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Radiation Hardened Foam Cold Test Plan – *Phase-I: Foam Adhesion, Contamination Fixation, Moisture Stresses, Pipe Cutting, and Thermal Profile Testing*

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LIST OF ABBREVIATIONS

SRNL	Savannah River National Laboratory
SRS	Savannah River Site
FIU	Florida International University
D&D	Deactivation and Decommissioning
2” WC	Two Inches Water Column (unit for pressure)
DOE-EM	Department of Energy Office of Environmental Management

1.0 Purpose

This document outlines the Phase-I test objectives and implementation plan for a down-selective 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 an intumescent, fire-retardant 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 in FIU's Outdoor Test and Evaluation Facility using a mock-up that replicates the operational conditions at the proposed hot test location at F/H labs. Results from the cold test plan 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 site and transported to a proper disposal facility. Phase-I testing will address eight test objectives: (1) evaluation of the adhesion and bonding properties of foam fixative in piping, (2) evaluation of the adhesion of the foam fixative in piping under varying moisture conditions, (3) determination of the heat profile of the foam fixative during curing, (4) determination of the relationship between pipe diameter and foam fixative quantity, (5) determination of the internal pipe pressure after foam deployment and curing, (6) development of a leak test standard operating procedure to test for the effectiveness of the foam plug, (7) initiation of a literature review to determine if using a hot tap is a viable method to deliver foam into piping, and (8) initiation of a 10-foot mock up test that will be used in Phase-II cold testing. The cold test will be conducted at FIU and all testing activities will comply with SRNL Conduct of R&D Protocols (SRNL-IM-2020-00019).

2.0 Background

Intumescent foam technologies are currently being tested at SRNL and FIU to support 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 intumescent foams has the potential to reduce the number of engineering/administrative controls currently needed to remove contaminated piping systems for disposal. Previous testing demonstrated that degradation of intumescent foam was possible at elevated temperatures and/or high humidities along with other external factors.[1-3] The cold test objectives will be conducted in open air conditions at FIU to determine if intumescent foam can be a reliable technology to mitigate the release of residual contamination in piping systems during D&D activities. The intumescent foam used in the cold test will be Hilti Firestop Foam CP-620. This research is funded as part of FIU-DOE Cooperative Agreement (Contract # DE-EM0000598).

3.0 Goal and Objectives

3.1 Goal

The purpose of Phase-I testing is to assess the mechanical properties of Hilti CP-620 commercial intumescent foam under select stressors common to a real-world operational environment and determine the suitability of Hilti CP-620 as a foam fixative that can be used to mitigate/reduce the release of residual contamination during D&D activities being planned at F/H labs. This testing is a necessary precursor to large-scale dry run/mock-up testing that will be conducted in Phase-II. Photos, illustrations, and characteristics of the proposed hot-test site location will be provided in the Phase-II cold test plan. All Phase-I testing will be done at FIU.

3.2 Objectives

Phase-I testing is being performed to characterize the mechanical properties of Hilti CP-620 intumescent foam when exposed to various stressors that may impact its effectiveness as a 3D fixative in Hastelloy C-22 pipes. Specifically, testing will address the following:

- Evaluation of the adhesion and bonding properties of the Hilti CP-620 foam plug to Hastelloy C-22 piping.
- Evaluation of the adhesion and bonding properties of the Hilti CP-620 foam plug to Hastelloy C-22 piping under various moisture conditions.
- Determination of the heat profile of Hilti CP-620 foam during curing in Hastelloy C-22 piping.
- Establish the relationship between piping diameter and necessary quantity of Hilti CP-620 foam.
- Determine the internal pipe pressure after Hilti CP-620 foam deployment and curing.
- Develop a leak test standard operating procedure to test for the effectiveness of the Hilti CP-620 foam plug
- Conduct a literature review to determine if using a hot tap is a viable method to deliver Hilti CP-620 foam into piping
- Gather information and reference material to initiate the construction of the mock-up test of the F/H labs courtyard.

4.0 Test Parameters

4.1 Evaluation of the adhesion and bonding properties of the Hilti CP-620 foam plug to Hastelloy C-22 piping

Adhesion capabilities of the Hilti CP-620 to Hastelloy C-22 piping will ultimately decide whether an intumescent foam fixative plug can 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 Hilti CP-620 foam to delaminate from Hastelloy C-22 piping, causing the release of contamination. The tensile adhesion, compressive and shear properties of the Hilti CP-620 foam itself are well understood and have been baselined by FIU and SRNL in accordance with *ASTM D1623: Test method for tensile adhesion properties of rigid cellular plastics*[4], *ASTM D1621: Standard Test Method for Compressive Properties of Rigid Cellular Plastics,* and *ASTM D3574 E Foam Tension Testing*. Furthermore, FIU and SRNL previously developed a series of testing practices specifically designed to evaluate the Hilti foam's ability to perform and function as a permanent plug in a pipe under normal operating conditions and when exposed to a variety of environmental and impact stressors. Using the MTS Criterion series 43 Tensile Tester with compression plates, the amount of force required to push a Hilti CP-620 foam plug out of a 3" D x 14" L, 304 stainless steel pipe segment was determined (Figure 7-1). Additional Hilti CP-620 foam samples were subjected to a variety of stressors, including water submersion (Figure 7-2) and drop testing (Figure 7-3). Results from these tests can be seen in Figure 7-4. The same battery of tests will be conducted on the Hilti CP-620 foam plugs in Hastelloy C-22 pipes, and the data will be compared with that obtained from the 304 stainless steel pipes to ascertain any differences in adhesion and bonding properties between the two pipe materials. After the testing is complete, collaborators from SRNL, FIU, and F/H labs will discuss the results and determine if they meet acceptance criteria for progressing the Hilti foam to Phase-II testing.

4.2 Evaluation of the adhesion and bonding properties of the Hilti CP-620 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 Hilti CP-620 foam is being injected into the pipes. It is important to understand the impact that residual moisture will have on the adhesion of the Hilti CP-620 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 liquid 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. The pipe will have a temporary cap at one end and the Hilti CP-620 foam will be injected into the pipe at the other end (Appendix A). If the Hilti CP-620 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. After the testing is complete, collaborators from SRNL, FIU, and F/H labs will discuss the results and determine if they meet acceptance criteria for progressing the Hilti foam to Phase-II testing.

4.3 Determination of the heat profile of Hilti CP-620 foam during curing in Hastelloy C-22 piping.

Section 7.10 of *ASTM E3191-18: Standard specification for permanent foaming fixatives used to mitigate spread of radioactive contamination*[5] states that "the foaming fixative shall not generate heat sufficient to compromise any of the components within the enclosure to which it is applied". Based on initial data from a series of experiments done at FIU, the curing temperature profile of Hilti CP-620 foam when applied in 1.5-inch diameter 304 stainless steel piping reached a maximum temperature range of 255.6°F to 276.0°F. Confirmation of the temperature profile of the curing process of the Hilti foam in Hastelloy C-22 piping on a larger sample size will be performed (Figure 7-5) using the Extech SDL200 datalogger and Extech TP870 Type K thermocouples (Figure 4-1). These thermocouples will be calibrated by a certified calibration facility. A similar experimental design as highlighted in FIU's 2019 Technical Progress Report titled "Intumescent Foams in Operational Scenarios"[6] will be used (Appendix B), and the heat profile (Figure 7-6) of the Hilti CP-620 foam curing over time will be recorded until the cured foam has cooled to a temperature at which the pipe can be safely handled by workers (Figure 7-7). The monitoring time of the Hilti CP-620 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.

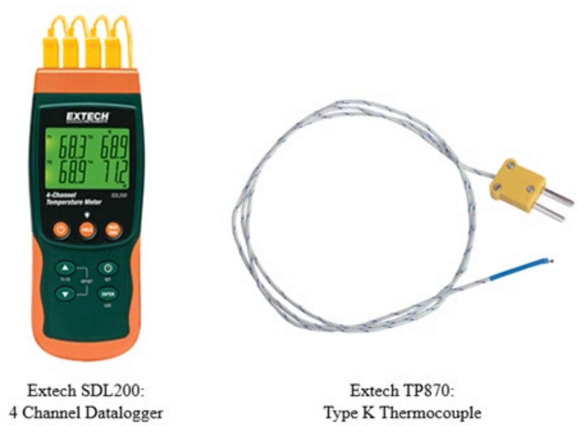

Figure 4-1. Extech Equipment.

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 4-2) 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 4-2. FLIR E53 Thermal Imaging Camera.

4.4 <u>Establish the relationship between piping diameter and necessary quantity of Hilti CP-620 foam.</u>

The Hastelloy C-22 piping used in the F/H labs courtyard has a diameter of both 2 and 3 inches. The variability in core piping diameter may influence the amount of intumescent foam that is needed to ensure a tight and secure seal plug. Previous studies conducted at FIU[6] have determined that in a 3" diameter pipe, one application of intumescent foam from a 110 in^3 canister can achieve 14 – 16 inches of plug. For a 2" diameter pipe, this distance expands to 2 – 3 feet. Additional studies may be conducted to look at various piping diameters (e.g., ½" to 5") to assess the plug size using one cannister of foam. After the foam has been injected into the piping and if it is cured properly, the piping will be cut at a fixed distance on both sides of the Hilti foam injection point and will be assessed to determine if a complete seal has been made.

4.5 <u>Determine the internal pipe pressure after Hilti CP-620 foam deployment and curing time.</u>

During Phase-I testing it is important to understand if Hilti CP-620 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 Hilti CP-620 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 Hilti CP-620 foams are injected and the pressure will be monitored for 24 hours. 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.

4.6 <u>Develop a leak test standard operating procedure to test for the effectiveness of the Hilti CP-620 foam plug</u>

During Phase-I, the Cold Test Team will develop 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 Hilti CP-620 foam plug after injection and curing. Leak testing on the Hilti CP-620 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*, 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).

Upon completion, the water leak test SOP developed by the Cold Test Team will be submitted for review as a formal testing practice to ASTM International's E10 Committee on Nuclear Technology and

Applications and its associated E10.03 Subcommittee on Radiological Protection for Decontamination and Decommissioning of Nuclear Facilities and Components. This will allow for the Cold Test Team to codify the leak test experimental design as an international standard for this technology.

4.7 Conduct a literature review to determine if using a hot tap is a viable method to deliver Hilti CP-620 foam into piping.

The Savannah River Site (SRS) has routinely used hot taps to make connections to existing piping. The hot tap method has been demonstrated for both stainless steel piping and carbon alloy piping; however, it is unknown if the standard hot tap method will be effective at (a) tapping into Hastelloy C-22 piping and (b) if the Hilti CP-620 foam can be injected into Hastelloy C-22 piping using a hot tap. This literature review will assess previous uses of hot taps on Hastelloy C-22 piping. The information will be compiled together and SRNL, FIU, and F/H labs will develop a Phase-II test plan to test for the implementation of Hilti CP-620 foam using hot taps in a mock-up facility at FIU.

4.8 Gather information and reference material to initiate the construction of the mock-up test of the F/H labs courtyard.

If the previous objectives have passed their pass/fail criteria (where applicable), FIU will begin construction of a mock-up test which will mirror the anticipated hot-test location at the F-area courtyard between buildings 772-F and 772-1F. During Phase-I testing, SRNL, FIU, and F/H labs will work to gather information and reference material (e.g., pictures, plans, processing history) of the hot-test location that will provide site information for the cold test at FIU during Phase-II testing. Results of this information gathering endeavor will be included in the Phase-II test plan.

5.0 Outcome

The result of these experiments will determine whether the Hilti CP-620 foam is capable of being deployed in non-ideal environments and quantify the impact on foam curing as well as post-curing performance. The Cold Test Team has received approval to use BOX software licenses as part of the early adopter program as a platform to upload project content. BOX is an online-based file sharing software which will be used to share/disseminate information between members of the Cold Test Team. Results will be disseminated through a formal technical report at the end of the activity.

6.0 Schedule

Table 6-1. Cold Test Phase-I Activity Schedule

TASKS	RESPONSIBLE PARTY	ESTIMATED COMPLETION
Order and shipment of supplies	FIU	Feb. 2022
Evaluate adhesion of Hilti Foam to Piping	FIU	Mar. 2022
Evaluate moisture impacts on Hilti Foam adhesion	FIU	July 2022
Determine heat profile of Hilti Foam during curing	FIU	Mar. 2022
Determine relationship between piping diameter & Hilti foam quantities	FIU	July 2022
Determine internal pipe pressure after Hilti foam curing	FIU	July 2022
Develop leak test procedure for Hilti foam plug	FIU / SRNL	Ongoing
Conduct literature review for hot tap delivery methods of Hilti foam	FIU / SRNL	Ongoing
Gather information and resources for cold test mockup at FIU	SRNL	Ongoing
Report preparation	FIU / SRNL	July 2022
Review and signatures obtained	FIU / SRNL	Sep. 2022
Report submission	FIU / SRNL	Sep. 2022

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5. ASTM Standard E3191-18, "Standard Specification for Permanent Foaming Fixatives Used to Mitigate Spread of Radioactive Contamination", ASTM International, West Conshohocken, PA.

6. Sinicrope, J., Komninakis, M., Donoclift, T., Lagos, L., Nicholson, J., and Washington, A. (2019). "Intumescent Foams in Operational Scenarios – Phase I", Technical Progress Report, Florida International University.

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Appendix A. Experimental Set-up for Adhesion, Bonding and Plug Strength of Hilti CP-620 Foam in Hastelloy C-22 pipes under Normal Operating Conditions and When Exposed to Stressors

As outlined under test objective 4.1 and 4.2, confirmation of the adhesion and bonding characteristics of the Hilti CP-620 foam on Hastelloy C-22 piping is required. This analysis is desired for the foam under normal operating conditions as well as when the foam plug is exposed to various environmental and impact stressors postulated in NRC 10 CFR 71.83. The initial portion of this test will be conducted simulating normal / ideal operating conditions to establish a baseline. A Hilti foam plug will be applied to three separate 14” lengths of 3” Hastelloy C-22, Schedule 40 pipe segments and allowed to cure. Then, using the MTS Criterion series 43 Tensile Tester (Figure 7-1) with compression plates, the amount of force required to push the foam plug out of each sample size will be determined. These results will be compared with those obtained during the same series of tests on the 304 stainless steel pipes as outlined in the Technical Progress Report.[6]

Figure A-1. These photos depict the process developed by FIU and SRNL and conducted by FIU on Hilti CP-620 foam plugs in 304 stainless steel. This Cold Test Plan outlines a similar experiment using Hastelloy C-22, Schedule 40 pipes.

Once a baseline has been established for the plugs under ideal conditions, additional samples of the same dimensions will be made and subjected to an environmental stressor (water immersion) and impact stressor (drop tests). For the water immersion test, the Hastelloy C-22, Schedule 40 pipes with Hilti CP-620 foam plugs will be submerged at a depth of 3’ in water for a period of 8, 12, and 24 hours, respectively (Figure 7-2). At the conclusion of each time period, the 3 samples will be extracted and tested using the MTS Criterion series 43 Tensile Tester with compression plates via the same methodology outlined above. For the drop test, samples will be made and dropped from heights of 4’, 8’ and 12’, respectively. Again, at the conclusion of each height, the 3 samples will be tested using the MTS Criterion series 43 Tensile Tester with compression plates via the same methodology.

Figure A-2. Depiction of 3 x Hilti plugs in 304 stainless steel submerged in 3' of water for 12 hours before being tested using the MTS.

Figure A-3. Hilti plugs in 304 stainless steel drop tested at designated heights before using the MTS.

Finally, as mentioned in the main text of the test plan, similar procedures will be utilized to evaluate how well the plug cures and holds when applied to moisture. All data will be collected and compared to results obtained when the plugs were tested under similar conditions in 304 stainless steel pipes.

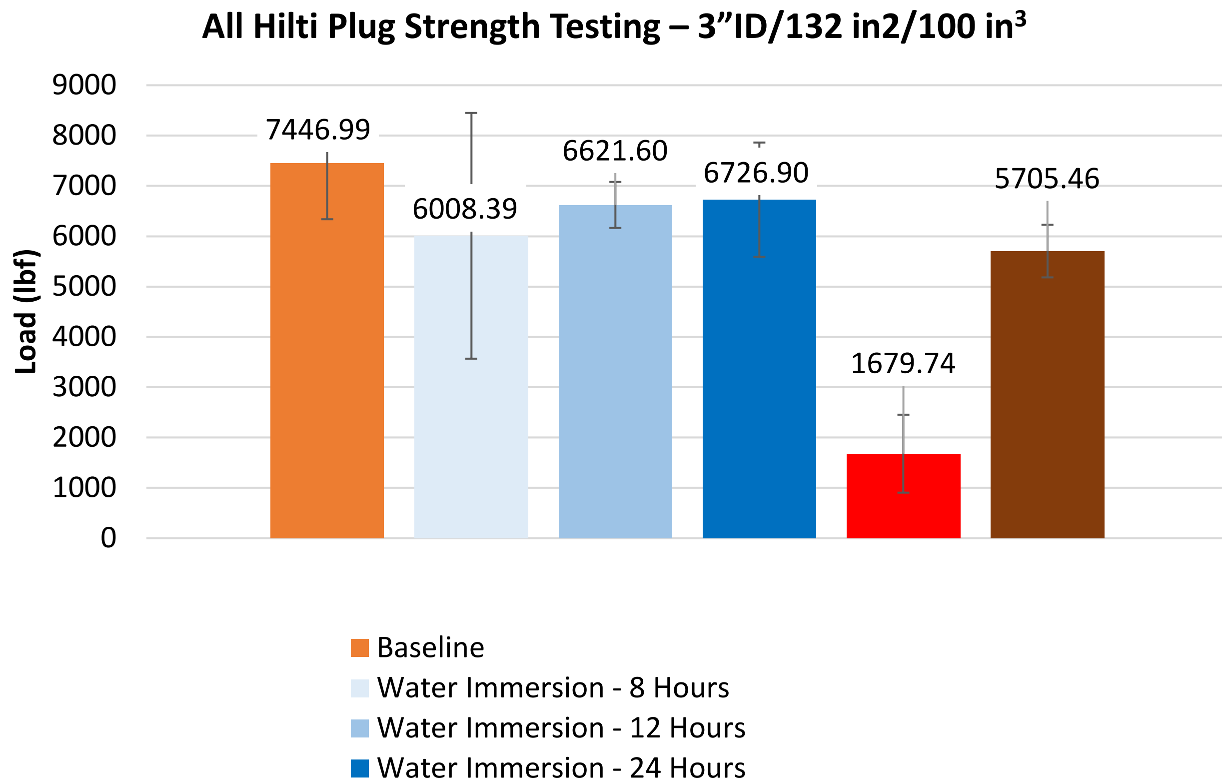

Figure A-4. Sample table of results from similar testing protocols for Hilti plugs in 304 stainless steel pipes.

Appendix B. Brief Description of Experimental Set-up for Curing Temperature Profile at FIU

As outlined under test objective 4.3, confirmation of the temperature profile of the curing process of the Hilti foam internal to the Hastelloy C-22 piping is required. This will be performed using the Extech SDL200 datalogger and Extech TP870 Type K thermocouples. The thermocouples will be placed at varying depths throughout the pipe segment, and then the foam will be applied in the pipe. Data measurements will be obtained over a 2-hour period and graphed.

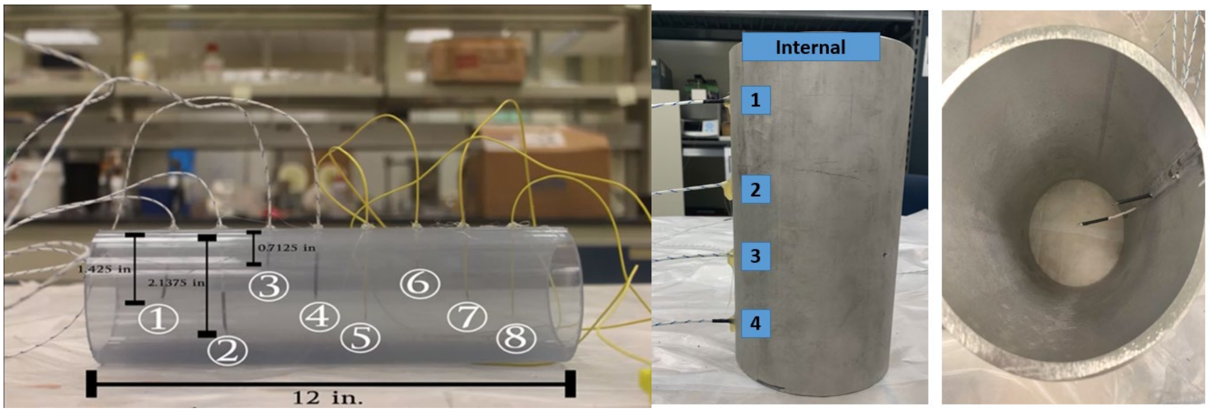

Figure B-1. Sample placement of Extech thermocouples at varying depths. Left photo uses clear PVC for ease of depiction, and middle and right photos demonstrate sample placement after holes are drilled into 304 SS pipe. Actual experiment will be using Hastelloy C-22, Schedule 40 pipes.

To obtain the temperature profile of the external area of the pipe a FLIR E53 thermal imaging system will be used. Simultaneous to the readings being obtained by the thermocouples internal to the pipe, the thermal imager will collect temperature data on the external components of the pipe over the same 2-hour period and graphed.

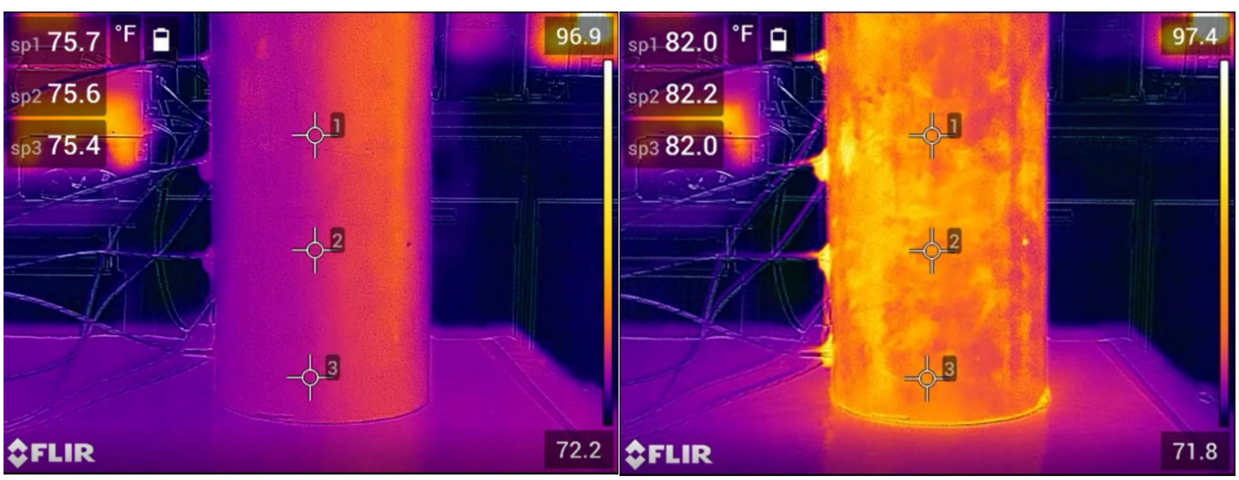

Figure B-2. Thermal image of 304 SS pipe before foam application (left) and during the curing process (right) using the FLIR E53 thermal imaging camera system.

A graphed curing temperature profile over a 2-hour period will be created from the data for both the internal and external areas of the Hastelloy C-22 pipes and be compared with those obtained from the 304 SS pipes.

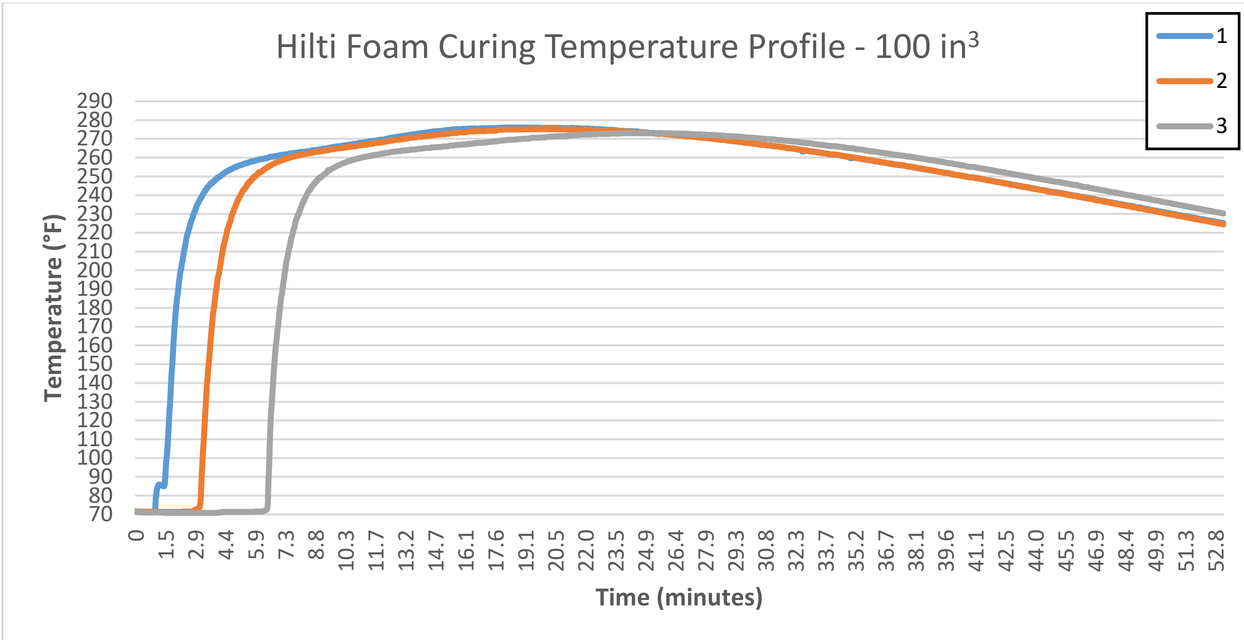

Figure B-3. Hilti CP-620 foam curing temperature profile obtained from 304 SS piping. The study was done in triplicate. Each line represents the heat profile of one cartridge of Hilti CP-620 foam.

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