

Contract No:

This document was prepared in conjunction with work accomplished under Contract No. DE-AC09-08SR22470 with the U.S. Department of Energy (DOE) Office of Environmental Management (EM).

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Stabilization of Residual Contamination with Alternative Materials - 19404

James C. Nicholson*, Joseph Sinicrope**, Aaron L. Washington, II*, Peggy Shoffner**,
Leonel Lagos **, and Michael G. Serrato *

*Savannah River National Laboratory

**Florida International University Applied Research Center

ABSTRACT

Since 2015, Savannah River National Laboratory (SRNL) in conjunction with Florida International University (FIU) have been investigating the applicability of intumescent coatings as radiological contamination fixatives for stabilization of residual materials after decontamination efforts have concluded. Intumescent coatings are designed to prevent heat transfer to a protected substrate and are typically used in both the aerospace industry to protect components from thermal shock and in building design to protect building joints that may fail in the event of a fire. For this work, these materials are examined for their ability to reduce worker risk by fixating residual contamination and lower the impact of a release in a scenario that breaches containment of a contaminated building and subsequently induces a large-scale fire by crediting their fire-retardant attributes. Initial studies of intumescent coatings and commercial fixatives included performance in direct flame, varied environmental conditions (20-110 F or -6.67 C, 10-95% relative humidity), adhesion studies to stainless steel after curing in varied environmental conditions, and radiological exposure. Of all the materials tested, many were found to be inadequate in one or more of these areas. These materials failed in one or more of three characteristic ways: 1) through delamination at reduced temperatures (≤ 40 F or 4.44 C) or from even moderate handling, 2) melting off the substrate at elevated temperatures (≥ 90 F or 32.22 C), 3) proving to be flammable and promote smoke propagation. Failure in these ways results in the potential for a subsequent release of the fixated contamination in the event of water presence/absence or direct flame. Using these results as a platform for the best in class identified material that did not experience failure in any of these tests, SRNL and FIU targeted the plutonium-238 contaminated hot cells within Savannah River Site (SRS) Building 235-F as a characteristic test bed to benchmark an intumescent coating used as a contaminant fixative. Through discussion with the SRS 235-F Risk Reduction personnel, appropriate hazards and performance metrics were discussed and a cold demonstration was performed in a hot cell mockup located at SRS. The application methods were proven functional in the cold mock up, and these intumescent coatings were subsequently utilized in the Savannah River Site Building 235-F's Plutonium Fuel Form (PuFF) Facility to aid in stabilization of plutonium-238 in both a contaminated hot cell and Entry Hood in September 2018. Presented in this work is the overview of the application process, associated working considerations for introduction of equipment and materials into the facility, lessons learned, and lifetime monitoring data to date.

INTRODUCTION

Nuclear facilities that are moving towards final disposition face enormous challenges to ensure no release of holdup material is released to the environment between the time the facility is no longer active until the final disposition is achieved. Workers actively seek to remove as much of the radioactive holdup; however, decontamination can only remove so much of the material before further efforts become time/cost prohibitive. As such, for contaminated areas, there is often residual contamination remaining after material removal and decontamination operations are completed. To overcome free standing contamination, it is

sometimes favorable to place a fixating layer over the spot to ensure no residual contamination can be released should any be left behind. In conjunction with Florida International University (FIU), it has been found through laboratory scale testing that commercial fixating platforms are inhibited in one or more of the following ways when considered for extended life performance:

- 1) They rapidly degrade in the presence of elevated temperatures or direct flame [1]
- 2) They are adversely affected by ambient moisture/temperatures beyond manufacturer specified operational regions after curing that can cause [2-3]
 - a. Moisture “breathing” (uptake and subsequent evaporation)
 - b. Delamination
 - c. Dissolution
- 3) They are adversely affected by ambient moisture/temperatures beyond manufacturer specified regions that inhibit curing processes [3]

To circumvent these problems, Savannah River National Laboratory (SRNL) in conjunction with Florida International University (FIU) have been investigating the applicability of intumescent coatings as radiological contamination fixatives for stabilization of residual materials after decontamination efforts have concluded. Intumescent coatings are designed to prevent heat transfer to a protected substrate and are typically used in building design to protect building joints that may fail in the event of a fire [4-5]. For this work, these materials are examined for their ability to reduce worker risk by fixating residual contamination and lower the impact of a release in a scenario that breaches containment of a contaminated building and subsequently induces a large-scale fire by crediting their fire-retardant attributes. SRS building 235-F Plutonium Fuel Fabrication Facility (PuFF), a building which manufactured Pu-238 radioisotope thermoelectric generators for NASA space missions, was targeted for this demonstration as it is actively undergoing decontamination activities and is a prime target for stabilization platform research. Presented in this work is the overview of the application process, associated working considerations for introduction of equipment and materials into the facility, lessons learned, and lifetime monitoring data to date.

METHODS

Prior to application of the chosen intumescent coating into SRS 235-F, two cold demonstrations were performed. The first cold demonstration was performed by FIU to determine the feasibility and best practices of application of the material onto the Process Cell walls. Once lessons learned were generated from this cold demonstration, a second cold demonstration was performed at Savannah River Site to train the operators that would be applying this material to the radiologically contaminated areas within SRS 235-F.

Florida International University Cold Demonstration

In close coordination with SRNL and SRS site personnel, FIU designed and developed a to-scale, combined corner cell and middle cell configuration that mirrors the operating conditions (dimensions, glove ports, surface materials, obstacles/obstructions, etc.) encountered in hot cells 6-9 at the SRS 235-F facility as shown in Figure 1. FIU completed the construction of the hot cell test bed in May 2017.

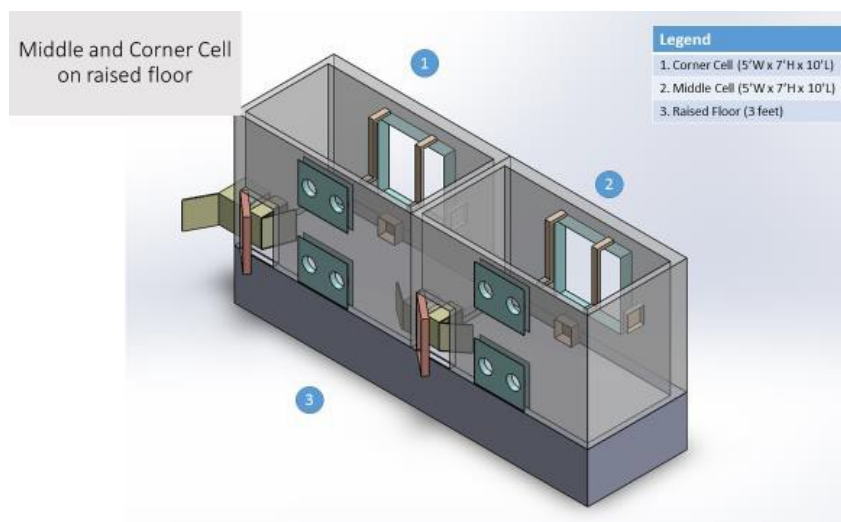


Figure 1: Computer design of the hot cell mockup constructed at FIU to represent SRS 235-F PuFF Process Cells 6 and 7.

The dimensions of each cell are 5' wide x 10' long x 7' high and include a 3' raised floor. Pass-through ports and glove ports were sized and positioned in collaboration with SRNL/SRS to closely mimic the actual conditions at the facility. Sheets of 304 stainless steel were installed to represent the surfaces found in the hot cells. Photos of the completed hot cell mockup interior and exterior can be seen in Figure 2.

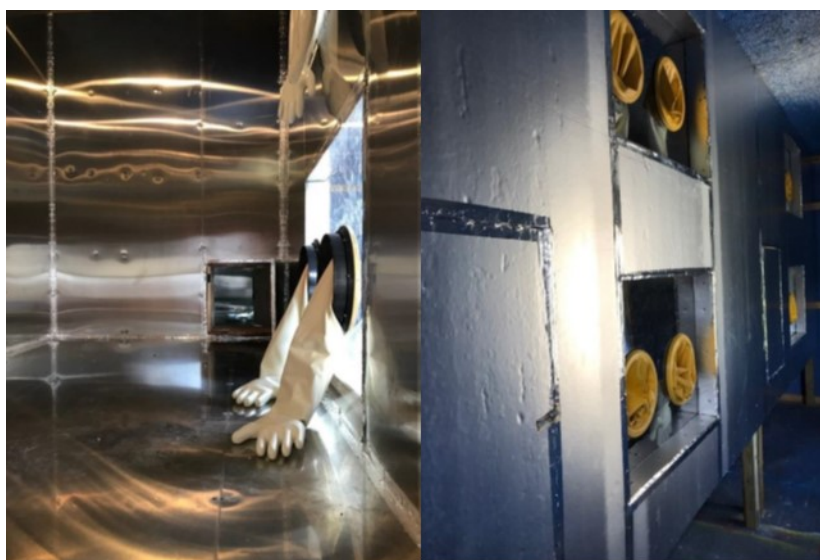


Figure 2: Completed interior (left) and exterior (right) photos of the hot cell mockup constructed at FIU to represent SRS 235-F PuFF Process Cells 6 and 7.

Once construction was complete, FIU began with the application of the intumescent coating using SRS approved tools for use in building 235-F [6], specifically the extension pole and a roller brush, along with monitoring of curing times, observations and recording of the ability of all required tools and materials to fit through the bag in/bag out port and pass-through port, and an evaluation of volume/surface area of intumescent coating required to cover all surfaces to the minimum thickness needed (1/8" or 3.2 mm).

All of the required tools and materials easily fit through the bag in/bag out port as well as the pass-through port between hot cells. No significant challenges were encountered in moving materials and supplies associated with the operation from one hot cell to another using the pass-through port. FIU used standard paint trays as well as 2.5-quart plastic containers to hold the intumescent coating. These options were used in order to mitigate any weight challenges that could potentially cause excessive fatigue in the operator during application.

FIU was able to reach all locations within the hot cell configuration from both the bottom set of glove ports as well as the top set of glove ports using the site approved tools. Application of the intumescent coating using the site approved tools, specifically the extension pole and a roller brush was uneventful with no significant challenges encountered. Manipulating the extension pole was relatively easy and allowed FIU to set the required length to reach all surfaces. FIU did not include obstacles in the hot cell configuration that could complicate access and application.

FIU proceeded with a larger-scale test and evaluation of the technique. FIU sectioned off a 5'x5' (1.52 m X 1.52 m) section of the hot cell to further evaluate the effectiveness of using a simple slow-pour method of applying an intumescent coating to the floor of the hot cell test bed. FIU used the following tools:

- Container – 2.5-quart-size (2.4 L) plastic bucket to hold/transport the IC
- Gripper - used to maneuver a 2.5-quart-size (2.4 L) plastic container of IC
- Custom wooden T-shaped extension tool - used to spread the IC. To develop this tool, FIU connected a 13" (33 cm) wooden head to the approved extension handle on the SRS tooling list.

FIU poured the IC into a 2.5-quart (2.4 L) plastic container outside the hot cell, filling the container with 1 to 1.25 quarts (0.95 to 1.2 L) of IC to keep the weight between 2 to 3 pounds (0.9 to 1.4 kg) to minimize worker fatigue. The container and tools were then passed through the bag in/bag out port. Maneuvering the 2.5-qt (2.4 L) container to various locations within the hot cell by manipulating the gripper from the glove-ports was relatively easy and allowed for targeted pouring of the IC. After marking the hot cell border at 1/8" (3.2 mm) above the floor, FIU poured the IC from the container at a height of about 1" to 3" (2.54 to 7.62 cm) above the floor to minimize any potential for splatter or disturbance of any residual contamination that may be present in a radioactive environment. FIU then spread and smoothed the IC across the floor area using the wooden T-shaped tool, using the 1/8" (3.2 mm) markings along the perimeter of the hot cell as a thickness guide. The custom wooden T-shaped extension tool allowed access to all locations within the hot cell test bed from the glove-ports and easily reached the corners. An area of 5' x 5' (1.52 m X 1.52 m) was coated to 1/8" (3.2 mm) thickness in approximately 15 minutes once the tools and IC were in the hot cell. Total curing time after the slow pour application was 48 hours under hot and humid weather conditions (temperatures 75-97 F or 23.9-36.1 C and humidity 45%-93%).

The slow pour method for the floor/horizontal surfaces proved very effective during application, significantly reducing time and effort while greatly facilitating application to the requisite thickness level. With an estimated 95% of the contamination residing on the hot cell floors at the SRS 235-F facility and given the composition and characteristics of the IC being tested, the slow pour method may be a viable

application method for horizontal surfaces that would expedite application and minimize disturbance of any residual contamination.

No international testing protocols for confirming curing of an intumescent coating at 1/8" (3.2 mm) or greater in a radioactive environment were identified. To confirm curing, FIU developed a basic pressure test based on previous experiments with the material. Using a white paper towel, FIU pressed firmly on the test area, remove the paper towel, and observed the surface for any indentation and/or any discoloration of paper towel. For areas within the hot cell test bed that were beyond arm reach, this test was accomplished using a gripper tool. Photographs are shown in Figure 3 of cured (no indentation, (a) and (b)) and not cured (indentation, (c) and (d)) surfaces coated with IC. All horizontal surfaces were cured at 48 hours after the slow pour application method.

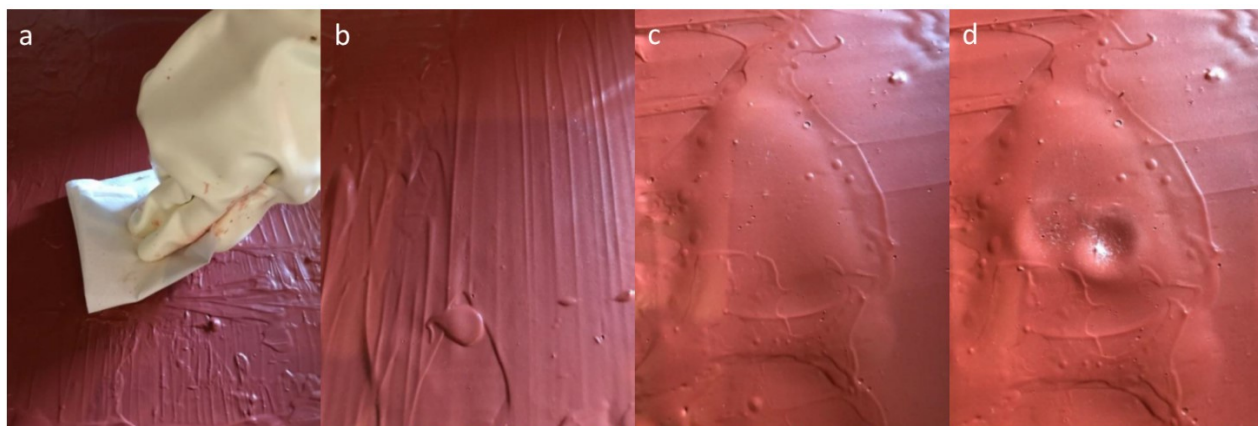


Figure 3: a) blot test on assumed dried IC, b) confirmation of full drying due to no indentation, c) pre-blot test on assumed dried IC, and d) indentation post-blot test showing material is not fully cured.

Thickness of the coating was confirmed using a Defelsko PosiTector-6000 FNTS (0-250 mils, Ferrous + Non- Ferrous). This instrument conforms to ISO 2178/2360/2 808, ASTM B499/D1186/D1400/D7091/E376/G12, BS3900-C5, SSPC-PA2 and others. After two applications using the slow pour application method for horizontal surfaces previously described, all areas of the floor coated met the requisite 1/8" (3.2 mm) thickness requirement for the IC fire rating. Representative measurements are shown in Figure 4.



Figure 4: Measurements of the IC thickness via Defelsko PosiTector-6000 FNTS. Results show that the final cured thickness is greater than the 1/8" (3.2 mm) required by the manufacturer.

FIU also benchmarked the use of a selected handheld sprayer (Graco Ultra Max Cordless) that demonstrated the capacity to spray the IC at various distances. These positive results indicated that this type of tool may be an acceptable application method for vertical surfaces and warrants further testing to evaluate this potential. The viscosity of the IC being tested is 120,000 centipoise (CPS). For context, the viscosity of water is 1 CPS and the viscosity of peanut butter is roughly 250,000 CPS. It was found for the chosen sprayer, the entire cartridge (1 qt) could be completely sprayed over the course of about a minute and a half, significantly reducing the amount of work time over traditional painting techniques that were initially explored for the vertical surfaces. Figure 5 shows the IC being sprayed by the selected handheld sprayer.



Figure 5: IC being sprayed from the Graco Ultra Max Cordless onto stainless steel.

Savannah River National Laboratory Cold Demonstration

Following the results of the FIU cold demonstration, lessons learned on application techniques were examined and a test plan was developed to outline objectives for a hot demonstration that needed to be benchmarked prior to facility entry in order to train operations personnel [7]. From the FIU results, it was found that spraying using the Graco Ultra brand sprayers was the best in class application method vs. traditional painting due to the time associated with coating: one quart of IC could be sprayed over the course of about a minute and a half with reloading time of cartridges averaging about two minutes whereas a roller method required significantly more physical effort and time to put equivalent material on the coated surface. Maintaining control of the spraying pattern was also found to be relatively easy with minimal overspray in the training environments. Likewise, for horizontal coating, the pour and spread method was found to be the best in class over spraying or rolling onto the surface. Due to the weight of the sprayer and the angles involved for the floor, the sprayer was disqualified as a means for coating. Traditional paint rolling also exhibited the same problems as with vertical surfaces: the amount of time and effort it took to coat the floor vs. the pouring method proved to be excessive.

From these lessons learned, SRNL and SRS personnel embarked on a cold demonstration in a prebuilt mockup that mimicked Process Cell 7 in 235-F. Initial efforts were performed outside of the mockup to familiarize operators with the new tooling (sprayer, roller, thickness gauge) they would be using to perform these operations. All cold demonstration operations were applied to either paper or cardboard to keep the mockup enclosure as clean as possible. All activities were performed from the maintenance gloveports within the hot cell due to manipulators in the 235-F building being inoperable. A summary of these activities is shown in Figure 6. Areas that were scrutinized were: 1) whether the coating method was able to achieve the requisite 1/8" (3.2 mm) coating from the gloveports, 2) how well the associated tooling (sprayer, cartridges, spreader, thickness gauge) could be manipulated from the gloveports, and 3) identifying any additional obstacles that came from working within the enclosed space through gloves.

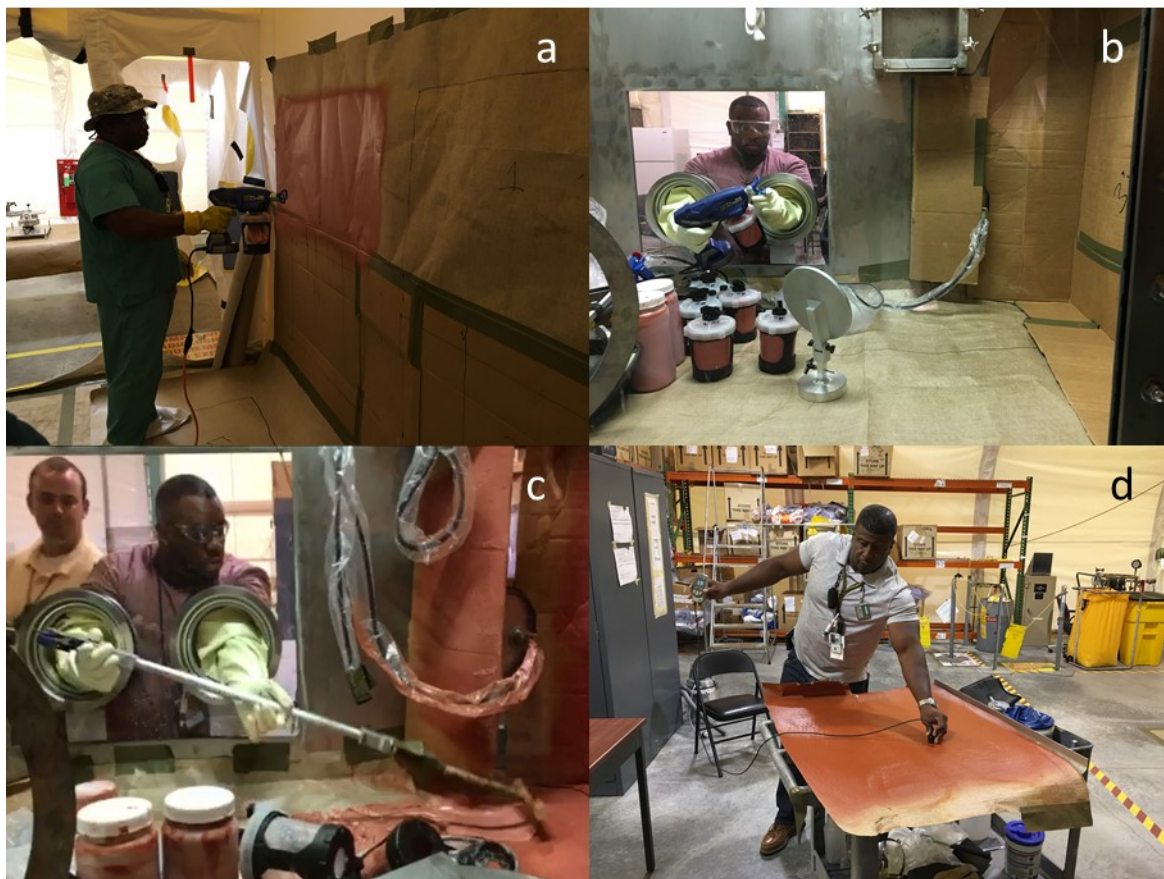


Figure 6: a) Vertical surface sprayed outside of cell mockup to familiarize operators with sprayer setup, functions, and cleaning. b) Vertical surface sprayed inside SRS cell mockup that is of the same geometry of the cell to be sprayed (235-F Process Cell 7). c) Horizontal pouring/spreading of the IC on the floor of the mockup. d) Material thickness measurement verification.

This cold demonstration proved successful and the hands-on training the operators received through this process was valuable as the team moved towards the hot demonstration. It was noted that adhesion to the cardboard and paper vs. stainless steel was significantly different and lead to sagging when too much material was applied. It was also found that if the material was applied in a stagnant environment, it took significantly longer to dry as compared to a system where active ventilation was circulating air. This was induced within the mockup through a simulated air venting system and proved to cure the material in approximately 48 hours once ventilation was turned on as opposed to the material still being completely uncured after 24 hours with no induced ventilation.

Savannah River Site 235-F Identified Hot Demonstration Areas

Taking the lessons learned from the SRNL/SRS cold demonstration, two application sites were identified within building 235-F: 1) Process Cell 7 which had already undergone decontamination efforts and was known to contain approximately 1g of Pu-238 holdup and 2) the Entry Hood leading into a glovebox train attached to Process Cell 1 that had not been decontaminated at all and was known to contain approximately 0.33g of Pu-238. Pictures of these two areas prior to application of the IC are shown in Figure 7.



Figure 7: (left) Entry Hood leading into Process Cell 1 glovebox train with ~0.33g of Pu-238 holdup and (right) Process Cell 7 with ~1g of Pu-238 holdup.

Prior to beginning work in these areas, masking had to be installed to protect certain assets that facility personnel wanted to retain access to. This included a door in the back of the Entry Hood (masked with plastic), the electrical passthrough and associated window (masked with plastic, right side of right picture in Figure 7), and the circular passthrough leading from Process Cell 7 to 8 (masked with cardboard). Also, to maintain confinement of any material that may be stirred up during activities, a flex panel glove wall was installed on the Entry Hood opening and can be seen on the left of Figure 7.

RESULTS AND DISCUSSION

Entry Hood

Initial efforts focused on the Entry Hood due to ease of access. The back wall was first coated completely through three applications to above the 1/8" (3.2 mm) thickness over the course of 3 weeks with a week drying time in between applications. This week drying time was found to be necessary due to inherent facility conditions such as minimal flow in the Entry Hood that rendered the environment nearly stagnant and a downed chiller that occurred twice that resulted in extreme moisture content in the air. Measurements were taken at various points across the coated surface and are shown in Figures 8 and 9. One benefit of taking measurements and recording them in a visual grid was that it allowed operators to visualize the areas that were not sufficiently thick and apply more material in a targeted manner. It also showed the operators areas where they sprayed from one glove position trying to maximize the amount of material applied that was subsequently resprayed by another operator in the second glove position.

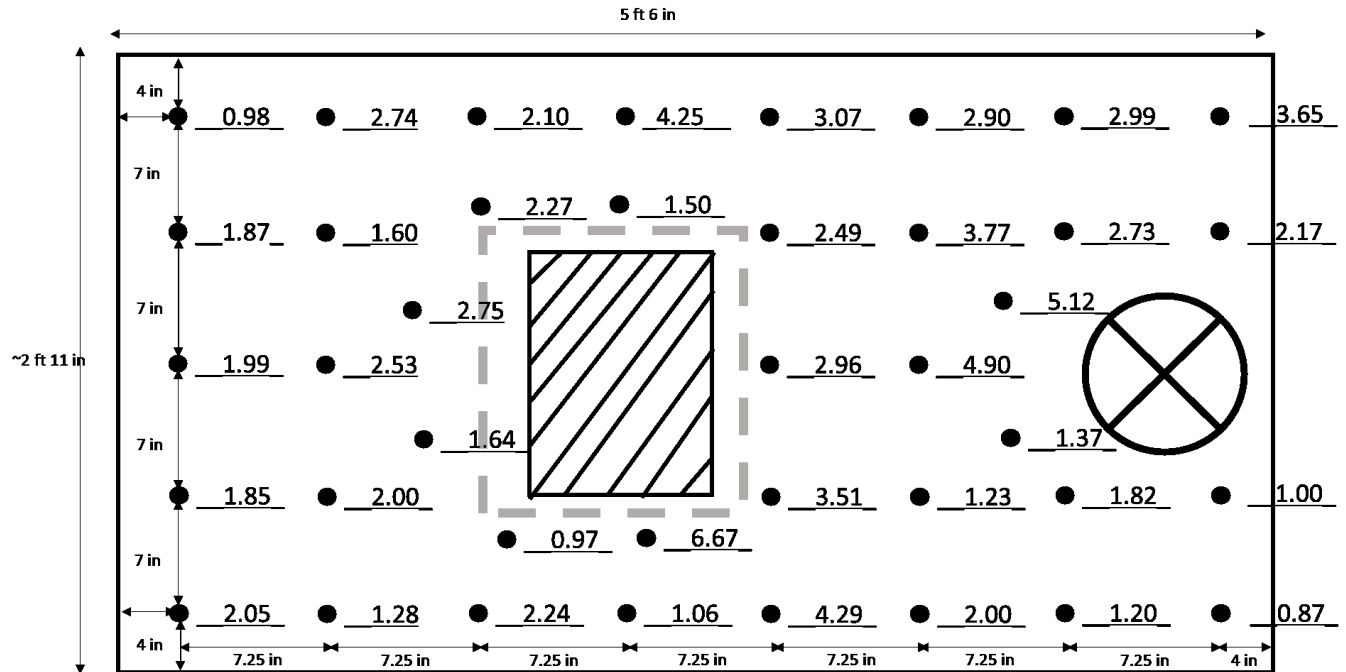


Figure 8: Measurements of Entry Hood back wall after first spray application. All values reported are in millimeters. Dashed line represents the mask over the square door.

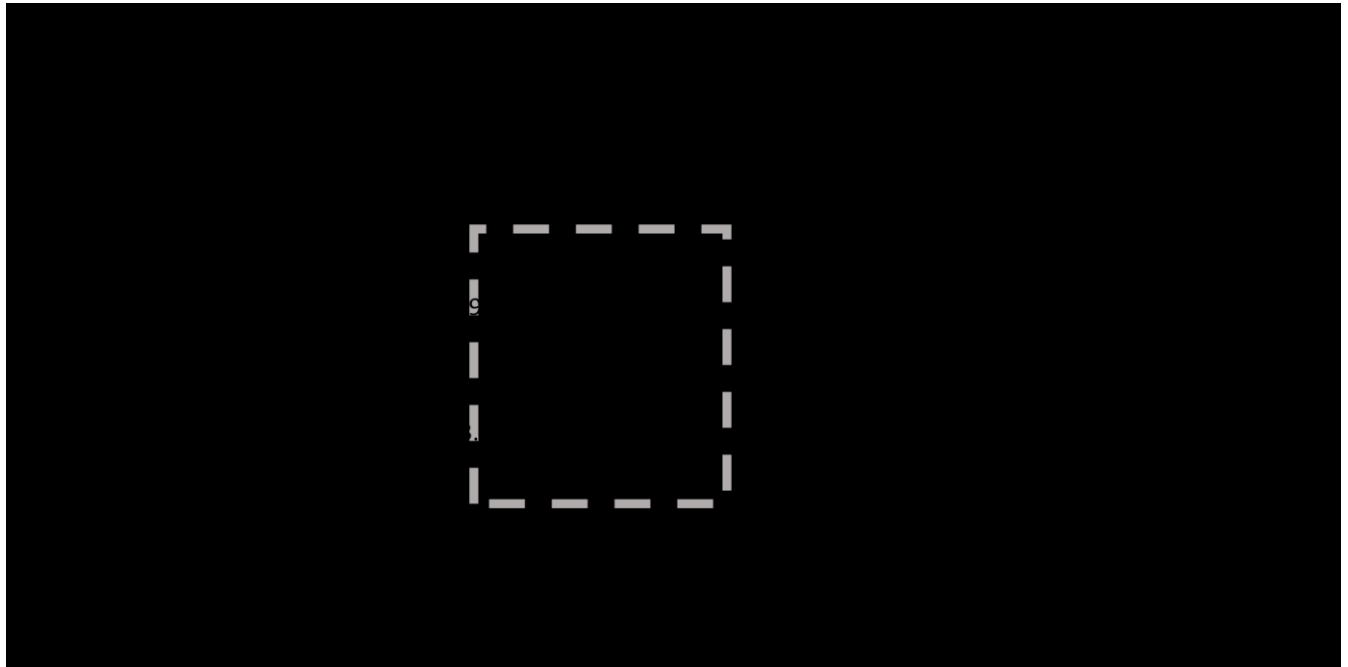


Figure 9: Measurements of Entry Hood back wall after final spray application. All values reported are in millimeters. Dashed line represents the mask over the square door.

Once the back-wall coating was complete, the floor was coated via a pour and spread method wherein the total volume necessary to coat the floor to 1/8" (3.2 mm) was poured and pushed along the floor. Some additional material was also poured at the same time to compensate for losses to container walls and

incomplete pouring from the host container. This process was performed in one application and all measured points were found to be of the correct thickness.

Process Cell 7

During drying times for material applied in the Entry Hood, activities were performed in parallel in Process Cell 7. As of the publication of this paper, only the vertical portion of the cell that was to be coated has been performed and was found to meet thickness specification after 2 coatings, with the coating of the floor still to be performed. While activities in the cold mockup were performed without any incident, significant impedance was found within the hot cell that was not considered in either of the mocked scenarios. Shown in Figure 10 is the spray coating performed in the mockup (left) and in the hot cell (right). Of particular note is the metal post with valves and hoses coming off of it in front of the glove coupled with the plastic covering on the smaller window on the right photo. This post limited range of motion of the operator who was spraying, leading to moderate difficulty in ensuring complete coverage of the area to be sprayed and induced some spray onto the plastic shielding the electrical connections. While the operation was still able to be successfully completed, these were operational difficulties that were not taken into account until the operation was going live.



Figure 10: Comparison of spraying activities performed in the cold cell mockup (left) and inside Process Cell 7 (right).

CONCLUSIONS

The downselected IC that was chosen from three years of benchmarking has been successfully applied through both spray and pour/spread methods to the back wall and floor, respectively, in the Entry Hood within SRS 235-F PuFF facility and through the spray method to the wall (to date) within Process Cell 7 and before and after photos of each area are shown in Figure 11. Throughout the process of these applications, the value of the cold demonstrations was found time and again in the operators' competencies with the tools and material that was being applied. Specifically, a time out was called when the operators noticed minimal running/sagging of the material to allow the material to cure for a time before more was applied.



Figure 11: Before and after photos of the SRS 235-F Cell 1 Entry Hood (top) and Process Cell 7 (bottom).

One key lesson learned from this work was the value of the flex wall in containing contamination within the Entry Hood. No contamination was found on any of the workers or the surrounding work area throughout the 3 weeks of activity performed in this location. Likewise, no air monitoring alarms sounded from this work which historically had not been the case with this location prior to the entry hood being installed; this entry hood was a known source of particulate that induced nearby air monitoring alarms.

From the coating of the entry hood, it was noted that the standoff distance from the flex wall to the back wall was closer than the suggested distance of 3 ft (36 in or 91.5 cm) for the Graco sprayer. This had some minimal impact on the spraying operations in that the amount of arm movement necessary to ensure a full coating was exaggerated over what could be expected from a farther standoff. Conversely, in Process Cell 7, the standoff was greater than the suggested distance of 3 ft (36 in or 91.5 cm), and lead to more than anticipated overspray of the designated spraying area. That said, the ease and timeliness of application from the sprayer more than made up for these short comings, and operators were able to easily adjust to ensure full coating resulted.

Also of note is the masking covering the electrical passthrough in Process Cell 7. Due to the constraints with the metal stand, this covering got more than anticipated material sprayed on it. This in turn cause the plastic coating to fall due to the increased weight of the IC layered on the plastic. It was suggested by the operations personnel from 235-F that the masking needed to be rethought and better designed to both a) be easier applied to the protected area and b) better withstand weight of any material that is sprayed onto it.

As of this writing, work is still ongoing to pour/spread coat the floor of Process Cell 7. Once this is complete and all measurements are above the requisite thickness, performance monitoring both within the Process Cell and in the Entry Hood will be performed. This will entail monitoring the thickness over this performance period to determine whether there is any subsequent shrinking/swelling induced from normal curing processes or induced from the Pu-238 that is immobilized beneath the IC. To benchmark this performance, control coupons have been created and are being stored both in controlled laboratory environments and outdoors to simulate any environmental considerations that may occur from the routine outages with the 235-F chiller.

REFERENCES

1. L. E. Lagos, et al., “Incombustible Fixatives – Adapting Intumescent Coatings as Fire Retardant Fixatives to Support D&D Activities,” Florida International University. Prepared for the Department of Energy under Cooperative Agreement No. DE-EM0000598. June 30, 2016.
2. J. C. Nicholson, A. L. Washington, II, “Evaluation of Commercial Fixative and Intumescent Coatings in Varied Environmental and Radiological Conditions,” Savannah River National Laboratory. SRNL-L3100-2016-00174, 2016.
3. J. C. Nicholson, J. A. Velten, “Evaluation of Environmental Conditions on the Curing of Commercial Fixative and Intumescent Coatings,” Savannah River National Laboratory. SRNL-L3100-2016-00230 (2016).
4. "Intumescent Paint, Fireproofing, and Firestopping." Archtoolbox. Arch Media Group LLC, n.d. Web. 12 Nov. 2018. <https://www.archtoolbox.com/materials-systems/thermal-moisture-protection/intumescent-paint-fireproofing-and-firestopping.html>.
5. "Association for Specialist Fire Protection Technical Guidance Documents (11-17)." Association for Specialist Fire Protection. ASFP Kingsley House, n.d. Web. 12 Nov. 2018. http://asfp.associationhouse.org.uk/default.php?cmd=210&doc_category=16.
6. J. C. Musall, “235-F Risk Reduction Tooling List,” Savannah River Site. SDD-2015-00002, 2015.
7. J. C. Nicholson and M. J. Siegfried, “Incombustible Fixative and ACE 2.0 Test Plan – Radiological Hot Field Test of Intumescent Coatings and Electrostatic Precipitators,” Savannah River National Laboratory. SRNL-TR-2018-00074, Rev. 1, 2018.