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Radiation Hardened Foam Cold Test Plan - Phase II: Foam Characterization Testing and Environmental Chamber Testing of FoamBag Fixative Foam

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2” WC	Two Inches Water Column (unit for pressure)
COTS	commercial-off-the-shelf
D&D	Deactivation and Decommissioning
DOE-EM	Department of Energy Office of Environmental Management
FIU	Florida International University
GC-MS	Gas Chromatography-Mass Spectroscopy
LLW WAC	Low Level Waste Acceptance Criteria
MSX	Multi-spectral Dynamic imaging
SOP	Standard Operating Procedure
SRNL	Savannah River National Laboratory
SRS	Savannah River Site
VOCs	Volatile Organic Compounds

1.0 Purpose

This document outlines the Phase II test objectives and implementation plan for a down-select foam fixative technology intended to facilitate activities in support of the Savannah River Site (SRS) F/H labs deactivation and decommissioning (D&D) efforts. It is a collaborative effort between Savannah River National Laboratory (SRNL), Florida International University (FIU), and the SRS F/H labs team intended to test and evaluate the potential of a polyurethane resin foam in mitigating the release of contamination during dismantling operations on radioactively contaminated piping in legacy facilities. The cold test plan addresses specific requirements highlighted by site and safety personnel and will be executed at FIU and SRNL test and lab facilities. Results from the cold tests will inform the hot test at F/H labs, which will use the foam fixative to confine and/or isolate residual contamination within a 3-dimensional void space of Hastelloy C-22 piping designated for removal from the area and transported to a designated disposal facility. Phase I testing was previously conducted using Hilti CP620 fixative foam. Results from Phase I testing indicate Hilti CP-620 fixative foam is incompatible with SRS site hot taps as an effective foam delivery method into Hastelloy C-22 piping. Phase II testing will be conducted using FoamBag™ fixative foam as an alternative fixative foam option. Phase II testing will address eight test objectives: (1) evaluation of the adhesion and bonding properties of FoamBag™ in Hastelloy C-22 piping, (2) evaluation of the adhesion of FoamBag™ in piping under various moisture conditions, (3) determination of the heat profile of FoamBag™ during curing, (4) determination of the internal pipe pressure after FoamBag™ deployment and curing, (5) conduct a leak test to determine if FoamBag™ is effective at creating a full seal within piping, (6) headspace testing of FoamBag™ to determine if there are any associated off-gas hazards during FoamBag™ curing, (7) conduct environmental chamber testing of FoamBag™ to understand how environmental parameters impacts FoamBag™ curing, and (8) conduct fire testing to evaluate FoamBag™ fire retardant characteristics.

2.0 Background

Intumescent and polyurethane foam technologies are currently being tested at SRNL and FIU to support Department of Energy Office of Environmental Management (DOE-EM) D&D operations, which includes the decommission of piping systems that are radioactively contaminated. These foam technologies are being evaluated for their ability to immobilize and/or isolate residual radioactive contamination within piping systems prior to their removal and transport to proper disposal facilities. The use of foam technologies has the potential to improve the engineering controls currently in place to remove contaminated piping systems for disposal. Prior to Phase I testing, four foams were selected for environmental chamber testing to identify a foam candidate for Phase I testing. Hilti CP-620 foam was down selected from the group of foams due to its resilience in different environments and its ability to withstand thermal stressors. Phase I testing was conducted to evaluate Hilti CP-620 intumescent foam. Testing comprised of open-air tests at FIU to evaluate Hilti CP-620 foam properties in piping and environmental chamber testing at SRNL to evaluate the impact of environmental parameters on Hilti CP-620 curing. Results from these tests indicate that, while Hilti CP-620 fixative foam creates an effective seal within Hastelloy C-22 piping, its fast-curing time is incompatible with the use of a hot tap as a deployment method into piping because the Hilti foam cures before it is completely injected into the pipe. The use of a hot tap to inject fixative foams into piping is necessary because it significantly reduces worker exposure to radiological contamination within piping system. During Phase I testing, a second fixative foam (FoamBag™) was identified as an alternative foam solution that may overcome the hot tap challenges facing Hilti CP-620 foam, and will therefore, be the focus for the Phase II cold test (FoamBag™, Steve Vick International, Avon, UK, highlighted in Appendix A). Phase II testing will consist of foam property testing and environmental chamber testing from Phase I to evaluate FoamBag™ as an effective fixative foam for D&D activities. Phase II testing objectives will be conducted in open air conditions at FIU and environmental chamber testing at SRNL to determine if FoamBag™ can be a reliable technology to mitigate the release of residual contamination in piping systems during D&D activities. This research performed by FIU is funded as part of FIU-DOE Cooperative Agreement (Contract # DE-EM0005213).

3.0 Goals and Objectives

3.1 Goals

The goals of Phase II testing are to assess the mechanical properties of FoamBag™ commercial foam (Appendix A) under select stressors common to a real-world operational environment and determine the suitability of FoamBag™ as a foam fixative that can be used to mitigate/reduce the release of residual contamination during D&D activities. This testing is a necessary precursor to large-scale dry run/mock-up testing that will be conducted in Phase III. Photos, illustrations, and characteristics of the proposed hot-test site location will be provided in the Phase III cold test plan. Mechanical property testing and thermal stressor testing of FoamBag™ will be conducted at FIU. Headspace analysis and environmental chamber testing of FoamBag™ will be conducted at SRNL.

3.2 Objectives

Phase II testing will be performed to characterize the mechanical properties and thermal properties of FoamBag™ foam fixative that may impact its effectiveness as a 3D fixative in Hastelloy C-22 pipes. Headspace testing and environmental chamber testing will be conducted in gloveboxes. Specifically, testing will address the following:

- Evaluation of the adhesion and bonding properties of the FoamBag™ foam plug to Hastelloy C-22 piping.
- Evaluation of the adhesion and bonding properties of the FoamBag™ foam plug to Hastelloy C-22 piping under various moisture conditions.
- Determination of the heat profile of FoamBag™ foam during curing in Hastelloy C-22 piping.
- Determination of the internal pipe pressure after FoamBag™ foam deployment and curing.
- Determination the effectiveness of FoamBag™ using a standard leak test
- Evaluation of off-gas formed during FoamBag™ curing
- Evaluation of the impacts of temperature and humidity on FoamBag™ stability during environmental chamber testing
- Evaluation of FoamBag™ resistance to thermal stressors

4.0 Test Parameters

4.1 Evaluation of the adhesion and bonding properties of the FoamBag™ foam plug to Hastelloy C-22 piping.

Adhesion capabilities of the FoamBag™ to Hastelloy C-22 piping will determine whether the FoamBag™ can function as an effective plug and confine and immobilize residual contamination in piping prior to transportation and disposition. The adhesion test will assess if any potential incidental impact could cause the FoamBag™ foam to delaminate from Hastelloy C-22 piping, causing the release of contamination. The FoamBag™ will be deployed into Hastelloy C-22 piping and be allowed to cure until it hardens and creates a full seal within the pipe. Using an adaptation of *ASTM D1621: Standard Test Method for Compressive Properties of Rigid Cellular Plastics* (ASTM, 2016), the cured plug in the Hastelloy C-22 pipe segment will be subjected to mechanical stressors (Figure 4-1) using the MTS Criterion series 43 Tensile Tester with compression plates and the amount of force required to push a FoamBag™ foam plug out of a 3” D x 14” L, Hastelloy C-22 steel pipe segment will be determined.

Figure 4-1. MTS Criterion Compression Testing of Foam Plug in Hastelloy C-22 Pipes

4.2 Evaluation of the adhesion and bonding properties of the FoamBag™ foam plug to Hastelloy C-22 piping under various moisture conditions.

The piping system within F/H labs was previously used for the transport of radioactive waste-bearing liquids. Residual moisture may be present within the Hastelloy C-22 piping when the FoamBag™ foam is being injected into the pipes. It is important to understand the impact that residual moisture will have on the adhesion of the FoamBag™ foam to the inner wall of the pipes. To evaluate this, FIU will saturate 14" segments of 3" Hastelloy C-22 pipe using two different scenarios: (a) spray a known amount of water uniformly to ensure an even coating of liquid around the entire surface area of the inner pipe wall, and (b) fill with liquid at the bottom of the pipe up to 1/8th, 1/16th, and 1/32nd inch depths (Figure 4-2). The pipe will have a temporary cap at one end and the FoamBag™ foam will be injected into the pipe at the other end. If the FoamBag™ foam properly cures, the temporary cap will be removed, as well as excess liquid. The adhesion test will be conducted in the same manner as the adhesion testing in section 4.1 using the MTS Criterion series 43 Tensile Tester with compression plates.

Figure 4-2. Impact of Moisture on Foam Stability in Hastelloy C-22 Pipes. Left photo shows the addition of residual moisture into the pipe. Right photo shows the formation of empty pores within foam due to the presence of moisture in the pipe.

4.3 <u>Determination of the heat profile of FoamBag™ foam during curing in Hastelloy C-22 piping.</u>

Section 7.10 of *ASTM E3191-18: Standard specification for permanent foaming fixatives used to mitigate spread of radioactive contamination* (ASTM, 2018) states that "the foaming fixative shall not generate heat sufficient to compromise any of the components within the enclosure to which it is applied". Evaluation of the temperature profile of the curing process of the FoamBag™ in Hastelloy C-22 piping on a larger sample size will be performed using the Extech SDL200 datalogger and Extech TP870 Type K thermocouples (Figure 4-3). These thermocouples will be calibrated by a certified calibration facility. The monitoring time of the FoamBag™ foam and the temperature needed to be reached for safe handling by workers will be determined by SRNL, FIU, and F/H labs during testing. Site personnel have also expressed interest in capturing the temperature profile of the outside of the Hastelloy C-22 piping during the curing process to further ensure worker safety. Testing protocols developed by FIU using a highly sensitive FLIR E53 thermal imaging camera (Figure 4-3) will be leveraged to achieve this objective. The FLIR E53 has the capacity to measure object temperatures up to 1200°F, has a thermal sensitivity of < 0.07°F (40 mK), and a measurement accuracy of ± 2%. The supplemental FLIR software provides insight in terms of temperature data analysis. Multi-spectral Dynamic imaging (MSX) mode will be used for all image analysis. MSX mode overlays both the thermal and digital images together and provides a more detailed thermal image.

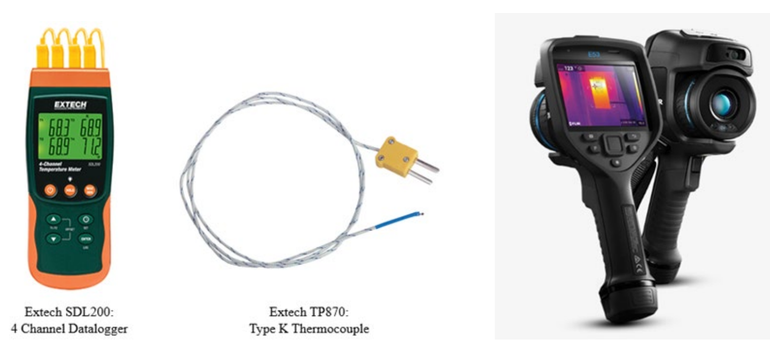


Figure 4-3. Extech Thermocouple Equipment and FLIR E53 Thermal Imaging Camera

4.4 Determination of the internal pipe pressure after FoamBag™ foam deployment and curing

During Phase II testing it is important to understand if FoamBag™ foam plugs will create internal pipe pressure in the void space between two foam plugs, as pipe segments will be capped by two foam plugs. The initial assessment is that the first time we inject FoamBag™ foam plug into piping there will be no pressure on either side of the plug so the foam will expand uniformly in both directions. We anticipate that for the second plug, the expanding foam will push air and off-gases against the first plug during curing, which may pressurize the sealed pipe segment. This research objective will determine how much internal pressure is formed inside 5-foot-long clear piping with a diameter of 2-3 inches. A pressure sensor will be placed within the pipe before the FoamBag™ foams are injected and the pressure will be monitored for 24 hours. (Figure 4-4). SRS Manual 1S LLW WAC section 5.3 identifies the maximum amount of allowable pressure within a pressurized container to be 1.5 atmospheres at 20°C. This manual criterion will be used as a pass/fail criterion for this objective. Previous studies with Hilti foam found the internal pipe pressure to be below this limit.

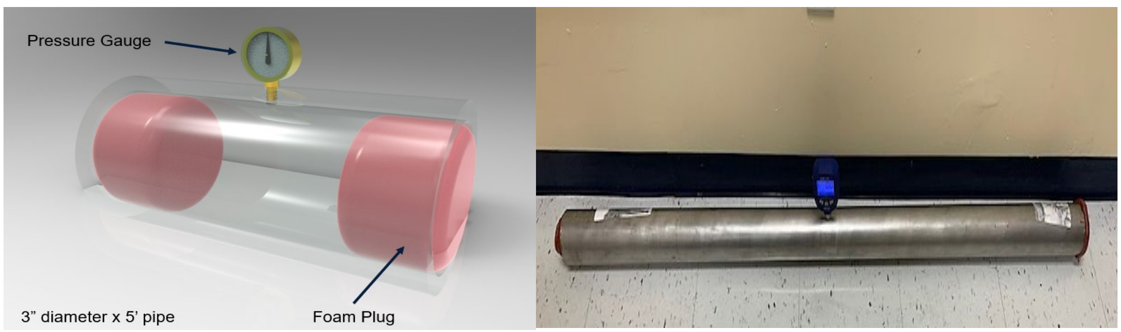


Figure 4-4. Determining Internal Pipe Pressure Between Foam Injection Points. The left picture shows a schematic for the pipe pressure test. The right figure shows the Hastelloy C-22 pipe fit with a pressure gauge prior to testing.

4.5 Determination of the effectiveness of FoamBag™ using a standard leak test

During Phase I testing, the Cold Test Team developed a consensus-based standard operating procedure (SOP) in consult with the respective stakeholders (site personnel, SRNL, DOE-EM, F/H labs, FIU, EH&S, etc.). The developed water leak SOP will be used in Phase II to evaluate the effectiveness of the FoamBag™ foam plug after injection and curing. Leak testing on the FoamBag™ foam plug in Hastelloy C-22 piping will be performed in accordance with *Engineering Standard 15889: Confinement Ventilation Systems Design Criteria, Section 5.2.1.1* (SRS, 2009), which states that the pass/fail criterion for leak tests should be comparable to solids/liquids applied to a cell or glovebox. The expected maximum permissible leak rate is expected to be 0.1% of pipe volume per hour at a differential pressure of 2 inches water column (2” WC). A fluorescent dye will be used under UV light to assess if water leaks through the foam-pipe seal (Figure 4-5).

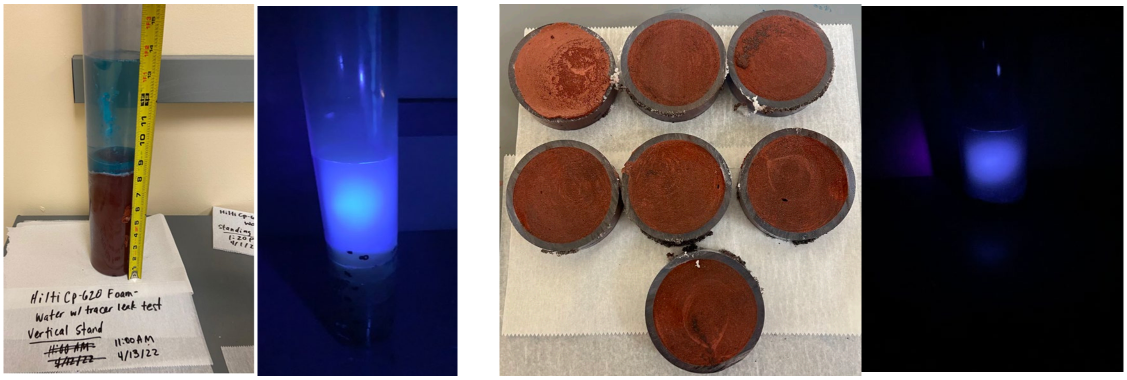


Figure 4-5. Water Leak Test and Fluorescent Dye Tracking for Foams. The left figure shows the leak test with fluorescent-dyed standing water. The right figure shows the cut foam segments after the leak test, to determine if fluorescent dye leaked through the foam.

4.6 Evaluation of off-gas formed during FoamBag™ curing

Headspace testing will be conducted on FoamBag™ during curing. To determine the safety factor for flammable and/or noxious emissions of FoamBag™ fixative foams, the headspace above the FoamBag™ will be measured using gas chromatography-mass spectroscopy (GC-MS) to test whether volatile organic compounds (VOCs) are off-gassed during or after the curing process. The FoamBag™ foam will be dispensed into 150 mL Wheaton bottles, lightly stirred and crimp sealed with a septum top. The curing time of the FoamBag™ foam is approximately 30 minutes. GC-MS samples will be taken after the FoamBag™ foam cures completely (Figure 4-6). The SOP for this experiment will follow the procedures outlined in SRNL-L2100-2018-00072 (SRNL, 2018). The outcomes from this test will be evaluated by SRNL, SRS F/H labs, and FIU and a pass/fail criteria will be determined.

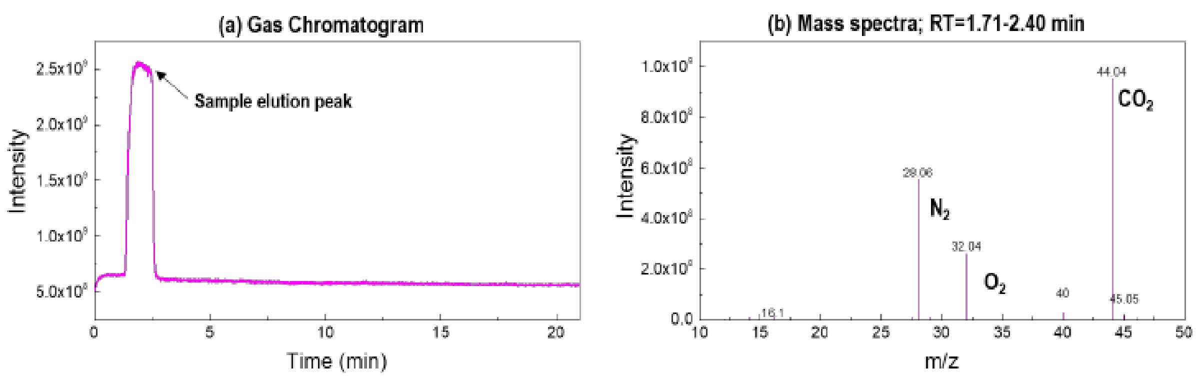


Figure 4-6. Gas chromatograms and mass spectra for foam after curing

4.7 Evaluation of the impacts of temperature and humidity on FoamBag™ stability during environmental chamber testing

The environmental chamber testing component of the Phase II cold test plan project relates directly to DOE-EM's deactivation objectives by testing commercial-off-the-shelf (COTS) foams for the potential leveraging of those products for D&D activities. There are currently no regulatory guidelines to accredit foam fixative technology and understanding how readily available foams will behave in various conditions is the first step in answering the question of efficacy and applicability of these foams. The FoamBag™ fixative foam will be evaluated for its ability to set, become dust-free, and dry to touch per ASTM D1640 specifications (ASTM, 2003) in different environmental conditions (temp, humidity). This test will also determine the percent mass gained or lost by absorption of water from the air over a 24-hour period. This will help with choosing appropriate foams for D&D processes based on typical environmental conditions found at DOE-EM sites throughout the U.S. The FoamBag™ foam will be tested at various temperatures (each with 40% relative humidity) and humidity (each at 25°C) to infer how these foams will behave at specific sites under varying environmental conditions (Figure 4-7). The various temperatures and humidities will range between high humidity, high temperature (indicative of South Carolina summer) and low humidity, low temperature (indicative of Nevada winter). The SOP for this experiment will follow the procedures outlined in SRNL-STI-2022-00004 (SRNL, 2022).

Figure 4-7. Environmental Chamber Testing of Foams. The left figure shows FlexFoam from the initial screening of foams prior to Phase I testing. The figure on the right shows the environmental chamber used in the initial screening test, populated with the four tested foams (Hilti CPw-620, FlexFoam 7, FlexFoam 14, FlexFoam 23).

4.8 Evaluation of resistance to thermal stressors

Though not specifically developed to test and evaluate polyurethane foams and their resistance to thermal stressors, the IEC 60695-11-10 Flammability Standard (IEC, 2013) has been identified as a potential "near fit / best fit" standard after extensive discussions between FIU and SRNL researchers. The standard specifies small-scale laboratory test procedures intended to compare the burning behavior of plastics and foams when vertically or horizontally oriented test bar specimens are exposed to a flame ignition source (Figure 4-8). The objectives of the Phase II thermal stressor test are to conduct a modified version of the IEC 60695-11-10 Flammability Test (both horizontal and vertical methods), a 30-minute direct exposure test of the FoamBag™ to a 2000° propane flame, and a mass loss test through exposure to incremental temperatures in a muffle furnace. The outcomes from this test will be evaluated by SRNL, SRS F/H labs, and FIU and a pass/fail criteria will be determined.

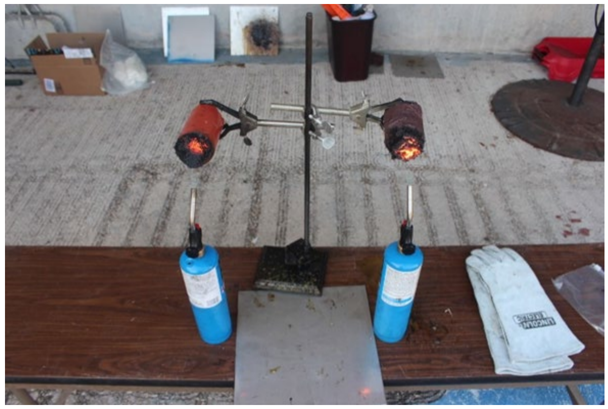


Figure 4-8. Foam Flammability Testing using Propane Flame

5.0 Outcome

The result of these experiments will determine whether the FoamBag™ foam is capable of being deployed in non-ideal environments and quantify the impact on foam curing as well as post-curing performance. The results will also dictate if FoamBag™ is capable of moving to Phase III testing in a dry-run mock-up cold test demo at FIU. Results will be disseminated through a formal technical report at the end of the activity.

6.0 Schedule

Table 6-1. Cold Test Plan Phase II Activity Schedule

TASKS	RESPONSIBLE PARTY	ESTIMATED COMPLETION
Evaluate adhesion of FoamBag™ Foam to Piping	FIU	Mar 2023
Evaluate moisture impacts on FoamBag™ adhesion	FIU	May 2023
Determine heat profile of FoamBag™ during curing	FIU	April 2023
Determine internal pipe pressure after FoamBag™ curing	FIU	Sep 2023
Conduct leak test procedure for FoamBag™ plug	FIU	June 2023
Evaluation of FoamBag™ resistance to thermal stressors	FIU	May 2023
Evaluation of off-gas formed during FoamBag™ curing	SRNL	July 2023
Evaluation of environmental chamber testing for FoamBag™	SRNL	July 2023
Report preparation	FIU / SRNL	Aug 2023
Review and signatures obtained	FIU / SRNL	Aug 2023
Report submission	FIU / SRNL	Sep 2023

7.0 References

[1] ASTM Standard D1621-16, "Standard Test Method for Compressive Properties of Rigid Cellular Plastics", Book of Standards Volume 08.01, ASTM International, West Conshohocken, PA, 2016.

[2] ASTM Standard E3191-18, "Standard Specification for Permanent Foaming Fixatives Used to Mitigate Spread of Radioactive Contamination", Book of Standards Volume 12.02, ASTM International, West Conshohocken, PA, 2018.

[3] WSRC-TM-95-1, *SRS Engineering Standards Manual,* Standard 15889, Confinement Ventilation Systems Design Criteria, 2009.

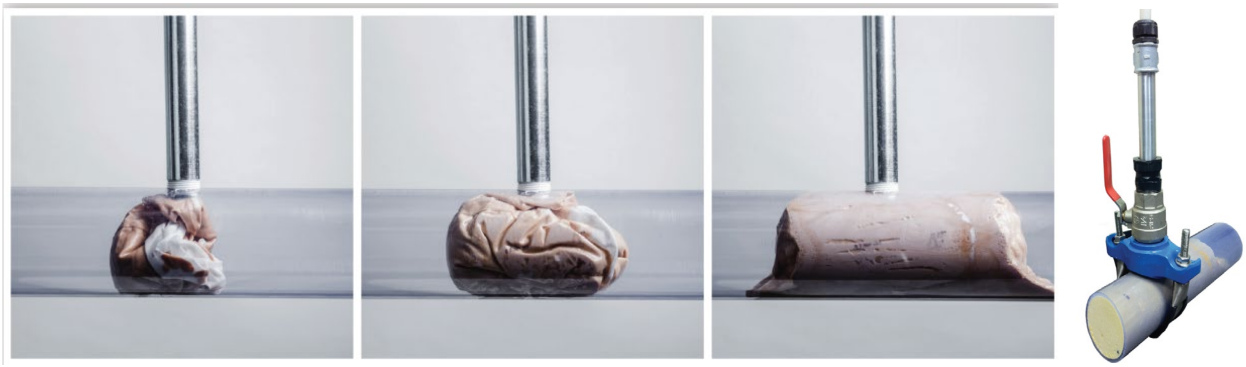
[4] SRNL-L2100-2018-00072, Headspace Analysis of Hilti Foam, 2018.

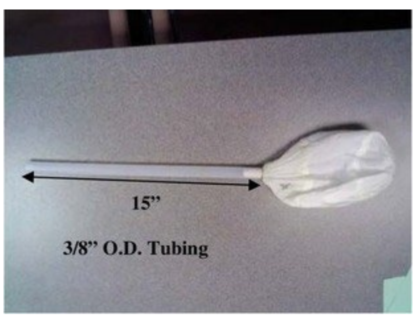
[5] SRNL-STI-2022-00004, Permanent Foam Fixatives for Radioactive Contamination Control: Environmental Chamber Testing, 2022.

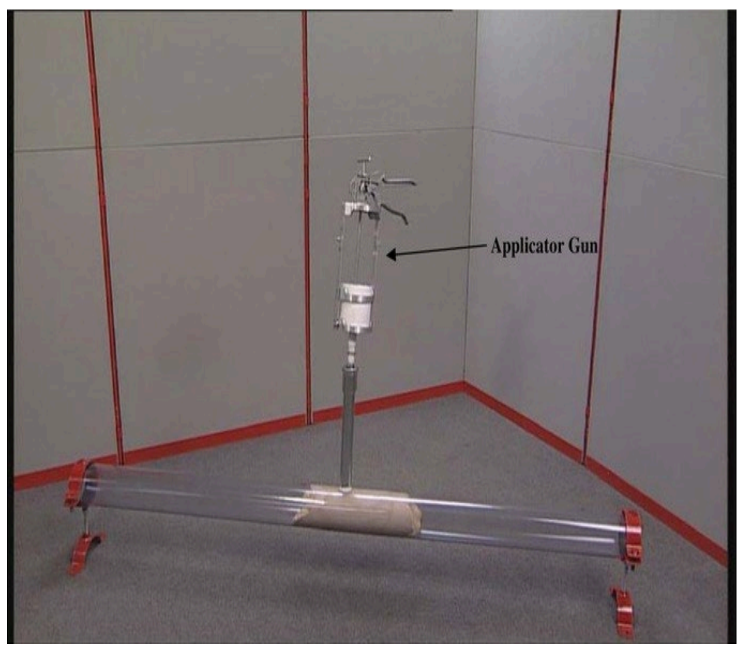
[6] IEC Standard 60695-11-10, 2013, "Fire hazard testing – Test Flames – 50 W Horizontal and Vertical Flame Test Methods". International Electrotechnical Commission, www.webstore.iec.ch/publication/2938

Appendix A.

The FoamBag™ technique has been in use in the UK in gloveboxes at Sellafield and meets the UK gas industry technical standard T/SP/E/59. The FoamBag™ holds resin foam in place as it expands. At full expansion, the foam seeps through the semi-porous panels of the bag to form an adhesive seal with the pipe. Information related to the FoamBag™ can be found in the FoamBag™ product sheet and brochure that are attached with the cold test plan document.







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