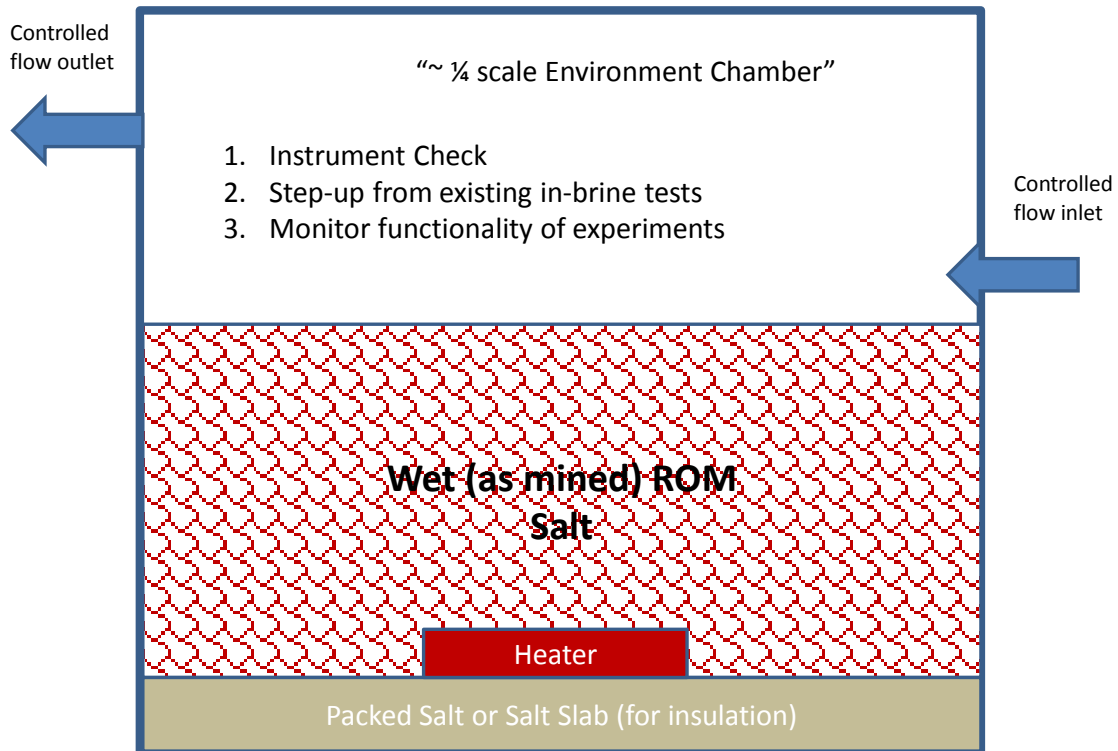
Figure 11. Schematic of planned benchtop scale experiments to develop protocols and to develop/finalize intermediate-scale experiments.

Detailed plans and test procedures for the intermediate-scale (m^3) tests will be developed during and after the benchtop experiments. They will apply lessons learned from these experiments to address model scale-up and the associated greater complexity in the actual coupling of the processes as well as the design/configuration of the monitoring and experimental protocols.

The overall design for these experiments is given in Figure 12. These will be run as multi-investigator experiments with themes that range along the following:

- Various heat loadings, salt mesh size (including solid slabs if available) and water content
- Various types of monitoring equipment
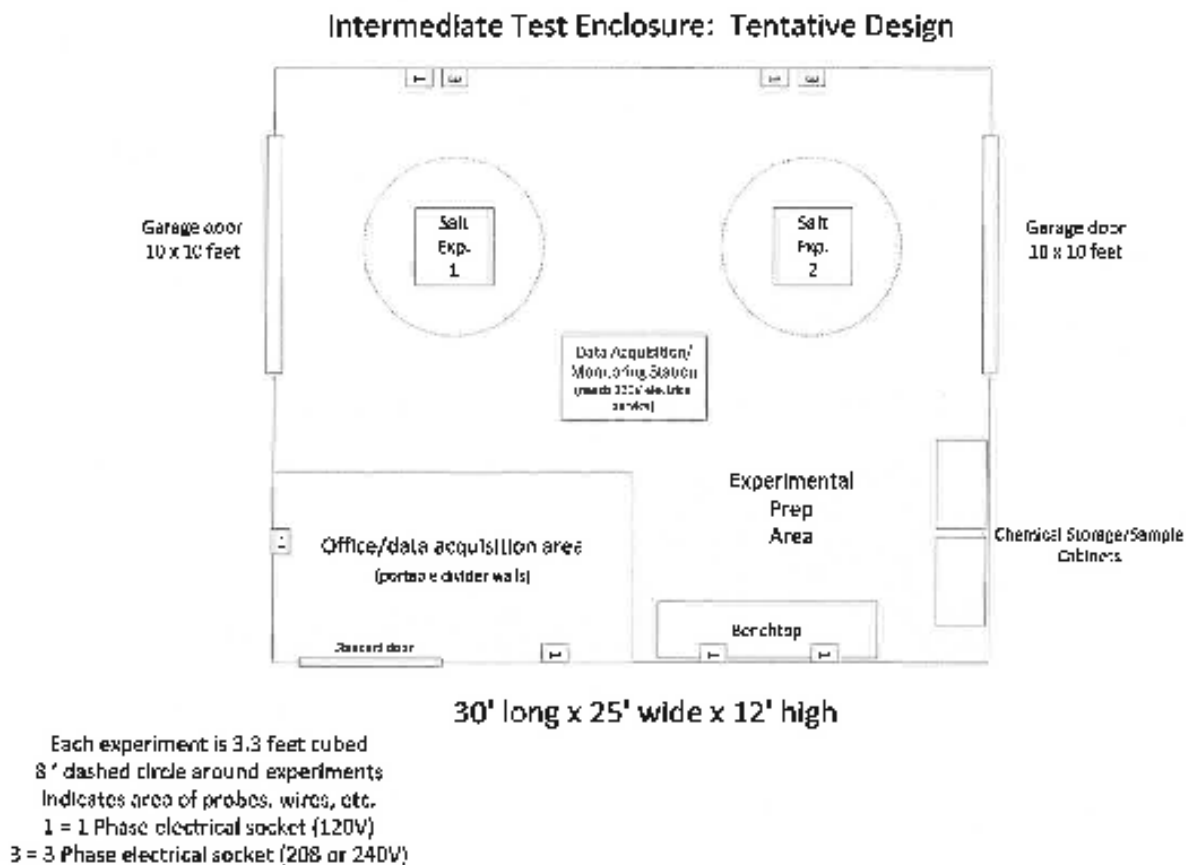- Various inflow and ventilation conditions

Figure 12. Schematic of intermediate-scale test configuration.

5.   Sample Control/Quality Assurance/Data integrity

A quality assurance plan will be developed prior to experiment initiation. Procedures for record-keeping, data validation, and sample control will be developed as the particular instruments are selected. Equipment will be purchased from the LANL-RSO Qualified Supplier List when possible.

6.   Health and Safety Planning

A LANL integrated work document (IWD) will be developed for this project. If neutron probes are used, that will be included in the IWD and involvement of LANL Radiation Protection staff will be required. Additional training for personnel may be necessary.

7.   Concluding Remarks

The bench- and intermediate-scale experiments proposed here will fill crucial gaps in understanding the heated porous RoM salt pile prior to the field-scale experiment. They will provide validation for the ongoing numerical modeling effort, and lessons learned from the small-scale experiments will be applied

during the larger-scale experiments. Finally, the focus on individual processes in the small experiments will aid in understanding the coupling between these processes in more complex scenarios. For the bench-scale experiments, low cost, quick turnaround time and the ability to do repeated tests are key features. The experiments may be modified from the plans described here under constraints of actual funding, and as a result of lessons learned during pre-test modeling/planning.

8.   References

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