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Experimental Test Plan for Grouting H-3 Calcine

A. K. Herbst

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Idaho
Cleanup
Project

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Calcine Disposition Project
Project No. 23582
Idaho Cleanup Project
Idaho Falls, Idaho 83415

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ABSTRACT

Approximately 4400 cubic meters of solid high-level waste called calcine are stored at the Idaho Nuclear Technology and Engineering Center. Under the Idaho Cleanup Project, dual disposal paths are being investigated. The first path includes calcine retrieval, package "as-is", and ship to the Monitored Geological Repository (MGR). The second path involves treatment of the calcine with such methods as vitrification or grouting. This test plan outlines the hot bench scale tests to grout actual calcine and verify that the waste form properties meet the waste acceptance criteria. This is a necessary sequential step in the process of qualifying a new waste form for repository acceptance. The archive H-3 calcine samples at the Contaminated Equipment Maintenance Building attached to New Waste Calcining Facility will be used in these tests at the Remote Analytical Laboratory. The tests are scheduled for the second quarter of fiscal year 2007.

NOMENCLATURE AND ACRONYMS

ALD	Analytical Laboratories Department.
ASTM	American Society for Testing and Materials.
BEA	Battelle Energy Alliance.
Blast Furnace Slag	A finely ground non-metallic waste product developed in the manufacture of pig iron, consisting basically of a mixture of lime, silica, and alumina, the same oxides that make up portland cement, but not in the same proportions or forms.
Calcination	The process of converting a liquid to a solid granular product called calcine.
Cement	Refers to type I/II Portland cement.
CFA	Central Facilities Area.
CPP	Chemical Processing Plant. Former name of INTEC. INTEC facilities still use CPP designations.
CRWMS	Civilian Radioactive Waste Management System.
DIAL-MSU	Diagnostic Instrumentation and Analysis Laboratory at Mississippi State University.
EDF	Engineering Design File.
DOE-ID	Department of Energy Idaho Operations Office.
Fly Ash	A pozzolan of finely divided residue that results from the combustion of ground or powdered coal. Class C fly ash may contain 10% lime, has cementitious properties, and reacts with water to form a solid. Class F fly ash does not use water and aids in grout flow.
Grout	A mixture of portland cement, other powdered additives, waste, and water. It may contain fine-grained sand and does not include large aggregate material. For this study, grouting is the process of solidifying and stabilizing low-level waste in cement-based materials.
HLW	High-Level Waste.
INTEC	Idaho Nuclear Technology and Engineering Center.
Leaching	The process whereby a liquid agent will dissolve hazardous materials within a waste mass and transport these materials through the mass and beyond. The most widely used laboratory leaching test is the TCLP (Toxic Characteristic Leaching Procedure) specified by the EPA in

	several regulations. For many treated and untreated wastes, the results of this test determines whether the EPA considers the material toxic or not.
MGR	Monitored Geological Repository.
NGLW	Newly generated liquid waste -- low-level waste projected to be produced that is not part of the existing tank farm inventory. Sources are the process equipment waste system, decontamination solutions, and filter leach solutions.
NWCF	New Waste Calcining Facility.
PCT	Product Consistency Test.
PEW	Process Equipment Waste.
Portland Cement	The product obtained by pulverizing clinker consisting essentially of hydraulic calcium silicates.
Pozzolan	A siliceous or siliceous and aluminous material that reacts with liquid calcium hydroxide in the cement gel to form compounds possessing cementitious properties.
RAL	Remote Analytical Laboratory.
RCRA	Resource Conservation and Recovery Act.
Solidification	The process of producing from liquid, sludge, or loose solids a more or less monolithic structure having some integrity. Occasionally, solidification may refer to the process that results in a soil-like material rather than a monolithic structure. Solidification does not necessarily reduce leaching of hazardous materials. However, when a waste is solidified, its mass and structure are altered, decreasing migration of solutions within the mass.
Stabilization	Generally refers to a purposeful chemical reaction that is carried out to make waste constituents less leachable. This is accomplished by chemically immobilizing hazardous materials or reducing their solubility by a chemical reaction.
Waste Form	The final product for long-term storage. This includes the solidified/stabilized waste as well as the container. The waste form must pass extensive qualification testing prior to release for storage.
Waste Loading	The mass weight percent of the waste in the total mass of the final waste form.
WAC	Waste Acceptance Criteria.
WGS	Waste Generator Services.

UTS	Universal Treatment Standards.
Vitrification	The process of placing waste material in a glass form. This is a thermal process where the waste material is placed in a melter with glass forming material (chemicals or frit), then heated together, poured into a storage container, and cooled to a solid form.

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EXPERIMENTAL TEST PLAN FOR GROUTING H-3 CALCINE

1. INTRODUCTION

Approximately 4400 cubic meters of solid high-level waste called calcine are stored at the Idaho Nuclear Technology and Engineering Center (INTEC). Under the Idaho Cleanup Project, dual disposal paths are being investigated.[1] The first path includes calcine retrieval, package “as-is”, and ship to the Monitored Geological Repository (MGR). The second path involves treatment of the calcine with such methods as vitrification or grouting. This test plan will demonstrate grouting of the calcine and evaluate the waste form properties.

1.1 Purpose and Scope

The primary purpose of the hot bench scale tests is to grout actual calcine and verify that the waste form properties are consistent with those observed using the cold calcine simulants. This is a necessary sequential step in the process of qualifying a new waste form for repository acceptance. The calcine will be collected from the archive samples at the Contaminated Equipment Maintenance Building attached to New Waste Calcining Facility (NWCF) and transferred to the Remote Analytical Laboratory (RAL) where it will be test grouted. The chemical properties of the calcine will be determined, i.e., chemical composition, radionuclide composition, bulk density, and moisture content. Once the calcine is grouted and allowed to cure, the waste form density, compressive strength, and leach resistance will be determined. The Product Consistency Test (PCT) will be run for comparison to vitrified waste forms. Finally, an encapsulated grout will be heated and the internal pressure and temperature will be monitored to simulate storage conditions. These tests are designed to meet many of the technology needs noted in Engineering Design File, EDF-5107.[2] These tests are planned for the second quarter of fiscal year 2007.

1.2 History

During 2004 and 2005, personnel at the Diagnostic Instrumentation and Analysis Laboratory at Mississippi State University (DIAL-MSU) experimented with various ceramic and cement formulations to grout pilot-plant calcine produced at INTEC.[3] Of the waste form alternatives tested, the grout formulation was the only one that met the performance goals of >500 psi for the cured form and passed the Universal Treatment Standard (UTS) leach limits for cadmium, chromium, mercury, and nickel in the Toxicity Characteristic Leaching Procedure (TCLP).[4] Through several iterations, a formulation of Portland cement, blast furnace slag, and coal fly ash was developed which could be used to grout calcine with up to 34.1 weight percent (wt%) loading and still meet the desired performance specifications. To aid in meeting the UTS leach limits for toxic metals, sodium sulfide was added to prevent leaching of mercury, chromium, and cadmium.

1.3 Test Objectives

The objectives of this test are to 1) characterize the calcine sample, 2) demonstrate that actual, radioactive calcine can be grouted, and 3) evaluate the disposal properties of the waste form, specifically:

1. Calcine characterization:
    - ∉ inorganic analyses
    - ∉ radiochemical analyses
2. Waste form
    - ∉ no free liquid

- compressive strength
- density
- leach resistance (TCLP)
- product consistency (PCT)
- head space gases
- grout integrity up to 400°C

3. Canister corrosion (non-radioactive test)

1.4 Theory/Approach

The basis of this test is to show grout as an alternative method to successfully treat calcine for high-level waste disposal. Therefore, the test should show that the grouted waste form can meet or exceed the waste acceptance criteria currently established for high-level wastes by the Department of Energy Radioactive Waste Management Office. Additionally, EDF-5107 on calcine technology needs is addressed.

The waste acceptance criteria for the Monitored Geologic Repository is based on "Civilian Radioactive Waste Management System Waste Acceptance System (CRWMS) Requirements Document," DOE/RW-0351.[5] Section 4.2.2 of this document states that the CRWMS shall only accept HLW that is not subject to regulation as hazardous waste under the Resource Conservation and Recovery Act (RCRA). This provision requires that the waste form pass the UTS limits for toxic metals using the TCLP. Section 4.8 of this document addresses "Specific Requirements for High-Level Waste." Several of the requirements apply only to the disposal canister; however, those that apply to the waste include:

1. Demonstrate control of waste form production by comparing samples to the Environmental Assessment benchmark glass using the PCT.
    - The PCT will be run on a grout waste form; however, the results may not be satisfactory since the test was designed for a vitrified waste form.
2. Show that canister materials preclude chemical, electrochemical, or other reactions (such as internal corrosion).
    - For the canister corrosion tests, it is recommended that this test be run "cold" using pilot-plant calcine due to the long-term nature of corrosion testing; thus a "cold" test would avoid long-term tracking and maintenance of radioactive samples. To simulate canister placement in the storage drift, the corrosion test should be run at 200°C (EDF-5107, Section 6).[2] It is thought that at this temperature, 6 months should be sufficient time for grout fluids/gases to react with the coupons in order to determine a corrosion rate. It is expected that the canisters will be 316L stainless steel[6] with 304L and Carpenter 22 as alternatives.
3. Filled canister weight is not to exceed 9260 pounds (4200 kg).
    - The density of the grouted calcine can be determined and extrapolated to canister volume to show filled canister weight. For example, the 15 foot spent fuel canister has a volume of 1.07 m^3 at 98% fill and with a grout density of 1800 kg/m^3, the grout weight would be 4242 pounds (1926 kg).
4. Sealed canisters shall contain no residual water beyond that condensing from water vapor inside the canister as it cools.
    - Visual observation of the curing grout in an enclosed container will be made to determine if any free liquid develops.
5. Thermal output shall not exceed 2540 watts per canister.
    - EDF-6258 documented decay heat from direct disposed calcine in various canisters and in no case did any canister fully loaded with calcine exceed the 2540 watts.[7] Since the grout will only be at 35 wt% loading, the decay heat will be even less. Therefore, due to low decay heat values, a thermal analysis test will not be run.

EDF-5107, "Technology Development Needs for the Calcine Disposition Project,"[2] presents several items to be evaluated in a hot bench scale study (Section 5.4) which include:

1. Confirming that the stabilizer containing actual calcine can meet the leach limits of the UTS and the environmental assessment (EA) borosilicate glass, which will be required for repository acceptance.
   - The PCT was noted above for EA glass leach testing. UTS refers to the Universal Treatment Standards which are part of Environmental Protection Agency's (EPA) TCLP as found in 40 CFR 268.48. TCLP can be run on the waste form to check for RCRA toxic metal leaching.
2. Confirming that the leach characteristics via the Materials Characterization Center's MCC-1P test on monolithic waste forms are similar between the cold and hot waste forms.
   - This test is not currently required by the MGR waste acceptance criteria and as such will not be utilized in this plan.
3. Determining if radiolysis causes out-gassing from the hot waste form.
   - It is not recommended that this test be pursued as the production of radiolysis gases takes considerable time and would require a long-term test beyond the scope of this bench test.
4. Determining the extent of physical degradation or phase changes up to 400°C.
   - The grouted calcine waste form could be heated in a laboratory furnace and visually examined for degradation. Further, comparison of the compression test, TCLP, and PCT results between the control and the heated samples will show whether or not the heating causes degradation of the waste form. The waste form should maintain a compressive strength of at least 500 psi as was required in the DIAL tests.[3]
5. Determining the composition and equilibrium pressure of gases in a sealed container up to 400°C.
   - Heating a grouted calcine sample in an enclosed cylinder creates excessive internal pressures for normal bench scale laboratory experimentation (3200 psi per steam table pressures [8,9]). In order to collect head space gases, the test cylinder, while in the furnace, will have a vapor tube connected to an external condenser and a liquid collection bottle. The collected liquid will be submitted for analysis to determine the components of the head space gas.
6. Designing tests for long-term stability observations of the hot waste form over a several year period.
   - Such long-term testing is desired, but laboratory hot cell space is not available to maintain highly radioactive samples for extended periods of time. However, the 5 cm diameter by 10 cm high test canister with calcine grout will be returned to Contaminated Equipment Maintenance Building (CPP-1659) for storage with the archived calcine.
7. Designing tests for long-term studies of corrosion on the canister material over a several year period due to the hot waste form.
   - As noted above, it is recommended that this test be done with non-radioactive samples.

2. EXPERIMENTAL CRITERIA AND PREPARATIONS

The radioactive portions of this test will be carried out by the Analytical Laboratories Department (ALD). The grouting of the radioactive calcine will be completed in the Remote Analytical Laboratory (RAL). Solutions requiring analyses will be transferred to the Laboratory/Offices Building (CPP-602). The "cold" canister corrosion test will be subcontracted to Battelle Energy Alliance (BEA) for completion at the corrosion laboratories in CF-612.

2.1 Analytical Procedures

All ALD work will be done according to ALD test procedures. As needed, new ALD procedures will be prepared and approved for use prior to starting these tests.

2.2 Quality Assurance/Quality Control Designation

This work will be Quality Level 3, such that procedures, operations, stock chemicals, sample tracking, calculations, and results are well documented and submitted for project records. Refer to Form 414.A06, "Quality Level and Requirements Determination," located at the end of this document.

The Calcine Disposition Project will appoint a Principal Investigator to oversee the treatability study in RAL. The PI will review and follow analytical procedures, maintain a daily logbook of work details, sample and data collection, data sheets, direct photography, ensure proper equipment setup, etc. The PI in conjunction with the RAL lead scientist will maintain and submit required treatability study forms, sample tracking forms, and waste disposition forms. Overall, the PI will ensure that the quality level is met and maintained.

The BEA lead scientist for the corrosion laboratory will serve as the PI for the corrosion study (Section 3.7). The BEA lead scientist will ensure that the quality level is met and maintained for the corrosion study.

2.3 Calcine Sampling

The calcine sample must be obtained in the Contaminated Equipment Maintenance Building (CPP-1659) attached to the NWCF and transferred to RAL (CPP-684). H-3 calcine will be used for these tests. Three types of calcine (Al, Zr, and H-3) are archived in CPP-1659. The Al and Zr calcine were stored in plastic sample bottles and over the years the bottles have degraded allowing the calcine to contaminate the inside of the storage cask. The H-3 calcine is in stainless steel sample bottles and is in a separate cask from the Al and Zr calcine; therefore, the H-3 calcine is acceptable for sampling. The H-3 calcine was obtained in 1993 during aluminum, sodium, and Fluorinel waste processing. Thus, the H-3 calcine provides a good blend of Al and Zr for these grout tests. A sample plan and associated procedures will be by separate documentation.

2.4 Calibrations

ALD calibration standards are to be followed and implemented. For new tests, such as the compression test, the American Society for Testing and Materials (ASTM) standards will be implemented into ALD procedures.

2.5 Waste Management

All waste management is to be done in accordance with ALD procedures and with Waste Generator Services (WGS) concurrence and approval. Liquid wastes such as rinses, solutions for analyses, or TCLP extracts are to be disposed as Process Equipment Waste (PEW). Solids are to be collected and disposed of as incidental radioactive waste.

2.6 Equipment

The following equipment shall be collected, purchased, or manufactured ahead of time for this test plan:

- Mixer
- Mixing bowl, spoons, spatulas, and wooden tongue depressor stick
- Grout molds (see 2.6.1)
- Silicon vacuum sealant compound
- Sample bottles
- Plastic closure bags (Zip-lock or slide closure type)
- Test canister (see 2.6.2)
- Arbor press (see 2.6.3)
- Compression tester (see 2.6.4)
- Laboratory balance
- Condenser with tubing and fittings
- Fusion crucibles and flux material
- TCLP extraction equipment
- TCLP extraction fluids
- PCT vials
- Hot plate
- 3 L beaker
- Watch glass (heat tempered for 400°C)
- Lab furnace (400°C needed)
- Corrosion coupons with weld seam (include material test reports)
- Bottles/jars, heat resistant with air-tight lids for corrosion tests
- Personnel protective equipment (PPE) specific to this test
- Waste disposal / shipping packages
- Digital camera

Note that all of the above equipment may be sacrificed due to radioactive contamination and as such will need to be disposed of properly as directed by ALD procedures and WGS.

2.6.1 Grout Molds

For the compression test, TCLP, and PCT, two molds containing 3 each 2 cm diameter by 3 cm high cylindrical samples will be prepared. These molds shall be made in accordance with Figure 1. This will give a total of 6 cylinders for testing (3 for compression test, 3 for a 400°C test, and fragments of all for the TCLP and PCT). To prepare the molds, the bottom plate is to be covered with silicon vacuum compound and pressure fitted to the bottom of the mold. The top lid is simply placed on the mold following placement of the waste grout. (NOTE: A method to affix the bottom plate to the mold to allow remote removal of the bottom plate will need to be determined.)

2.6.2 Test Canister

A stainless steel test canister shall be manufactured in accordance with Figure 2. The canister should be about 5 cm in diameter by 10 cm in height. It is planned to fill this canister about three-fourths full to leave head space. The lid will have a port to allow any vapor to be collected as a liquid via a condenser. Additionally, a Teflon o-ring shall be in place between the canister top and lid to prevent any gas leakage in the furnace.

2.6.3 Arbor Press

The arbor press is to be used to push the grout samples out of the molds. A manual arbor press is available from McMaster-Carr, part # 244A61 at www.mcmaster.com. The arbor press has a 3/4" square ram to which a 1.8 cm diameter by 3 cm long rod will need to be welded to permit the ram to push through the holes in the molds and extract the grout samples.

2.6.4 Compression Test Device

The compressive strength tester will need to be capable of remote operation to physically crush the grout cylinders to destruction and determine the break force. The device must be small enough to fit through the hot cell loading tunnel which is 11 inches wide by 22 inches tall. Compression testers are available from ELE International or Carver Laboratory Equipment. The exact model will need to be determined, such that it will fit through the tunnel and can be operated by the manipulators. Refer to ASTM C39 for compressive strength test requirements for cylinders.[10]

3. TEST PROCEDURE

3.1 Experiment Setup

Pre-stage the mixer, compression tester, lab furnace, molds, and condenser in the hot cell. Pre-blend the measured amounts (refer to Table 1) of Portland cement, blast furnace slag, and fly ash outside of the hot cell in a separate container. Place the blended cements, water, and sodium sulfide in the hot cell. Use Data Sheet #3 to record digital photograph descriptions.

3.2 Calcine Transfer and Characterization

Complete the transfer of the archival calcine from CPP-1659 and place in the RAL hot cell. Photograph the calcine. Weigh out the 5 samples of calcine: 1) 2.0 g calcine characterization via fusion (run in triplicate), 2) < 0.1 g for gamma/beta scan, and 3) 133.7 g for calcine grouting.

Submit the < 0.1 g sample to radiochemistry for gamma/beta scan.

Pre-weigh the 2 g calcine sample and heat the sample at 400°C until a stable mass is achieved to indicate the moisture has been driven off. Record initial and final mass and the percent of moisture driven off. Maintain sample is a dry environment for fusion tests.

From the 2 g sample use aliquots as needed to complete the fusion dissolution for both cation and anion elements/compounds (run each in triplicate) and submit the solutions for analyses.

3.3 Calcine Grout Mixing

One batch of calcine grout shall be prepared as noted in Table 1 below. Use Data Sheet #1 to record actual amounts. The waste form ingredients shall be mixed in the following order:

1. Add the water to the mixing bowl
2. Dissolve the sodium sulfide in the water
3. Add the calcine to the water and stir for 3 to 5 minutes
4. Add the cement mixture to the calcine and water and mix for 10 to 15 minutes

Table 1. Calcine Grout Waste Formulation. *

Component	Amount (grams)	Weight Percent
Water	120.3	30.7
Sodium Sulfide	3.9	1.0
Calcine	133.7	34.1
Portland Cement	67.0	17.1
Blast Furnace Slag	33.5	8.55
Coal Fly Ash	33.5	8.55

* Based on a waste loading of 34.1 wt% and a density of 1.8 g/cm3. This formulation allows for 25 g residual grout that may remain in the mixing bowl and on the tools.

Following mixing, photograph the mix and then divide the batch into the various molds as noted in Table 2. Use the wooden tongue depressor stick to smooth/level off the top of each mold. Photograph the prepared molds.

Table 2. Calcine Grout Samples

Samples	Waste Form Mass	Use
Control Set – Mold #1	50.9 g	Compression test, TCLP, and PCT
Thermal Set – Mold #2	50.9 g	400°C test with compression test, TCLP, and PCT
Canister	265.1 g	400°C test, head space gases, and visual integrity

Place each of the cylinder molds in plastic closure bags and allow the grout to cure for 28 days prior to further testing. Curing is to be at ambient conditions with normal room temperature and no humidity requirements.

3.4 Control Tests – Mold #1

After the 28 day cure time, the three small cylinders may be removed from the Mold #1. Remove the top and bottom covers from the mold and use the Arbor press extract the cylinders from the mold. Note that the cylinders are made with a small taper such that the top diameter is slightly larger than the bottom diameter. Thus, place the mold in the Arbor press with the bottom up and press the cylinders out the top which is facing down. Weigh each cylinder and place each in a separate plastic bag. Arbitrarily label the small cylinders as S1-1, S1-2, and S1-3.

Weigh and record the final mass on Data Sheet #1. Photograph the cylinders. Complete a compression test on each cylinder and record the compression force on Data Sheet #1.

Place about one-half of each cylinder in a sample bottle and submit for TCLP analysis. Place the remaining one-half of each cylinder in a sample bottle and submit for PCT analysis. Thus, there will be 3 samples for TCLP and 3 samples for PCT. Record the sample bottle tare and total mass on Data Sheet #1.

Collect any solid residues in a common waste bag for later disposal.

3.5 Thermal Tests – Mold #2

After the 28 day cure time, the three small cylinders may be removed from the Mold #2. Remove the top and bottom covers from the mold and use the Arbor press extract the cylinders from the mold. Note that the cylinders are made with a small taper such that the top diameter is slightly larger than the bottom diameter. Thus, place the mold in the Arbor press with the bottom up and press the cylinders out the top which is facing down. Weigh each cylinder and place each in a separate plastic bag. Arbitrarily label the small cylinders as S2-1, S2-2, and S2-3.

Weigh each cylinder and record on Data Sheet #1. Using a black marker put a 1, 2, or 3 on each cylinder to match the S2-1, S2-2, and S2-3, respectively. Photograph the 3 cylinders. Place the 3 cylinders on a heat treated watch glass and place in the furnace at 400°C. Allow the samples to heat at temperature for 7 days.

Following the 7 day "bake" period, visually examine each cylinder for physical changes or waste form degradation. Photograph the post-test condition of the cylinders. Complete a compression test on each cylinder and record the compression force on Data Sheet #1.

Place about one-half of each cylinder in a sample bottle and submit for TCLP analysis. Place the remaining on-half of each cylinder in a sample bottle and submit for PCT analysis. Thus, there will be 3 samples for TCLP and 3 samples for PCT. Record the sample bottle tare and total mass on Data Sheet #1.

Collect any solid residues in a common waste bag for later disposal.

3.6 Canister Sample Tests

The test cylinder should be about three-fourths full allowing space for any gases to develop or permeate from the waste form. Photograph the internal condition of the cylinder with the grout. Weigh and record the total mass on Data Sheet #2. Weigh and record the mass of the liquid collector bottle. Connect the test cylinder vapor outlet to the condenser and to the sample collection bottle (see Figure 3). The cylinder is then placed in a furnace set at 400°C, record start date and time. (NOTE: This test can run concurrently with the above 400°C test if there is sufficient space in the furnace.) The cylinder is to remain in the furnace at temperature for a total of 14 days. Check the furnace and condenser daily to verify proper operation.

Following the 14 days, the cylinder is taken from the furnace, record end date and time. Weigh and record the post-test total mass of the filled cylinder and the liquid collection bottle. Remove the lid and photograph the internal condition of the cylinder with the grout. Submit the collected liquid for mass spectroscopy analysis and for beta/gamma scan.

Collect any solid residues and place in the common waste bag for later disposal. Retain the test cylinder with grout for return to the H-3 calcine archive in CPP-1659 along with any unused calcine.

3.7 Corrosion Testing (non-radioactive)

Clean, dry, and weigh 3 - Carpenter 22 corrosion coupons, 3 - 304L stainless steel corrosion coupons, and 3 - 316L corrosion coupons. Record the pre-test coupon specifications on Data Sheet #4. Photograph the initial condition of the coupons. Procure 2 heat resistant, wide-mouthed jars with air-tight lids that can be sealed to maintain internal steam pressures inside the jars at 200°C. Jars should have about 300 ml volume and will be filled to about 200 ml.

The pilot plant calcine will be provided by the Calcine Disposition Project. It is planned that this calcine will be the same as used by DIAL-MSU for their grout studies. The calcine is a blend of 30% Run 17, 10% RSH-1, 10% Run20, and 50% Run 74 pilot plant calcines.[11]

Using the non-radioactive pilot plant calcine, mix a batch of grouted calcine as noted in Table 3. Place the grout in 3 jars. In one jars, place 3 Carpenter 22 corrosion coupons such that half of each coupon is in the grout and half is in the air space above the grout. In the second jar, place 3 304L stainless steel corrosion coupons in the same method as the carbon steel coupons. In the third jar, place 3 316L stainless steel corrosion coupons in the same method as the carbon steel coupons. Photograph the pre-test conditions inside each jar. Seal the lids on each jar and allow the grout to cure for 24 hours.

Table 3. Simulated Calcine Grout Formulation for Corrosion Tests

Component	Amount (grams)	Weight Percent
Water	245.6	30.7
Sodium Sulfide	8.0	1.0
Pilot Plant Calcine	272.8	34.1
Portland Cement	136.8	17.1
Blast Furnace Slag	68.4	8.55
Coal Fly Ash	68.4	8.55

Place the grout jars with coupons in a laboratory furnace heated to 200°C. “Bake” the jars for a total of 6 months maintaining 200°C. Following the test period, open the jars and photograph the corrosion coupons in-place. Remove each coupon, breaking the sample if needed. Clean and weigh each coupon. Record the post-weights on Data Sheet #4. Photograph the post-condition of the coupons. Calculate and record the corrosion rate for each coupon.

Place each corrosion coupon set in a closable plastic bag and forward to the PI. Discard the grout samples in accordance with approved WGS plans.

4. DATA COLLECTION AND REPORTING

The Principal Investigator (PI) shall witness the treatability testing at RAL and record daily observations and progress in a logbook. All data sheets and analytical results are to be approved by the RAL lead scientist, checked by QA, and sent to the PI. Electronic copies of the digital photographs and Data Sheet #3 are to be forwarded to the PI. Additionally, electronic copies of the analytical procedures used in these tests are to be sent to the (PI).

The corrosion test laboratory will likewise utilize a logbook to document daily observations and measurements. The corrosion test laboratory will also submit the corrosion coupons, data sheets, test results, photographs, coupon material test reports, and a copy of the logbook pages to the PI.

The PI will collect all of the above information and submit a final data package to the Calcine Disposition Project file. The PI will prepare and issue report documenting the results of the calcine grouting and corrosion testing.

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DATA SHEET #1 – CALCINE GROUT MIXING
(Refer to Test Plan Sections 3.3, 3.4 and 3.5)

Mix Date: ____________________

Mix Start Time: __________________ Mix Completion Time: ____________________

Component	Tare (g)	Total Mass (g)	Net Mass (g)	Residual Tare (g)
Mixing Bowl & Tools				
Water				
Sodium Sulfide				
Calcine				
Blended Cements				
Mold #1 (set S1)				
Mold #2 (set S2)				
Canister				

Sample	Cured Mass (g) at 28 Days	Post 400°C Test Mass (g)	Compression Force (lbf)
S1-1		n/a	
S1-2		n/a	
S1-3		n/a	
S2-1			
S2-2			
S2-3			

TCLP Samples	Sample Bottle Tare (g)	Filled Bottle Mass (g)	Net Mass (g)
S1-1-T			
S1-2-T			
S1-3-T			
S2-1-T			
S2-2-T			
S2-3-T			

PCT Samples	Sample Bottle Tare (g)	Filled Bottle Mass (g)	Net Mass (g)
S1-1-P			
S1-2-P			
S1-3-P			
S2-1-P			
S2-2-P			
S2-3-P			

______________________________ ______________________________

Experimenter – Signature/Date PI Review – Signature/Date

DATA SHEET #2 – Canister Sample 400°C Test and Head Space Gas Test
(Refer to Test Plan Sections 3.3 and 3.6)

Start Date: ___________________ Start Time: __________________

Canister and grout total mass at start: _____________ g

Liquid collection bottle initial mass: _______________ g

Heat cylinder at 400°C for 14 days.

End Date: ____________________ End Time: __________________

Canister and grout total mass at end: ______________ g

Liquid collection bottle final mass: ________________ g

___________________________________ ___________________________________

Experimenter – Signature/Date PI Review – Signature/Date

DATA SHEET #3 – Digital Photograph Log

Photo	File Name/#	Date	Time	Description
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				
11				
12				
13				
14				
15				
16				
17				
18				
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21				
22				
23				
24				
25				
26				
27				
28				
29				
30				
31				
32				
33				
34				
35				
36				
37				
38				
39				
40				

Experimenter – Signature/Date

PI Review – Signature/Date

DATA SHEET #4 – Corrosion Tests
(Refer to Test Plan Section 3.7)

Start Date: ________________________ Time: ____________

Completion Date: __________________________ Time: _________

Coupon Type	ASTM Type	Serial #	Surface Area (cm^2)	Initial Mass (g)	Final Mass (g)
Carpenter 22 #1					
Carpenter 22 #2					
Carpenter 22 #3					
304L Stainless #1					
304L Stainless #2					
304L Stainless #3					
316L Stainless #1					
316L Stainless #2					
316L Stainless #3					

Results:

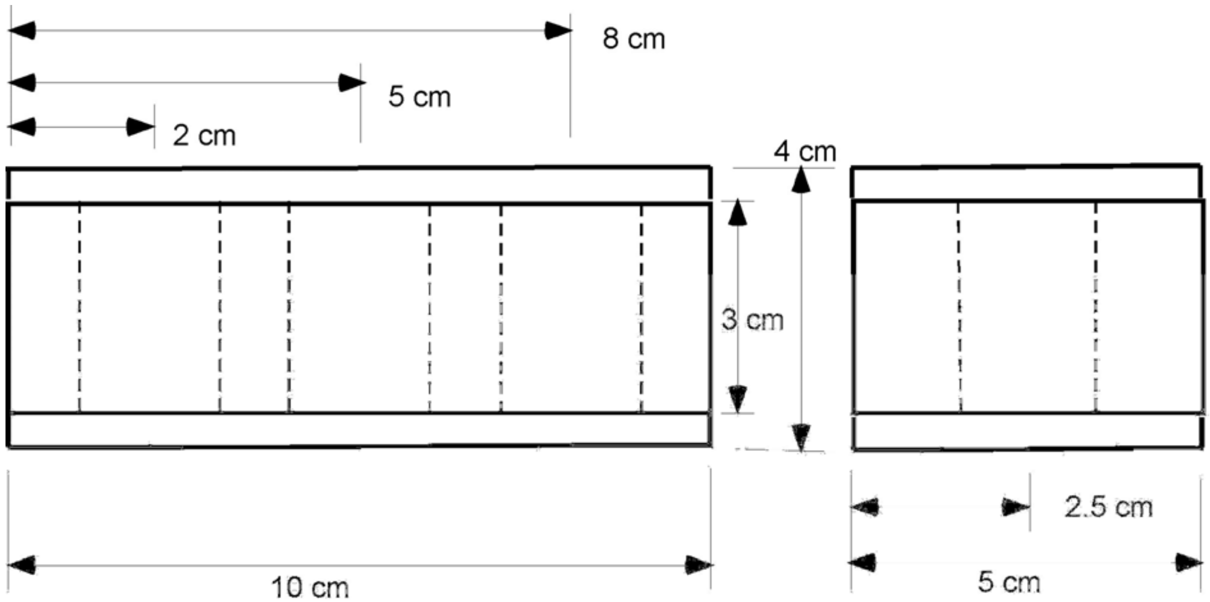
Coupon Type	Exposure Time (hours)	Corrosion Rate (mpy)	Average (mpy)	Standard Deviation (mpy)
Carpenter 22 #1				
Carpenter 22 #2				
Carpenter 22 #3				
304L Stainless #1				
304L Stainless #2				
304L Stainless #3				
316L Stainless #1				
316L Stainless #2				
316L Stainless #3				

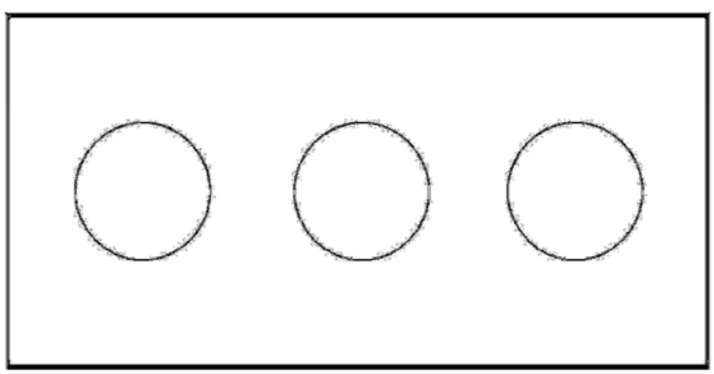
mpy = mils per year

_________________________________ _________________________________

Experimenter – Signature/Date PI Review – Signature/Date

FIGURE 1 – Mold Fabrication Sketch





NOTES:

1. Mold block to be made out of teflon, 10 X 5 X 3 cm.
2. Base and top plates to be made of plexiglass, 10 X 5 X 0.5 cm.
3. Holes are to be 2 cm diameter, nominal. The holes should taper slightly such that the top is ~1-2 mm larger than the bottom diameter.

FIGURE 2 – Test Canister Fabrication Sketch

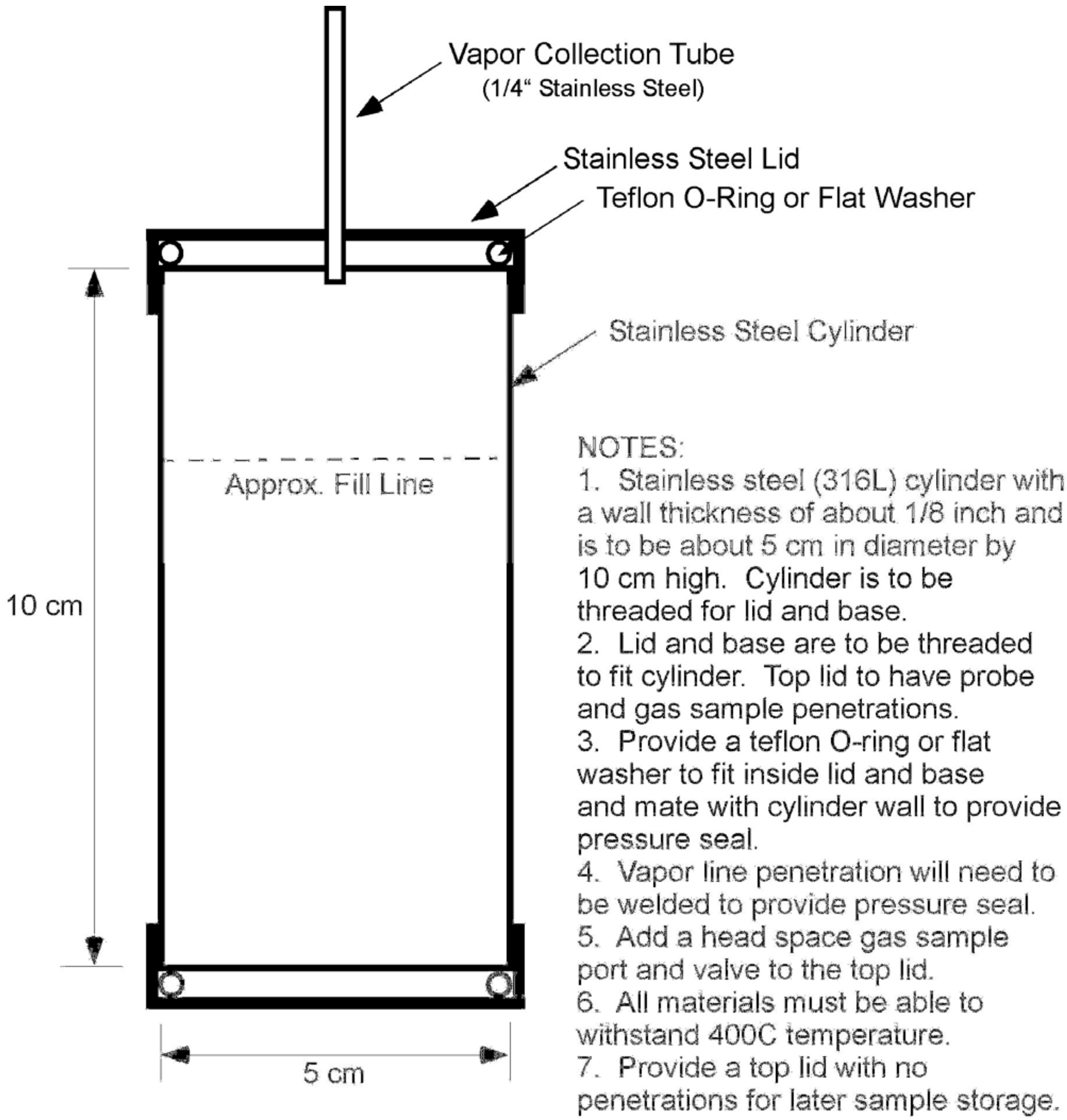
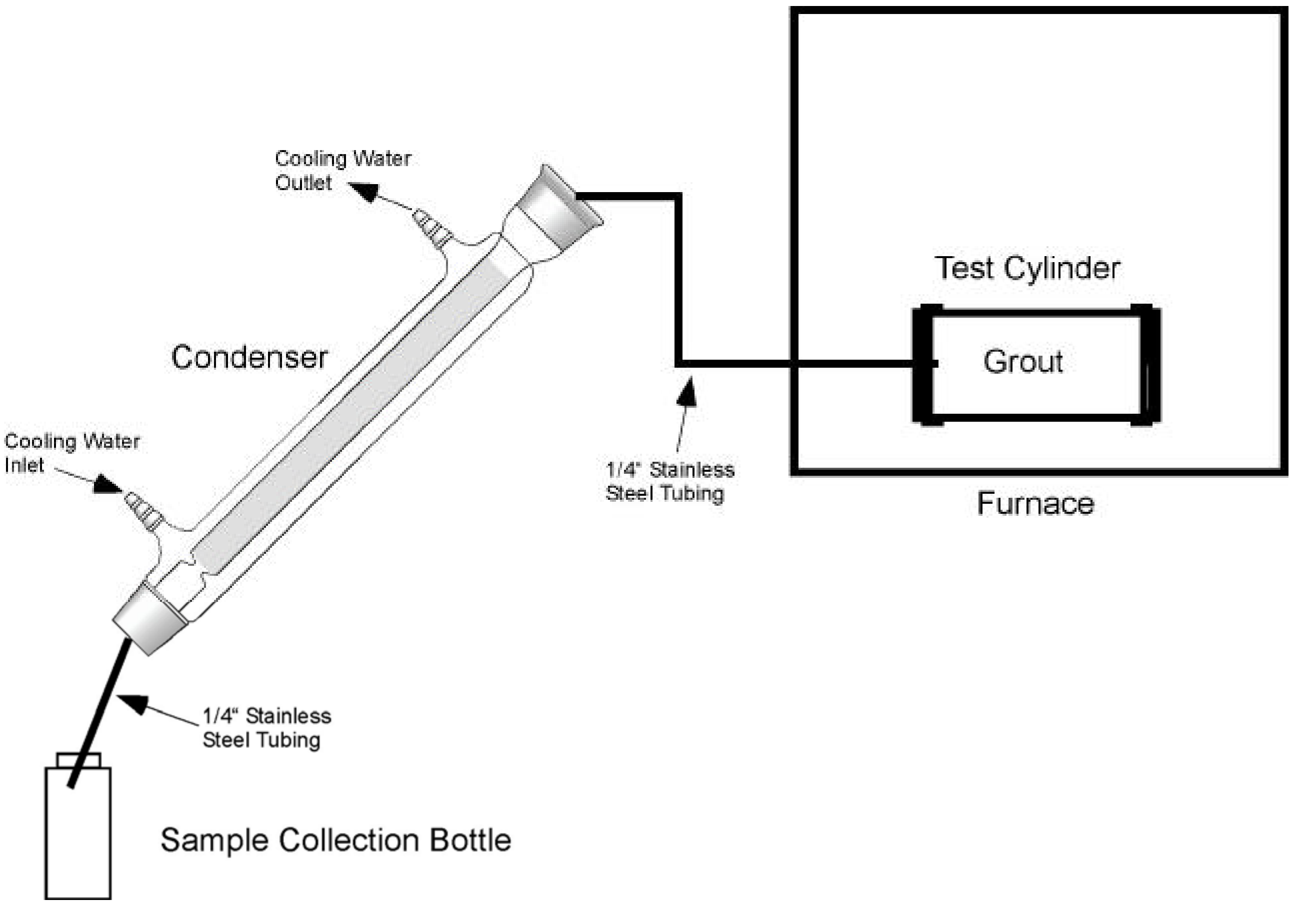


FIGURE 3 – 400°C Test Equipment Setup



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	Date Prepared:	Dec. 20, 2005

	Revision No.: 0
Activity or Work Package No(s).:	**Activity or Work Package Title(s):** Hot Bench Test for Grouting Calcine

For R&D work with multiple components, which have different quality levels assigned, attach a list of items by name with their assigned quality levels to this form.

If the work scope is complex and cannot be evaluated on a single form, or if there are activities that multiple quality levels, then a separate form shall be completed for each activity.

Review and Approvals for Quality Level Assignment and Quality Requirement selection:

Quality Level Assigned: 3 Date: 12/20/05

Print/Type Name	Signature	Date
A. K. Herbst Quality Level Selection Made By Print/Type Name	AK Herbst Quality Level Selection Made By Signature	1/27/06 Date
A. K. Herbst Principal Investigator, Approval Print/Type Name	AK Herbst Principal Investigator, Approval Signature	1/27/06 Date
M. W. Patterson Manager Approval Print/Type Name	MW Patterson Manager Approval Signature	1/27/06 Date
A. S. Salcido Quality Engineer (If Reviewed) Print/Type Name	AK Herbst for A.S. Salcido Quality Engineer (If Reviewed) per telecon Signature	1/30/06 Date

CONVERSION TABLE

This table shall be used to convert a safety category to an equivalent quality level.

QUALITY LEVEL	SAFETY CATEGORY
1	Safety Class - **SC**
2	Safety Significant - **SS**
3	Low Safety Consequence - **LSC**
4	Consumer Grade - **CG**

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Quality Level	Quality Level 4	Quality Level 3	Quality Level 2	Quality Level 1
Quality Requirements To Be Applied	☒ PI is responsible for the design of the fabrication/assembly of the laboratory prototype using commercial catalog items. MCP-9272 ☒ Approved industry standards will be used in any fabrication to ensure safety of design, MCP-9272 ☐##Documents prototype production/data results in a lab notebook, or Engineering Design File or Project File, MCP-9272 ☒##Statement of Work or Specification for procured items or services to include specific requirements (such as product/service deliverables, milestones, acceptance criteria, data collection and data review requirements and equipment calibration requirements), MCP-9359 for Specification and TEM-101 for Template for Statements of Work ☐##Quality Level 4 (consumer grade) Procurement Requirements, MCP-593, MCP-1185 ☐##Customer requirement as specified ☒##Peer review performed in accordance with MCP-9272 ☐##Work performed to approved procedures and Statement of Work/Specification requirements ☒##Provide documentation of process for calibration of M&TE including tolerances, MCP-2391, MCP-9272, App. B ☒##Design control implemented per MCP-2811 ☐##System Operability Test and Integrated Tests per MCP-3056 ☐#Special processes specified (welding, brazing, NDE, etc.) per MCP-37, if required by governing codes, standards and regulations	☐##Document inspections per MCP-195 and selected test TPR ☐##Special processes specified (welding, brazing, NDE, etc.) per MCP-37 ☐##Quality Level 3 Procurement Requirements specified (vendor qualification, receipt inspections, etc.) MCP-590, MCP-591, MCP-1185 ☒##Documented training/certification of personnel performing special processes and inspections as per MCP-37 ☒##Documents prototype production/data results in a lab notebook, or Engineering Design File or Project File, MCP-2875.	☐##Quality Level 2 Procurement Requirements specified (vendor qualification, receipt inspections, etc.) MCP-590, MCP-591, MCP-1185, MCP-3512, MCP-3513 ☐##Software Control per MCP-3039.	☐##Quality Level 1 Procurement Requirements specified (vendor qualification, receipt inspections, etc.) MCP-590, MCP-591, MCP-1185, MCP-3512, MCP-3513.