

PROCESS AND EQUIPMENT DEVELOPMENT FOR HOT ISOSTATIC PRESSING TREATABILITY STUDY

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ABSTRACT

Battelle Energy Alliance (BEA), LLC, has developed processes and equipment for a pilot-scale hot isostatic pressing (HIP) treatability study to stabilize and volume reduce radioactive calcine stored at Idaho National Laboratory (INL). In 2009, the U. S. Department of Energy signed a Record of Decision with the state of Idaho selecting HIP technology as the method to treat 5,800 yd³ (4,400 m³) of granular zirconia and alumina calcine produced between 1953 and 1992 as a waste byproduct of spent nuclear fuel reprocessing. Since the 1990s, a variety of radioactive and hazardous waste forms have been remotely treated using HIP within INL hot cells. To execute the remote process at INL, waste is loaded into a stainless-steel or aluminum can, which is evacuated, sealed, and placed into a HIP furnace. The HIP simultaneously heats and pressurizes the waste, reducing its volume and increasing its durability.

Two 1-gal cans of calcine waste currently stored in a shielded cask were identified as candidate materials for a treatability study involving the HIP process. Equipment and materials for cask handling and calcine transfer into INL hot cells, as well as remotely operated equipment for waste can opening, particle sizing, material blending, and HIP can loading have been designed and successfully tested. These results demonstrate BEA's readiness for treatment of INL calcine.

INTRODUCTION

This paper presents the equipment designed and process to be used by BEA for a HIP treatability study of calcine that will be shipped from INL's Idaho Nuclear Technology and

Engineering Center (INTEC) to the Materials and Fuels Complex (MFC), also located on the INL Site.

INL calcine was produced between 1953 and 1992 and is currently stored at INTEC in six calcined solids storage facilities (CSSFs), reinforced concrete vaults that each contain between three and seven stainless steel vessels (Fig. 1) (Patterson and Prather 2004). Because of its source, as defined by the Nuclear Waste Policy Act (42 USC § 10101) and DOE Order 435.1 Chg. 1, "Radioactive Waste Management," calcine is considered high-level waste. Additionally, calcine exhibits hazardous waste characteristics for metals toxicity.

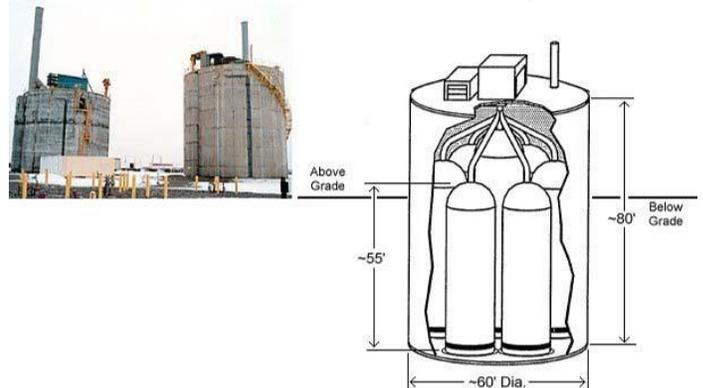


FIG. 1. TYPICAL CSSF CALCINE STORAGE CONFIGURATION

For waste treatment performed via the HIP process, a stainless steel or aluminum can is filled with waste material, evacuated, sealed via welding, and placed in a HIP furnace

where it is simultaneously heated and pressurized in order to produce a size-reduced material with increased durability. This volume reduction allows for long-term repository and interim-storage cost savings as compared to direct loading of granular calcine into storage/disposal casks. According to a study conducted by the Australian Nuclear Science and Technology Organisation, using the HIP process for volume reduction of INL granular calcine and solidified sodium-bearing waste would save an estimated \$2 billion in disposal costs (a 50% reduction) over the U.S. Department of Energy baseline treatment of immobilization in a borosilicate glass matrix (Begg et al. 2005).

In December 2009, the U.S. Department of Energy signed a Record of Decision with the state of Idaho selecting HIP technology for calcine treatment to “provide a volume reduced monolithic waste form that is suitable for transport outside Idaho...” (75 FR 1). The BEA HIP and welding equipment have been used at INL since the 1990s for waste form stabilization experiments. The equipment is operated remotely within a shielded hot cell at the Hot Fuels Examination Facility (HFEF) at MFC. Electrorefining waste salts containing fission products (Bateman, Rigg, and Wiest 2002) and simulated calcine waste materials (Begg et al. 2005; Bateman 2011) have been successfully treated by HIP processing.

CALCINE MATERIAL FOR TREATABILITY STUDY

BEA has identified two 1-gal (3.78 L) calcine waste paint cans as candidate materials for a HIP treatability study. The material had been removed from CSSF 2 in 1978 and placed in Calcine Sample Storage (CSS) Cask 978 (Patterson and Prather 2004), a concrete shielded cask with an inner pipe sleeve that contains the material (Fig. 2). CSS 978 is currently stored on a pallet at INTEC. The cans themselves are highly radioactive and have loose calcine contamination on their surfaces. Special



FIG. 2. CALCINE PAINT CANS STORED IN CSS 978

controls must be used when handling the material to ensure worker safety and prevent release of contamination to the facility or environment. Alumina and zirconia INTEC calcine have mean particle diameters of approximately 0.4 mm and 0.3 mm, respectively, with a standard deviation of <4% for both calcine forms (Patterson and Prather 2004).

CALCINE CASK TRANSFER

Upon initiation of the treatability study, CSS 978 will be shipped from INTEC to MFC. Integral to the design of CSS casks are carbon steel ears to which the three turnbuckles are attached. Each ear has a large-diameter hole to be used as a vertical pick point. However, the ears cannot be adequately load tested and are unacceptable for lifting the cask greater than a distance of 1 ft (0.3 m), nominally. Therefore, a lift fixture (Fig. 3) was designed, fabricated, and tested for CSS transfer within the HFEF facility (Hallgren et al. 2009).

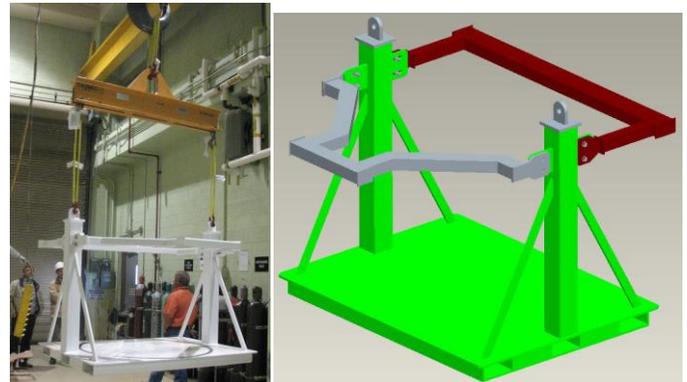


FIG. 3. CSS CASK LIFT FIXTURE (HALLGREN ET AL. 2009)

The lift fixture consists of a base and removable front and rear braces, all constructed from carbon steel. When not loaded, the lift fixture can be moved via forklift using rectangular tubing channels within its base. The cask rests on a 3/8-in.-thick (9.52 mm) carbon steel plate on the fixture base. Vertical members are attached to the plate to provide lift lugs above the cask’s center of gravity. Two braces attached near the top of the vertical member slide against the cask to create a snug fit against the cask with three points of contact (Hallgren et. al 2009).

After the cask is removed from its shipping trailer with a forklift, it will be rigged in a three-point pick and transferred to the lift fixture. The cask will be lifted to the minimum possible height to allow safe transfer. The lift fixture is designed to transport the CSS cask when it is centered, with a maximum tolerance of 1 in. (25.4 mm) off center. A circle painted on the lift fixture’s base plate guides rigging personnel in centering the cask (Hallgren et. al 2009).

The loaded lift fixture can be transported within HFEF using a 48-in.-wide (122 cm) spreader bar to transmit the vertical force to the lift points (Fig. 3). The vertical members are lower than the cask lid and allow access to the entire lid and

cask cavity for contamination control efforts during unloading operations (Hallgren et. al 2009).

CALCINE CONTAMINATION CONTROL

Because of high levels of loose calcine within Cask 978, a collaborative study (Demmer) was conducted to evaluate techniques to minimize contamination spread during cask unloading. Three fixatives: Bartlette Polymetric Barrier System (PBS), Firedam (AS-205), and a proprietary INL formulation (INL-FX1) were evaluated for their ability to limit airborne particulate. All three of these materials, which have been successful in similar past operations at MFC, were tested for their ability to control simulated calcine (a mixture of sand and talcum powder) applied to the surface of paint cans. A 33% solution of PBS was chosen as the preferred fixative, based on its fast penetration into the simulant, ability to encapsulate particulate, quick drying capability, and ease of application.

Further quantitative testing was conducted on the effectiveness of the 33% PBS solution. A handheld particle counter was used to measure the effect of the fixative. Calcine surrogate (approximately 10 grams) was placed onto a small can. A particulate concentration of 15 mg/m³ was detected in the vicinity of the canister after a blast of air was directed toward the calcine. Immediately after applying a spray of PBS, the concentration reduced to 0.165 mg/m³. Within 30 seconds, the concentration was 0.04 mg/m³, further demonstrating the effectiveness of PBS fixative.

As part of the collaborative study, a mockup test procedure was conducted to simulate calcine unloading from the CSS cask. A number of tools were tested during the mockup (Fig. 4). A gripping reach rod allowed manipulation of a hook, brush, or putty knife inside of the cask while the operator maintained a distance of 8 ft. A commercial garden sprayer was used to apply the fixative. A mirror attached to the end of a reach rod provided operators with a view of the cask contents while maintaining a safe distance. To begin the test, fixative was sprayed for approximately 20 seconds to cover all potentially contaminated surfaces. The putty knife and brush tools were then used to remove the simulated calcine from the top of the can and place it further inside of the cask. After visual inspection showed that the can top appeared dry, additional fixative was applied. A hook mounted on the reach rod was then used to remove the can by the lifting bale.

Operators had little difficulty completing the evolutions in the mockup procedure and the tests demonstrated a viable method for contamination control during calcine unloading from the CSS cask.



FIG. 4. CLOCKWISE FROM TOP LEFT: GRIPPING REACH ROD WITH HOOK TOOL, LONG REACH MIRROR TOOL ATTACHMENT, LONG REACH GARDEN SPRAYER FOR FIXATIVE APPLICATION

CALCINE TRANSFER INTO HOT CELLS

To prepare for calcine transfer into the HFEF hot cell, Cask 978 will be moved within the facility using its lift fixture. The cask will be opened and fixative applied using the techniques discussed in the previous section. Using the gripping reach rod, the calcine can bail will be manipulated to allow a crane to hook the calcine and move it into a transfer container (Fig. 5), which was designed and fabricated by BEA.



FIG. 5. CLOCKWISE FROM TOP LEFT: CALCINE TRANSFER CONTAINER AND HANDLE ASSEMBLY, TRANSFER CONTAINER HANDLE QUICK RELEASE PINS, TRANSFER CONTAINER LID WITH O-RING AND ALIGNMENT PIN

The transfer container assembly consists of a transparent section of polycarbonate piping with flanged ends. A removable aluminum bottom is bolted onto the pipe and the top is left open. The flanged ends are designed to be compatible with other modules and containers used for the sieving and blending operations required prior to undergoing HIP. A handle connected to the top flange interfaces with the overhead crane and hot cell manipulators to enable material transfer into and within the hot cell. Quick release pins securing the handle to the transfer vessel are readily detachable to allow the handle to be rotated or removed, allowing access to the container contents to complete HIP processing steps. The overhead crane will lower the loaded transfer container through a roof hatch into the HFEF decontamination hot cell.

IN CELL CALCINE PROCESSING

After the calcine is loaded into the decontamination cell, it will be transferred to a suitable hot cell window for visual observation of processing operations. Prior to undergoing HIP, the calcine container must be opened and the contents size reduced, blended with treatment additives, and loaded into a canister to be sealed via welding and treated with HIP. These operations and relevant equipment are described below. All operations will take place remotely using the decontamination cell overhead crane and hot cell master slave manipulators (MSM). Equipment for HIP treatability study hot cell operations has been designed and mockup tested outside of a hot cell. Some common design features of all relevant equipment include:

- Processing equipment designed to be lifted and secured using standard tooling in conjunction with hot cell MSMs
- O-rings used to seal equipment connections to prevent spread of contamination to the hot cell
- Container lids aligned with pins and secured with hex-head socket capscrews.

Opening the Can

To begin processing, the transfer container loaded with the calcine paint can is moved to the Calcine HIP can punch stand. The calcine can punch is a modified Edlund Model 700 manual crown punch (Fig. 6). The container bottom flange is secured with holding blocks that were added to the base design of the can punch stand. A hold-down ring is installed to the top of the transfer container to secure the can for the punching process.



FIG. 6. FROM TOP: CROWN PUNCH FRONT VIEW, CROWN PUNCH SIDE VIEW WITH LOCKING SCREWS, BOTTOM PLATE WITH HOLD-DOWN BLOCKS SECURED, BOTTOM PLATE WITH HOLD-DOWN BLOCKS OPEN

Additional modifications were made to the vertical portion of the stand to allow the punch head to be removed and reinstalled for positioning of the calcine transfer container. A cable bail and lift rod were added to the punch head to aid in positioning. Locking screws were added to allow the MSM to secure the punch head to the vertical portion of the stand.

After the container and punch head are secured, the MSM lowers the punch handle to open the paint can. The lid is removed from the can with a magnet (Fig. 7). The crown punch head is removed to allow access for removal of the transfer container from the crown punch stand.

To initially size the calcine material, the paint can hold-down ring is removed and a polycarbonate lid with integral breakup tool is installed to the transfer container (Fig. 8).



FIG. 7. TOP: CROWN PUNCH WITH HANDLE, BOTTOM: MAGNET FOR PAINT CAN LID REMOVAL

Particle Sizing

As stated previously, alumina and zirconia INTEC calcine have mean particle diameters of approximately 0.4 mm and 0.3 mm, respectively (Patterson and Prather 2004), which exceeds the 0.25 mm (250 μm) desired size for HIP. The particle size distribution must be determined and calcine must be sized as appropriate. The handle at the end of breakup tool



FIG. 8. TOP: TRANSFER CONTAINER WITH INSTALLED BREAKUP TOOL, BOTTOM: BREAKUP TOOL AND LID

allows use of the MSM to manipulate the tool. The serrated rod end size reduces agglomerated calcine that may be present and removes any calcine hold up from the sides of the calcine paint can. The transparent lid allows visual inspection of the calcine within the can.

Particle Sieving

After the initial size reduction step, the breakup tool and lid are removed and replaced by a sieve plate with mesh openings of 0.06 in. (1.524 mm) (Fig. 9). A smaller transparent container with similar design to the transfer container is bolted

in an inverted configuration to the sieve plate. The entire assembly is then inverted using the MSM in conjunction with the transfer container handle such that the smaller container is on the bottom of the assembly, allowing smaller calcine particles to pass through the sieve mesh. The bottom of the transfer container is then replaced by a bagging sleeve ring so that the paint canister containing the calcine can be bagged and removed with a master slave manipulator

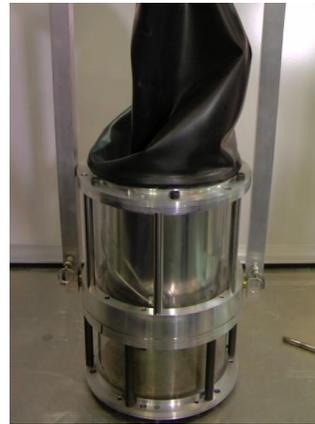
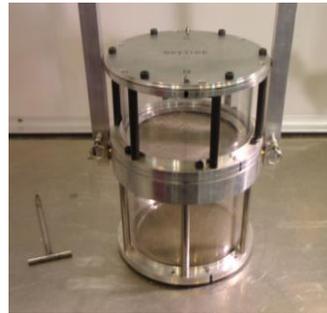


FIG. 9. TOP TO BOTTOM: SIEVE PLATE INSTALLED ON TRANSFER CONTAINER, SMALL CONTAINER INSTALLED INVERTED ON TOP OF TRANSFER CONTAINER, INVERTED ASSEMBLY WITH BAGGING RING INSTALLED FOR PAINT CAN REMOVAL

After module inversion, smaller calcine particles pass through the sieve mesh to the smaller container. To sieve the remaining calcine, a sieve tool is installed to the transfer container and used to direct material through the mesh (Fig. 10). The sieve tool is controlled by the manipulator and is identical to the breakup tool except the tool head is smooth, rather than serrated. This design prevents the wire mesh from

becoming damaged during sieving operations. For the initial sieving step, a coarse mesh (0.06 in. [1.524 mm]) approximately 4 to 5 times the mean particle diameter of stored granular calcines is used.

In order to achieve the HIP process-required particle sizes, progressively smaller mesh sizes will be used in conjunction with the sieve tool to pass material between the transfer and smaller containers. The final size-reduced calcine product will be achieved when all material has passed through a 0.01 in. (250 μm) mesh and is held inside the transfer container. This graded approach to sieving is used to prevent clogging of sieves with large particles.



FIG. 10. CALCINE SIEVING TOOL

Calcine Proportioning and Mixing

As discussed in a previous section, a more stable HIP-processed product is achieved when additives are blended with calcine. An intermediate container (Fig. 11) was designed to apportion a known volume of calcine for blending operations while maintaining containment of the radioactive calcine.

To fill the intermediate container, a funnel assembly with integrated ball valve is installed on the transfer container containing the calcine. The intermediate container is connected to the funnel ball valve via a pipe adapter. Another ball valve is connected to the opposite end of the intermediate container. The assembly is inverted such that the intermediate container is on the bottom. The funnel side ball valve is opened while the valve on the opposite side of the intermediate container remains closed to transfer calcine into the intermediate container.



FIG. 11. TOP: INTERMEDIATE CONTAINER AND FUNNEL INSTALLED ON THE TRANSFER CONTAINER, BOTTOM: INVERTED CONFIGURATION

A mixing container is then connected to the intermediate container (Fig. 12). The apportioned calcine is transferred to the mixing container by closing the funnel side ball and opening the intermediate container ball valve.



FIG. 12. MIXING CONTAINER ATTACHED TO INTERMEDIATE CONTAINER

If necessary, additives are introduced to the calcine using a material additive container (Fig. 13). The container's design features a large cap secured by a clamp opposite a butterfly valve. The cap can be removed to load a prescribed amount of additives to the container. To transfer the additives to the mixing container with the calcine, the cap is secured and the ball valve side of the container is connected to the ball valve of the mixing container. Both valves are opened to introduce the material.



FIG. 13. TOP: MATERIAL ADDITIVE CONTAINER: WITH LID INSTALLED, MIDDLE: CONTAINER WITH LID REMOVED, AND BOTTOM: LIDDED MATERIAL ADDITIVE CONTAINER ATTACHED TO THE MIXING CONTAINER.

To load and blend material in the mixing container, the receiving container is vibrated. An aluminum frame is secured to the receiving container (Fig. 14), and a high-strength 1566 steel handle on the frame is grasped by the MSM to shake the container. The container can be rotated clockwise or counterclockwise and pinned in place so that it can be angled during the mixing process to promote better material blending.



FIG. 14. MIXING CONTAINER WITH ATTACHED VIBRATION FRAME

Hip Can Loading

The final calcine storage canister operation is loading of the HIP can. After blending operations, a fill tube used for transferring blended material to the HIP can is installed to connect the mixing container ball valve to the HIP can (Fig. 15). After the mixer assembly is uprighted, the ball valve is opened and calcine is dispensed into the HIP can. Previously designed and used in-cell equipment is used to seal, weld, and press the waste.

CONCLUSION

BEA has designed and performed out of cell mockup testing of specific equipment designed for a HIP treatability study using INTEC calcine. Mockup testing was successful, and only minor revisions to equipment were suggested. The work completed to date demonstrates BEA's readiness for receipt and treatment of INTEC calcine at INL's MFC.

ACKNOWLEDGEMENT

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FIG. 15. HIP CAN LOADING

NOMENCLATURE

BEA	Battelle Energy Alliance
CSS	calcined sample storage (cask)
CSSF	Calcined Solids Storage Facility
HFEF	Hot Fuels Examination Facility
HIP	hot isostatic press(ing)
INL	Idaho National Laboratory
INTEC	Idaho Nuclear Technology and Engineering Center
MFC	Materials and Fuels Complex
MSM	master slave manipulator
PBS	Polymetric Barrier System

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