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# Permanent Foam Fixatives for Radioactive Contamination Control: Environmental Chamber Testing

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- J. D. Sinicrope
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- R. Walker
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February 2022

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#### **EXECUTIVE SUMMARY**

The Department of Energy (DOE) Office of Environmental Management (EM) is tasked with closing cleanup sites throughout the DOE-EM complex. A major component of successfully closing the sites is the deactivation and decommissioning (D&D) of radioactively contaminated buildings in nuclear facilities. Deactivation consists of the process of placing a contaminated facility in stable condition in order to minimize any risks that could affect workers, the public, and the environment. Successful deactivation and decommissioning consists of leaving the buildings in an agreed upon end state to provide future protection against hazards. Hazards include radiation but can also include asbestos, polychlorinated biphenyls, and other environmental and physical hazards.

Discussions with representatives from the Department of Energy's Office of Environmental Management (DOE-EM) and sites across the complex have identified an operational requirement for a fixative technology to immobilize and/or isolate residual contamination within a 3-dimensional void space of various volumes that might result from anticipated to unanticipated events. Potential applications for a light weight, rigid, permanent foam fixative technology abound to include supporting decommissioning activities related to gloveboxes, heating ventilation and air conditioning systems, etc. In particular, the decommissioning of contaminated pipework on legacy nuclear sites remains a challenging process. A technology that can establish an internal barrier or "plug" to mitigate the release of residual contamination prior to cutting operations of contaminated pipework, would bring several benefits to decommissioning efforts. The potential for commercial-off-the-shelf (COTS) polyurethane foams to meet this requirement and support deactivation and decommisioning activities is being investigated through a collaborative effort by SRNL and FIU. Foams can encapsulate large irregular void volumes whereas coatings can be difficult to apply in these scenarios.

Phase I of this research activity involves the down selection of a COTS foam technology based upon mechanical, chemical, environmental, and performance properties outlined in ASTM E3191-18, Standard Specification for Permanent Foam Fixatives, specifically developed by the ASTM International E10.03 Subcommittee to support this broader activity. Building upon parallel work conducted by Florida International University (FIU) in its mechanical property testing of foam technologies, Savannah River National Laboratory (SRNL) tested four foams to better understand how variation in temperature and relative humidity affects the time it takes to achieve set-to-touch, dust-free, and dry-to-touch conditions. Additionally, water uptake was measured to determine potential negative effects due to increased relative humidity.

The first foam tested was Firestop Foam CP 620 by Hilti, Inc., herein referred to simply as Hilti, which was determined to be the ideal foam after FIU's rigorous testing of adhesion capabilities and mechanical properties. Hilti consistently passed set-to-touch, dust-free, and dry-to-touch tests within 60 seconds regardless of temperature or humidity. Cross-sections of four Hilti foams showed consistency in application and drying time throughout (e.g. similar pores, no large voids). Hilti absorbed more water than the other foams (11 out of 16 samples gained mass), but the percent uptake was low and ranged from 0.04% to 0.26% mass gained.

The other three foams that were tested are two-part polyurethane flexible foams procured from Smooth-On, Inc. and were tested alongside Hilti CP 620 Firestop Foam: FlexFoam-iT!<sup>TM</sup> 7 FR, FlexFoam-iT!<sup>TM</sup> 14, and FlexFoam-iT!<sup>TM</sup> 23 FR. The FR accompanying FlexFoam-iT!<sup>TM</sup> 7 and 23 refer to the fire resistant properties.

FlexFoam-iT!<sup>TM</sup> 7 FR is the easiest to mix but took the longest time to achieve the desired characteristics (60 minutes). It also showed some inconsistencies including a hardened, porous top, and the cores seen in

the cross-sections indicate variations in drying. While it is the least expensive, the observation showing an 8x expansion is a cause for concern due to possible pressure increase when applied to a closed system.

FlexFoam-iT!<sup>TM</sup> 14 had the quickest setting, dust-free, and drying times and is easy to mix, but is not fire resistant. The faster setting, dust-free, and drying times is advantageous, but does not overcome its lack of fire resistance. However, testing of FlexFoam-iT!<sup>TM</sup> 14 was still necessary to understand if FlexFoam-iT!<sup>TM</sup> 14 would be superior without flame resistant chemicals.

The final foam, FlexFoam-iT!<sup>TM</sup> 23 FR is the hardest to mix, expands the least, and is very dense, which could be beneficial to decommissioning efforts. However, there were observed inconsistencies in curing when cross sections were observed.

Based on the results of this series of testing, Hilti is the most promising candidate for effective contamination fixation. Hilti has been selected for follow-on testing at a non-radiological mock-up facility at FIU in preparation for a future hot test in the F/H lab courtyard located at the Savannah River Site.

Based on where DOE-EM sites are located, different foams could be selected for optimal use. It is important to identify foams that can be used in multiple applications. Certification of these foams will be required to allow widespread use of these materials in diverse applications throughout the wider DOE-EM complex. These materials will help streamline the decommissioning of buildings and associated infrastructure in a safe and efficient way. This preliminary testing effort was focused on identifying performance under conditions that are present at the SRS and LANL facilities.

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#### LIST OF ABBREVIATIONS

ASTM American Society for Testing and Materials

D&D Deactivation & Decommissioning

DOE Department of Energy

EEC Environmental Evaluation Checklist
ELN Electronic Laboratory Notebook
EM Environmental Management

FIU Florida International University

FR Fire Resistant
HA Hazard Analysis

Hilti CP 620 Firestop Foam

M&TE Measurement and Test Equipment

pbv parts-by-volume pbw parts-by-weight

PPE Personal Protective Equipment

RH Relative Humidity

SRNL Savannah River National Laboratory

#### 1.0 Introduction and Background

The Department of Energy (DOE) Office of Environmental Management (EM) is tasked with closing cleanup sites throughout the DOE-EM complex. A major component of successfully closing the sites is the deactivation and decommissioning (D&D) of previously used and contaminated buildings in nuclear facilities. Deactivation consists of the process of placing a contaminated facility in stable condition in order to minimize any risks that could affect workers, the public, and the environment. Successful deactivation and decommissioning consists of leaving these buildings in agreed upon end states to provide protection against hazards, typically radiation hazards, but also can include asbestos, polychlorinated biphenyls, and other environmental and physical hazards.

The environmental chamber testing component of the Radiation Resistant Foam Environmental Chamber Testing & Material 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 ultimate outcome of the FIU team who routinely work with incombustible fixative technologies is to develop a process in conjunction with the ASTM International E10.03 subcommittee to have a process for accrediting the fixative technologies.

Four foams were selected and tested as foam fixatives for this project. These four COTS foams were evaluated for their ability to set, become dust-free, and dry to touch per ASTM D1640 specifications (ASTM, 2003). This project also determined 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 foams were testing at various temperatures (each with 40% relative humidity [RH]) and humidity (each at 25°C) to infer how these foams will behave at specific sites under varying environmental conditions.

The objective of the environmental chamber portion of this project was to assess the impact of various environmental conditions on the curing behavior of foams selected for D&D activities. OTS polyurethane foams were selected based on relevant data collected by Florida International University (FIU) as well as previous research done at Savannah River National Laboratory (SRNL).

Hilti was investigated previously by FIU (Lagos et al., 2019) due to its resiliency in fire events and other extreme environmental conditions. Based on results from testing performed at FIU, Hilti was the initial front-runner for durability in various temperatures and humidity conditions. In addition to Hilti, three Smooth-On FlexFoam-iT!<sup>TM</sup> variations, two fire resistant and one not, were selected for this testing. SRNL did investigative studies with this set of foams and selected these foams from an extensive literature search focused on fire resistance. Although FlexFoam-iT!<sup>TM</sup> 14 is not fire resistant, it was rated high in previous experiments (Rivas, 2018). Table 1-1 summarizes important characteristics of the selected foams.

Table 1	1_1	Details	of select	ted foams
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Foam	Mixing Details	Manufacturer's Recommended Curing Time	Recommended Product Application Conditions	Approximate Volumetric Expansion	Fire Resistant	Fire-Specific Standards Tested by Manufacturer
Hilti CP 620 Firestop Foam	Mixes while dispensing	< 10 minutes	10°C to 30°C	1.5 X	Yes	UL 1479. ASTM E814, CAN/ULC – S115
FlexFoam- iT!™ 7 FR	1:1 parts by volume	2 hours	23°C < 50% RH	8 X	Yes	FBMSS-302
FlexFoam- iT! <sup>TM</sup> 14	1:2 parts by volume	2 hours	23°C < 50% RH	4 X	No	N/A
FlexFoam- iT! <sup>TM</sup> 23 FR	85:100 parts by weight	2 hours	23°C < 50% RH	2 X	Yes	UL-94 HB

Figure 1-1 displays the range of temperature and humidity combinations tested in the Binder KMF 240 Environmental Chamber (marked with black circles inside transparent black rectangle). These temperature and humidity combinations were selected to monitor trends in changing humidity (with constant temperature) and temperature (with constant relative humidity).

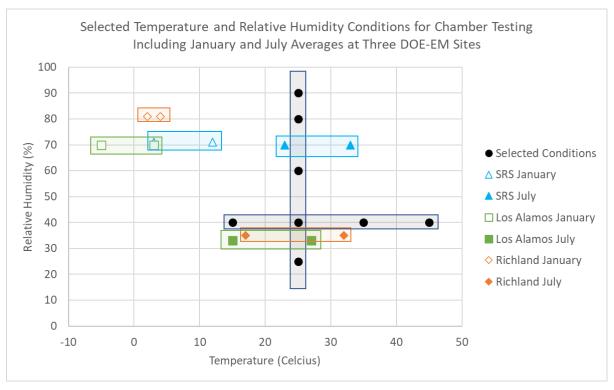


Figure 1-1. Temperature and relative humidity conditions selected for environmental chamber testing

Temperatures and relative humidity vary between DOE-EM sites and understanding how the foams respond to this variation will assist in the selection of ideal foams. For reference, Table 1-2 shows average temperatures and relative humidity at DOE-EM locations where this data can be applied. January and July were selected as they are six months apart and generally good indicators of low and high temperatures. This

has also been incorporated into Figure 1-1. This project was on a constrained timeline and it was not possible to calibrate the combined hygrometer and thermometer device to be confident in recording humidity and temperature values listed for January.

Table 1-2. Average temperature and relative humidity in January and July for three DOE-EM sites (www.weather-us.com, 2021)

City	Site	Average Monthly Temperature Range	Average Relative Humidity
Aiken, SC	Savannah River	January: 3°C to 12°C	January: 71%
Aikeii, SC	Site	July: 23°C to 33°C	July: 70%
Los Alamos,	Los Alamos Site	January: -5°C to 3°C	January: 70%
NM	Los Alaillos Site	July: 15°C to 27°C	July: 33%
Richland, WA	Hanford Site	January: 2°C to 4°C	January: 81%
Kiciliand, WA	Hainord Site	July: 17°C to 32°C	July: 35%

The original intention was to test 5°C and -5°C at 40% RH, but both the calibration of the combined hygrometer/thermometer device and the capabilities of the environmental chamber were unable to accommodate the lower temperatures at any humidity with confidence. While it would have been advantageous to have been able to test temperatures between -5°C and 15°C at a percent RH of 70% to account for January conditions, our equipment was not able to accommodate those combinations so these conditions were dropped from testing, and 25°C and 25% RH was added as the lowest temperature and humidity combination that could be confidently tested.

#### 2.0 Experimental Procedure

The R&D Directions developed at SRNL are based on manufacturer recommended instructions for mixing, curing, and applying foams. The procedure for determining set-to-touch, dust-free, and dry-to-touch have been adapted from the ASTM D1640 Standard for test methods for drying, curing, or film formation of organic coatings at room temperature (ASTM International, 2003).

#### 2.1 Hazard Analysis

The Hazard Analysis (HA), SRNL-HA-01924 titled "Developing and Testing Radiation Resistant High Density Polyurethane Foam" received work authorization approval prior to initiating hands-on lab work.

#### 2.2 Equipment and Materials

- Environmental chamber and associated parts (e.g. hygrometer/thermometer)
- PPE: gloves, safety glasses/goggles, lab coat
- FlexFoam-iT!<sup>TM</sup> 7 FR parts A & B
- FlexFoam-iT!<sup>TM</sup> 14 parts A & B
- FlexFoam-iT!<sup>TM</sup> 23 FR parts A & B
- Hilti CP 620 Firestop Foam and applicator
- Craft sticks for mixing
- Plastic cups (500mL for mixed foams and 250mL for foam parts)
- Syringes for measuring and dispensing parts A and B of FlexFoam-iT!<sup>TM</sup> foams
- Analytical balance and weights for calibration
- Computer for access to electronic lab notebook (ELN)
- Glass slide for determining set to touch time

- Cotton balls for determining dust-free time
- Table covering to avoid spills directly on the lab bench
- Permanent marker to label plastic cups and syringes

#### 2.3 <u>Initial Steps for Consideration</u>

- 1. PPE was properly worn by all hands-on workers (gloves, lab coat, and safety glasses)
- 2. The balance was calibrated with the weight set
- 3. The environmental chamber was set to the desired temperature and humidity conditions

#### 2.4 Preparation of Foams

#### 2.4.1 Preparation to dispense Hilti Firestop Foam CP 620

- 1. Two 500mL containers were labeled with the foam being tested, date, temperature, and relative humidity. Designated each replicate by adding R1 or R2.
- 2. Each cup was weighed, and the mass was recorded on the data sheet or ELN to two significant figures
- 3. A 250mL cup was labeled as "Hilti Prep"
- 4. The Hilti cartridge was put in the dispenser and the lid was removed.
- 5. The mixer was placed on the Hilti tube

#### 2.4.2 Preparation to mix FlexFoam-iT!<sup>TM</sup> 7 FR

- 1. Two 500mL containers were labeled with the foam being tested, date, temperature, and relative humidity. Replicates were designated by adding R1 or R2.
- 2. Each cup was weighed, and the mass was recorded on the data sheet or ELN
- 3. Four syringes without needles were obtained
- 4. The syringes were labeled "7 FR Part A R1–15 mL" and "7 FR Part A R2–15 mL"
- 5. The other two were labeled "7 FR Part B R2–15 mL" and "7 FR Part B R2–15 mL"
- 6. The mass of each syringe was recorded on the data sheet or ELN
- 7. A 250mL cup was labeled with 7 FR Part A
- 8. A 250mL cup was labeled with 7 FR Part B
- 9. After securing lids, the FlexFoam-iT!<sup>TM</sup> 7 FR was carefully shaken before dispensing
- 10. A small amount of FlexFoam-iT!<sup>TM</sup> 7 FR Part A was poured into the cup labeled A, enough to complete steps 12 and 13
- 11. A small amount of FlexFoam-iT!<sup>TM</sup> 7 FR Part B was poured into the cup labeled B, enough to complete steps 12 and 13 below
- 12. 15 mL of part A was taken up in the corresponding syringe and this was repeated with the second syringe labeled Part A
- 13. 15 mL of part B was taken up in the corresponding syringe and this was repeated with the second syringe labeled Part B
- 14. Each filled syringe was weighed, and the mass recorded the data sheet or ELN
- 15. Syringes were set aside until ready to mix

#### 2.4.3 Preparation to mix FlexFoam-iT!<sup>TM</sup> 14

- 1. Two 500mL containers were labeled with the foam being tested, date, temperature, and relative humidity. Replicates were designated by adding R1 or R2.
- 2. Each cup was weighed, and the mass was recorded on the data sheet or ELN
- 3. Four syringes without needles were obtained
- 4. The syringes were labeled "14 Part A R1–10 mL" and "14 Part A R2–10 mL"
- 5. The other two were labeled "14 Part B R2– 20 mL" and "14 Part B R2– 20 mL"
- 6. The mass of each syringe was recorded on the data sheet or ELN

- 7. A 250mL cup was labeled with 14 Part A
- 8. A 250mL cup was labeled with 14 Part B
- 9. After securing lids, the FlexFoam-iT!<sup>TM</sup> 14 container was carefully shaken before dispensing
- 10. A small amount of FlexFoam-iT!<sup>TM</sup> 14 Part A was poured into the cup labeled A, enough to complete steps 12 and 13
- 11. A small amount of FlexFoam-iT!<sup>TM</sup> 14 Part B was poured into the cup labeled B, enough to complete steps 12 and 13 below
- 12. 10 mL of part A was taken up in the corresponding syringe and was and repeated with the second syringe labeled Part A
- 13. 20 mL of part B was taken up in the corresponding syringe and was repeated with the second syringe labeled Part B
- 14. Each filled syringe was weighed, and the mass was recorded the data sheet or ELN
- 15. Syringes were set aside until ready to mix

#### 2.4.4 Preparation to mix FlexFoam-iT!<sup>TM</sup> 23 FR

- 1. Two 500mL containers were labeled with the foam being tested, date, temperature, and relative humidity. The replicates were designated by adding R1 or R2.
- 2. Each cup was weighed, and the mass was recorded on the data sheet or ELN
- 3. Two syringes were obtained without needles
- 4. One syringe was labeled with "23 FR Part A"
- 5. The other was labeled with "23 FR- Part B"
- 6. A 250mL cup was labeled with "23 FR Part A" and a second was labeled "23 FR Part B"
- 7. After securing lids, the FlexFoam-iT!<sup>TM</sup> 23 FR container was carefully shaken prior to dispensing
- 8. A small amount of FlexFoam-iT!<sup>TM</sup> 23 FR Part A was poured into cup A and Part B was poured into the cup labeled B
- 9. A syringed labeled 23 FR Part A was used to take up Part A, approximately 40mL
- 10. The second syringe labeled 23 FR Part B was used to take up Part B, approximately 40mL
- 11. The syringes were set aside until they were ready to be mixed



Figure 2-1. Hands-on workers preparing to mix the two-part polyurethane flexible foams

#### 2.5 Mixing of Foams

For each test, an empty cup was weighed, and the mass was recorded. It was then placed in the chamber with the foams to account for any mass lost or gained from the cup being in the chamber for a prolonged period of time.

#### 2.5.1 Dispense of Hilti Firestop Foam CP 620

- 1. The cartridge was primed (if using a new cartridge) and discarded in the labeled 250mL container
- 2. Hilti was immediately dispensed into the two labeled 500mL containers in the amount of 2-3 full pumps
- 3. The cups with Hilti were immediately placed in the environmental chamber and the time was recorded

#### 2.5.2 Mixing of FlexFoam-iT!<sup>TM</sup> 7 FR

- 1. The syringe labeled 7 FR Part B R1 was dispensed into the cup labeled 7 FR R1
- 2. The syringe labeled 7 FR Part B R2 was dispensed into the cup labeled 7 FR R2
- 3. The second syringe labeled A R1 was dispensed into cup labeled 7 FR R1 and mixed for at least 15 seconds with a craft stick; the sides were scraped as needed
- 4. The mixture was visually inspected to ensure it was homogeneous before moving on
- 5. The syringe labeled A R2 and was dispensed into the cup labeled 7 FR R2 and mixed for at least 15 seconds with a craft stick; the sides were scraped as needed
- 6. The mixture was visually inspected to ensure it was homogeneous before moving on
- 7. The cups were immediately placed in the environmental chamber and the time was recorded

#### 2.5.3 Mixing of FlexFoam-iT!<sup>TM</sup> 14

1. The syringe labeled 14 Part B – R1 was dispensed into the cup labeled 14 – R1

- 2. The syringe labeled 14 Part B R2 was dispensed into the cup labeled 14 R2
- 3. The second syringe labeled 14 Part A R1 was dispensed into the cup labeled 14 R1 and mixed for at least 15 seconds with a craft stick; the sides were scraped as needed
- 4. The mixture was visually inspected to ensure it was homogeneous before moving on
- 5. The syringe labeled 14 Part A R2 and was dispensed into the cup labeled 14 R2 and mixed for at least 15 seconds with a craft stick; the sides were scraped as needed
- 6. The mixture was visually inspected to ensure it was homogeneous before moving on
- 7. The cups were immediately placed in the environmental chamber and the time was recorded

#### 2.5.4 Mixing of FlexFoam-iT!<sup>TM</sup> 23 FR

- 1. The 500mL cup labeled 23 FR R1 was placed on a balance and zeroed out
- 2. 20g of 23 FR Part B from the syringe labeled 23 Part B was squeezed into the cup and the final mass was recorded
- 3. The cup was removed and replaced with the cup labeled 23 FR R2
- 4. 20g of Part B from the syringe labeled 23 FR Part B was squeezed into the cup and the final mass was recorded
- 5. 23 FR Part A was added to the cup with Part B until it reached a total of 37g
- 6. This was repeated with the second 500mL cup with Part B already in it
- 7. Both were mixed with a craft stick for a minimum of 15 seconds, and the sides were scraped down as necessary
- 8. The mixture was visually inspected to ensure it was homogeneous
- 9. The cups were immediately placed in the environmental chamber and the time was recorded



Figure 2-2. Replicates of the four selected foams after being placed in the Binder KMF 240 Environmental Chamber

#### 2.6 Testing for Set-to-Touch, Dust-Free, and Dry-to-Touch (ASTM, 2003)

1. After 60 seconds, the Hilti container was removed from the chamber and tested to see if it was dry-to-touch, set-to-touch, and/or dust-free

- a. **Set-to-touch time**: The surface was touched with a finger in a nitrile glove, then the finger rolled across a glass slide. The sample was considered dry when no residue was transferred.
  - Note: If the foam did not pass this test, there was no need to test for dry-to-touch.
- b. **Dust-free time**: A small amount of cotton was dropped from approximately one inch above the surface. Gently blown parallel to the substrate surface. It was considered dust-free if the cotton could be gently blown off the surface. The cotton was not pressed into the foam's surface.
- c. **Dry-to-touch time**: The substrate was touched gently with nitrile gloves and considered dry when the material no longer adheres to the finger and did not move when the finger was lightly rubbed across the surface. Please note that this was not a tack-free test and it was considered dry when no material adhered to the finger when wiped.
- 2. FlexFoam-iT!™ foams were removed from the environmental chamber every 12 minutes to undergo testing listed above.
- 3. Each cup was checked for set-to-touch, dust-free, and dry-to-touch. Note: dry-to-touch only was tested if the foam passed the set-to-touch test.
- 4. When a foam passed each test, the elapsed time was recorded
- 5. Foams that did not pass were returned to the chamber and testing was repeated every 12 minutes until all the foams passed all the tests (12 minutes was selected as it is 10% of the recommended cure time)

#### 2.7 Determining Percent Water Uptake

- 1. After mixing the foams, cups were placed in the chamber and the time was recorded
- 2. The cup containing Hilti was removed after ten minutes and the total mass of the cup and foam was weighed
- 3. Cups were returned to the chamber
- 4. After a full two hours, the FlexFoam-iT!<sup>TM</sup> cups were removed from the chamber and the total mass of the cup and foam was recorded
- 5. Cups were returned to the environmental chamber
- 6. After 24 hours from initial placement into the chamber, the cups were removed and masses of all eight cups with foam and the empty test cup were weighed and recorded.

#### 2.8 Quality Assurance

The environmental chamber used in 723-A Lab 162 is routinely calibrated by Measurement and Test Equipment (M&TE) and was up-to-date on the calibration requirements at time of use. The combination hygrometer and thermometer used to monitor the relative humidity and temperature of the environmental chamber in 999-1W Lab 102 was calibrated by the Standards Lab according to SRNL's M&TE standards.

#### 3.0 Results and Discussion

#### 3.1 Set-to-Touch, Dust-Free, and Dry-to-Touch

As seen in Table 3-1 below, Hilti foam always cured within 60 seconds regardless of conditions. FlexFoam-iT!<sup>TM</sup> 14 was also consistent throughout, curing at 12 minutes under for all conditions except when set to the coldest temperature. The fire resistant flexible foams were far less consistent, but never exceeded their manufacturer specified cure time. The set-to-touch, dust-free, and dry-to-touch times of these foams at 40% RH with (considered within ideal conditions) with varying temperatures can be seen below in Figure 3-1 through Figure 3-3.

Table 3-1. Full environmental chamber testing results for set-to-touch, dust-free, and dry-to-touch testing

	Hilti Fi	irestop Fo 620	oam CP	FlexFo	oam-iT! <sup>TM</sup>	7 FR	FlexI	Foam-iT!T	м 14	FlexFo	oam-iT! <sup>TM</sup>	23 FR
	Set-To- Touch (min)	Dust-Free (min)	Dry-to- Touch (min)	Set-to- Touch (min)	Dust-Free (min)	Dry-to- Touch (min)	Set-to- Touch (min)	Dust-Free (min)	Dry-to- Touch (min)	Set-to- Touch (min)	Dust-Free (min)	Dry-to- Touch (min)
25°C/	1	1	1	36	36	36	12	12	12	24	48	36
25% RH	1	1	1	36	36	36	12	12	12	24	48	36
15°C/	1	1	1	36	60	60	24	24	24	36	48	36
40% RH	1	1	1	36	60	60	24	24	24	36	48	36
25°C/	1	1	1	24	48	24	12	12	12	24	36	36
40% RH	1	1	1	24	48	24	12	12	12	24	36	36
35°C/	1	1	1	24	36	36	12	12	12	24	36	36
40% RH	1	1	1	24	36	36	12	12	12	24	36	36
45°C/	1	1	1	12	12	12	12	12	12	12	24	12
40% RH	1	1	1	12	12	12	12	12	12	12	24	12
25°C/	1	1	1	24	24	24	12	12	12	24	24	24
60% RH	1	1	1	24	24	24	12	12	12	36	36	36
25°C/	1	1	1	24	24	24	12	12	12	24	24	24
80% RH	1	1	1	24	24	24	12	12	12	24	24	24
25°C/	1	1	1	12	24	12	12	12	12	12	24	24
90% RH	1	1	1	12	24	12	12	12	12	12	24	24

Note: Colors selected get darker as time increases to better visualize elapsed time.

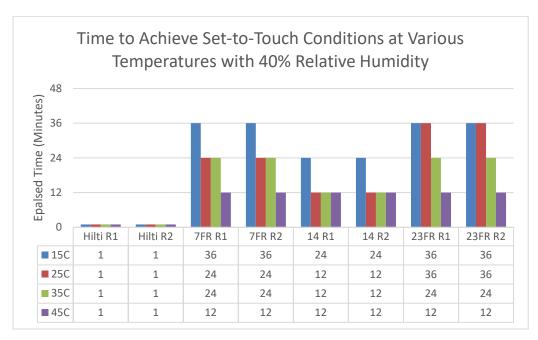


Figure 3-1. Time to achieve set-to-touch conditions at 40% relative humidity at varying temperatures

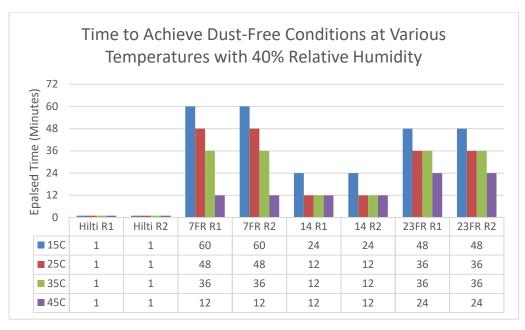


Figure 3-2. Time to achieve dust-free conditions at 40% relative humidity at varying temperatures

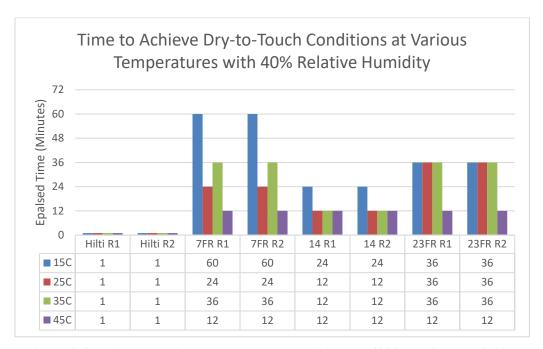


Figure 3-3. Time to achieve dry-to-touch conditions at 40% relative humidity at varying temperatures

The following graphs (Figure 3-4 through Figure 3-6) show the response to different humidities at a constant 25°C. The humidity value that had the fastest set-to-touch time and dry-to-touch time was 90% RH. Dust-free occurred fastest at humidity values 60% and higher.

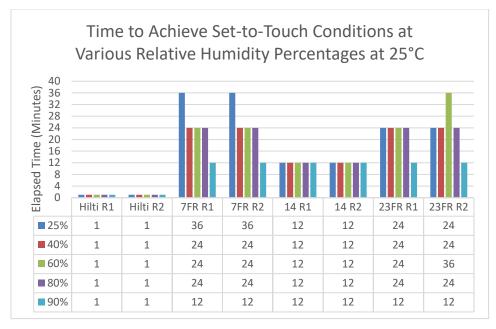


Figure 3-4. Time to achieve set-to-touch conditions at the set temperature of 25°C with varying relative

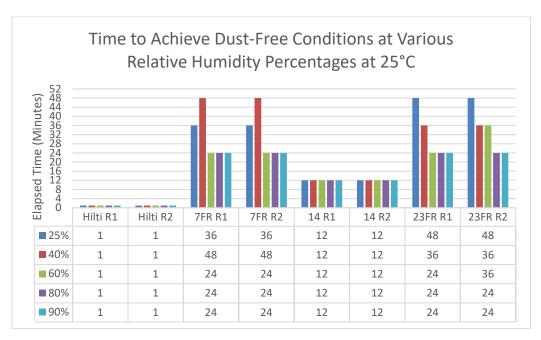


Figure 3-5. Time to achieve dust-free conditions at the set temperature of 25°C with varying relative humidity percentages

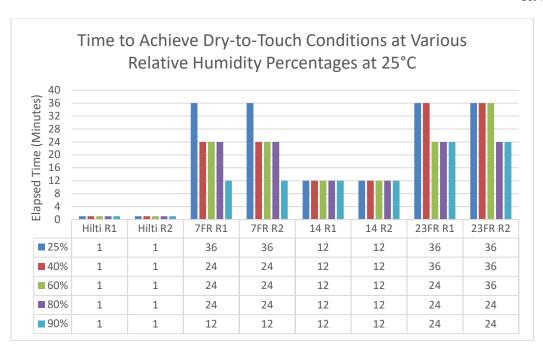


Figure 3-6. Time to achieve dry-to-touch conditions at the set temperature of 25°C with varying relative humidity percentages

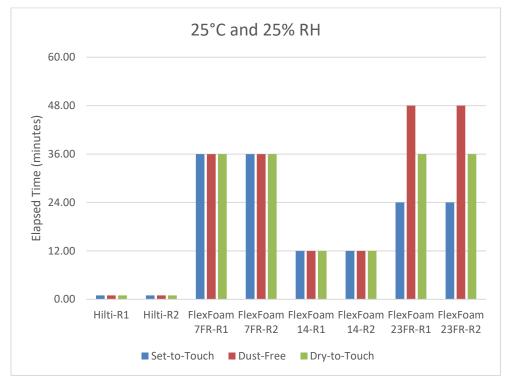


Figure 3-7. Set-to-touch, dust-free, and dry-to-touch performance at 25°C and 25% relative humidity

15°C and 40% RH yielded the longest cure times for all foams except Hilti. FlexFoam-iT!™ 7 FR was setto-touch, dust-free, and dry-to-touch at 60 minutes. This is the longest time that any foam took to cure out

of all the trials. FlexFoam-iT!<sup>TM</sup> 14 took longer than any other test, curing in 24 minutes. FlexFoam-iT!<sup>TM</sup> had the longest dust-free time (Figure 3-8).

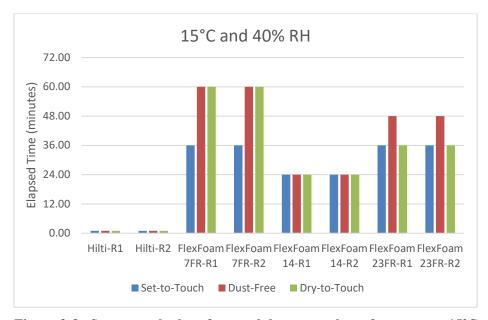


Figure 3-8. Set-to-touch, dust-free, and dry-to-touch performance at 15°C and 40% relative humidity

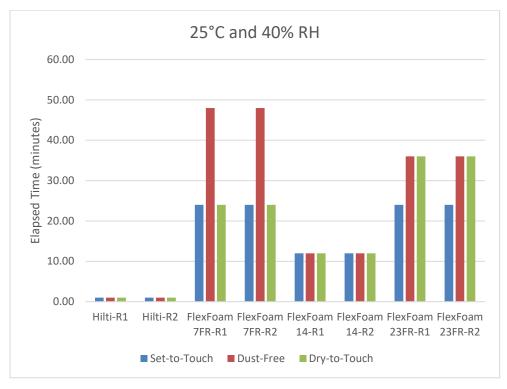


Figure 3-9. Set-to-touch, dust-free, and dry-to-touch performance at 25°C and 40% relative humidity

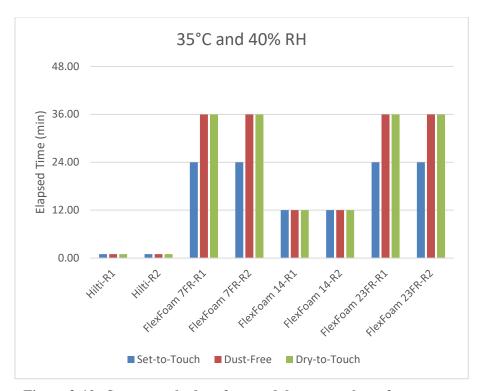


Figure 3-10. Set-to-touch, dust-free, and dry-to-touch performance at 35°C and 40% relative humidity

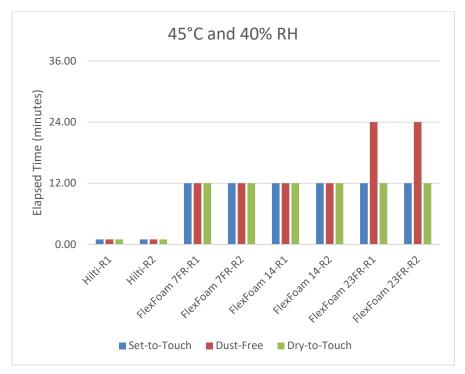


Figure 3-11. Set-to-touch, dust-free, and dry-to-touch performance at 45°C and 40% relative humidity

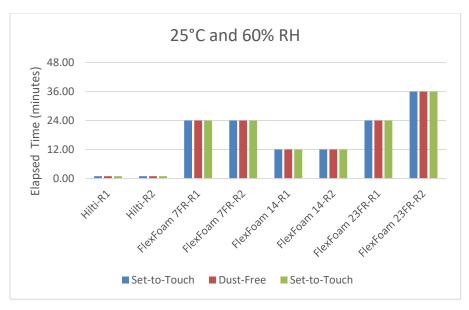


Figure 3-12. Set-to-touch, dust-free, and dry-to-touch performance at 25°C and 60% relative humidity

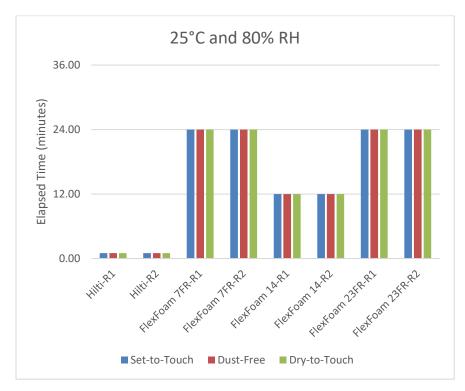


Figure 3-13. Set-to-touch, dust-free, and dry-to-touch performance at 25°C and 80% relative humidity

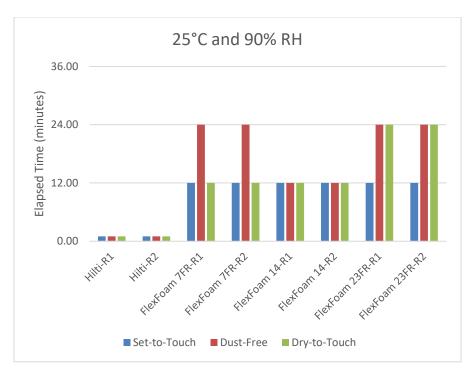


Figure 3-14. Set-to-touch, dust-free, and dry-to-touch performance at 25°c and 90% relative humidity

Hilti consistently passed set to touch, dust-free, and dry-to-touch milestones at 60 seconds. This not only meets, but exceeds the manufacturer listed cure time of three minutes even under conditions that lie far outside ideal conditions. Unfortunately, since it does begin its curing process so quickly, even with preparation and planning, there was no way in the experimental design to get the Hilti cups in the chamber faster than 5-10 seconds after ejection. They were prepared at room temperature and the environmental chamber door was opened minimally to gain access to the chamber. This expected flaw was mitigated as best as possible by performing the Hilti chamber testing first until it was completed before starting the FlexFoam-iT!<sup>TM</sup> tests to avoid opening the door for an extended period of time.

FlexFoam-iT!<sup>TM</sup> 14 was similarly consistent under changes in humidity, drying in 12 minutes except when the temperature was lowered to 15°C. FlexFoam-iT!<sup>TM</sup> 7 FR and 23 FR both had a wider variation in their dry-to-touch times but neither exceeded the manufacturer specified cure time. All three Smooth-On foams experienced their longest dry-to-touch times at 15°C and 40% humidity. It is expected that results would continue to vary with decreasing temperatures.

#### 3.2 Water Uptake

Table 3-2. Environmental chamber testing results for water uptake with an error value of 1 standard deviation

	Hilti Fir	estop Foai	m CP 620	FlexF	oam-iT!T	<sup>M</sup> 7 FR	Flex	Foam-iT!	гм 14	Flexl	Foam-iT! <sup>T</sup>	M 23 FR
Conditions	Initial Foam Mass (g)	Final Foam Mass (g)	Percent Mass Change	Initial Foam Mass (g)	Final Foam Mass (g)	Percent Mass Change	Initial Foam Mass (g)	Final Foam Mass (g)	Percent Mass Change	Initial Foam Mass (g)	Final Foam Mass (g)	Percent Mass Change
25°C/	$50.65 \pm 4.30$	$50.63 \pm 4.29$	<b>-0.04%</b> ± 0.01	$31.94 \pm 0.33$	$31.82 \pm 0.34$	-0.38% ± 0.04	$\begin{array}{c} 29.33 \\ \pm \ 0.08 \end{array}$	29.23 ± 0.08	-0.34% ± 0.00	$36.23 \pm 0.35$	$36.18 \pm 0.35$	<b>-0.14%</b> ± 0.01
25°C/ 25% RH	44.57	44.56	-0.02%	31.48	31.34	-0.44%	29.45	29.35	-0.34%	36.73	36.67	-0.16%
	± 4.30	± 4.29	± 0.01	$\pm 0.33$	$\pm 0.34$	± 0.04	$\pm 0.08$	$\pm 0.08$	$\pm 0.00$	$\pm 0.35$	$\pm 0.35$	± 0.01
	16.28	16.24	-0.25%	31.29	31.20	-0.29%	29.25	29.16	-0.31%	27.27	27.08	-0.70%
15°C/	± 2.92	± 2.93	± 0.10	± 0.13	± 0.12	± 0.02	± 0.29	± 0.29	$\pm 0.00$	± 0.94	± 0.93	± 0.02
40% RH	20.41	20.39	-0.10%	31.11	31.03	-0.26%	28.84	28.75	-0.31%	28.60	28.39	-0.73%
	$\pm \ 2.92$	$\pm 2.93$	$\pm 0.10$	$\pm 0.13$	$\pm 0.12$	$\pm 0.02$	$\pm 0.29$	$\pm 0.29$	$\pm 0.00$	± 0.94	$\pm 0.93$	± 0.02
	29.02	29.04	0.07%	32.52	32.49	-0.09%	28.16	28.07	-0.32%	36.52	36.52	0.00%
25°C/	$\pm 0.35$	$\pm 0.35$	$\pm 0.02$	$\pm 0.24$	$\pm 0.25$	$\pm 0.05$	$\pm 0.22$	$\pm 0.24$	$\pm 0.08$	$\pm 0.18$	$\pm 0.18$	$\pm 0.00$
40% RH	$29.51 \pm$	29.54	0.10%	32.18	32.13	-0.16%	28.47	28.41	-0.21%	36.27	36.27	0.00%
	0.35	$\pm 0.35$	$\pm 0.02$	$\pm 0.24$	$\pm 0.25$	$\pm 0.05$	$\pm 0.22$	± 0.24	$\pm 0.08$	$\pm 0.18$	$\pm 0.18$	$\pm 0.00$
	$21.68 \pm$	21.70	0.09%	31.07	31.06	-0.03%	30.13	30.10	-0.10%	37.26	37.37	0.30%
35°C/	2.54	$\pm 2.53$	± 0.04	$\pm 0.60$	± 0.62	$\pm 0.05$	± 1.53	± 1.53	$\pm 0.16$	$\pm 0.47$	$\pm 0.48$	± 0.02
40% RH	25.27 ±	25.28	0.04%	30.22	30.19	-0.10%	27.97	27.93	-0.14%	36.59	36.69	0.27%
	2.54	± 2.54	± 0.04	$\pm 0.60$	± 0.62	± 0.05	± 1.53	± 1.53	± 0.16	$\pm 0.47$	± 0.48	± 0.02
	22.15 ±	22.18	0.14%	30.84	30.84	0.00%	29.43	29.38	-0.17%	36.47	36.60	0.36%
45°C/	1.62	± 1.63	± 0.03	± 0.47	$\pm 0.08$	± 0.02	± 0.36	± 0.35	± 0.02	± 0.31	± 0.22	± 0.35
40% RH	19.86	19.88	0.10%	30.17	30.72	0.03%	29.94	29.88	-0.20%	36.91	36.91	0.00%
	±1.62	±1.63	± 0.03	± 0.47	± 0.08	± 0.02	± 0.36	± 0.35	± 0.02	± 0.31	± 0.22	± 0.35
	47.86	47.72	-0.29%	31.25	31.41	0.51%	30.29	30.36	0.23%	36.31	36.50	0.52%
25°C/	±1.83	± 1.70	± 0.28	± 0.69	± 0.75	± 0.20	± 0.50	± 0.48	± 0.08	± 0.46	± 0.43	± 0.08
60% RH	45.27	45.32	0.11%	30.28	30.35	0.23%	29.58	29.68	0.34%	36.96	37.11	0.41%
	±1.83	± 1.70	± 0.28	± 0.69	± 0.75	± 0.20	± 0.50	± 0.48	± 0.08	± 0.46	± 0.43	± 0.08
2.50.01	51.72	51.85	0.25%	30.33	30.70	1.22% ± 0.20	29.46	29.69	0.78%	36.22	36.55	0.91%
25°C/ 80% RH	± 2.59	± 2.60	± 0.00	± 0.40	± 0.35		± 0.04	± 0.04	± 0.00	± 0.24	± 0.23	± 0.04
00/0 KII	55.38 ± 2.59	$55.52 \pm 2.60$	<b>0.25%</b> ± 0.00	$30.90 \pm 0.40$	$31.19 \pm 0.35$	<b>0.94%</b> ± 0.20	$29.41 \pm 0.04$	29.64 ± 0.04	<b>0.78%</b> ± 0.00	$36.56 \pm 0.24$	$36.87 \pm 0.23$	<b>0.85%</b> ± 0.04
	± 2.39 41.31	± 2.60 41.41	0.24%	29.51	± 0.33 29.85	± 0.20 1.15%	29.15	29.41	0.89%	37.14	37.58	± 0.04 1.18%
2500/	± 4.43	$\pm 4.44$	± 0.01	$\pm 1.27$	$\pm 1.32$	± 0.11	$\pm 0.23$	$\pm 0.23$	± 0.01	$\pm 0.18$	$\pm 0.20$	± 0.03
25°C/ 90% RH	35.04	35.13	0.26%	$\frac{\pm 1.27}{31.30}$	$\pm 1.32$ 31.71	1.31%	$\pm 0.23$ 28.82	29.08	0.90%	36.88	37.30	± 0.03 1.14%
7070 KII	± 4.43	55.15 ± 4.44	± 0.01	$\pm 1.27$	$\pm 1.32$	± 0.11	$\pm 0.23$	$\pm 0.23$	± 0.01	$\pm 0.18$	$\pm 0.20$	± 0.03
			te water loss							± 0.10	⊥ 0.∠0	± 0.03

Note: Red cells indicate water loss, blue cells indicate water gain, and green cells reflect no change

Table 3-2 shows the initial mass of the foams, final mass of the foams, and percent change of the mass after 24 hours. These values have been adjusted for the initial mass of each individual cup as well as any changes in mass in an empty cup. The standard deviation between each pair of replicates have been calculated and added as an error in the table.

For the water uptake testing, an increase in mass is assumed to signify water uptake as the data indicates a clear correlation between mass gain and heightened humidity. At 25°C with 25% RH and 15°C with 40% RH all samples experienced a mass loss. Of the foams tested, FlexFoam-iT!<sup>TM</sup> 14 had the most instances of mass lost and FlexFoam-iT!<sup>TM</sup> 23 FR had the least. Hilti had the most instances off water uptake (11 out of 16). However, these are only a small changes (20mg change in 20g).

It was also noticed that over 95% of samples underwent water uptake at humidity values over 60%. The most mass gained was FlexFoam-iT!<sup>TM</sup> 7 FR with 1.31% gain at 25°C and 90% RH. The most mass lost was 0.73% at the coldest temperature, 15°C and 40% RH. Samples at 40% RH with increasing temperatures had a lower mass percentage loss as temperatures increased. There was 100% loss at 15°C, 50% of the samples lost mass at 25°C, 50% of the samples lost mass at 35°C, and 25% of the samples lost mass at 45°C. This indicates that an increase in curing temperature beyond the manufacturer's recommendation can result in mass loss.

The general trend showed Hilti absorbing water in all instances except for the coldest temperature (15°C and 40%) and lowest humidity (25°C and 25%). There was one combination where Hilti lost mass where it was not expected: a loss of 0.29% mass at 25°C and 60%. It is unknown why there was an anomaly, but further testing would be advantageous to determine if additional replicates would yield similar results, particularly if Hilti is the desired foam to be used and similar climate conditions are found at the site in question.

#### 3.3 <u>Discussion of Foam Performance and Applicability</u>

The objective was to determine which foams should be selected for further testing. Table 3-3 lists some advantages and disadvantages to the four foams tested. Three of the four foams tested are fire resistant (excludes FlexFoam-iT!<sup>TM</sup> 14). While FlexFoam-iT!<sup>TM</sup> 14 had the most consistent results, both in set-to-touch, dust-free, and dry-to touch testing and water uptake testing, along with visual inspection of the foams, the test team has elected to not select FlexFoam-iT!<sup>TM</sup> 14 for additional projects as it lacks fire resistance, which is desirable for long-term D&D activities.

Hilti is by far the easiest to prepare and is cured in 60 seconds or less in low and high temperatures and low and high humidities. FIU has selected Hilti as their front runner COTS foam from previous testing. It has superior adhesion and other mechanical properties that make it ideal for continuing to test.

A significant portion of time in the lab was dedicated to mixing the two-part foams, which was not an issue for Hilti as no mixing is required. While speed isn't necessarily the goal for D&D work, manually mixing appropriate ratios did take quite a bit of time and also opened up room for errors. If it was required to fill an entire pipe with Hilti in real-world applications, it would be difficult. This is mostly because of the requirements of manually ejecting the foam from the cartridges. Additionally, it only is available in limited-size cartridges, approximately 116 in<sup>3</sup> expanded. However, the plan for future work is to utilize a hot tap and inject Hilti into portions of the pipe and let it cure. From there, the pipes will be cut in half with the expectation of Hilti being cured and plugging both ends of the cut pipe. This will be used to decrease the release of residual contamination from these pipes during routine D&D activities. Work is in the planning stages to determine the amounts needed and if the limitations on size will be a factor.

FlexFoam-iT!<sup>TM</sup> 7 FR expands 8x its original volume, resulting in the cheapest option (i.e. lowest cost per unit volume). Mixing FlexFoam-iT!<sup>TM</sup> 7 FR in large quantities is simple as it requires equal volumes of parts A and B. There was one instance where FlexFoam-iT!<sup>TM</sup> 7 FR did not respond as expected (Figure 3-15). This process was closely monitored, and it is not thought to be the result of improper mixing. Repetition of this test yielded the originally expected result. While it also had the longest wait time for two tests (60 minutes), expected cure time per Smooth-On is three hours. Since it passed set-to-touch, dust-free, and dry-to-touch conditions within that time in all tested conditions, it is still considered a good option for future work.

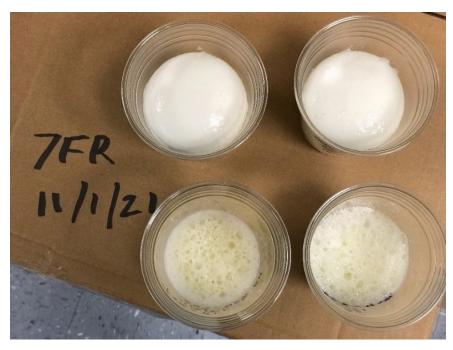


Figure 3-15. FlexFoam-iT!<sup>TM</sup> 7 FR set in the chamber on same day in the same temperature and humidity conditions (25°C and 40% RH) with one success (top set) and one failure (bottom set)

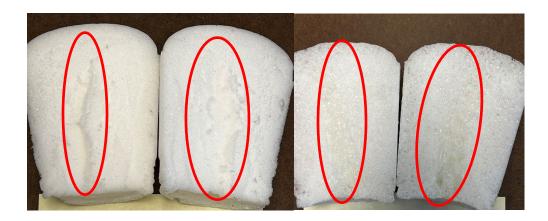


Figure 3-16. FlexFoam-iT!<sup>TM</sup> 7 FR cross-section at 25°C and 40% RH (left), 25°C and 90% RH (right) and with visible variation noted by red circles in the core

The final Smooth-On foam tested was FlexFoam-iT!<sup>TM</sup> 23 FR. It had consistent set-to-touch, dust-free, and dry-to-touch times and had the most instances of no mass gain or lost along with being fire resistant. It is the most expensive of all the FlexFoam-iT!<sup>TM</sup> foams tested, but it is still almost four times less expensive than Hilti. The main disadvantage to this product is the mixing ratio. It is an 85:100 parts by weight (pbw) ratio, which is more difficult to measure due to the need for a scale and more complex scaling depending upon the targeted amount of foam. FlexFoam-iT!<sup>TM</sup> 23 FR also had an instance of large pores inside, only visible by cutting (Figure 3-17). The top of the foam and all outward characteristics did not indicate there

were large voids inside. It is important to note that none of the other FlexFoam-iT!<sup>TM</sup> 23 FR samples examined showed similar voids.



Figure 3-17. Cross section of FlexFoam-iT!<sup>TM</sup> 23 FR at 15°C and 40% RH

Table 3-3. Summary of the pros and cons for foams tested

Foam	Advantages	Disadvantages	Cost Per Cubic Inch
Hilti Firestop Foam CP 620	<ul> <li>Completely dry in 60 seconds in all conditions tested</li> <li>No mixing required</li> <li>Consistent cross-sections (Figures 3-18 through 3-21).</li> <li>Low expansion rate to limit pressure build up in closed systems</li> </ul>	<ul> <li>Most expensive of the four foams</li> <li>Limited to one size canister</li> <li>Releases the most heat (250°C peak expansion temperature)</li> </ul>	\$0.56
FlexFoam- iT! <sup>TM</sup> 7 FR	<ul> <li>1:1 parts by volume (pbv) ratio for easy mixing in large quantities</li> <li>Fire resistant</li> <li>Expands the most, so less product would be required for the same amount of space filled</li> </ul>	<ul> <li>Had one instance of large pores on top (Figure 3-15) and could be indicative of more likelihood of failure</li> <li>Had the longest dust-free and dry-to-touch times recorded (60 minutes at 15°C and 40% RH.</li> <li>Two of the four cut in half showed inconsistency in drying (Figure 3-16)</li> </ul>	\$0.04
FlexFoam- iT! <sup>TM</sup> 14	Became set-to-touch, dust- free, and dry-to-touch faster than other Smooth-On foams	Not fire resistant	\$0.07

Foam	Advantages	Disadvantages	Cost Per Cubic Inch
	• Easy ratio for mixing large quantities (1:2 pbv)		
FlexFoam- iT! <sup>TM</sup> 23 FR	<ul> <li>Consistent dry-to-touch, dust-free, and set-to-touch times</li> <li>Fire resistant</li> <li>3 instances of no mass gained or lost</li> </ul>	<ul> <li>Weight ratio rather than by volume</li> <li>Has lowest expansion rate and more product would be required to fill large spaces</li> <li>Significant void space at lowest temperature (Figure 3-17)</li> </ul>	\$0.13



Figure 3-18. Hilti Firestop Foam CP 620 cross-section from 25°C and 40% RH testing.



Figure 3-19. Hilti Firestop Foam CP 620 cross-section from 45°C and 40% RH testing.



Figure 3-20. Hilti Firestop Foam CP 620 cross-section from 25°C and 90% RH testing.



Figure 3-21. Hilti Firestop Foam CP 620 cross-section from 15°C and 40% RH testing.

#### 4.0 Conclusions

The Hilti Firestop Foam CP 620, FlexFoam-iT!<sup>TM</sup> 7 FR, and FlexFoam-iT!<sup>TM</sup> 23 FR should be considered for continued study. There are no large anomalies that extend the drying time of the foams leading to the assumption that the set-to-touch, dust-free, and dry-to-touch times are not a point of concern. Water uptake was seen consistently at humidities 60% and over, with additional water gain in Hilti in lower humidity ranges. The percent water uptake was only between 0.04% to 0.14% for humidity values under 60%. Overall, Hilti was the most consistent in respect to set-to-touch, dust-free, and dry-to-touch times and visually uniform after cross-section examination (Figures 3-18 through 3-21) and has the most potential for success in future applications.

#### 5.0 Recommendations, Path Forward or Future Work

There is currently preliminary planning underway for testing Hilti Firestop Foam CP 620 to characterize its mechanical properties when exposed to various stressors. The first phase of testing will investigate the following:

- adhesion and bonding properties with and without moisture
- determination the heat profile during curing
- development of a leak test standard operating procedure
- development of a literature review to determine if a hot tap is appropriate to deliver Hilti to the piping
- establishment of the relationship between pipe diameter and necessary quantity of Hilti
- determination of pipe pressure after deployment and curing

In addition to the Cold Test Plan that FIU will be executing, there is interest in this work from Los Alamos National Laboratory. There are upcoming D&D activities at Los Alamos that includes continuing work on TA-21 Building 257 and the Ion Beam Facility. These facilities were vacated in the late 1990s and are cold and dark. Successful stabilization of previously radionuclide-contaminated piping will be essential for completion of the D&D of these buildings.

More work will be done on the impact of pipe moisture on Hilti adhesion in the proposed cold test, which will take place at a full-scale mockup facility at FIU's Applied Research Center in 2022. It is recommended that FIU aim to get the final selected COTS foam certified to be used throughout the complex by meeting regulatory specifications for stabilization of radionuclides. This regulatory hurdle is necessary for the application of these foams throughout the complex and should be considered a priority.

#### 6.0 References

- 1. ASTM Standard D1640. 2003. Standard Test Methods for Drying, Curing, or Film Formation of Organic Coatings at Room Temperature. ASTM International, West Conshohocken, PA.
- 2. ASTM E3191-18. 2018. Standard Specification for Permanent Foaming Fixatives Used to Mitigate Spread of Radioactive Contamination. ASTM International, West Conshohocken, PA.
- 3. Hilti Firestop Foam CP 620 Product Information. 2020. <u>Technical-information-ASSET-DOC-LOC-1543328.pdf</u> (hilti.com)
- 4. Lagos, L.E., Sinicrope, J., Komninakis, M., Donoclift, T., Simoes-Ponce, T., Rendon, J. 2019. "Intumescent Foams in Operational Scenarios Phase I." Florida International University.
- 5. Rivas, A. 2018. Characterization of Radiation Hardened Polyurethane Foams for DOE Decontamination and Decommissioning (D&D) Operations. SRNL-STI-2018-00406. Aiken, SC.
- 6. Smooth-On, Inc. FlexFoam-iT!<sup>TM</sup> Series Technical Bulletin. 2019. <u>FLEXFOAM-IT!<sup>TM</sup> SERIES.pdf</u> (Smooth-On, Inc..com).
- 7. Weather-us.com. 2021. Long-term Weather Forecast for Aiken, SC, Los Alamos, NM, and Richland, WA. Accessed: December 8, 2021. Weather Atlas | South Carolina, USA weather forecast and Climate information (weather-us.com).

#### 7.0 Appendix

#### 7.1 Hilti Firestop Foam CP 620 Product Information

Note: The following images can be double clicked for PDFs of the full information.



#### FIRESTOP FOAM CP 620

#### Product description

 Rigid and fast-curing firestop foam with excellent water resistance to help create a fire, smoke and moisture barrier around cable and mixed penetrations

#### Applications for use

- · Concrete, drywall and masonry
- Multiple and mixed penetrations
- · Metal pipes, single cables, cable bundles and cable trays
- · Suitable for irregular and difficult-to-reach openings
- Sealing small to medium size openings
- · Where cables, steel, copper, cast iom or plastic pipes all pass through the same opening

#### Advantages

- · Adheres to concrete, drywall and masonry
- Approval for virtually any transit type cables, trays, conduits and pipes (metal
- Up to 6 times expansion
- Suitable for complex and difficult-to-reach applications single-sided installation possible and no reinforcing material needed
- Cures within 60 seconds
- · Mold, mildew, water and vapor resistant
- Future-proof re-penetrable to allow future cable capacity increases
- Optimal smoke tightness according to DIN EN 1026 and ASTM E 283-04

#### Installation instructions

See Hilti literature or third-party listings for complete application and installation details

#### Product Information





Chemical basis	Two component polyurethane				
Color	Red				
Recommended product application temperature	50" to 86"F (10" to 30"C)				
Recommended substrate temperature	32* to 104*F (0* to 40*C)				
Storage and transportation temperature range	41" to 77"F (5" to 25"C)				
Temperature resistance range	-22 to 212°F (-30° to 100°C)				
Expansion ratio	1:1.5 Class 0 (A8TM G21-96)				
Mold and mildew performance					
Tested in accordance with	UL 1479, ASTM E814, CAN/ULC-\$115				
Surface burning characteristics (ASTM E84)	Flame Spread 0 Smoke Development 15				
Minimum curing time ready to cut	3 min				
LEED VOC	1.3 g/L				
Volume per cartridge & foam yield (up to)	300ml & 116 in <sup>3</sup>				
Acoustics performance	50 (relates to specific construction) ASTM E90				
Shelf Life	9 months				
Expansion tempature)	482°F (250°C)				

Order designation	Sales pack quantity	Item number	
CP 620 Firestop Foam	12	338725	
CP 620 + Dispenser	12	371883	









Mixer CP 620-M















#### 7.2 FlexFoam-iT!<sup>TM</sup> Series Product Information

# FlexFoam-iT!™ Series

SMOOTH-ON

Flexible Polyurethane Foams

3lb., 4 lb., 5lb., 6 lb., 7 lb., 8 lb., 10 lb., 14 lb., 15 lb., 17 lb., 23 lb. or 25 lb.

www.smooth-on.com

#### PRODUCT OVERVIEW

FlexFoam-IT!\* Series foams are premium quality water blown flexible foams that can be used for a variety of industrial, special effects and art & crafts and projects. With several to choose from, uses include making theatrical props (swords, knives, hammers, etc.), industrial gaskets, custom padding and cushioning, and more. FlexFoam-IT!\* foams are also used as repair materials for seats, cushions and archery targets. SO-Strong\* colorants can be added for color effects.

Part A and B liquids are combined, mixed and poured into a mold or other form (apply release agent if necessary). Mixture will rise and cure quickly to a solid, flexible foam. Foams vary by density and offer good physical properties. The lower the number, the more the foam expands. FlexFoam-IT!\*\* It is the lowest density foam and expands the most. FlexFoam-IT!\*\* 15 has a relatively long, 2 minute pot life.

- FlexFoam-IT!" IV and FlexFoam-IT!" 15 are "Tuff Stuff" foams which are exceptionally strong.
- FlexFoam-IT!<sup>™</sup> 6 and FlexFoam-IT!<sup>™</sup> 8 are "Pillow Soft" foams with a similar softness to pillow or cushion foam.
- FlexFoam-IT!™ 7 FR is Flame Rated to FMVSS-302 specification
- . FlexFoam-IT!™ 23 FR is Flame Rated to UL-94 HB specification



#### TECHNICAL OVERVIEW

	A.B Mix Ratio by Volume	A:B Mix Ratio by Weight	Specific Gravity	Specific Volume	Pot Life (Commitme (ASTMD-2007)	Handling Time	Cure Time	Approximate Volumetric Expansion	Approximate Lbs./Cu. Foot≈ Kgs./Cu. Meter
FlexFoam-iT!™ III	1:2 pbv	57.5:100 pbw	0.05	504	35 sec.	25 min.	2 hrs	15 times	$3 \text{ lb/ft}^3 = 48 \text{ kg/m}^3$
FlexFoam-iT!™ IV	N/A	80:100 pbw	0.06	420	30 sec.	45 min.	2 hrs	13 times	$4 \text{ lb/ft}^3 = 64 \text{ kg/m}^3$
FlexFoam-iT!™ V	1:1 pbv	105:100 pbw	0.08	315	50 sec.	45 min.	2 hrs	11 times	$5 \text{ lb/ft}^3 = 80 \text{ kg/m}^3$
FlexFoam-iT!™ 6	1:1 pbv	105:100 pbw	0.09	280	35 sec.	60 min.	2 hrs	10 times	$6 \text{ lb/ft}^3 = 96 \text{ kg/m}^3$
FlexFoam-iT!™ 7 FR	1:1 pbv	100:88 pbw	0.11	229	35 sec.	60 min.	2 hrs	8 times	7 lb/ft <sup>3</sup> = 110 kg/m <sup>3</sup>
FlexFoam-iT!™ VIII	1:2 pbv	52.6:100 pbw	0.13	194	35 sec.	25 min.	2 hrs	7 times	$8 \text{ lb/ft}^3 = 128 \text{ kg/m}^3$
FlexFoam-iT!™ X	1:1 pbv	105:100 pbw	0.16	157	50 sec.	45 min.	2 hrs	6 times	$10 \text{ lb/ft}^3 = 160 \text{ kg/m}^3$
FlexFoam-iT!™ 14	1:2 pbv	100:190 pbw	0.22	114	60 sec.	45 min.	2 hrs	4 times	$14 \text{ lb/ft}^3 = 220 \text{ kg/m}^3$
FlexFoam-iT!™ 15	1:2 pbv	100:185 pbw	0.24	115	2 min.	90 min.	4 hrs	4 times	15 lb/ft3 = 240 kg/m3
FlexFoam-iT!™ 17	1:2 pbv	100:185 pbw	0.27	93	60 sec.	30 min.	2 hrs	3.5 times	17 lb/ft³ = 270 kg/m³
FlexFoam-iT!™ 23 FR	N/A	85:100 pbw	0.37	68	90 sec.	60 min.	2 hrs	2 times	23 lb/ft3 = 370 kg/m3
FlexFoam-iT!™ 25	N/A	1:2 pbw	0.40	63	90 sec.	25 min.	2 hrs	2 times	$25 \text{ lb/ft}^3 = 400 \text{ kg/m}^3$

Mixed Viscosity (ASTM D-2393): 1000 CPS

Color: White

\* Values measured at room temperature (73°F/23°C)

IMPORTANT: Shelf life of product is reduced after opening. Remaining product should be used as soon as possible. Immediately replacing the lids on both containers after dispensing product will help prolong the shelf life of the unused product. XTEND-IT\*\* Dry Gas Blanket (available from Smooth-On) will significantly prolong the shelf life of unused liquid urethane products.

#### 7.3 ASTM D1640



Designation: D 1640 - 03

#### Standard Test Methods for Drying, Curing, or Film Formation of Organic Coatings at Room Temperature<sup>1</sup>

This standard is issued under the fixed designation D 1640; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (c) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by apencies of the Department of Defense

#### 1. Scope

- 1.1 These test methods cover the determination of the various stages and rates of film formation in the drying or curing of organic coatings normally used under conditions of ambient room temperature.
- 1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information
- 1.3 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

#### 2. Referenced Documents

2.1 ASTM Standards: 2

D 823 Practices for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels

D 1005 Test Methods for Measurement of Dry-Film Thickness of Organic Coatings Using Micrometers

#### 3. Significance and Use

3.1 These test methods are used to determine the various stages and rates of drying, curing, and film formation of organic coatings for the purpose of comparing types of coatings or ingredient changes, or both. This is significant in the development of organic coatings for various end uses and also for production quality control.

#### 4. Coatings and Recommended Film Thicknesses

4.1 Whenever tests are to be performed on coatings not listed in Table 1, there should be a prior agreement between the

TABLE 1 Recommended Film Thickness of Materials to be Tested\*

Material	Dry Film Thickness
Drying oils	1.25 ± 0.25 mll (32 ± 6 µm) <sup>®</sup>
Vamishes	1 ± 0.1 mll (25 ± 2 µm)
Lacquers	0.5 ± 0.1 mll (12.5 ± 2 µm) (8ee 7.5.2)
Resin solutions	0.5 ± 0.1 mll (12.5 ± 2 µm)
Enamels	1.5 ± 0.25 mll (36.5 ± 6 µm)
Oil paints	1.8 ± 0.2 mll (45 ± 2.5 µm) (8ee 6.1.2 )
Water paints	1 ± 0.1 mll (25 ± 2 µm)

AThis table is a general guide to be used when nothing more specific is agreed upon between the purchaser and the seller.

<sup>8</sup>See 6.1.2 and 7.5.1. Add driers a minimum of 24 h before test.

purchaser and seller as to the substrate, film thickness, and application method for testing the specific coating involved.

#### 5. Test Conditions

- 5.1 Conduct all drying tests in a well-ventilated room or chamber, free from direct drafts, dust, products of combustion, laboratory fumes and under diffused light (see 5.4). Make all measurements at a temperature of 23  $\pm$  2°C and 50  $\pm$  5% relative humidity with the coated panels in a horizontal position while drying.
- 5.2 Tests should be carried out at practical viscosities at which films can be applied to the proper film thickness with resultant good flow and leveling properties. In the absence of any specific material specification, instructions for preparation of the film should be determined and agreed upon between the purchaser and the seller.
- 5.3 Films to be tested should have practical thicknesses commensurate with performance characteristics expected under actual usage for the type under test. All testing should be done within an area, any point of which is not less than 15 mm (1/2 in.) from the film edge.
- 5.4 Light Conditions-Illumination of the films during the entire drying test period from normal laboratory or sky sources, never from direct sunlight or other sources high in nonvisible radiant energy.

#### 6. Preparation of Test Specimens

6.1 Carry out all tests as described in 6.1.1, 6.1.2 and 6.1.3, unless otherwise noted

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<sup>&</sup>lt;sup>1</sup> These test methods are under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and are the direct responsibility of Subcommittee D01.23 on Physical Properties of Applied Paint

Current edition approved Dec. 1, 2003. Published December 2003. Originally

approved in 1959. Last previous edition approved in 1995 as D 1640 −95 (1999).

<sup>3</sup> For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For Annual Book of ASTM Standard's volume information, refer to the standard's Document Summary page on the ASTM website.

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