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Physical Interaction Between Sodium and Standard Insulation

by

Jordan Harley

A thesis

submitted in partial fulfillment of

the requirements for the degree of

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To the Graduate Faculty

The members of the committee appointed to examine the thesis of Jordan Harley find it

satisfactory and recommend that it be accepted.

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Physical Interaction Between Sodium and Standard Insulation

Thesis Abstract — Idaho State University (2022)

The purpose of this project was to obtain the modes in which molten sodium interacts with standard fiberglass insulation. This type of interaction can be representative of a leaking pipe inside of a sodium fast reactor. Knowing the modes of interaction will help with future design work in sodium leak detection. Two different experiment iterations were undertaken. The first iteration was a drip assembly that heated the sodium and dripped it onto the surface of the insulation sample. The second iteration was a furnace, which maintained the sodium in a molten state for an extended period while in contact with the insulation. When in contact, it was found that the sodium did not permeate the insulation, but rather degraded the insulation at higher temperatures.

Key Words: molten, sodium, fiberglass, insulation, degradation, ingress, permeate, fast, reactor, leak, detection

Introduction

Sodium-cooled Fast Reactors (SFRs) are currently being developed and pursued as candidates for the next generation of nuclear reactors, Gen IV. Sodium is a unique coolant in that it has excellent heat transfer properties with a large thermal conductivity, making sodium roughly 100 times more efficient at transferring heat than water, the coolant of choice for all powergenerating reactors in the U.S. Sodium also has a low density of 0.97 g/cm³ and a large liquidus state (98 - 883 °C) [1]. Sodium is the sixth most abundant element in the earth's crust [2] and allows for a fast neutron spectrum in a fast reactor. These fast reactors utilize more fuel than traditional thermal reactors and reduce the amount of nuclear waste produced. Sodium fast reactors have had success in the past in the United States, such as the two prototype reactors known as the Experimental Breeder Reactor II (EBR-II) and the Fast Flux Test Facility (FFTF). These successes were also accompanied by many growing pains such as sodium leaks, fires, and failed fuel pins. It has been almost three decades since EBR-II and FFTF were shut down in the 1990's, and the information/experience gap that was left since then has been large and growing. Newfound needs in the United States have put SFRs back on the radar, with one need being the proposed Versatile Test Reactor (VTR), a high flux test facility needed for the acceleration of testing new technologies, fuels, materials, and much more. With new momentum behind SFR research and the advantages they pose, there are many gaps to fill.

This thesis addresses the mechanisms of interaction between liquid sodium and standard fiberglass insulation. The United States Nuclear Regulatory Commission (NRC) in 2019 came out with a comprehensive report outlining the current operational experience regarding SFRs. In this report, the NRC outlined many 'significant findings' pertaining to SFRs in which it listed several

design parameters learned through the years such as, "Shrink-fit parts should be avoided because they could loosen during thermal transients" or "Oil-based lubricants should be avoided in SFRs." [3] The report also outlines several areas where further research is needed if SFRs are to move forward. The area of interest that will be focused on in this thesis will be, "Accurate detection methods of corrosion and leaks are necessary, particularly in regions coated with insulation... Inadequate or unreliable leak detection systems have resulted in extensive shutdowns caused by sodium contamination and excessive sodium leaks with consequent fires." [3] The French Atomic Energy Commission (CEA) also recognize this need of sodium leak detection when they published a report in 2016 and stated there is a "need for leak detection systems, and for provisions to manage sodium fires quickly and efficiently in all premises housing sodium circuits..." [4]

Knowledge on how to safely operate the next generation of SFRs is of great importance. This thesis supplements a specific need outlined by the NRC, where accurate leak detection is needed in regions coated with insulation. The risks associated with undetected sodium leaks are sodium fires, which pose risks to personnel and equipment, and unintended shutdowns which cause loss of time and resources. To accurately detect sodium leaks in these regions, knowing the interaction mechanism between sodium and insulation is vital. The objective of this research was to discover this interaction mechanism. The scope of this research focused on testing one type of insulation under a variety of temperatures while in contact with a small amount of molten sodium (less than two grams). The insulation type used was rigid fiberglass piping insulation.

Two types of experiments were performed that brought sodium into contact with insulation. The first experiment was an apparatus that melted the sodium and dripped it onto insulation samples, simulating a leaking pipe. The second experiment was performed inside of a jeweler's furnace, which melted the sodium for an extended period of time while in contact with the insulation sample.

The first section of this thesis outlines the literature review performed and depicts how previous research performed was inadequate at addressing current day needs. The sections for designing, pre-testing, and executing experiments will then outline each experiment performed. The results from each experiment and their significance will then be discussed in depth in the results and conclusion sections.

Literature Review

Even though the United States took the route of the pressurized water reactor as their major nuclear fleet, other countries have had ongoing SFR operation and research. These reactors include the Phénix and Superphénix in France, Joyo and Monju in Japan, the BN-x series of reactors in Russia, and the Prototype Fast Reactor (PFR) in the UK. All of these reactors have each had their own challenges and successes. Challenges often come in the form of welds that can fail due to both thermal stresses and cyclic fatigue from vibrations, valves that have failed due to improper design or installation, or corrosion of materials if sodium purity is not prioritized. Sodium leak detection has been unreliable and led to several fires. Many successes, however, have also been documented. From EBR-II specifically, fifteen years of under-sodium-operation of components such as pumps was accomplished, with only minor repairable damage. Passive safety for this unique reactor was demonstrated at one point by shutting off the reactors coolant flow, heat sink, and scram system, and then watching the reactor shut itself down from 100% power [5].

Sodium is a very reactive element when it comes in contact with water and oxygen due to the single valence electron in sodium's outer shell, giving it an oxidation state of +1. The most reactive combination is sodium with water, followed by molten sodium with oxygen and solid sodium with oxygen [6]. Thus, careful attention should be made to keep sodium inside of an enclosed system where it is kept under an inert atmosphere. Consequently, when sodium leaks occur, they are often accompanied by sodium fires. Accurately and quickly detecting these leaks when they do occur is the next priority. In order to accurately detect these sodium leaks when they do occur, one must understand how sodium behaves and interacts with its surroundings. In this case, in order to accurately detect leaks in regions coated with insulation, one must understand how the sodium behaves and interacts with insulation.

Pipe insulation plays a key role in the heat transport system of reactors. A few basic criteria that insulation should meet include "good resistance to heat loss, sufficiently high maximum service temperature, compatibility with piping and piped fluid, adequate personnel protection, and maintenance of a satisfactory thermal environment in areas adjacent to the piping" [7]. Insulation for SFRs has come in many different types and forms. Wherever possible, the insulation should be non-hygroscopic and chemically inert if sodium contact does occur. A report from 1969 outlined several different types of insulation that have been used in SFRs and can be found in Table 1 [7].

Material	Maximum Service Temperature, F	Density LB/CU FT
Amosite asbestos	1500	16
Mineral wool	1900	17
Diatomaceous silica	1900	24
Ceramic wools	2300	3 - 24
Calcium silicate	1200	11
Unbonded fiberglass	1000	9 - 11
Reflective insulation	1500	

Table 1. Different insulation types used in SFRs.

In a more recent study performed at Los Alamos National Laboratory, it was found that a majority of U.S. PWRs utilize fiberglass insulation. Many plants also utilize Reflective Metallic Insulation but are updating them to fiberglass [8]. As fiberglass is one of the most popular insulation types, this illustrates the need for further knowledge regarding the interaction between sodium and fiberglass insulation for SFRs.

Insulation Testing, Russian Institute of Physics and Power Engineering

This need for insulation testing was acted upon by the Russian Institute of Physics and Power Engineering (IPPE). In 1996, IPPE conducted a number of experiments concerning, "[t]he effect of thermal insulation on the mechanism of sodium leaking through the crack in the pipe wall..." [9] In this experiment a horizontal pipe was insulated with fiberglass. Holes were drilled into the pipe with diameters of either 3 or 8 mm. Gas was used to pressurize liquid sodium in the pipe and the leak rate was observed, along with the temperature. The tests used a 50 mm layer of insulation in the case of the 3 mm orifice. Three 40 mm layers of insulation were used for the 8 mm orifice, 2 layers being fiberglass and the third being kaolin wool. The results of these tests are summarized in Table 2 [9].

						T _{max} under	
Test No.	T_{Na}, C	P _{Na} , MPa	$\tau_{Na,} \min$	G _{Na} , kg/s	V _{Na} , 1	insulation, °C	Notes
1	520	4	7.5	0.08	40	800	*)
2	515	5.5	8.0	0.09	50	850	*)
3	400	5.5	10	0.1	70	600	*)
4	450	5.5	2	1.0	120	600	**)

Table 2. Experimental results and sodium behavior under multiple conditions.

*) 3 mm diameter orifice. Thermal insulation: one 50 mm layer of glass wool;

**) 8 mm diameter orifice. Thermal insulation: two 40 mm glass wool layers plus one 40 mm kaolin wool layer [9].

After the tests were complete it was noted that the area of insulation affected most was located right at the defect area, and that the sodium leaked through the insulation with a very active interaction while no insulation was burst. Other than these few statements, this experiment did not give a lot of physical data or observations about how the sodium and insulation interacted. More experimental work must be done if sodium and insulation interactions are to be fully understood.

Leak Detection Methods

Leak detection is a very important part for SFR safety and operation. When leaks do occur, they need to be quickly detected, located, and stopped before further leakage occurs. Leaks that are not detected early enough can lead to sodium fires, which can cause issues such as property damage, reactor shut down, and personnel risk. Leaks can occur due to a number of different reasons as outlined by the CEA [4]:

- Fabrication flaw;
- Design flaw;
- Material-related problem;
- Thermal striping damage, as a result of the incomplete mixing of fluid jets leading to temperature fluctuations and to surface-breaking cracking;
- Corrosion, e.g., as a result of air ingress into the circuits;
- Operator error (e.g. during circuit defreezing and the related sodium expansion).

Most leaks occur because of either fabrication flaws or thermal striping damage. Some common fabrication flaws where most leaks occur are pipe welds, when excess weld material and weld constraints have led to operational problems. Residual stresses have led to reheat cracking and have been caused by, "[1]owering the threshold or quality in welds and secondary loops..."

[3]

A variety of leak detection methods have been designed and tested throughout the history of SFRs. Some have proven successful, while others were seen to be very unreliable. Most of the leak detection methods fall under a few different categories: aerosol detection, physical leak detection, smoke detection, and camera detection. The CEA has had extensive SFR experience with the Rapsodie, Phénix, and Superphénix reactors, and determined that a redundant leak detection system involving multiple methods has had the most success. By their assessment, the two best options were aerosol detection and "insulated electrical conductors (bead-fitted wire, metallic tissue...); [where] the conductor is put in contact with the pipe, and this short circuit triggers an alarm." [4] Leak detection methods for several different reactors will be discussed below along with their failures and successes.

Superphénix Reactor

The Superphénix was a sodium fast reactor built in France in the Creys-Malville area with its construction beginning in 1976. The Superphénix was meant to demonstrate that large sodium reactors were capable of economically producing electricity, with an electric capacity of 1,242 MWe [10]. The reactor ran until political opposition forced it to close in 1997 [11]. The Superphénix reactor was equipped with fire detectors, spark plug devices, and aerosol detectors coupled with spectrophotometers. The spark plug devices were used as short circuit devices when they came in contact with liquid sodium, but were unreliable for two reasons: "(1) the degassing of products in the thermal insulation deposited on the spark portal electrodes caused them to short to ground and (2) keeping the spark plugs at high temperatures caused a loss of electrical insulation by degrading the spark plug's internal insulation." [3]

Another detection method was accomplished by installing beaded wires underneath thermal insulation. If the sodium leaked it would contact the wires and short circuit them, thus sounding an alarm for actions to be taken. This wire method also proved to be unreliable and a new method was developed which consisted of placing a steel sheet electrode between two layers of insulation. The sheet operated agreeably, but before it could be implemented and further vetted in the system, Superphénix was shut down.

Sodium Advanced Fast Reactor

The Sodium Advanced Fast Reactor (SAFR) was a United States SFR designed as a coalition between Rockwell International Corporation and Argonne National Laboratory. Although it was never built, an in-depth design went into the passive safety of SAFR. Its sodium piping and leak detection system can be seen in Figure 1 [12]. The sodium piping is surrounded by an annular gas gap, followed by two layers of insulation and a metal sleeve. The gas gap allows space for electrical heaters and other instrumentation, such as an aerosol sniffer. There also exists a drainpipe that can capture any leaked sodium and transfer it into a storage tank via gravity. Since the SAFR reactor was never built, the leak detection it proposed is still only concept.



Figure 1. SAFR leak detection and drainage [12].

Clinch River Breeder Reactor

The Clinch River Breeder Reactor (CRBR) was a prototype reactor that was in its final stages of approval and had begun construction before being defunded due to political reasons. Sodium leak detection was a large part of the CRBR project and several different ideas came out of its design. Two of these leak detection designs were the 'cable detector' and the 'contact detector.' The cable detector was made from a stainless-steel coaxial cable insulated with magnesium oxide, and the outer sheath was grounded and perforated. Once liquid sodium

penetrates the outer sheath it causes a low resistance path to ground, increasing the voltage drop and sounds the alarm. This cable detector can be seen in Figure 2 [13].



Figure 2. CRBR cable detector for liquid sodium leaks [13].

The contact detector is very similar to the cable detector in that it is stainless steel sheathed and insulated with magnesium oxide. The detector contains a coaxial cable joined as an exposed junction. When liquid sodium flows into the junction, it creates an electrical short between the junction and sheath and sounds the alarm. The contact detector acts more like a spark plug then the cable detector and can be seen in Figure 3 [13].



Figure 3. CRBR contact detector for liquid sodium leaks [13].

Sodium leak detection is a priority for the next generation SFRs, particularly in locations of insulated piping. Leak detection systems have historically relied on electrode systems that are set to short circuit when sodium contacts them. In areas of insulation, sodium can leak into the insulation material before it even comes in contact with uncovered electrodes (such as spark plugs). It is important to understand how the sodium will physically react and behave with the insulation. Knowledge of how the sodium interacts with the insulation can provide insight on developing advanced sodium leak detection systems.

Manufacturing Process of Fiberglass Insulation

Fiberglass is one of the most popular types of insulation used for thermally insulating pipes in nuclear reactors [8]. Fiberglass can be formed into a variety of different shapes, such as "fluffy soft board, rigid hard board, or rigid molded material" [14]. This experiment will focus on the rigid molded material in the form of pipe insulation.

Fiberglass is made from molten silicate glass mixed with an organic binder. The silicates are first melted and then drawn into small fibrous strands through a rotating orifice. As the fibers are drawn out they harden and break away onto a conveyor belt as new fibers are formed. The length of each individual fiber varies from a few inches down to ¹/₄ inch.

Once on the conveyor belt, the fibers are sprayed with an organic binder typically comprised of a phenolic resin. The weight percent of binder present depends on the desired final state of the insulation and ranges from 1 - 15%. Once the binder and fiberglass have been combined, they form a semi-solid undefined mat that can easily break apart. The conveyor belt takes this mat and shapes it into pipes by rolling it and curing the organic binder in an oven, which also acts to

fluff the mat to the desired thickness. During this rolling and curing process, the fiberglass tends to form itself into noticeable layers that can later be manipulated and carved out if desired (e.g. for pipes with a low profile flange, a few layers of the insulation can be cut and removed to snugly fit around it). These layers formed in the insulation can be seen in Figure 4. The density of the cured insulation depends on the percent of binder present, the fluffing technique of the oven, and the speed at which the insulation was formed.

Density plays a critical role in the workings of insulation, as it is often used as a barrier to control heat transfer. Fiberglass insulation is very effective because as the fibers are bound together and fluffed to a certain thickness and density, voids are created throughout the insulation material in the form of air pockets. The amount of voids present in the insulation can be quantified by porosity, which is the volume fraction of the voids present over the total volume. The porosity of insulation can range anywhere from 0.9 for denser insulation to 0.99 for low density insulation. In other terms, the density of fiberglass insulation can range from 0.6 to 12 pounds per cubic foot. With fiberglass insulation having such a high porosity and being mostly air, the thermal conductivity of the insulation is dependent on the thermal conductivity of air, which is a poor conductor of heat. This is what makes fiberglass a very good insulation material.

The concept of fiberglass having a high porosity is important to this experiment, as the porosity can have a significant effect on the ability for fluid to flow into a material. In principle, the sodium will flow into the insulation and displace the voids. The question to be answered by this study is how the sodium flows through the insulation, if at all.



Figure 4. During the manufacturing process layers are formed in the insulation.

The specific type of insulation used for testing in this research was a rigid core, 2-inch piping insulation manufactured by "Owens Corning" with a 1 inch thickness. The insulation had an operating working temperature of 1000 °F and the data specification sheet can be found in Appendix A: Fiberglass Insulation Data Specification Sheet.

Fluid Flow in Porous Media

Before performing any experiments with molten sodium, the behavior of liquids when in contact with the porous insulation needed to be understood. A pioneer of researching flow of fluids through porous media was Henry Darcy, a French engineer who lived in the mid-19th century. Darcy performed a series of tests and published his work in 1856 while he was the "Dean of the School of Bridges and Roadways." Darcy performed his work to better understand the filtration of water and its distribution systems, especially in the city of Dijon where he lived [15].

Darcy's research included an ingenious apparatus using simple filtration systems made of layers of sand, stones, and gravel inside of a vertical column. The various grain sizes of the media used in the filter was determined and the average porosity was found for the sand filter. Utilizing gravity, Darcy would regulate water flowing through this sand column until an equilibrium was reached and the flow rate going in and out was equal. The water pressure was captured on both sides of the filtration column using mercury manometers. Darcy presented these results in the form of volumetric flow rates and the pressure drop through the sand filter [15].

While Darcy played a key role in pioneering the research for fluid flow through porous media, his research focused on bulk fluid flow and the resulting flow rates and pressure differences. Darcy's experiments also only looked at flows through media that were already saturated with the working fluid. The experiment performed for this thesis , on the other hand, is concerned with the transient flow of fluid and its progressive ingression into porous media, especially one that is dry (unsaturated). To better understand this phenomenon, several experiments were looked at.

The first experiment looks at a simulation of fluid flow through soil as performed by Cueto-Felgueroso and Juanes [16]. As it turns out, the invasive patterns seen inside of soil vary based on factors including a balance "between capillary, viscous, and gravitational forces at the pore scale" [16]. Tests have repeatedly shown that flows into homogenous soil, which behaves as a porous media, "takes the form of preferential flow paths (fingers)." It has been shown that when a fluid is introduced into a homogenous porous media, that a path of least resistance is found, and the fluid adheres to that. While Darcy's experiments did not look at flow patterns in porous media, others did and related these flow patterns using extensions to the Richardson's equation, which is "the traditional model of unsaturated flow" [16]. These extensions compensate for Darcy's law and considers water permeability in porous media to change with the proportion of water saturation currently in the media. Permeability is a quantification of how well a material allows fluids to pass through them. The extensions also help explain the phenomenon of gravity finger formation during fluid penetration. Figure 5 depicts fingers forming in dry soil, with S_0 indicating the initial water saturation of the media.



Figure 5. As water ingresses into the dry, porous soil, fingers are created as the water finds its path of least resistance [16].

In these simulations, it was observed that as the water penetrated the soil, gravity fingers were formed in various sizes. The simulations also illustrated the role that the initial saturation played in the finger formations [16]. Larger degrees of initial saturation led to the fluid to penetrate at a more even rate, with less prominent fingers being manifested. Lower degrees of initial saturation showed how the fluid would create very prominent fingers and penetrate deep down into the media much faster than the main waterfront. These finger formations as they are related to saturation levels can be seen in Figure 6. In the experiment performed for this thesis, the initial saturation of the insulation is nearly zero so it would be expected to have large fingers forming as the sodium ingresses into the insulation.



a. Long, pronounced fingers are formed as the water penetrates.



b. Short, less pronounced fingers are formed as the water penetrates.

Figure 6. Comparison between finger formation as water penetrates porous media. All variables are the same for the two simulations except (b) has twice the initial saturation as (a) [16].

It can also be expected that this fingering pattern will occur in the insulation tests performed because the insulation sample is fairly homogenous, with the exception of small layers appearing inside the insulation itself during its manufacturing process. In the tests presented hereafter, it is found that these layers play a large role in the flow pattern of liquid in the insulation. To provide further background information about how these layers might affect the flow patterns, a second experiment was considered.

The second experiment performed by Huppert et al. looks at how differing permeabilities for media affect the flow of fluids [17]. This experiment was set up by layering two media one atop the other, each layer having a different permeability. The experiment can be seen in Figure 7, where k_U and k_L represent the permeabilities of the upper and lower regions, respectively. The different layers were made of glass ballotini (small beads of glass). Changing the bead size of the ballotini and making them larger positively changed the permeability of the layers.



Figure 7. Depiction of experiment set up by Huppert et al [17]. Two layers exist, with the lower layer having a different permeability (k_U) than the upper layer (k_L) .

Before the test began, the two layers of media were created, each with a different bead size. Once in place, the bead bed was filled with fresh water while keeping the top open to the atmosphere. A dense fluid that was previously dyed blue was then carefully released into the lower layer at a constant flux controlled via an elevated tank.

During the testing, Huppert et al. demonstrated that the differing permeability of the layers affected the way that the dense fluid saturated the media. When the lower layer was more permeable it was seen that the fluid flow focuses along the bottom of this layer. This can be seen in Figure 8. When the upper layer was more permeable, the fluid flow focuses on that layer. This is true at higher fluxes, even when the fluid was introduced into the lower, less permeable layer. It seems that the fluid finds the more permeable layer and then stays in that layer as long as it can, which is referred to as "flow focusing" [17]. One point of interest is when you look at how the fluid behaved after it has been allowed to flow in the upper, more permeable layer for an extended period of time. The fluid continued to flow along the base of this more permeable layer for a time. Gravitational effects eventually overcome the flow focusing effect and fingering began to appear in the lower, less permeable media. The upper layer flow pattern along with the eventual fingering can be seen in .



Figure 8. The lower layer is more permeable than the upper layer, causing the fluid to dominantly flow in this region along its base [17].



Figure 9. Dense fluid that was previously dyed is introduced in the lower, less permeable layer (top image). At higher fluxes the fluid finds the upper layer and focuses its flow at the base of this layer (middle image). Eventually, the fluid is overtaken by gravity and fingering begins to occur down into the lower layer (bottom image) [17].

This fingering effect correlated with the fingering found in the experiment performed by Cueto-Felgueroso and Juanes and exemplifies how a boundary of differing permeability can direct a fluid to flow horizontally along the bottom of a layer before the fluid invades into lower layers. In the testing hereafter, it will be shown how the water penetrated the insulation and would flow along a layer horizontally before it would start fingering and penetrate into lower layers, much like the fluid in this second experiment.

Experiment Design

The purpose of these experiments was to bring molten sodium and insulation together and observe the mechanisms of their interaction. Different environments were created by changing the temperature and applied pressure to see how the sodium and insulation would react under each scenario. Two different designs were developed for this experiment, the first design was a drip assembly and the second was a jeweler's furnace. The drip assembly experiment heated sodium to a molten state inside of an enclosed space. Once the sodium was molten it was then dripped onto a sample piece of insulation. The jeweler's furnace experiment heated the sodium and insulation inside of a crucible for an extended period of time.

Drip Assembly

The purpose of the drip assembly was to house the sodium, heat it up to a molten state, and then drip the sodium onto a piece of insulation. The assembly parts were mostly made of 316 stainless steel (SS) because of its excellent material characteristics and compatibility with liquid sodium. 316 SS has been well vetted and used in a variety of SFRs, including Rapsodie, Phénix, Superphénix, and others. If a necessary part was not available in 316 SS material, a close material match was found such as 416 SS or 304L SS. Table 3 outlines the drip assembly parts along with the part description, the part material, the company the part was purchased from, and the associated part number.

A depiction of the completed drip assembly can be seen in Figure 10. Each part in the assembly is identified and an exploded view depicts how the assembly connects. All parts contained NPT (National Pipe Tapered) threads to provide a tighter seal.

Part Description	Material	Company	Part #
Cap Fitting	316 SS	Swagelok	SS-200-1-2
Cap	316 SS	McMaster-Carr	4452K244
Vessel	304L SS	McMaster-Carr	2161K23
Ball valve	316 SS	McMaster-Carr	45395K118
Valve Fitting	316 SS	Grainger	1DFY3
Needle Valve	316 SS	Grainger	2KLC3
Straight Nozzle	416 SS	McMaster-Carr	3234K312

Table 3. Sodium drip assembly components along with the part description, material, company purchased from, and part number.



Figure 10. Completed drip assembly with each part being identified. An exploded view of the assembly shows how each part was connected.

The cap for the vessel helped enclose the system and have a more isolated environment for the heated sodium. It would prevent any unwanted objects from falling into the vessel, and also help prevent sodium from leaking out in the event the assembly fell over. The cap had a fitting placed into its top to provide an orifice to insert a thermocouple. The thermocouple was used to measure the temperature of the sodium inside of the vessel. The cap fitting also provided a way for argon gas to flow into the vessel during testing. This would help prevent suction from occurring inside the apparatus, allowing the sodium to flow more easily and consistently. The cap did not originally have a threaded hole to attach the fitting to, so an 1/8-inch NPT threaded hole was tapped into the center of the cap.

The vessel was made from a threaded pipe nipple reducer made of Schedule 80 SS. The pipe reducer was chosen because it had a large 2-inch opening on the top and a small ¹/₂-inch opening on the bottom. The larger orifice on the top allowed for easier access to the vessel for inserting sodium and instrumentation such as thermocouples. The small orifice on the bottom was good for reducing to smaller piping to allow better flow control and drip rates.

Two valves were used to provide an extra barrier and prevent unwanted sodium drippage and were connected through the valve fitting. The upper valve was a ¹/₂-inch ball valve that was mainly used as an on/off switch for the liquid sodium. The lower valve was a ¹/₄-inch needle valve that was used as a gradual open/close switch to achieve a more precise and accurate drip rate than a simple ball valve, while also reducing unwanted releases of sodium. A straight nozzle was chosen to facilitate a solid stream drip pattern. The nozzle had a small orifice diameter of 0.06 inches which helped control the drip rate. The experiment was designed to remove as much sodium as possible through gravity drainage when the experiment was over. This was done by using a cone shaped sodium vessel and making the entire assembly vertical. However, horizontal surfaces were present at each of the connection points from the vessel downward. These horizontal surfaces act as hydraulic low points and will retain residual sodium even when the bulk sodium has been drained. A depiction of these low points can be seen in Figure 11. The surface area of these horizontal points is minimal but should still be considered when taking apart the assembly for cleaning.



Figure 11. Cut-away view of the sodium drip assembly. Four low points are depicted in the assembly, which represent possible locations of sodium build-up after the sodium was drained.
The drip assembly was heated via a silicone pipe heating tape, measuring 4 feet long and a $\frac{1}{2}$ inch wide. The heat tape maximum temperature rating was 232 °C (450 °F) and contained a temperature controller box. This model of heat tape was chosen because it was flexible and could fit around the odd-shaped drip assembly while still providing maximum heat load to the surface.

Fiberglass insulation was chosen to wrap the drip assembly to prevent heat loss. The bottom half of the assembly was covered with ½-inch pipe insulation with a ½-inch thickness. This insulation had a maximum working temperature of 850 °F (454 °C) and was manufactured by 'Johns Manville.' The top half of the assembly was covered by 2-inch pipe insulation with a 1-inch thickness. This insulation had a max working temperature of 1000 °F (538 °C) and was manufactured by "Owens Corning." Pieces of the 2-inch insulation were also used as the receiver samples for the sodium testing. With the independent measurements that were performed, the insulation used in this experiment had an approximate density of 4.5 lb/ft³.

Fabricating the Drip Assembly

Before the drip assembly was constructed, each individual part was tested for leak tightness against air and water. The detailed testing process can be found in Appendix B: Leak Testing the Drip Assembly. Once the assembly was fully tested for leak tightness, the final fabrication work began, including assembling the apparatus, wrapping and securing the heat tape, and installing the insulation.

The first step was to bake the assembly parts in the box furnace at 80 °C for one hour. This was to bake off residual water left from the leak testing process. Latex gloves were used when handling the parts to minimize getting oils and dirt on the metal surfaces. Polytetrafluoroethylene

(PTFE) pipe tape was used to wrap each thread according to the specifications in Table 8. The apparatus was assembled from the top down by attaching the ball valve to the top vessel, then the valve fitting, followed by the needle valve and nozzle. Lastly, the 1/8-inch fitting was attached to the cap which was then attached to the top vessel. Each part was first fastened hand tight, and then mechanically tight using an adjustable or crescent wrench. Three parts needed to be tightened using an adjustable strap wrench because they were either too big for the available wrenches (valve fitting) or had only smooth surfaces (cap and top vessel).

Once the apparatus was assembled it was helically wrapped with the heating tape. The heating tape was secured to the surfaces of the assembly using high temperature aluminum adhesive tape. The heat tape was wrapped tightly to create the best possible connection. Depictions of assembling the apparatus prior to installing the insulation can be seen in Figure 12. The temperature control box needed to be ground to the surface it was heating, so good contact with the surface was required. This was accomplished by holding the temperature control box in place using the insulation itself, along with aluminum tape. Before the insulation was installed, the heat tape, along with its temperature control box, were tested. This testing process can be found in Appendix C: Testing the Heat Tape.

Piping insulation was installed onto the apparatus to prevent excess heat loss and to protect surrounding equipment from the high temperatures of the heating tape. The ¹/₂-inch insulation was put on first. The insulation needed hole cutouts to allow the ball valve handle to be accessible as well as wrap around the protruding stem of the needle valve.



a. Parts were wiped down with 'Kimwipes' to remove any excess oils or residues.



b. The assembly was fastened together by hand before using wrenches.



c. The heat tape was helically wrapped around the final assembly and secured using aluminum tape.

Figure 12. Assembling the final apparatus in preparation for installing the insulation.

To achieve these requirements, the insulation was first cut to length. Then, using calipers, measurements were made and marked for the locations of cutouts for the ball valve handle and needle valve stem. The locations were marked with a black marker and then cut out with a box cutter knife and scissors.

It was found that the insulation was too small and would only wrap around ³/₄ of the piping, leaving an open gap due to the excess material present from the heat tape and valve bodies. An extra piece of insulation was cut to fill in the gap, and the high temperature aluminum tape was used to wrap and secure the insulation in place. Small pieces of insulation were shaved off around the ball valve handle to make sure it had adequate room to open and close. Installation of this insulation along with creating the valve cutouts can be seen in Figure 13.

The 2-inch piping insulation was attached to the drip assembly next. As with the ¹/₂-inch insulation, the insulation was not able to fit around the entire vessel due to excess material present from the heat tape. This caused a gap that was filled by cutting an extra piece of insulation. The outer diameter of the ¹/₂-inch insulation was also small enough to allow the 2-inch insulation to fit snuggly over top and eliminate any possible gaps. Two criteria had to be met when putting this insulation on: (1) the heat tape temperature control box had to be physically touching and grounded to the assembly, and (2) the control box hadle had to be visible and accessible. To accomplish this, an opening was cut into the insulation where the control box was going to be physically touching the top vessel. The insulation was then cut at an angle to allow the control box to protrude outward while still allowing the insulation to contact the vessel.



a. The insulation was measured and marked for the valve handle and stem cutout locations.



b. A box cutter knife and scissors were used for making the cutouts.



c. The ¹/₂-inch insulation was installed and an extra piece of insulation was cut to fill the gap.



d. The ball valve handle is seen protruding through fully installed ¹/₂-inch insulation.

Figure 13. Installing the ¹/₂-inch insulation and creating the valve cutouts.

The vessel cap insulation was formed from both the 2-inch and ¹/₂-inch insulation. The larger insulation was cut to length where it would just cover the cap body and leave the cap flange exposed. The top of the cap was covered by a cut piece of ¹/₂-inch insulation which not only fit inside the outer 2-inch insulation but also had enough room for the cap fitting to poke through. A depiction of assembling the top half of the insulation can be seen in Figure 14.

After the apparatus was assembled, it was transferred to the Radiochemistry Lab (RCL) located in the Center for Advanced Energy Studies facility in Idaho Falls. The apparatus was placed into the large glovebox antechamber and flushed with argon gas three times. With the apparatus still inside, the antechamber was then evacuated to -25 in. Hg and left for three days. This process was to help degas the apparatus and remove any excess oxygen or moisture that could be present inside the insulation or attached to the apparatus walls. Once the process was complete, the apparatus was transferred into the glovebox. A depiction of this process can be seen in Figure 15.



Figure 14. Installing the 2-inch pipe insulation used for the vessel and cap. The insulation also acted as a support structure to keep the temperature control box in place.



a. The assembly was placed inside the antechamber and left over the weekend with signage.



b. The assembly was then transferred into the glove box and put on a stand.

Figure 15. The completed assembly was placed inside the glove box's large antechamber to degas for three days. The antechamber was evacuated to -25 in. Hg and signage was placed to inform anyone wanting to use the chamber during off hours.

Testing the Final Drip Assembly

The completed apparatus was rigorously tested once it was place in the glovebox to ensure the heat tape could consistently regulate the temperature and provide enough heat to the apparatus while it was fully assembled. The apparatus was prepared by suspending it from a lab stand using a chain clamp to secure the top and a three fingered clamp to hold the bottom. For safety, a grounding wire was attached to the apparatus on the needle valve body and grounded to the glovebox in case the heating tape short circuited to the apparatus. The temperature of the heat tape was verified by placing a thermocouple between the heat tape and the apparatus. A second thermocouple was placed inside the vessel through the cap fitting to verify the inside temperature of the apparatus.

The first test was performed to check if the heat tape could regulate its temperature for extended periods of time. To start, the heat tape was set to a low setting of about 50 °C. Gradually, the heat tape was turned up until the tape reached 130 °C and was left at this temperature for 1 hour. After an hour, the heat tape was turned up to 150 °C and then 170 °C, each temperature being held for an hour. The heat tape temperature would fluctuate roughly \pm 5 °C. To accurately re-create these temperatures in the next test, the control box dial was marked with adhesives that corresponded with specific temperature settings.

Extra caution was taken during the heating process while monitoring the oxygen and water contents inside the glovebox. It was not known if heating up the apparatus and insulation would cause oxygen or water to be released into the glovebox. During the process, however, the levels did not rise above normal operating conditions, which were between 0.1 and 20 ppm for both oxygen and water, separately.

A second test was performed to verify that the heat tape could replicate temperatures from the first test, as well as heat the nozzle to a temperature above the melting point of sodium. During this test it was found that gradually turning up the heat to the desired set point was better than starting at your final temperature, as this caused overshooting your desired temperature. The heat tape was plugged in and gradually turned to 150 °C. After about 1.5 hours, thermal equilibrium had been reached and the tip was stabilizing at a temperature of about 70 °C. This was well below the melting temperature of sodium and was a problem because after the first test using the sodium, residual sodium could remain in the nozzle. Once you try to conduct a second experiment the residual sodium would have solidified and blocked the opening of the nozzle and could not be melted due to a lack of sufficient heat to the nozzle. To address this problem, the apparatus was allowed to completely cool off, and the bottom half of the insulation was removed. The bottom half of the heating tape was undone and re-wrapped so that the end of the heating tape was surrounding the bottom of the needle valve right near the nozzle. The insulation was re-installed, as well as putting a new piece of insulation around the bottom half of the needle valve. After these measures were taken, the nozzle was able to get to 112 °C with a heat tape temperature of 173 °C. The process of taking the apparatus apart and rebuilding it can be seen in Figure 16.

The drip assembly was the original design, but, during testing of the sodium it was found that the sodium droplets would solidify too quickly on top of the insulation. Because of this, the second design involving the jeweler's furnace was created to keep the sodium molten. The furnace was readily available inside of the glovebox and could easily reach 500 °C, the temperatures needed for these tests.



a. The heat tape was re-wrapped so its end was surrounding the bottom half of the needle valve.



b. A new piece of insulation was placed on the bottom half of the exposed needle valve.



c. Nozzle temperature reached 112 $^{\circ}$ C with a steady heat tape temperature of 173 $^{\circ}$ C.

Figure 16. Modifying the drip assembly to ensure the nozzle got above 100 °C.

Jeweler's Furnace

Due to the challenges with maintaining sodium droplets in a molten state once they dripped from the custom assembly, an alternate experimental design was developed that would keep the sodium in a molten state for an extended period of time. This experiment used a jeweler's furnace to heat both the sodium and insulation sample to relevant temperatures, while providing a longer window for the molten sodium to interact with the insulation. The jeweler's furnace was a large Kerr Lab Automatic Maxi Electro-Melt [18] with a maximum temperature rating of 1120 °C. The furnace was digitally controlled with a Johnson 3216 controller. A depiction of the jeweler's furnace can be seen in Figure 17. A large graphite crucible served as both the sample holder and heat transfer medium for the furnace. A smaller alumina crucible was used as the container vessel for the insulation sample, and was lined with a stainless-steel foil sheath. The foil provided a barrier between the sodium and insulation and the inner crucible, preventing any contamination between the molten sodium and the alumina or graphite crucibles.



Figure 17. A depiction of the jeweler's furnace used in these experiments.

The insulation samples were prepared by taking the 2-inch pipe insulation and cutting small circular pucks that would fit snuggly inside the foil within the furnace beaker. The beaker and insulation sample were then placed inside of the furnace crucible. The preparation of the insulation pucks as well as the foil liners can be seen in Figure 18.



a. The insulation was cut into pucks that fit inside the alumina crucible.



b. Six-inch squares were cut from the stainless-steel foil.



c. The foil was pressed to form a liner inside the crucible.



d. View of the foil liner removed.



e. The alumina crucible, along with its liner, was then placed inside of the furnace crucible.



f. The alumina crucible fit snugly inside the furnace for good contact.

Figure 18. Depiction of making the insulation pucks used for testing samples as well as the foil liners for the alumina beakers.

Met-L-X Bags

Before the sodium could be placed into the glove box in the RCL, a fire suppressant needed to be present inside the glove box. This was needed to be able to extinguish any possible sodium fires that could occur inside the glovebox. The glovebox contained an inert atmosphere consisting of recirculating argon gas with maintained oxygen and moisture levels below 10 ppm. While the inert atmosphere reduces the risk of sodium reactivity, it was suggested that a source of direct fire suppression be incorporated into the experimental design. The fire suppressant of choice was Met-L-X powder [19]. Met-L-X is a Class D fire extinguishing agent used for extinguishing reactive metal fires, and was determined to the preferred material for extinguishing sodium fires.

Portions of Met-L-X powder were placed into small plastic bags for eventual (as needed) distribution near the sodium experimental equipment. Each small plastic bag contained 20 grams of powder and was made by first massing the powder on a scale and then transferring it into a bag. The excess air inside the bag was then removed by pinching the bag and squeezing the air out. The bag was then zipped shut. This bagging process was done inside a fume hood in the RCL, and a total of fifteen 20-gram bags were made. This bagging process can be seen in Figure 19.

After the Met-L-X bags were finished, they were then transferred into the glove box through the anti-chamber on a large plastic tray. A paper towel was draped over the bags and tucked underneath the tray on both sides. In case one of the bags popped inside the anti-chamber while it was being evacuated, the paper towel would catch the powder and prevent it from spraying everywhere.



Figure 19. Preparing the Met-L-X bags before being placed inside the glovebox.

Once inside, each bag of Met-L-X was opened with the help of tweezers and allowed to outgas with the argon atmosphere. This was to allow any trapped moisture or oxygen inside the bags to release. After outgassing occurred, the small bags were then split into two groups of 6 and 7 and placed into two larger plastic bags. These larger bags were then sealed and placed into a part of

the glovebox that was easily accessible. The transfer and outgassing process can be seen in Figure 20. In the case of a sodium fire, the researcher could quickly grab the bags of powder and simply drop them onto the flame and smother it. The heat from the flame would melt the plastic and release the Met-L-X powder which will extinguish any fire. This bag method was chosen due to its simplicity as well as its convenience since handling objects inside the glovebox can be arduous at times.



Figure 20. Transferring the Met-L-X bags into the glovebox via the antechamber.

Pre-Testing the Insulation Using Water

The purpose of these tests was to obtain an understanding of how a liquid with similar properties to molten sodium would permeate the insulation. It was originally supposed that water would be a good indicator of what liquid sodium would do since both fluids have similar densities. Liquid sodium also has a lower viscosity than water, indicating that sodium would flow easier. Distilled water was readily available and therefore used before sodium was tested inside the glove box

Two different tests were performed with the insulation. First, a penetration test was performed to specifically look at the inside layers of the insulation to see how the fluid travelled through these layers. Second, a simulation test was performed using a small size of insulation (1-inch cube) that would be the same size as the samples used for the sodium drip assembly test. Both tests used specimens cut from the same piece of 2-inch pipe insulation to ensure they were of the same make. Each piece of insulation was used for only one test to ensure the physical characteristics would be similar. A volumetric burette was used and acted as the drip assembly to supply water to the insulation at a controlled drip rate. The burette had a rubber tube attached to its end that acted as the straight nozzle on the drip assembly. The tube had an inner diameter of 0.27 inches. The burette and insulation set up can be seen in Figure 21.



Figure 21. General set up of a cut piece of insulation resting underneath a volumetric burette with its attached tube. Insulation specimens were placed with their center point beneath the burette.

Water Penetration Test

The penetration test was to see how the fluid travelled through the insulation and was performed with a piece of insulation that was 4.35 inches long. This test highlighted the comparison between how the fluid travelled horizontally along the insulation's layers and how it travelled vertically into the insulation. The dry weight of the insulation weighed 26.77 grams at the start of the test. The center of the insulation was placed underneath the burette and the test was started by filling the burette with water.

Within 20 seconds of starting the test, water began to appear just below the top layer of insulation as the water spread quickly out in a circular pattern. Soon afterward, a wet patch appeared on the top layer of the insulation. At the same time the water was observably moving

underneath the top layer of insulation towards the front edge. After 2 min 37 seconds the water reached the front edge of the insulation, and the test was stopped. The insulation was weighed to be 32.81 grams, which meant a total of 6.04 grams of water diffused into the insulation at an average drip rate of 2.3 g/min.

The insulation was cut in half and the water penetration was observed. The water penetrated the first layer to a depth of 0.12 inches. However, in the horizontal direction it travelled all the way to the front edge of the insulation, which was a distance of 2.17 inches from the drip point. The water also spread horizontally 1.63 inches up towards the back edge, which indicates that water found its path of least resistance and favored that direction. In the direction along the curvature of the insulation, the water was measured to travel up 1.15 inches from the drip point. A depiction of the cut insulation, the water penetration, and diffusion pattern measurements can be seen in Figure 22.



a. The insulation was cut to observe the water diffusion and penetration.



b. Water penetrated 0.12 inches vertically into the insulation then exclusively travelled horizontally along the layers path.



c. Water spread 1.15 inches along insulation curvature.



d. Water at first diffused in a circular pattern (seen at drip point) then quickly traveled horizontally to the front edge.

Figure 22. The water penetration and diffusion path. The water finds the path of least resistance and favors that direction.

Insulation Simulation Test

During the drip assembly experiment a small cube of insulation was used as the sample. A similar 1-inch cube of insulation was cut and tested for this water test. The sample had a dry weight of 1.02 grams and was placed under the burette, as seen in Figure 23. It can be seen that a smaller burette was used for this test as the piece of insulation was smaller. The tube attached to the burette still had an inner diameter of 0.27 inches, the same as the larger burette used.



Figure 23. Depiction of the test set up using a 1-inch cube of insulation.

Once the sample was positioned underneath the burette, the water was poured into the burette and allowed to drain. During the test, the top of the burette was lightly pressed upon to put pressure onto the insulation and compress it. This was to help the water ingress into the insulation and simulate how insulation would be compressed against the pipes they are insulating. This slight pressure was not needed for the previous insulation test because the larger burette had enough weight to slightly compress the insulation on its own.

After the water started to flow, the water quickly diffused into roughly half of the top layer and proceeded to penetrate down about 0.11 inches into the insulation and then spread horizontally out along that layer. After 3 minutes, the test was stopped and the insulation weighed to be 4.12 grams, giving a drip rate of about 1.4 g/min.

The insulation layers were progressively taken apart and the diffusion patterns were observed. These patterns can be seen in Figure 24. From the figure it can be seen that the water traveled straight through the top layer, and started diffusing more as it penetrated deeper and deeper. Once it reached a certain layer it was easier for the water to diffuse out into the entirety of this layer rather than penetrating deeper. Once this layer was saturated, the water began to form a finger as it started to penetrate deeper.



a. Water diffused into roughly half of the top layer before penetrating deeper.



c. Water diffuses along the entirety of the wet layer 0.11 inches from the top surface.



b. Water continues to penetrate deeper into the insulation.



d. Beneath the wet layer the tip of a finger is being manifested as the water penetrates deeper into the insulation.

Figure 24. Depiction of the insulation layers being progressively removed from the top layer down to the bottom of the wet layer.

Water Test Results

The predictions of the two experiments reviewed earlier as performed by Cueto-Felgueroso et al and Huppert et al are manifested in the fingering behavior depicted in the insulation tests done for this report. As water finds its path of least resistance in a homogeneous, non-saturated, and porous media it will penetrate via the formation of fingers. However, the insulation seems to provide varying degrees of permeability because of the presence of layers in the insulation.

We have seen in each of the tests performed that the water will most likely diffuse along a layer before penetrating deeper into the insulation. This was seen in the water penetration test as the water penetrated the layer just below the surface, and then diffused out along this layer to the insulation's edges. However, as was seen in the insulation simulation test, after an extended period of time, the effects of gravity take over and fingers begin to formulate as the water penetrated deeper into the insulation.

Each of the insulation tests performed here acted in similar manners with each other. The average drip rate for each test was slightly different, however, with the available equipment and the results obtained the insulation samples performed as predicted. As water and sodium have similar densities, this testing should be a good indication of how the sodium will behave with the insulation.

Drip Assembly Experiment

Before testing could begin, the sodium needed to be opened out of its original shipping package from Sigma Aldrich. The sodium was kept under an argon atmosphere during shipping and came fully oxidized in chunks of around 10 grams each. Before using the sodium for any experiment, the oxide was scraped off using a metal spatula.

To start the experiment, a piece of sodium was cut in half using a small metal spatula and the outside oxide was scraped off as best possible. The oxide was placed inside of a designated waste container that consisted of a small paint can with MET-L-X powder placed in the bottom. The sodium piece was massed to be 5.754 grams and was placed inside the drip assembly vessel. The thermocouples were put into place, with one inside the vessel through the cap fitting and the other placed between the heat tape and ball valve housing.

A 1-inch cube insulation sample was placed beneath the drip assembly nozzle by placing it on an upside-down 40 mL glass beaker that was placed inside of a 16 oz. paint can. The paint can was used to contain any sodium that might splatter or drip off of the insulation during testing. The top of the insulation piece was raised to just a few millimeters below the tip of the nozzle using a lab jack stand. The small MET-L-X bags that were prepared earlier were placed on either side of the apparatus to allow easy access in case they were needed. During the test, heat resistant gloves were used while handling the apparatus to prevent burning the glove box gloves. A depiction of preparing the sodium and the paint can holding the insulation sample can be seen in Figure 25.



a. The sodium had a layer of white oxide powder on its surface.



b. A spatula was used to break the sodium apart and scrape off the oxide.



c. The sodium had a dull gray appearance once the oxide was removed.



d. A paint can was placed underneath the drip assembly nozzle to hold the insulation sample for testing.

Figure 25. Preparing the sodium and insulation sample for testing.

The heat tape was turned on to a low temperature of 50 °C for 10 minutes to slowly heat up the apparatus. Every 20 minutes or so, the heat tape was slowly increased until it reached a steady 175 °C. After roughly 2.5 hours, the inside of the vessel was at 160 °C, and the test was started.

The needle valve was first opened to the smallest flow setting, and then the ball valve was then opened all the way. The nozzle was monitored closely, but no sodium appeared to be coming out. After a few minutes, the side of the drip assembly was gently tapped to see if it needed to overcome the surface tension of the sodium. The needle valve was then iteratively opened all the way to see if was preventing the sodium from flowing, but still no sodium droplets appeared.

Initially, it was supposed that the sodium was not coming out due to sodium freezing (or lack of thawing, technically) either within the nozzle or another part of the assembly. Temperature values were measured at a variety of locations along the length of the assembly; it was found that the housing of the needle valve was only reaching 77 °C while the tip of the nozzle was at a steady 107 °C. Additionally, the housing of the ball valve and needle valve were also exposed due to a lack of overlapping insulation. With this information, it was decided that the valves needed to be better insulated. The heat tape was turned off and the apparatus was allowed to completely cool. The ball valve was left open during the cooling process, as well as the needle valve. The insulation and paint can were kept in place, just in case any sodium might come out, although it was unlikely.

The next day, the drip assembly was removed from the lab stand and the cap was taken off. The inside of the vessel was visualized and it was noted that the sodium had melted and was now frozen inside of the body of the ball valve. It was also noted that a small amount of sodium oxide, which had initially been adhered to the chunk of sodium, was now resting on the top of the neck of the vessel, separate from the melted sodium metal. This indicated that the sodium was not flowing through the assembly because the sodium was most likely frozen inside the assembly rather than the oxide creating a blockage in the valve or nozzle.

Another test was performed after insulating the valves with quartz wool. A depiction of the drip assembly after the quartz wool was applied can be seen in Figure 26. The heat tape was turned on and increased to a temperature of about 175 °C over the course of a couple of hours. Though the ball valve and needle valve were still open from the previous test, no sodium droplets appeared. The heat tape was then turned up to 195 °C and the assembly was given time to reach thermal equilibrium

Temperature spot checks were then performed along the length of the drip assembly. It was noted that the needle valve body was at 120 °C, but the opening of the nozzle tip was only 93 °C, which is still several degrees below the melting point of sodium. The heat tape was turned off, and once cooled, was again re-wrapped around the assembly in an attempt to provide better heating throughout the entire assembly. A more significant portion of the heat tape was brought into contact with the nozzle and valve bodies to provide a higher heat load at these points. The heat tape was secured using high temperature aluminum tape, and the insulation was re-installed. The tip of the nozzle was protruding several millimeters from the insulation, to ensure that any sodium droplets that could form on top of the insulation sample during testing would not get soaked up by the apparatus insulation.



Figure 26. The drip assembly after quartz wool was applied to insulate the exposed needle and ball valves.

The insulation sample was put in place a few millimeters below the nozzle, along with the thermocouples and MET-L-X bags, and the heat tape was turned up in a similar matter as the first test. After about 2.5 hours, the heat tape was about 195 °C and the nozzle was reading a temperature of about 99.5 °C. With no visible signs of molten sodium exiting the nozzle, the insulation sample was raised (using the lab jack) to lightly touch the insulation to the nozzle tip. This would help to insulate the nozzle tip, and also provide slight pressure to help the sodium ingress into the fibers

of the insulation. The insulation was left in this position for an hour. After the hour, the insulation was lowered and observed. The top of the insulation showed no signs of sodium droplets. After a few minutes, however, a sodium droplet appeared. The droplet sat on top of the insulation sample in a spherical shape and did not ingress. After about 30 seconds, another droplet came out, combined with the first droplet, and then fell of the side of the insulation sample.

A few minutes later, another droplet came out, followed by two more droplets within a few minutes of time. Two of the droplets combined into one, and the third stayed separate. It took roughly 30 seconds for the sodium droplets to solidify on top of the insulation. This was discerned by observing the sheen of the fresh sodium droplets fading into a dull silver. The assembly and nozzle were observed for another hour, however, no further droplets were formed. Given time constraints within the laboratory, the test was stopped at this point and the heat tape was turned off.

After 15 minutes to allow the insulation sample and sodium to cool, the paint can was removed from underneath the assembly and observed. The two solidified droplets of sodium that had landed onto the top of the insulation sample promptly fell off the side and onto the bottom of the paint can when the sample was moved. This, along with no visible sodium residue left on the sample, indicated that the sodium did not ingress into the insulation.

The bottom of the paint can was observed, and it was found that a puddle of solidified sodium was present. Since no sodium was present prior to the start of the test, the logical solution is that the sodium had indeed formed droplets during the hour that the insulation was pressed up against the nozzle, and had rolled off the side into the bottom of the paint can. Even with slight pressure, the sodium did not ingress into the insulation sample. Figure 27 shows the formation of the droplets on top of the sodium as well as the pool of sodium at the bottom of the paint can.

After deducing that the sodium was not going to ingress in the current set up, it was decided that a different approach was needed to create an environment that encouraged the sodium to infiltrate the sample. The two likely scenarios to get the sodium to permeate the insulation were either to provide an environment where the sodium could remain in a molten state longer, or the system needed to be pressurized. Pressurizing the sodium was not a feasible option based on the facilities available, so the path forward was to devise a method to keep the sodium in a molten state longer in the hopes of allowing it time to ingress into the insulation. This was accomplished by using a jeweler's furnace.



a. The first two droplets combined on top of the insulation and had a silver sheen before they solidified.



b. The third droplet did not combine with the first two. The smaller, shiny droplet is the third one that had not solidified yet.



c. A small puddle of solidified sodium was found at the bottom of the test paint can indicating sodium had been flowing straight off the insulation sample.

Figure 27. The molten sodium either flowed straight off the insulation sample or formed droplets on top of the insulation and solidified.

Jeweler's Furnace Experiment

After realizing that the sodium was not going to ingress under the conditions set forth by the drip assembly, a new approach was developed using a jeweler's furnace. The furnace heated the sodium and insulation together at higher temperatures, which could encourage the penetration of the molten sodium into the pores of the insulation. The jeweler's furnace was an ohm resistance heater with a maximum temperature of 1120 °C. The furnace lid had a pre-existing hole carved out in its insulation to allow for instrumentation to be put in place and visualize samples during testing. This hole was covered during testing with a piece of solid furnace insulation to conserve heat. The insulation samples used for testing were made by cutting the 2-inch pipe insulation into small circular pucks that would fit snuggly in the alumina crucible inside the furnace. The alumina crucible was lined with steel foil to create a barrier in case any sodium melted off the surface of the insulation.

The first four furnace tests performed used insulation samples that were freshly cut from the pipe insulation. After the first four tests were completed, the insulation samples were altered since no interaction or ingression between the sodium and insulation occurred up to that point. In a typical reactor, insulation can become thermally aged over time due to the heat load applied from the pipes they are insulating. This thermal aging can change the insulation properties and possibly burn off the organic binders present in the insulation. To see if thermal aging had any effect on the sodium ingressing into the insulation, the insulation samples were baked in an oven at 300 °C for 6 hours. During the aging process the insulation samples turned a grey-white color, contrasted by the yellow color of new insulation from the factory. These aged insulation samples were used for the rest of the tests. Depictions of the new and pre-baked insulation samples can be seen in Figure

28.



Figure 28. Comparison between the new insulation and pre-baked insulation.

Over the course of the furnace tests performed, four different methods were used to bring the sodium and insulation together. The first and most basic method was placing the sodium on top of the insulation sample and heating it up. This would indicate whether gravity alone would cause the sodium to permeate the insulation. The second method was splitting the insulation sample in half in the horizontal direction and placing the sodium in between the two pieces of insulation in a 'sandwich' formation. There was also a small beaker placed on top of the sandwich insulation sample to provide pressure to the sodium. The sandwich method was to simulate a small amount of pressure that the sodium could be under as it leaked from a pipe into the insulation. The small beaker provided about 0.14 psi of pressure to the sample.
The third method was cutting a shallow hole into the insulation and allowing the sodium to melt and take the shape of the hole. This was to simulate how the sodium might behave once it has already passed the surface and was found inside of the body of the insulation. This method had a small beaker placed on top to provide a direct pressure of 0.14 psi to the sodium The fourth method was placing the sodium on the bottom of the foil sleeve and pressing the insulation into it from above utilizing a small beaker. This beaker provided 0.45 psi of pressure.

Sodium on Top Method

Four tests were performed by placing the sodium on top of the insulation and seeing if gravity alone had a strong enough effect to cause the sodium to ingress into the insulation. These tests were all performed at either 150 or 200 °C. Three of the tests used new insulation, while one of them used pre-baked insulation. In all four cases the sodium was seen to not ingress into or interact with the insulation sample. After each test, the sodium would easily peel away from the insulation sample and there appeared to be no interaction. A depiction of the sodium easily peeling away from the surface of the insulation after a test can be seen in Figure 29. The results from each of these tests can be found in Table 4. From the results of the table, it can be seen that there was no difference between the results that used the new versus the pre-baked insulation samples.



Figure 29. Depiction of the tests performed by placing the sodium on top of the insulation sample and seeing if ingression occurs during heating. After each test, the sodium would easily peel away from the surface of the insulation, indicating no ingression occurred.

Sodium Mass (g)	Insulation Mass (g)	Temperature (°C)	Test Duration (hr)	Ingression?	Notes
1.27	2.58	150	2	No	The sodium easily peeled away from the insulation.
1.27	2.58	200	5.5	No	During the test, the molten sodium was probed with a spatula, and it appeared that there was a thin shell of oxide on the outside layer of sodium.
0.64	2.27	200	5.5	No	There was no indication to the naked eye that the sodium had interacted with the insulation.
0.43	2.69*	200	8	No	The pre-baked insulation showed no signs of interaction.

Table 4. Test results for placing the sodium on top of the insulation and allowing gravity to be the driving force. All tests used new insulation samples except for one.

* Indicates the insulation was pre-baked at 300 °C for 6 hours before testing.

Sandwich Method

The sandwich method consisted of splitting a sample of insulation in half in the horizontal direction and placing a piece of sodium in between the two pieces of insulation. After seeing no ingression when the sodium was placed on top of the insulation, it was determined that perhaps the sodium was not ingressing because it needed some pressure to force it in. The surface tension of the sodium could have been playing a bigger role than was previously anticipated and preventing the sodium from ingressing. The surface tension of sodium at its melting temperature is 191 dynes/cm, roughly 2.5 times higher than that of water, which is 72.8 dynes/cm at room temperature [20].

To provide pressure to the sample during testing, a small alumina beaker weighing 107.139 grams was placed on top of the insulation. The bottom diameter of the beaker was 3.75 cm, giving an approximate pressure of 0.14 psi onto the insulation sample. The small beaker was tall enough that its top rested outside of the furnaces hole. Several pieces of insulation had to be stacked around the top of the beaker to keep it insulated.

Five tests were performed with a temperature range of 200 - 400 °C. All tests except for one used pre-baked insulation samples. No ingression of the sodium occurred at any temperature during this sandwich testing, however, once the furnace temperature reached 300 °C, the insulation began to discolor in locations that were directly contacting the sodium. As the temperature was increased to 350 and 400 °C, the insulation began to discolor and blacken even more as well as degrade and break apart in locations that were contacting the sodium. A depiction of this discoloration can be found in Figure 30. The results from each test can be found in Table 5.



Figure 30. During the sandwich method, at temperatures of 300 °C and higher, the insulation began to blacken and degrade in locations directly contacting the sodium.

Sodium Mass (g)	Insulation Mass (g)	Temperature (°C)	Test Duration (hr)	Ingression?	Notes
0.641	2.27*	200	5	No	Sodium was flattened by the pressure and travelled up the side of the foil.
0.94	2.81	250	5.5	No	Loose fibers had broken off and froze into the sodium during testing.
0.94	2.81	300	6	No	The insulation began to blacken where it contacted the sodium.
0.94	2.81	350	6	No	Insulation continued to blacken.
0.79	2.37	400	6	No	Insulation began to degrade and break apart in places that were touching the sodium.

Table 5. Test results for the sandwich method. During each test 0.14 psi of pressure was applied to the sample using a small beaker. All tests except for one used pre-baked insulation.

* Indicates the insulation was new and not pre-baked.

Hole Method

This method was designed to see how the sodium behaved after it had broken past the surface of the insulation. This might occur after a sodium leak occurs and the pressure from inside the pipe initially jets the sodium into the pipe insulation. To simulate this scenario, a shallow hole was cut into the center of the insulation sample, about ½ inch deep and ¼ inch wide. A small beaker was then placed on top of the sodium sample and provided a direct pressure of 0.14 psi to see if the sodium would ingress.





b. The sodium that was beneath the surface of the insulation inside the hole appeared to be unoxidized.

Figure 31. A depiction of the first hole test.

Five tests were performed with a temperature range of 200 - 400 °C, all of which used prebaked insulation. During the first test at 200 °C, the sodium was not pressurized to allow the sodium sample to melt and take the shape of the hole cutout in the insulation. After this first test was complete, it was observed that the top of the sodium that was exposed to the glovebox atmosphere appeared to have oxidized, while the sodium beneath the surface of the insulation was unoxidized. The results from the first test can be seen in Figure 31.

The four tests that followed all had the small beaker placed on top to apply pressure and were performed at temperatures of 250, 300, 350, and 400 °C. During these tests, no interaction between the sodium and insulation occurred until a temperature of 400 °C, at which point the insulation began to blacken. Small pieces of insulation had also broken off and were frozen into the sodium. A depiction of these test results can be seen in Figure 32. A description of each test can be found in Table 6.



a. The insulation was showing signs of discoloration around location of the sodium.

b. Pieces of blackened insulation froze into the insulation.



Up to this point, the sodium still showed no signs of ingression into the insulation. For further guidance, Mr. Randall S. Fielding of Idaho National Laboratory was contacted, who had direct experience working with sodium. His advice was to run the tests at a higher temperature. During Mr. Fielding's experience, he had to run the sodium at around 500 °C in order to handle the sodium and break its surface tension. He suggested that the surface tension was just too great and preventing any sort of infiltration into the insulation samples.

Sodium Mass (g)	Insulation Mass (g)	Temperature (°C)	Test Duration (hr)	Ingression?	Notes
1.51	2.95	200	7	No	Oxide appeared to form on the top surface of the sodium that was exposed to the glovebox atmosphere.
1.51	2.95	250	5.5	No	No sign of interaction.
1.51	2.95	300	6	No	No sign of interaction.
1.51	2.95	350	6	No	No sign of interaction.
1.51	2.95	400	6	No	The insulation began to discolor and blacken. Small bits of insulation had also broken off and were frozen into the sodium.

Table 6. Test results for the hole method. All tests were performed with pressure applied except for the first test. Each test utilized a pre-baked insulation sample.

Sodium on Bottom Method

Taking the advice from Mr. Fielding, it was decided to not only go to higher temperatures but also apply more pressure to the sample. This was accomplished by placing the sodium at the bottom of the steel foil and pressing the insulation into the sodium from above with a weighted beaker. This would be making it so the sodium would have a solid backing and force the sodium to either ingress into the insulation or squeeze out the sides.

The weighted beaker was created by taking the small alumina beaker used for the previous tests, wrapping its bottom in a protective steel foil sheath, and placing it on top of the insulation to provide pressure. The beaker was then filled with 234.783 grams of 316 stainless steel bolt nuts, making the total weight of the small beaker, the beaker sheath, and nuts 349.775 grams. With this added weight, the small beaker was now applying around 0.45 psi of pressure onto the insulation and sodium sample.

Three tests were performed at temperatures of 400, 450, and 500 °C. After testing the insulation at 400 and 450 °C, the insulation was blackened and brittle, especially in locations directly contacting the sodium. No ingression occurred during these tests, however, small bits of blackened insulation broke off during each test and would freeze into the sodium, indicating that some mixing was occurring as the insulation degraded. After the last test performed at 500 °C, the entirety of the insulation was blackened with the most degraded portion the part that was touching the sodium. The results from the 500 °C test can be seen in Figure 33. The specific parameters of each of the three tests performed can be found in Table 7.



a. The recovered sodium was covered by small bits of insulation that had broken off during testing. The bottom of the insulation that was in direct contact with the sodium was very brittle and blackened.



b. The top of the insulation was blackened, but not as degraded as the insulation in direct contact with the sodium.



Sodium Mass (g)	Insulation Mass (g)	Temperature (°C)	Test Duration (hr)	Ingression?	Notes
1.71	2.32	400	5	No	The insulation blackened where it was contacting the sodium.
1.71	2.32	450	6	No	Small pieces would break off and stick to the solidified sodium.
1.71	2.32	500	6	No	The entirety of the insulation was blackened, not just areas in contact with the sodium.

Table 7. Test results for placing the sodium on bottom with an applied pressure of 0.45 psi.

Experiment Results

This series of experiments was meant to investigate the migration patterns of molten sodium as it permeated typical fiberglass insulation. The experiment began with a drip assembly that liquified the sodium and released it into the top surface of insulation samples. It was discovered that the sodium would solidify quickly while on top of the insulation and not have enough time to ingress into the insulation. The experimental design then shifted to a standard jeweler's furnace that would keep the sodium in a liquified state for several hours while in contact with the insulation. Four different configurations were carried out under varying temperatures and applied pressures.

At temperatures above 350 °C, the insulation in contact with the molten sodium began to blacken and degrade, causing the insulation to become more fragile and brittle. It was also observed that once the sodium cooled, small pieces of brittle insulation were frozen within the cooled sodium, which would indicate that the insulation was breaking up on its surface and mixing into the molten sodium.

Initially, it was suspected that the molten sodium would permeate through the insulation much like the water did in the preliminary tests that were performed due to the similar densities of water and molten sodium. Molten sodium also has a lower dynamic viscosity than water which suggests that it might permeate the insulation better than what was observed in the water tests. However, sodium has a much higher surface tension than water, which is likely playing a role in inhibiting the sodium from ingressing into the pore structure of the insulation. From the observations in these experiments, the sodium did not permeate the insulation, even when pressure was applied at elevated temperatures for extended periods of time. The insulation would burn and degrade in areas where it made direct contact with the molten sodium, but the sodium did not appear to permeate the insulation layers. The maximum rated temperature for the insulation was 538 °C (1000°F), so the highest temperature used in testing was 500 °C. The insulation started to blacken and become brittle at temperatures around 350 °C in places that were in direct contact with the sodium. At 500 °C, the entirety of the insulation was blackened and burned.

Inside the secondary heat transport system of a SFR, sodium temperatures can reach upwards of 500 °C. If a leak is present, the leaking sodium temperature can be above 300 °C, which would be hot enough to start this degradation process of insulation at the location of the leak. One hypothesis is that the high-temperature molten sodium degrades and then erodes the insulation, causing it to become brittle and fail prematurely, instead of permeating through the insulation as was originally suspected.

Another finding was that in a furnace temperature range of 350 - 450 °C, the insulation would mostly only blacken in spots that were in direct contact with the sodium. At 500 °C, however, the entire piece of insulation would blacken. The sodium could have acted as a localized heating element due to its high thermal conductivity and provided a large heat load directly to the insulation at 350 - 450 °C. This heat load was large enough to start the degradation process of the insulation. At the higher temperature of 500 °C, the heat load from the furnace was high enough to degrade the entirety of the insulation.

Pipe leaks often occur long after the piping insulation has been installed. Over time, the piping insulation is thermally aged and begins the degradation process even before any leaked sodium comes into contact with the insulation. Differences in the interaction between sodium and new vs. aged insulation was investigated by artificially aging pieces of insulation in an oven for 6 hours at 300 °C. When the insulation was aged, it turned from its original yellow color to a white – grey color as if it had chemically changed, possibly attributed to the volatilization of the organic binders due to the excessive heat. Interestingly, the sodium did not permeate the insulation regardless of whether the insulation was thermally aged or not.

After the insulation was artificially aged in the oven, the insulation continued to age during the testing process inside the furnace. With every test performed, the insulation would be subjected to 5 - 6 hours of high heat as it was in contact with the sodium inside the furnace. This continual aging process may have attributed to the eventual breakdown of the insulation and caused it to become brittle. However, the tests at 350 - 450 °C showed that the insulation only blackened and became brittle in locations that were touching the sodium and not elsewhere, indicating that the sodium magnified the thermal degradation of the insulation in locations it was contacting.

Conclusion

Sodium Fast Reactors are among the candidates for the next generation of nuclear reactors. With newfound interest in SFRs and the advantages they possess, further research is needed in this field of study. Safety is a priority to the next generation of SFRs, especially when it comes to sodium leaks and leak detection. This work addresses a specific need outlined by the NRC, where accurate leak detection is needed in regions coated with insulation. Knowing how sodium interacts with fiberglass insulation during a leak scenario is important to the safe operation of these reactor facilities, by possibly reducing the risk of sodium fires and unanticipated shutdowns. Knowledge on how sodium and insulation interact during a leak scenario will help pave the way to future leak detection design and implementation.

This collection of experiments was developed to investigate the physical mechanisms of interaction between molten sodium and fiberglass insulation. It was demonstrated that independent of differing temperatures and applied pressures, molten sodium did not permeate the porous structure of the fiberglass insulation. Testing temperatures ranged from 150 – 500 °C, and the applied pressure ranged from atmospheric to ~0.5 psig. Both fresh and thermally aged insulation samples were used in this study, however, this did not result in substantial permeation of the sodium into the pores. At temperatures between 350 - 450 °C, the insulation began to degrade and become brittle in locations where it was in direct contact with the sodium. Rather than the sodium permeating through the insulation, it instead thermally degrades the insulation over time.

Future Research

These experiments only considered the possible interaction mechanisms for one type of insulation – standard fiberglass insulation. Other types of insulation have been used in SFRs, such as mineral wool, reflective insulations, and ceramic wools. The working temperature of these insulation types are higher than the fiberglass being tested and may interact with molten sodium differently than fiberglass

Another part of this experiment was the sodium being packaged and shipped in the form of chunks that were covered in powdered oxide. Before testing, the oxide was removed as best as possible through scraping it off. Even in small amounts, oxide was still present in the sodium throughout the testing process and may have caused the sodium to behave differently than pure sodium when interacting with the insulation. However, sodium will naturally oxidize once it has leaked from a pipe onto insulation in an SFR and should act similar to the results found in these experiments.

These experiments dealt with small sample sizes of sodium and insulation inside of a drip assembly and jeweler's furnace. The sodium was stationary as it was in contact with the insulation in the case of the jeweler's furnace. In a real scenario, the sodium would be leaking from a pipe at various flow rates into the surrounding insulation. These experiments provide an initial attempt to recreate a molten sodium leak scenario but could be improved in several ways. It is worth considering how pressure from inside of a sodium pipe might affect the properties of leaking sodium, as well as the mobility of the sodium once it has leaked. In most SFRs, the sodium will be leaking into insulation that is exposed to the atmosphere, which contains oxygen and water vapor. Inside the glovebox there was an inert argon environment. How the sodium reacts with the insulation in a non-inert environment will likely be different than how it reacted in these experiments. These experiments provide a fundamental understanding of how a sodium leak will behave when it contacts fiberglass pipe insulation.

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Appendix A: Fiberglass Insulation Data Specification Sheet

The following specification sheet is referring to the insulation that was used for sodium testing throughout this thesis. The specific insulation used was 2-inch piping insulation with a thickness of 1 inch. The type of insulation was rigid core with a maximum operating temperature of 1000 $^{\circ}$ F.





SSL II[®] WITH ASJ MAX FIBERGLAS[™] PIPE INSULATION FIBERGLASS INSULATION

Owens Corning[®] SSL II[®] with ASJ Max Fiberglas[™] pipe insulation is molded of heavy-density resin-bonded inorganic glass fibers that come in one-piece, 36-inch-long (914 mm), hinged sections. The insulation is tailored to fit for copper and iron pipe applications.

Features

- ASJ Max is an all-service jacket with a polymer film exterior surface that is smooth, durable, cleanable, wrinkle-resistant, resists water staining, and doesn't support mold or mildew growth¹
- ASJ Max can resist short durations of water exposure that may occur during construction
- SSL II® Positive Closure System is an advanced double adhesion that fastens and installs with no need for staples or mastic
- Insulation is tailored to fit with:
 - a flexible core to compress over copper and some small-bore iron pipes and fittings, saving time by eliminating the need to fillet
 - a rigid core for fast and easy fabrication on larger pipes
- The product has a maximum operating temperature of: Flex Core 850°F (454°C); Ridge Core 1,000°F (538°C) (with heat-up schedule)
- The product does not contain Polybromodiphenyl ethers (PBDE) (penta-, octa-, or deca-brominated diphenyl)
- 1 ASJ Max jacket does not support mold growth as tested in accordance with ASTM C1338.

Standards, Codes Compliance

- ASTM C547, Mineral Fiber Pipe Insulation:
 - Flex Core Type I, Grade A
 - Ridge Core Type IV, Grade B
- ASTM C585, Inner and Outer Diameters of Thermal Insulation for Nominal Sizes of Pipe and Tubing
- ASTM C1136, Flexible Low Permeance Vapor Retarders for Thermal Insulation: Types I, II, III, IV, X
- UL Labeled for Flame Spread Index of 25 or less and Smoke Developed Index of 50, and is fully building code compliant
- UL Listed and Labeled for use over PVC and other polymer pipes; UL Category BSMP; see Technical Bulletin Pub. No. 10023253
- ASTM C795, Thermal Insulation for Use in Contact with Austenitic Stainless Steel²
- Nuclear Regulatory Commission Guide 1.36, Non-Metallic Thermal Insulation²
- NFPA 90A and 90B
- 2 Preproduction qualification testing complete and on file. Chemical analysis of each production lot required for total conformance. Certification needs to be specified at time of order.

Physical Properties

PROPERTY	TEST METHOD	VALUE
Density (size dependent)	ASTM C303	3.5 to 5.5 pcf
Operating Temperature Range	ASTM C411	Flex Core – 0°F to 850°F (-18°C to 454°C) Ridge Core ³ – 0°F to 1,000°F (-18°C to 538°C)
Water Vapor Sorption	ASTM C1104	Less than 5% by weight
Jacket Temperature Limitation	ASTM C1136	-20°F to 150°F (-29°C to 66°C)
Jacket Permeance	ASTM E96, Proc. A	0.01 perm
Burst Strength, min	ASTM D774/D774M	100 psi
CORROSION RESISTANCE	TEST METHOD	VALUE
Corrosion to Copper and Aluminum	ASTM C665	Pass – cooper and aluminum
Corrosion to Steel	ASTM C1617	Pass
Stress Corrosion Evaluation on External Stress Corrosion Cracking Tendency of Austenitic Stainless Steel	ASTM C795 and ASTM C6922	Pass
Chemical Analysis for Cl-, Fl-, Na+, SiO3	ASTM C795 and ASTM C871 ²	Results fall within acceptability limits
FIRE	TEST METHOD	VALUE
Composite Surface Burning Characteristics ⁴	UL 723, ASTM E84, or CAN/ULC-S102	Flame Spread 25 Smoke Developed 50

3 With heat-up schedule when operating temperatures between 850°F and 1,000°F.

4 The surface burning characteristics of these products have been determined in accordance with UL 723, ASTM E84, and CAN/ULC-S102. Values are reported to the nearest 5 rating.

Applications

- Used to insulate iron, copper, PVC, and other polymer pipes with operating temperatures between 0°F (-18°C) and 1,000°F (538°C) in commercial and institutional buildings and industrial facilities
- When temperatures are above 650°F (454°C), maximum installed insulation thickness shall be no greater than 6 inches as a single layer or nested
- Flex Core Rated per ASTM C547, Type I, Grade A Pipe insulation can be installed on in-service/hot pipes with an operating temperature up to 850°F (454°C)
- Rigid Core Rated per ASTM C547, Type IV, Grade B When operating temperatures will be between 850°F (454°C) and 1,000°F (538°C), a heat-up schedule needs to be followed per the Installation Instructions, Pub. No. 10021355
- When installed outdoors, an additional weather-protective jacket is required

Thermal Conductivity

MEAN TEMPERATURE °F	k Btu•in/hr•ft²•°F	MEAN TEMPERATURE °C	λ ₩/M∙°C
50	0.22	10	0.032
75	0.23	25	0.034
100	0.24	50	0.037
150	0.27	100	0.043
200	0.29	125	0.047
250	0.32	150	0.051
300	0.35	175	0.056
350	0.39	200	0.062
400	0.43	225	0.068
450	0.48	250	0.075
500	0.54	275	0.082

Apparent thermal conductivity values determined in accordance with ASTM practice C1045 with data obtained by ASTM Test Method C335. Values are nominal, subject to normal testing and manufacturing tolerances.

Thickness to Prevent Surface Condensation

Owens Corning® ASJ Max Jacket for up to 16 inches NPS (400 mm DN)^{5,6}

AMBIENT TEMPERATURE		RELATIVE HUMIDITY	SYSTEM OPERATING TEMPERATURES					
°F	°C		35°F	(2°C)	45°F	(7°C)	55°F	(13°C)
		70%	1	(25)	1	(25)	1	(25)
110	(43)	80%	11/2	(38)	11/2	(38)	11/2	(38)
		90%	31/2	(89)	31/2	(89)	3	(76)
		70%	1	(25)	1	(25)	1	(25)
100	(38)	80%	11/2	(38)	11/2	(38)	1	(25)
		90%	31/2	(89)	3	(76)	21/2	(64)
		70%	1	(25)	1	(25)	1	(25)
90	(32)	80%	11/2	(38)	1	(25)	1	(25)
		90%	31/2	(89)	3	(76)	21/2	(64)
80	(27)	80%	11/2	(38)	1	(25)	1	(25)
		90%	3	(76)	21/2	(64)	2	(51)
70	(21)	80%	1	(25)	1	(25)	1	(25)
70	(21)	90%	21/2	(64)	2	(51)	1	(25)

5 Calculations estimated using NAIMA 3E Plus version 4.0 software. Fixed design conditions: Steel Horizontal Piping, 16* NPS, 0 mph wind speed, Outer Surface Jacket Emittance of 0.9.

6 Thermal conductivity values used in these calculations are subject to normal manufacturing tolerances.

Acoustic – Insertion Loss in dB per ASTM E1222

Fiberglas[™] Pipe Insulation with ASJ Max and SSL II[®]

	AT 1" THICKNESS	AT 2" THICKNESS
FREQUENCY (HZ)	INSERTION LOSS (dB)	INSERTION LOSS (dB)
315	-3	-2
400	2	0
500	1	0
630	3	1
800	0	-3
1000	6	8
1250	б	7
1600	10	13
2000	11	13
2500	16	20
3150	18	23
4000	19	23
5000	18	22

Availability

Our Fiberglas[™] pipe insulation portfolio is available in thicknesses up to 5 inches. Contact your local Owens Corning area sales manager for product availability.

Refer to "Fiberglas[™] Pipe Insulation Sizing Manual" for more information (Pub. No. 10018078).

Installation

Ambient application temperatures are from 25°F (-4°C) to 110°F (43°C).

For complete installation instructions and recommendations, see "Fiberglas™ Pipe Insulation Installation Instructions" (Pub. No. 10021355).

Environmental and Sustainability

Owens Corning is a worldwide leader in building material systems, insulation, and composite solutions, delivering a broad range of highquality products and services. Owens Corning is committed to driving sustainability by delivering solutions, transforming markets, and enhancing lives. More information can be found at <u>www.owenscorning.com</u>.

Certifications and Sustainable Features

- Certified by SCS Global Services to contain an average of 53% recycled glass content, 31% pre-consumer, and 22% post-consumer.
- For faced products: GREENGUARD certified products are certified to GREENGUARD standards for low chemical emissions into indoor air during product usage. For more information, visit <u>ul.com/gg</u>.
- Environmental Product Declaration (EPD) has been certified by UL Environment.
- RainBarrier[®] products have a published Health Product Declaration (HPD).



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Notes

For additional information, refer to the Safe Use Instruction Sheet (SUIS) found in the SDS Database via http://sds.owenscorning.com.

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Appendix B: Leak Testing the Drip Assembly

This testing was performed by Jordan Harley and Morgan Robbins

This appendix describes the methods used to leak test the drip assembly. The drip assembly was assembled outside of the glovebox first to ensure its capabilities and test its leak tightness. Each part of the assembly was thoroughly tested for leak tightness with air and water. Leak testing was necessary to ensure that the assembly would be able hold liquid sodium that would eventually be placed inside. The testing was done in two stages, first an air test and then a water test.

Stage 1: Air Test

The air test was performed to check that the threads and valves were airtight and would not allow any unwanted gases into or out of the vessel and attached parts. The valves were tested to check their integrity by pressurizing them with air from a pressurized air tank. The valves were then submerged under water to observe for air bubbles. The same process was used on the top vessel. A ¹/₂-inch male, ¹/₄-inch female NPT bushing was needed to connect the pressurized air tank hose to the ball valve since the air tank hose connection was ¹/₄-inch male connection. PTFE pipe tape was applied to all the threads in this test as this is the tape that was used for the final assembly.

Most parts had air bubbles of some kind appear during testing. The majority of the bubbles appeared to be coming through the threads. This was remedied by either tightening the part or reapplying the pipe tape to get a better seal. Once a good seal was found, the number of layers of pipe tape used to make the seal at each connection point was recorded so it could be referenced when making the final assembly. The number of pipe tape layers required for each part is recorded in Table 8.

ers of pipe tape
3 1/2
5
5
4 1/2
3 1/2
3 1/2

Table 8. Layers of pipe tape needed for each thread.

Throughout the testing process, it was found that applying the pipe tape was not trivial. If too few layers of pipe tape were applied, air would seep out through the threads due to a poor seal. If too many layers of pipe tape were applied, the parts would only get a few threads of connection due to the NPT taper. Generally, the larger threads needed more layers since their threads were deeper.

Air Test Procedure

- 1 Charge the air tank to 30 psi. (This was accomplished by using a compressor at a local gas station).
- 2 Fill a five-gallon bucket with water, about ³/₄ full.
- 3 Wrap all threads with sufficient pipe tape (in accordance with Table 8).
- 4 Attach the needle valve to the air tank hose, hand tight (make sure the valve is oriented correctly with the air flowing in the proper direction).
- 5 Tighten the valve with an adjustable wrench until tight.
- 6 Make sure the valve is CLOSED.
- 7 Slowly open the air tank and pressurize the valve.

- 8 Submerge the valve into the bucket full of water.
- 9 Observe for bubbles (if bubbles are observed, see below for next steps *, otherwise go to next step).
- 10 Close the air tank.
- 11 Slowly open the needle valve and release the residual pressurized air.
- 12 Detach the needle valve from the tank hose.
- 13 Attach the ¹/₂"-male, ¹/₄"-female bushing to air tank hose, hand tight.
- 14 Tighten bushing using an adjustable wrench until tight.
- 15 Attach the ball valve to the bushing, hand tight.
- 16 Tighten the valve with an adjustable wrench until tight.
- 17 Repeat steps 6 -12 to test the ball valve.
- 18 Attach the vessel cap to the top vessel.
- 19 Attach the vessel to the ¹/₂-inch globe valve (the valve should still be attached to the bushing)^a.
- 20 Torque the vessel 5 N-m beyond hand tight.
- 21 Make sure the valve is OPEN.
- 22 Slowly open the air tank to pressurize the pipe vessel and cap.
- 23 Submerge the entire assembly into the bucket full of water.
- 24 Observe for bubbles (if bubbles are observed, see below * for next steps).
- ^a A ¹/₂ inch female NPT globe valve was available that was used to attach the vessel to the air tank

* Bubbles may appear either at the <u>threads</u> or at the <u>valve opening</u>. Depending on where the bubbles appear, see below

Threads

- Take the apparatus out of the water.
- Retape the threads with pipe tape.
- Repeat steps to check for bubbles.

Valve Opening

- Check and make sure valve is completely closed.
- Repeat steps and check for bubbles.
- If valve is still leaking, the valve is defective.



a. Air tank was pressurized to about 30 psi.



b. Threads were wrapped with PTFE pipe tape.



c. Parts were connected to the air tank hose and pressurized.



d. Pressurized parts were submerged under water and observed for bubbles.

Figure 34. Steps to testing parts via pressurized air and water submersion.

Stage 2: Water Test

The water test was performed to check that the seals and valves were water-tight and would not allow the flow of liquid substances into or out of the apparatus. This test was not pressurized and only gravity fed, which correlates with the actual experiment which did not involve pressurizing the sodium. This fact, along with the previous pressurized air test, was sufficient to make sure no sodium leaks will occur during the experiment. The top vessel was used to hold the water and provide the gravity positive pressure for the thread tests. Figure 35 illustrates the various steps taken in this water test.

Water Test Procedure

- 1 Wrap all threads with sufficient pipe tape (in accordance with Table 8).
- 2 Attach the cap to the top vessel, hand tight.
- 3 Fill the vessel with water through the smaller end.
- 4 Invert the vessel upside down and place on level surface (so the vessel cap is on bottom).
- 5 Observe for leaks at threads for at least 10 minutes (if a leak is detected, see below **).
- 6 Drain out water and remove the cap.
- 7 Attach the vessel to the ball valve, hand tight.
- 8 Tighten valve with adjustable wrench.
- 9 Secure the apparatus to the ring stand using the chain clamp in an upright-vertical position (vessel on top, valve on bottom).
- 10 Make sure the valve is CLOSED.
- 11 Fill the vessel with water.
- 12 Observe for leaks for at least 10 minutes (if a leak is detected, see below **).

- 13 Drain the water out of the apparatus.
- 14 Attach the ¹/₄-inch male, ¹/₂-inch female fitting to the ball valve, hand tight.
- 15 Tighten fitting using an adjustable wrench.
- 16 Attach the needle valve to the fitting, hand tight.
- 17 Tighten valve with adjustable wrench.
- 18 Make sure the ball valve is OPEN.
- 19 Make sure the needle valve is CLOSED.
- 20 Fill the assembly with water.
- 21 Observe for leaks for at least 10 minutes (if a leak is detected, see below **).

** Water may leak through either the <u>threads</u> or the <u>valve opening</u>. Depending on where the leak appears, see below.

Threads

- Drain the apparatus of the water
- Retape the threads with pipe tape
- Repeat steps to check for water leaks

Valve Opening

- Check and make sure valve is completely closed
- Repeat steps and check for water leaks
- If valve is still leaking, the valve is defective



a. Fill vessel with water to maximum level.



b. Orient the filled apparatus to allow positive gravity water pressure towards threads and valves.



c. Inspect threads to make sure water is not seeping through.



d. Inspect valve openings to make sure water is not leaking.

Figure 35. Steps to testing parts for water leaks.

Appendix C: Testing the Heat Tape

This testing was performed by Jordan Harley and Morgan Robbins

This appendix describes the methods of testing the heat tape to ensure that it worked outside of the factory. Data was also needed to know how the drip assembly was going to react to the temperature transients that it was going to be put under while heating up in the glove box. The heat tape was rated to reach temperatures as high as 218 C, but for this test a temperature of 80 °C was reached.

The heating tape was a multi-wire stranded; glass knitted heating element meant for heating pipes. To protect the heating filament from short circuiting or accidently being ground, its entrapped in a silicone rubber matrix. The silicone also acts as a second dielectric protective layer that lets the heating element keep its flexibility and make it possible to handle safely without any protections. The heating element temperature was controlled by a temperature control box, with a simple knob acting as the user interface. The control box had a thermostat to help regulate the filament temperature, but only worked if the control box was grounded and physically touching the piping it was heating. The heat tape was equipped with a 6 ft power cord with a standard 2 prong plug (NEMA 1-15). The plug was rated for 1.2 A, 120 V, so it can be plugged into most standard electrical outlets.

The heat tape was tightly wrapped in a helical shape around the apparatus to keep a good contact between the metal surface and the heating filament. The high temperature aluminum tape was used in several locations to secure the heat tape and make sure it didn't move. A clamp secured the temperature control box to the vessel and made sure the box had constant contact with the

assembly. The assembly was then suspended on a lab stand using the chain clamp. Two thermocouples were used during the heating process, one placed on the outside surface of the assembly and the other placed on the inside surface. The apparatus and heat tape testing set up can be seen in Figure 36.

The heat tape was turned on to about half power and the surface temperatures were continuously monitored. While the apparatus was heating, the temperature gradient between the outside and inside surfaces was consistently between 13-14 °C. After several minutes, the outside surface temperature reached the desired 80 °C, and the heat tape was promptly turned off. Due to residual heat in the heat tape, the outside temperature rose to 81 °C, and the inside temperature equalized to 70 °C. The apparatus stayed at this temperature for a short period and then slowly cooled off to room temperatures over the course of about 30 minutes.

During heating, there was at one point a small whisp of smoke seen to appear, but it quickly vanished and was not seen again. After inspecting the apparatus and heat tape it was concluded that the smoke may have come from the heating of residual oil the testers' skin that the heat tape contacted. To prevent any excess oils or residues being present in the final assembly, the surfaces of each part were handled with gloves as much as possible, as well as wiped down with 'Kimwipes'.



Figure 36. Heat tape being tested on the apparatus. The temperature control box was secured to the top of the vessel with a clamp, ensuring good contact with the vessel surface. Two thermocouples can be seen, one on the inside surface of the vessel, and the other being placed on the outside surface of the ball valve.