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Chemical Interaction of Standard Insulation with Molten Sodium

by

Morgan Robbins

A thesis

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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 MORGAN ROBBINS find it satisfactory and recommend that it be accepted.

---

Daniel LaBrier, Ph.D.,  
Major Advisor

---

Chad Pope, Ph.D.,  
Committee Member

---

Bruce Savage, Ph.D.,  
Graduate Faculty Representative

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# Chemical Interaction of Standard Insulation with Molten Sodium

## Thesis Abstract -- Idaho State University (2022)

The interaction between fiberglass insulation and sodium metal was investigated. The sodium metal was heated up to a maximum temperature of 500°C while in contact with fiberglass insulation inside a jeweler's furnace. Samples were procured from the insulation near the contact points to the sodium metal. The acquired samples were analyzed by a Focused Ion Beam (FIB) apparatus with an Energy Dispersive X-ray Spectroscopy (EDS) unit for visual inspection and to obtain elemental composition of the insulation samples. Insulation samples displayed visual signs of fiber deviation from controls by sodium metal interaction. Elemental constituents for control samples were found to be in line with fiberglass compositions. Increases in the sodium content were observed and the sodium compounds of sodium oxide ( $\text{Na}_2\text{O}$ ) and sodium carbonate ( $\text{Na}_2\text{CO}_3$ ) were identified as candidates to be present in the experimental samples.

Key Words: fiberglass insulation, sodium metal, sodium compounds, sodium fast reactors

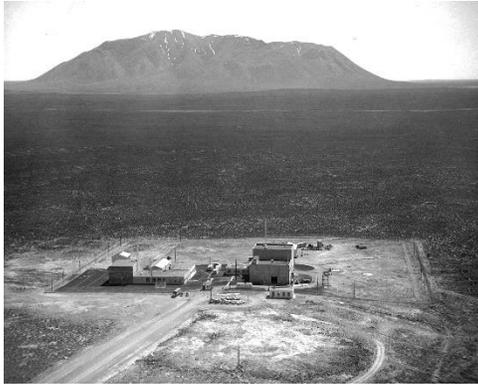
# Chapter 1: Introduction

## 1.1 Sodium Fast Reactors

### 1.1.1 History in the United States

The United States has had ample experience with sodium fast reactors (SFRs) in the past 70 years with Experimental Breeder Reactor – I (EBR-1), Fermi I, and Experimental Breeder Reactor – II (EBR-II). Each of the aforementioned showed that a reactor could use liquid sodium as a coolant. The reactors were eventually shutdown for separate reasons ending sodium fast reactors in the United States.

EBR-I was fabricated to test the design and feasibility of a breeder reactor to maximize energy output from natural uranium. EBR-I was the first liquid metal cooled fast reactor that used uranium metal pins as fuel and a sodium-potassium alloy (NaK) as its coolant. On December 20, 1951 EBR-I became the first power-producing reactor when it provided the energy to power four lightbulbs and subsequently provided power for its building as shown in Figure 1 (1; 2).



a) EBR-I location in Idaho.



b) Light bulbs lit by the world's first electricity generated from atomic power.

FIGURE 1 PHOTOS OF THE EBR-I FACILITY AND LIGHT BULBS LIT (1).

The successful operation of the EBR-I demonstrated not only that a reactor could generate electricity but also that a reactor could generate more fuel than it consumed. The reactor went through many core design changes to increase the generation ratio. After the design and implementation of the Mark-IV core in 1963, the reactor was shut down and decommissioned proving that a breeder reactor was possible (1; 2).

The Enrico Fermi Nuclear Generating Station (Fermi 1) located in Michigan had a sodium fast reactor in its facilities, as shown in Figure 2. The reactor reached criticality in 1963 with a subsequent power operating license in 1965. The reactor had a maximum generation capacity of 430 megawatts-thermal ( $MW_{th}$ ). Fermi I was shut down in 1972 when the core was nearing its burnup limit and subsequently decommissioned (3).



a) Enrico Fermi Generation Station Fermi II site.



b) Outside of photo (a) to the left, Fermi I site.

FIGURE 2 ENRICO FERMI GENERATION STATION FERMI I AND FERMI II SITES.

The most recent sodium fast reactor was EBR-II (Experimental Breeder Reactor II) which operated from 1964 to 1994. EBR-II was a pool-type reactor design in which the core was submerged in liquid sodium and coolant was pumped through the core to a heat exchanger as shown in Figure 3. The heat would be transferred to a secondary loop of sodium and heat transfer again with the water loop to go to the turbine for electricity generation. EBR-II was eventually shut down due to a loss of federal funding and ended the sodium fast reactor sector within the United States (1).

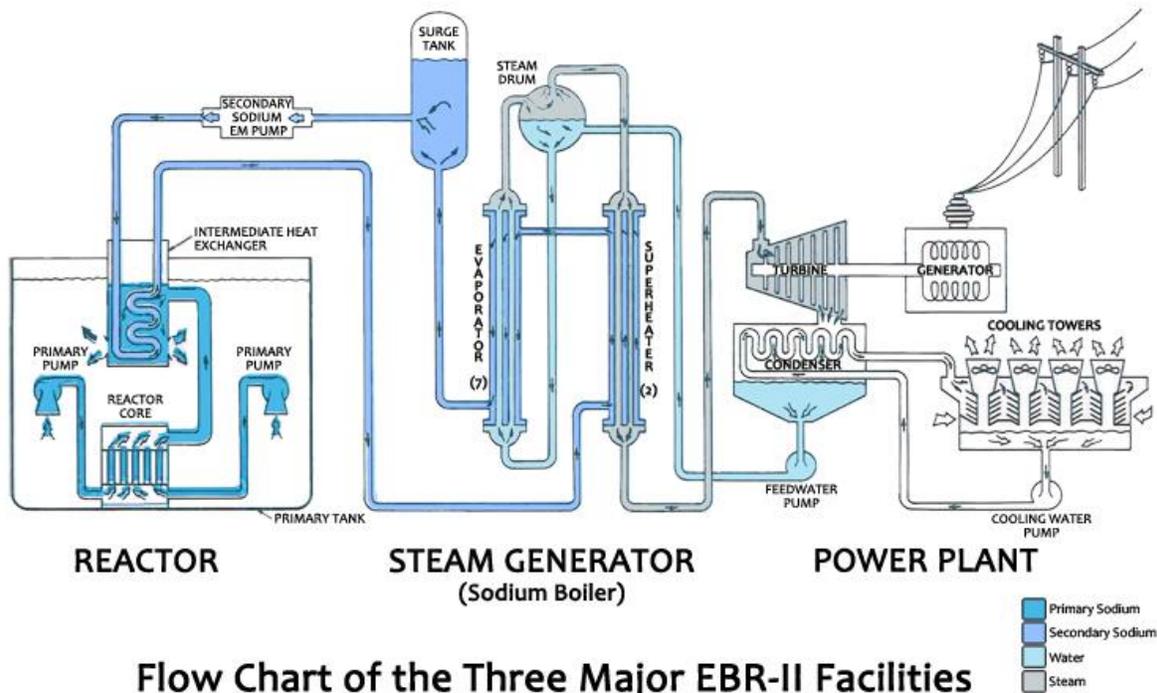


FIGURE 3 EBR-II FLOW CHART (1).

### 1.1.2 Renewed Interest

In the year 2000, the US Department of Energy (DOE) initiated an international cooperation to address energy needs and climate concerns for the future that nuclear energy systems could provide. In 2001, the international cooperation was chartered as the Generation IV International Forum (GIF) with Argentina, Brazil, Canada, France, Japan, the Republic of Korea, the Republic of South Africa, Switzerland, the United Kingdom, and the United States as founding members (4) with Australia, China, Euratom, and the Russian Federation joining later (5; 6). The GIF created a set of goals that would need to be achieved by nuclear energy systems to address the concerns of the future, including the following (4):

Sustainability-1: Generation IV nuclear energy systems will provide sustainable energy generation that meets clean air objectives and provides long-term availability of systems and effective fuel utilization for worldwide energy production.

Sustainability-2: Generation IV nuclear energy systems will minimize and manage their nuclear waste and notably reduce the long-term stewardship burden, thereby improving protection for the public health and the environment.

Economics-1: Generation IV nuclear energy systems will have a clear life-cycle cost advantage over other energy sources.

Economics-2: Generation IV nuclear energy systems will have a level of financial risk comparable to other energy projects.

Safety and Reliability-1: Generation IV nuclear energy systems operations will excel in safety and reliability.

Safety and Reliability-2: Generation IV nuclear energy systems will have a very low likelihood and degree of reactor core damage.

Safety and Reliability-3: Generation IV nuclear energy systems will eliminate the need for offsite emergency response.

Proliferation Resistance and Physical Protection: Generation IV nuclear energy systems will increase the assurance that they are very unattractive and the least desirable route for diversion or theft of weapons-usable materials, and provide increased physical protection against acts of terrorism.

The nuclear energy systems that were selected to fulfill the goals set are referred to as Generation IV. As shown in Figure 4 below, there are five generations of nuclear energy systems. Generation I systems were early prototype reactors like that of Fermi I discussed earlier in the 1950s and 60s. Generation II covers commercial power plants that still operate today from the 1970s. Generation III systems are from the 1990s with significant improvements to safety and economics. Generation III+ are advancements on Generation III systems with active development after 2000. Generation IV systems are designs for 2030 and beyond when the Generation II systems reach their end of license (4; 5).

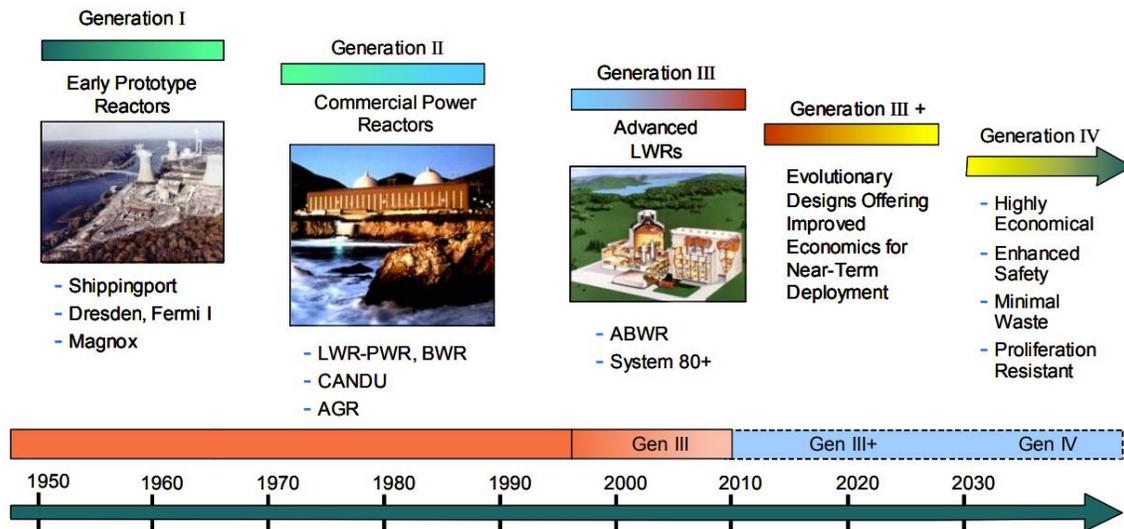


FIGURE 4 TIMELINE DISPLAYING NUCLEAR ENERGY SYSTEM GENERATIONS (4)

The GIF selected six prospective nuclear energy systems to be focused on and researched as Generation IV. The nuclear energy systems are:

- Gas-Cooled Fast Reactor System
- Lead-Cooled Fast Reactor System
- Molten Salt Reactor System

- Sodium-Cooled Fast Reactor System
- Supercritical-Water-Cooled Reactor System
- Very-High-Temperature Reactor System

The motivation behind the selection of these systems were: [1] to accomplish advancements in technology goals; [2] address electricity generation, hydrogen and process heat production, and actinide management; [3] overlapping coverage of capabilities to bring commercial deployment; and [4] accommodate national priorities of GIF countries (4; 5).

### **1.1.3 Sodium Insulation Concern**

As part of the push for improved nuclear systems the U.S. Nuclear Regulatory Commission (U.S. NRC) published a report addressing non-light-water reactor materials and experience. Within the report, several areas of further interest and need for research were addressed, including the need to “confirm the chemical compatibility between molten sodium and insulation material” (7).

The concern of chemical compatibility between molten sodium and insulation material stems from an event that occurred at the Phénix reactor in France. During operation on May 5, 1986 the beaded wires at the inlet to the steam generator reported an electric contact. There were no visual signs of a leak on the outside of the insulation so the plant continued operating. The Phénix reactor was shut down as scheduled on May 19 with the insulation at the inlet to the steam generator being partially removed on May 21. The removal of the insulation revealed solidified sodium. Analysis showed that the weld for the t-piece cracked and had leaked sodium for several

thousand hours resulting in a mixture of sodium and insulation that corroded the stainless-steel pipe (8).

## 1.2 Sodium

### 1.2.1 Element Properties

Sodium (Na) is a chemical element known as the alkali metal, located in the first column and on the third row with an atomic number of 11, as shown in Figure 5. The elemental properties of sodium can be found in Table 1. Sodium has a melting point of 97.81 °C (208 °F) and a boiling point of 882.9 °C (1,621 °F). The density of sodium is very similar to that of water at 0.971 grams per cubic centimeter (g/cm<sup>3</sup>).

Sodium has an electron configuration of  $1s^22s^22p^63s^1$  where the numbers '1' and '2' denote energy level as well as the row on the periodic table, the 's' and 'p' denoting which electron shell type, and the superscript numbers following denoting how many electrons are in that shell. The 's' shell can hold a maximum of two electrons with the 'p' shell having a maximum of six. The  $3s^1$  electron is a valence electron that makes sodium highly reactive with other elements. Sodium is the sixth most abundant element on Earth but is never found in its elemental form due to its high reactivity.

## Periodic table of the elements

group	1*	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
1	H																	He
2	Li	Be											B	C	N	O	F	Ne
3	Na	Mg											Al	Si	P	S	Cl	Ar
4	K	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br	Kr
5	Rb	Sr	Y	Zr	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Te	I	Xe
6	Cs	Ba	La	Hf	Ta	W	Re	Os	Ir	Pt	Au	Hg	Tl	Pb	Bi	Po	At	Rn
7	Fr	Ra	Ac	Rf	Db	Sg	Bh	Hs	Mt	Ds	Rg	Cn	Nh	Fl	Mc	Lv	Ts	Og
lanthanoid series	6	58	59	60	61	62	63	64	65	66	67	68	69	70	71			
		Ce	Pr	Nd	Pm	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu			
actinoid series	7	90	91	92	93	94	95	96	97	98	99	100	101	102	103			
		Th	Pa	U	Np	Pu	Am	Cm	Bk	Cf	Es	Fm	Md	No	Lr			

\*Numbering system adopted by the International Union of Pure and Applied Chemistry (IUPAC). © Encyclopædia Britannica, Inc.

FIGURE 5 PERIODIC TABLE OF THE ELEMENTS (9).

TABLE 1 SODIUM PROPERTIES

Elemental properties	Value
Atomic Number	11
Atomic Weight	22.9898 g/mol
Melting Point	97.81 °C (208 °F)
Boiling Point	882.9 °C (1,621 °F)
Density	0.971 g/cc
Electron Configuration	1s <sup>2</sup> 2s <sup>2</sup> 2p <sup>6</sup> 3s <sup>1</sup> or [Ne]3s <sup>1</sup>

### 1.2.2 Compounds

There are three chemical forms of sodium compounds that are of interest: hydroxide (OH), carbonate (CO<sub>3</sub>), and oxide (O). Hydroxide formation occurs when sodium (Na) comes into contact with water (H<sub>2</sub>O), creating sodium hydroxide (NaOH), hydrogen gas (H<sub>2</sub>), and a release of energy as shown in Equation (1). This is of concern since the volatility of the reaction increases

with increased temperature and the hydrogen gas can build up, if allowed to, which can lead to ignition. An alternate way to create sodium hydroxide is the reaction between sodium oxide ( $\text{Na}_2\text{O}$ ) and water as shown in Equation (2). Sodium hydroxide is a colorless liquid that is denser than water and can severely irritate the skin and eyes if come into contact with. It is also corrosive to metals and tissue.

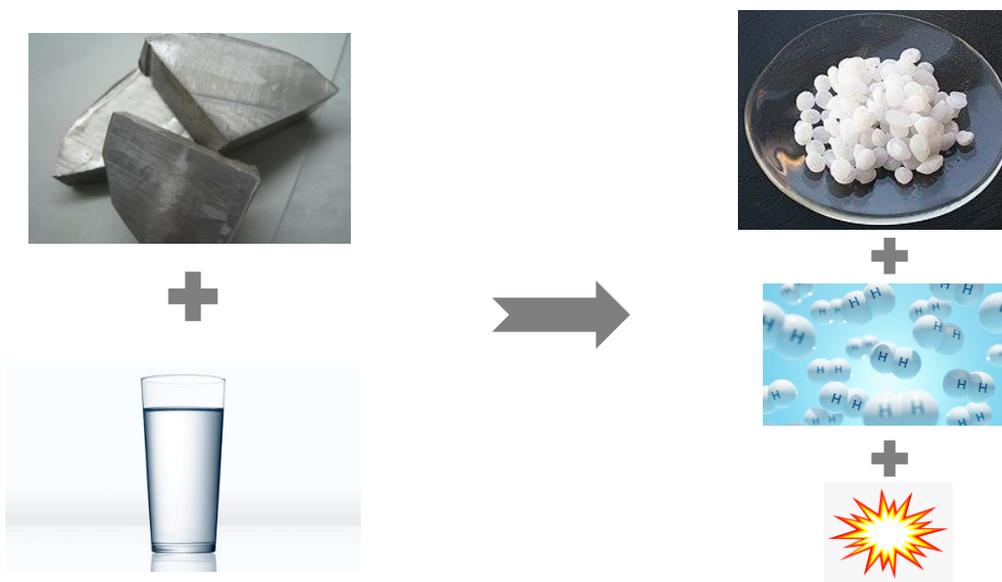
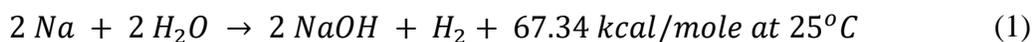


FIGURE 6 SODIUM METAL AND WATER REACTION INTO SODIUM HYDROXIDE, HYDROGEN, AND ENERGY RELEASE.



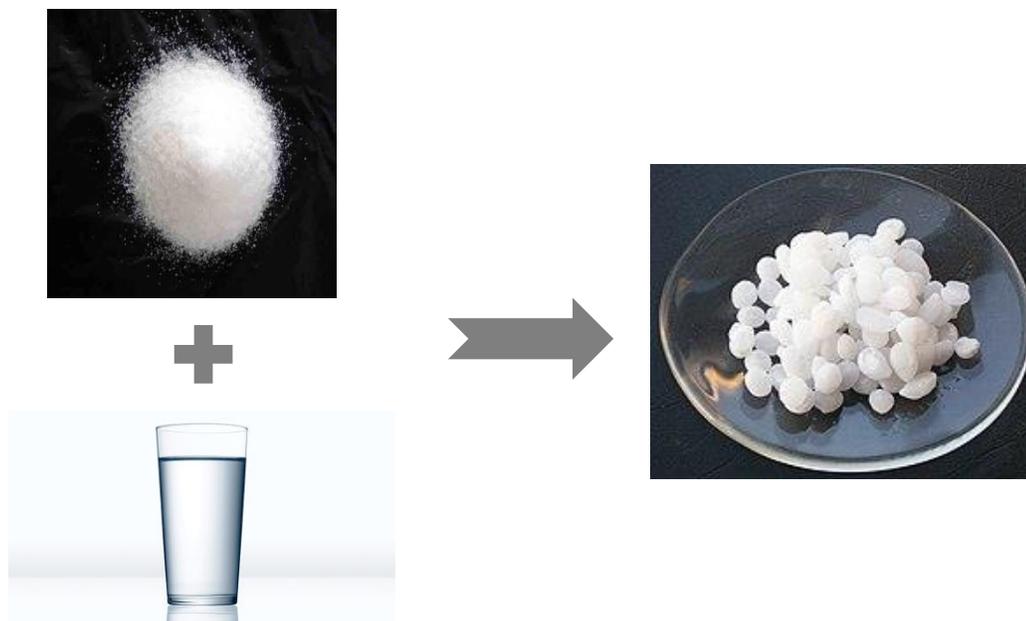


FIGURE 7 SODIUM OXIDE AND WATER REACTION INTO SODIUM HYDROXIDE.

Carbonate formation occurs when sodium hydroxide and carbon dioxide ( $\text{CO}_2$ ) react resulting in sodium carbonate ( $\text{Na}_2\text{CO}_3$ ) and water as shown in Equation (3). Sodium carbonate, also known as washing soda, is a white, odorless, water-soluble salt that is used in many domestic cleansing agents. Sodium carbonate can react with carbon dioxide and water to create sodium bicarbonate ( $2\text{NaHCO}_3$ ), known as baking soda, as shown in Equation (4).



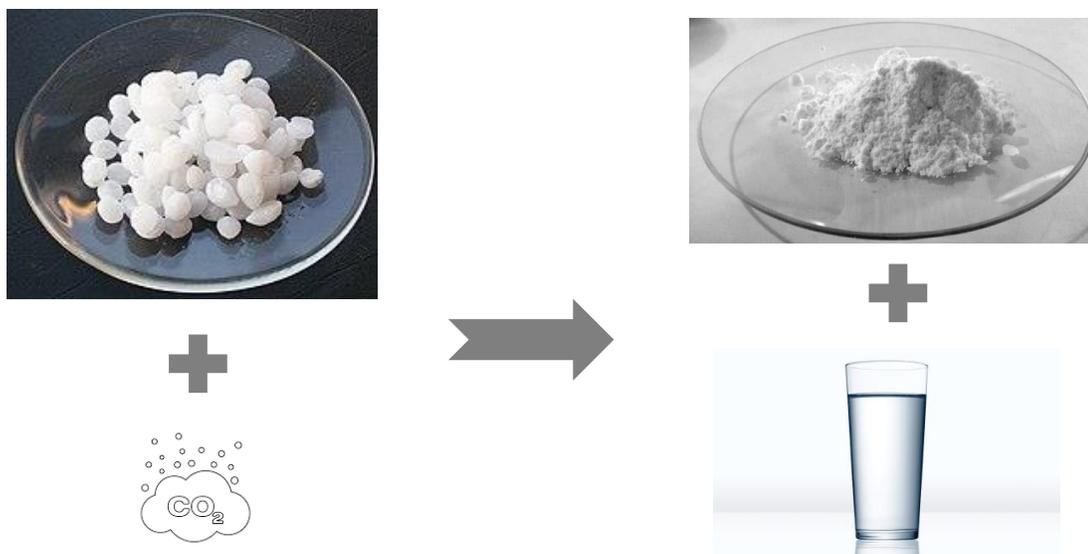


FIGURE 8 SODIUM HYDROXIDE AND CARBON DIOXIDE REACTION INTO SODIUM CARBONATE AND WATER.



FIGURE 9 SODIUM CARBONATE, CARBON DIOXIDE AND WATER REACTION INTO SODIUM BICARBONATE.

Oxide formation can happen when sodium hydroxide reacts with sodium resulting in sodium oxide ( $Na_2O$ ) and hydrogen gas as shown in Equation (5). Sodium oxide also forms from

the reaction of sodium and oxygen ( $O_2$ ) with sodium peroxide ( $Na_2O_2$ ) as a byproduct in Equation (6). Sodium peroxide can further react with sodium to produce sodium oxide in Equation (7). Sodium peroxide is an oxidizer and corrosive appearing as a yellow-white or yellow granular solid. Sodium oxide can react with water to produce sodium hydroxide as shown in Equation (8).

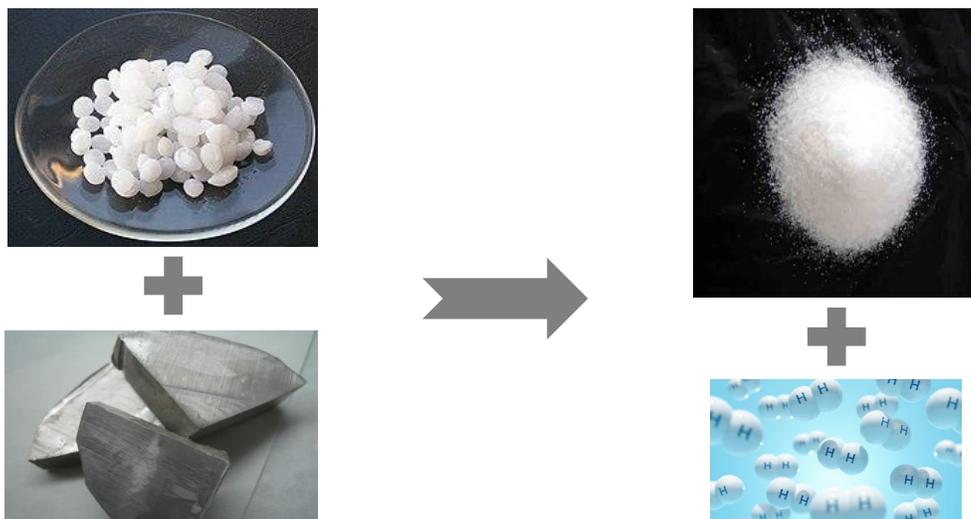
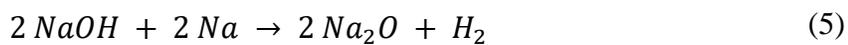
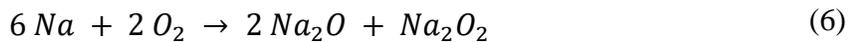


FIGURE 10 SODIUM HYDROXIDE AND SODIUM METAL REACTION INTO SODIUM OXIDE AND HYDROGEN.



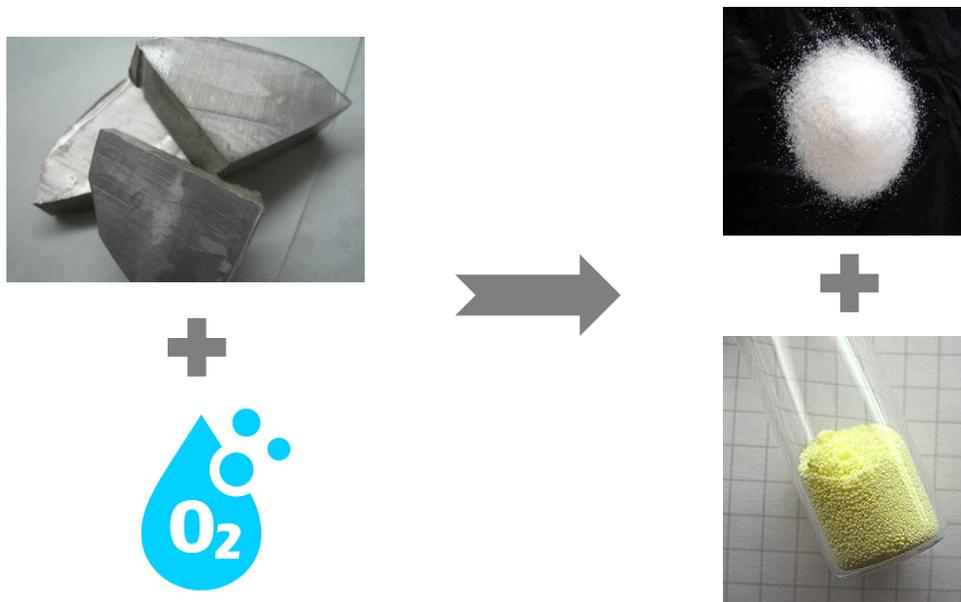


FIGURE 11 SODIUM METAL AND OXYGEN REACTION INTO SODIUM OXIDE AND SODIUM PEROXIDE.

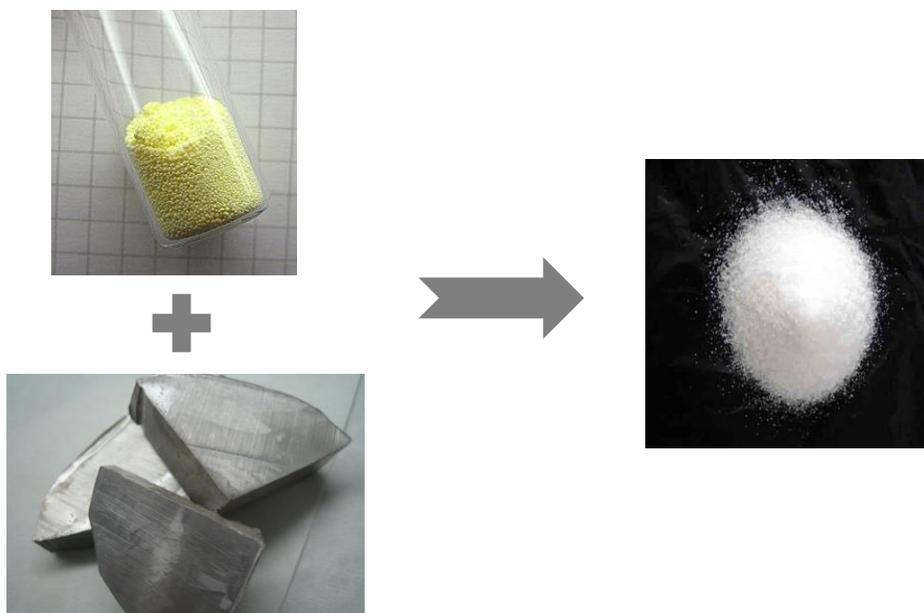


FIGURE 12 SODIUM PEROXIDE AND SODIUM METAL REACTION INTO SODIUM OXIDE.



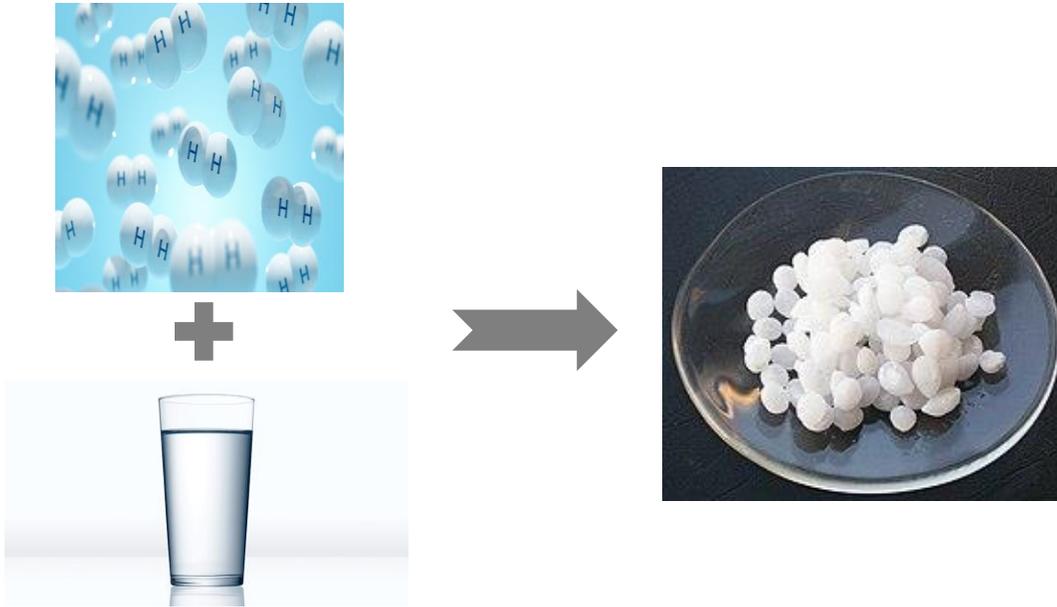


FIGURE 13 SODIUM OXIDE AND WATER REACTION INTO SODIUM HYDROXIDE.

### 1.3 Insulation

Insulation materials are used throughout the world in many applications to reduce thermal losses. In the case of nuclear reactors, insulation material is used on piping that transport working fluids such as water, sodium, and gas. There are four types of insulating materials that are used; fibrous, reflective metal, particulate, and foam (10). A brief discussion of these types is presented here along with results from an industry survey.

#### 1.3.1 Fibrous

Fibrous insulation consists of low-density fiberglass (LDFG), high-density fiberglass (HDFG) and mineral wool. In the case of LDFG and HDFG, the insulation material is crafted through melting a composition of silica, aluminum, calcium, magnesium, boron, soda, potassium, iron, titanium, and strontium with some manufacturers adding fluorine. The composition is then spun into fibers ranging from 1.25-10 microns in diameter with lengths ranging from ¼-inch to

infinite length (11). The same process is followed with mineral wool though the composition is derived from melted rock and/or slag. The mineral wool is spun to 3-10 microns in diameter with lengths of ¼-inch to several inches (10; 11). Figure 14 depicts fiberglass and mineral wool insulation.



a) Fiberglass insulation



b) Mineral wool insulation

FIGURE 14 FIBERGLASS AND MINERAL WOOL INSULATION.

The LDFG, HDFG, and mineral wool are then coated with an organic binder to bind the fibers together. The binder used is phenolic resin which is uncured making the insulation material tacky (10; 11). The insulation with the resin is cured and ready to be used. The fibrous insulation works through reducing air convection and thermal radiation heat transfer. High temperatures result in the degradation of the insulation material with the resin degrading into CO<sub>2</sub> and water vapor at temperatures above 232°C.

### 1.3.2 RMI

Reflective metal insulation (RMI) focuses on the radiant heat transfer process and reducing its effect. RMIs are made with aluminum or stainless steel with layers of paper, plastic, or foam

(10; 12). The effectiveness of RMI systems is dependent on the emittance of the materials used. With the creation of small air spaces to reduce heat transfer by convection, RMIs can reduce the radiant heat transfer up to 97% (12). Figure 15 shows a RMI setup.



FIGURE 15 METAL REFLECTIVE INSULATION.

### 1.3.3 Particulate

Particulate insulation is comprised of calcium silicate, Min-K, and Microtherm (10; 13). Particulate insulation is made through amorphous silica, lime, reinforcing fibers and additives mixed with water to create a slurry. The slurry is then boiled and poured into molds. The molds are placed in an indurator (steam pressure cooker) to let the chemical reaction happen to form calcium silicate. Once finished, the newly formed calcium silicate is placed in a drying oven. Once dried, the calcium silicate is trimmed and packaged. The insulation is a crystalline formation with more air than solid space (>90% air) (14). Calcium silicate insulation is shown below in Figure 16.



FIGURE 16 CALCIUM SILICATE INSULATION.

### 1.3.4 Foam

Foam insulation comes in the forms of foamglas, foamed plastic, and neoprene (10). Foam insulation is made from recycled glass that is crushed and melted. The glass is mixed with additives to allow the glass to expand into a foam. Foam insulation create cells that share walls thus not allowing any convection to occur within the material. Depicted below in Figure 17 is foamglas insulation.



FIGURE 17 FOAMGLAS INSULATION.

### 1.3.5 Industry Survey

A survey of the insulation materials used in PWR facilities was performed to assess the types and locations of insulation used (15). Most of the responders to the survey indicated that they use fibrous insulation in the form of low-density fiberglass and mineral-wool. Of the 40 PWR units that provided percentages of insulation, about 30 reported the use of fibrous material (e.g., Nukon, mineral wool, and generic fiberglass). Five units responded with at least 30% of the piping insulation as calcium-silicate (15).

## Chapter 2: Methods

The primary goal of this project was to investigate the chemical interaction between molten sodium and a representative type of insulation that might be used in a SFR facility. Therefore, an experiment was devised to allow molten sodium to interact with fiberglass insulation through the use of a drip assembly. The drip assembly was then placed into an argon environment glovebox. During the testing, it was realized that the drip assembly could not achieve what was originally desired and the experiment switched to using a furnace within the glovebox. Once testing was complete, samples of the insulation were prepared and extracted from the glovebox to oxidize before analysis.

### 2.1 Drip Assembly

#### 2.1.1 Assembly Design

An assembly was constructed to allow for the containment of sodium to be heated and then deposited onto a piece of fiberglass insulation. The material used for the assembly to drip the sodium was 316 Stainless Steel because of its excellent characteristics and compatibility with liquid sodium. If a necessary part was not available in 316 SS material, a close material match was found. Table 2 outlines each part that the drip assembly is comprised of, along with the part description, the material the part is made of, the company the part was purchased from, and the associated part number.

TABLE 2 SODIUM DRIP ASSEMBLY COMPONENTS

<b>Part Description</b>	<b>Material</b>	<b>Company</b>	<b>Part #</b>
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

A rendering of the completed drip assembly can be seen in Figure 18. Each part in the assembly is identified with an exploded view depicting how the assembly connects together. The drip assembly was designed to be as simple as possible while still exceeding expectations for what was needed. All parts contained NPT (National Pipe Tapered) threads to have a tighter seal against leaks.

The cap for the vessel enclosed the system to provide a more isolated environment for the heated sodium. The cap would prevent any unwanted objects from falling into the vessel and 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 cap fitting also provided a way for argon gas to flow into the vessel during testing. The cap fitting would help prevent the system from burping during the drip testing. The removal of burping allowed the sodium to flow more easily and consistently. The cap did not originally have a threaded hole to attach the fitting to so a 3.175 mm (1/8 in.) NPT threaded hole was tapped in the caps center.

The vessel was made from a threaded pipe nipple reducer made of schedule 80 stainless steel. The pipe reducer was chosen because it had a large 50.8 mm (2 in.) opening on the top and a small 12.7 mm (½-inch) opening on the bottom. The top allowed for easy access to the vessel for inserting sodium and instrumentation such as thermocouples. The small 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 to prevent unwanted sodium drippage and were connected through the valve fitting. The upper valve was a 12.7 mm (½ in.) ball valve and was mainly used as an on/off switch for the liquid sodium. The lower valve was a 6.35 mm (¼ in.) needle valve and was used as a gradual open/close switch to achieve a more precise and accurate drip rate than a simple ball valve. A straight nozzle was chosen to facilitate a solid stream drip pattern. The nozzle had a small orifice diameter of 1.524 mm (0.06 in.) which helped with controlling the drip rate.

The drip assembly was designed to deposit sodium via gravity onto the insulation samples. To drip sodium onto the insulation, a cone shaped sodium vessel and making the entire assembly vertical were utilized. 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 19.

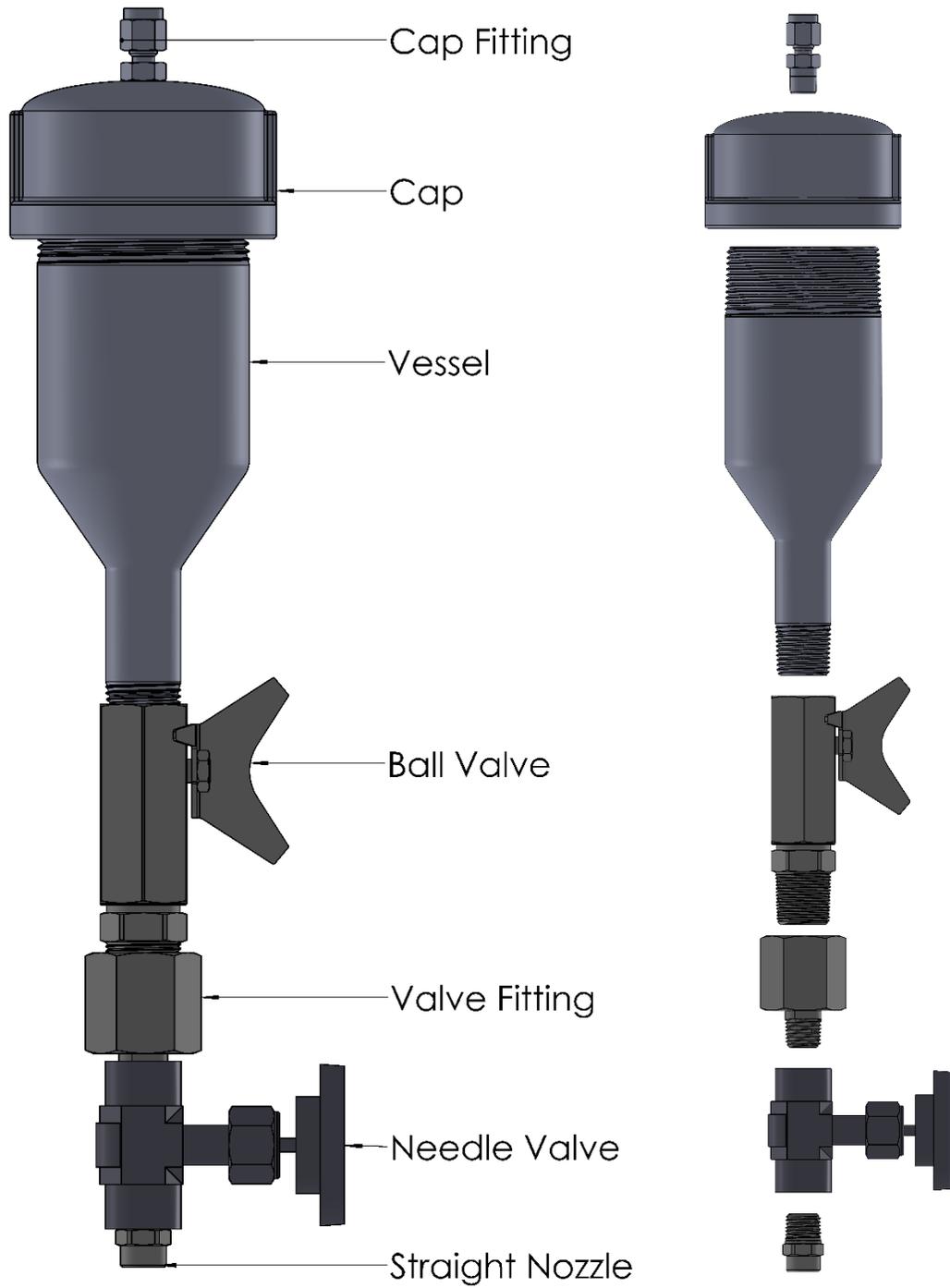


FIGURE 18 COMPLETED DRIP ASSEMBLY RENDERING.

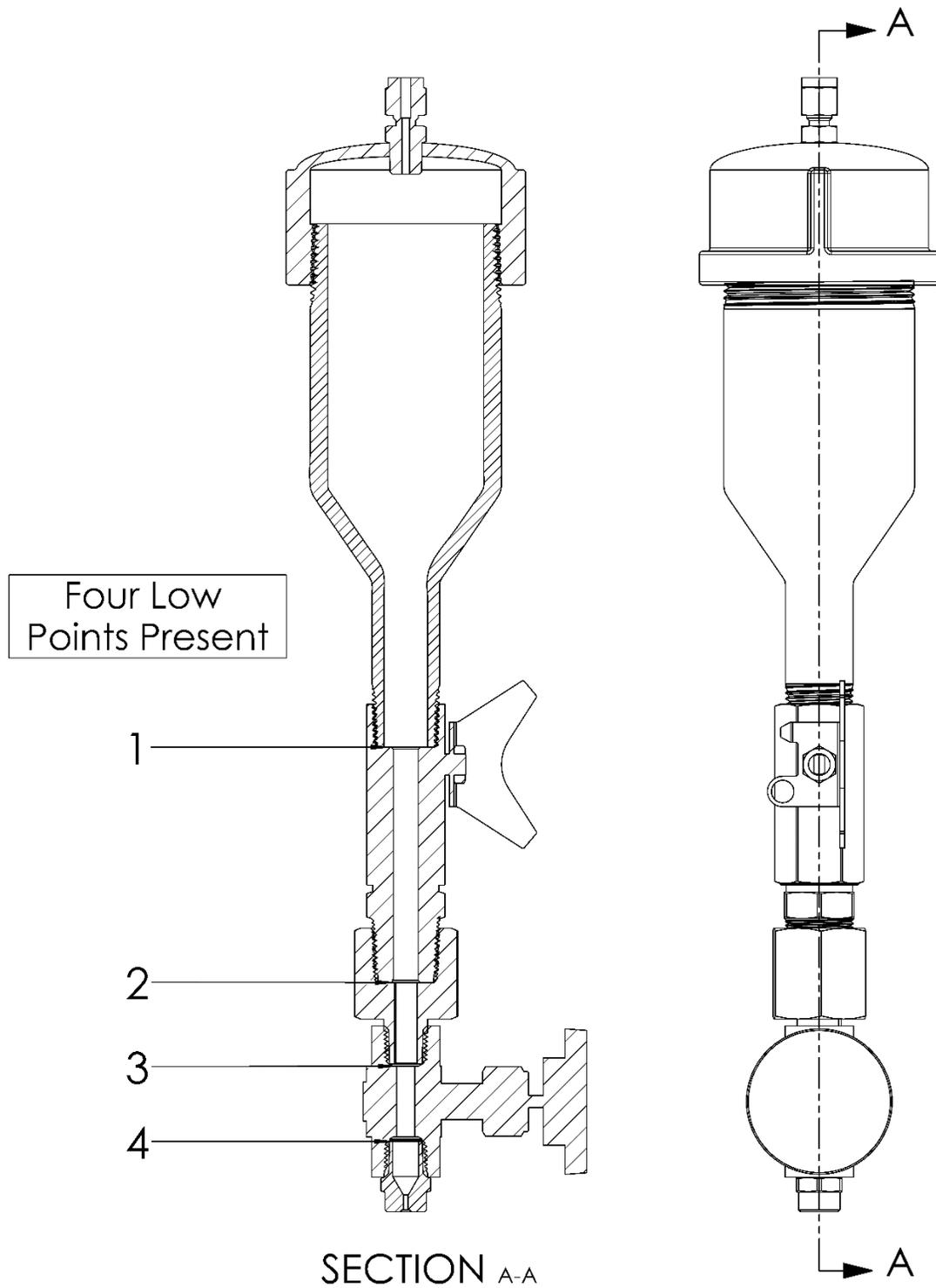


FIGURE 19 CUT-AWAY VIEW OF THE SODIUM DRIP ASSEMBLY. FOUR LOW POINTS ARE DEPICTED IN THE ASSEMBLY.

### **2.1.2 Air and Water Tests**

The drip assembly was assembled outside of the glovebox first to ensure its capabilities and tested for leaks. Each part of the assembly was thoroughly tested for leaks 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 with the air test first and the water test following.

The air test was performed to check that the threads and valves were air tight 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 12.7 mm (½ in.) male, 6.35 mm (¼ in.) female NPT bushing was needed to connect the pressurized air tank hose to the ball valve since the air tank hose connection was 6.35 mm (¼ in.) 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 as shown in Figure 20.



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 20. STEPS TO TESTING PARTS VIA PRESSURIZED AIR AND WATER SUBMERSION.

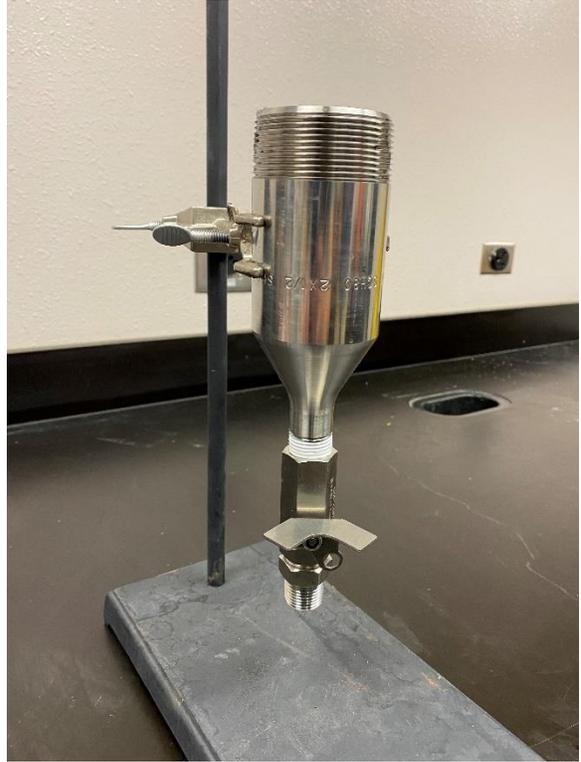
Most parts had air bubbles of some kind appear during testing. The majority of the bubbles appeared to be coming through the threads. This was remedied by either tightening the part or reapplying the pipe tape to get a better seal. Once a good seal was found, the number of layers of pipe tape used to 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 provided in Appendix A.

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. An air test procedure was developed to ensure the process was reproducible, and is provided in Appendix A.

A 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 assembly. This test was not pressurized and relied on gravity for flow, which mimics the experiment that would be performed using molten sodium. This fact, along with the previous pressurized air test, is sufficient to make sure no sodium leaks would occur during the experiment. The top vessel was used to hold the water and provide the gravity positive pressure for the thread tests. Figure 21 illustrates the various steps taken in this water test. A set of procedures was developed as with the air testing for reproducibility, and is provided in Appendix A.



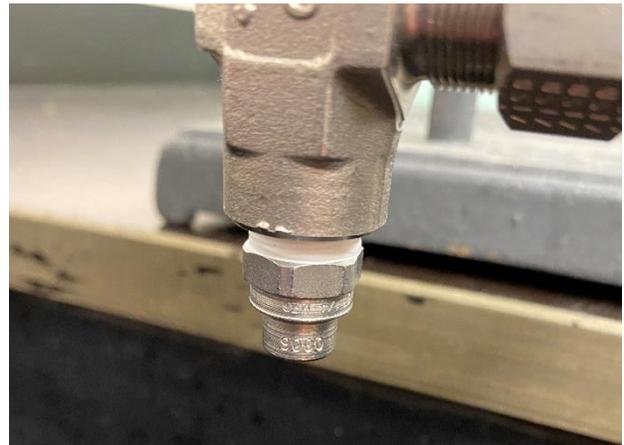
a) Fill vessel with water to maximum level.



b) Orient the filled assembly 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 21. STEPS TO TESTING PARTS FOR WATER LEAKS.

### 2.1.3 Needle Valve Mass Flow Test

The needle valve in the assembly was tested for mass flow rates that could satisfy the conditions for operation. The assembly was put together and placed on a lab stand over a digital scale with a plastic beaker to catch the water as seen in Figure 22. Water was placed in the beaker and the assembly placed close to prevent splashing out of the beaker that would affect the measurement of the mass dripped from the assembly. The digital scale was zeroed with the beaker and excess water on it to only measure what mass came from the assembly. Tests were performed over a 15-minute time frame and in sets of four to see how the mass flow rate changed over the course of an hour. A slow and fast setting were determined to simulate a loss of mass over a long period of time and within a shorter time frame respectively. Markings were placed on the stem of the valve to indicate the slow and fast settings for the tests.

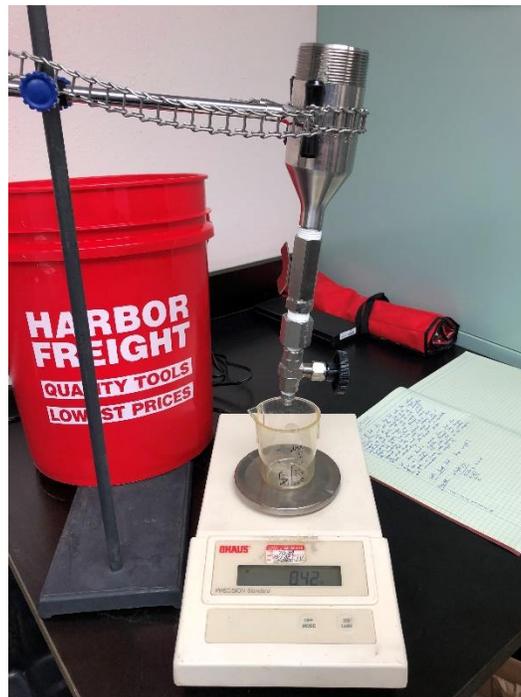


FIGURE 22 MASS FLOW TEST SETUP WITH THE ASSEMBLY PUT TOGETHER TO DRIP INTO A BEAKER ON A SCALE.

The vessel was filled with 25 mL of deionized (DI) water with the ball and needle valves closed. The needle valve was then set to the slow setting and the ball valve was opened afterwards to allow the water to drip through. It was observed that no water was dripping from the assembly and it was determined that an air bubble was in the system causing the water to have difficulty in passing through. A flush at the beginning of the test was implemented to remove the air bubble. This involved keeping the needle valve closed first and opening the ball valve to let the water fill in, then the needle valve was quickly opened beyond the two settings to flush the air bubble out and closed again. The ball valve was closed afterwards to prevent water from flowing through as the needle valve was set to the slow setting. After set, the ball valve was opened and the time began for the test. Once the 15 minutes were up, the ball valve was closed and the mass dripped was measured. The mass that was dripped was then measured out in a graduated cylinder and placed into the vessel to replace what was lost. Then the process was repeated without the flush so that the needle valve did not move from its initial setting after the flush. As can be seen in Table 3, the initial volume of each test was measured or calculated along with the mass lost from dripping and the flush.

TABLE 3 DATA FOR SLOW SETTING WITH A FLUSH AT THE BEGINNING OF TESTING

<b>Initial vol (mL)</b>	<b>Flush (g)</b>	<b>Time (min)</b>	<b>Total mass (g)</b>	<b>Vol added (mL)</b>
25.0	0.75	15	10.45	10.5
25.05	-	15	6.72	7.0
25.33	-	15	4.73	5.0
25.6	-	15	3.06	-

Further tests were performed to see how flushing prior to the test would affect the mass dripped from the assembly. The results can be seen in Table 4 which show that the mass deposited

is more consistent but results in a higher overall mass deposited which is counter to the slow setting. Even though the flush removes the air from the assembly and provides a more consistent mass deposit, it is too fast for what the setting should be. Therefore, a single flush was performed at the start to remove the initial gas pocket so that the liquid sodium could drip out in the experiment.

TABLE 4 DATA FOR SLOW SETTING WITH FLUSH AT THE START OF EACH TIME

<b>Initial vol (mL)</b>	<b>Flush (g)</b>	<b>Time (min)</b>	<b>Total mass (g)</b>	<b>Vol added (mL)</b>
25.0	1.29	15	16.22	16.5
25.28	0.83	15	8.75	9.0
25.53	0.78	15	10.65	11.0
25.88	0.65	15	8.02	-

The tests for the fast setting were the same as the slow setting in which the vessel was filled with 25 mL of water and flushed prior to running the tests as seen in Table 5. The effects of flushing were also explored and can be seen in Table 6. The total mass deposited is nearly the same with the mass deposits being more consistent with a flush prior to each run of the test. For simplicity, the approach to flush once at the beginning was taken.

TABLE 5 DATA FOR FAST SETTING WITH A FLUSH AT THE BEGINNING OF TESTING

<b>Initial vol (mL)</b>	<b>Flush (g)</b>	<b>Time (min)</b>	<b>Total mass (g)</b>	<b>Vol added (mL)</b>
25.0	2.30	15	22.30	22.5
25.2	-	15	12.67	13.0
25.53	-	15	7.50	7.5
25.53	-	15	9.13	-

TABLE 6 DATA FOR FAST SETTING WITH FLUSH AT THE START OF EACH TIME

Initial vol (mL)	Flush (g)	Time (min)	Total mass (g)	Vol added (mL)
25.0	1.65	15	11.27	11.5
25.23	1.03	15	17.71	18.0
25.52	0.94	15	11.08	11.0
25.44	0.82	15	13.24	-

### 2.1.4 Heat Tape Test

The heat tape was tested to ensure its safe operation while providing data needed to predict how the assembly would react to the temperature transients that it would undergo 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 accidentally being grounded, it is entrapped in a silicone rubber matrix. The silicone also acts as a second dielectric protective layer that lets the heating element maintain 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 has a thermostat to help regulate the filament temperature, but only worked if the control box was grounded and physically touching the piping it is heating. The heat tape was equipped with a 6 ft power cord with a standard 2 prong plug (NEMA 1-15). The plug is rated for 1.2 A at 120 V so it can be plugged into most standard electrical outlets.

The heat tape was tightly wrapped in a helical shape around the assembly to keep a good contact between the metal surface and the heating filament. High temperature aluminum tape was

used in several locations to secure the heat tape and ensure a stable position. A clamp secured the temperature control box to the vessel and ensured that 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 assembly and heat tape testing set up can be seen in Figure 23.

The heat tape was turned on to approximately 50% power and the surface temperatures were continuously monitored. While the assembly 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 assembly stayed at this temperature for a few minutes and then slowly cooled off to room temperatures over the course of about 30 minutes.

During the heating process, there was an instance where a small wisp of smoke was noted, but it quickly dissipated and was not seen again. After inspecting the assembly and heat tape, it was concluded that the smoke likely came from the heating of residual oil from our skin that the heat tape contacted, or entrapped dust on the surface of the assembly vessel. To prevent any excess oils or residues being present in the final assembly, all surfaces were handled using nitrile gloves as much as possible, as well as wiped down with Kimwipes.



FIGURE 23. HEAT TAPE BEING TESTED ON THE ASSEMBLY.

### **2.1.5 Assembly Bake-Out in Furnace**

Once the heat tape test was completed, the assembly was placed in a furnace to bake off any residual water vapor and oils. The furnace used was a Carbolite HTF 1800 which allowed for controlled temperatures up to 1800°C which was more than adequate for baking the assembly. The assembly was taken apart in order to fit into the furnace and wiped down with Kimwipes to remove any oils and dirt from the outer surface of the assembly. The assembly was placed inside the furnace to be baked out as shown in Figure 24.



a) Carbolite HTF 1800 used for baking



b) Inside chamber of the furnace



c) Wipe down of the assembly prior to entry into the furnace



d) Assembly pieces inside furnace chamber

FIGURE 24 DISPLAY OF THE FURNACE AND ASSEMBLY PLACED INSIDE.

The furnace was turned on once the assembly was placed inside and the front lid closed. The display showed the current temperature inside the furnace as well as the 'setpoint' below the desired temperature. The set point was changed using the arrow keys to 60°C and then stepped up to 80°C to sit for an hour. After the hour-long bake was done, the set point was dropped to 0°C to ensure the coils were off and the furnace turned off. The larger pieces were removed by hand using thermal gloves, while the smaller pieces were removed using tongs. All pieces were placed into a stainless-steel pan, which was then placed in a fume hood to help cool them off as seen in Figure 25.



a) Front display showing the temperature and set point just after being turned on.



b) The temperature and set point during the 1-hour bake out.



c) Small pieces removed via tongs into stainless steel container.



d) Larger pieces removed via an oven mitt into the container.

FIGURE 25 FURNACE DISPLAY AND REMOVAL OF ASSEMBLY PIECES.

### 2.1.6 Final Fabrication

Once the assembly had been fully tested and proven to be sufficient, the final fabrication work began. Materials had been purchased prior to the fabrication, and all that needed to be done was put it together. Fabrication included assembling the assembly, wrapping and securing the heat tape, and installing the insulation. All of these steps were performed in a research lab in the basement of the Center of Higher Education (CHE) building on the Idaho Falls campus.

The first step was to reassemble the parts after having baked in the box furnace. Nitrile 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 prior specifications. The assembly 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 3.175 mm (1/8 in.) 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 assembly was constructed it was helically wrapped with the heating tape. The heating tape was secured to the surfaces of the assembly by using high temperature aluminum adhesive tape. The heat tape was wrapped tightly to create the best possible connection with the assembly. Depictions of assembling the assembly prior to installing the insulation can be seen in Figure 26. As discussed previously, the temperature control box needed to be ground to the surface it was heating, so good contact with the surface was required. During the testing of the heating tape, the control box was secured to the vessel using a clamp. However, a clamp did not work in this case because the vessel was going to have its cap on while inside the glovebox. The temperature control box was instead held in place by the insulation itself and aluminum tape.

Piping insulation was installed onto the assembly to prevent excess heat loss and to protect surrounding equipment from the high temperatures of the heating tape. The insulation material was made of fiberglass rated up to 538°C. Two sizes of insulation were chosen for the assembly: for the top vessel and cap, 50.8 mm (2 in.) piping insulation was chosen with a 25.4 mm (1 in.)

thickness; for the bottom half of the assembly, 12.7 mm (½ in.) piping insulation was chosen with a 12.7 mm (½ in.) thickness.

The 12.7 mm (½ in.) insulation was put on the assembly 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. 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.

After wrapping the 12.7 mm (½ in.) insulation around the valves and nozzles, it was found that the insulation was too small and would only wrap around three-quarters of the piping, leaving an open gap. This was 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 27.



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



b) The assembly is assembled using wrenches.



c) Heat tape is helically wrapped around the final assembly and secured using aluminum tape.

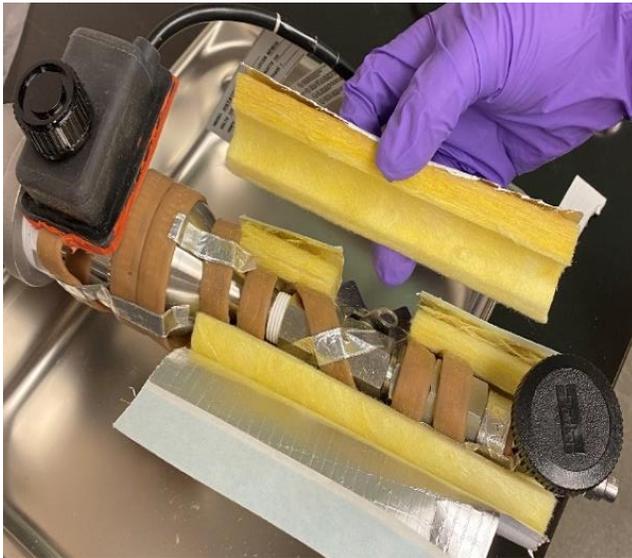
FIGURE 26. ASSEMBLING THE FINAL ASSEMBLY IN PREPARATION FOR INSULATION TO BE INSTALLED.



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 12.7 mm ( $\frac{1}{2}$  in.) insulation is installed and an extra piece of insulation is cut to fill the gap.



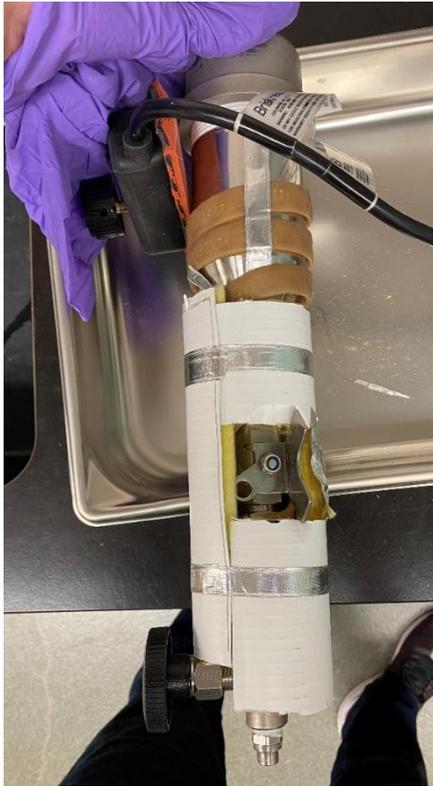
d) Ball valve handle is seen protruding through fully installed 12.7 mm ( $\frac{1}{2}$  in.) insulation.

FIGURE 27. INSTALLING THE  $\frac{1}{2}$ -INCH INSULATION AND CREATING THE VALVE CUTOUTS.

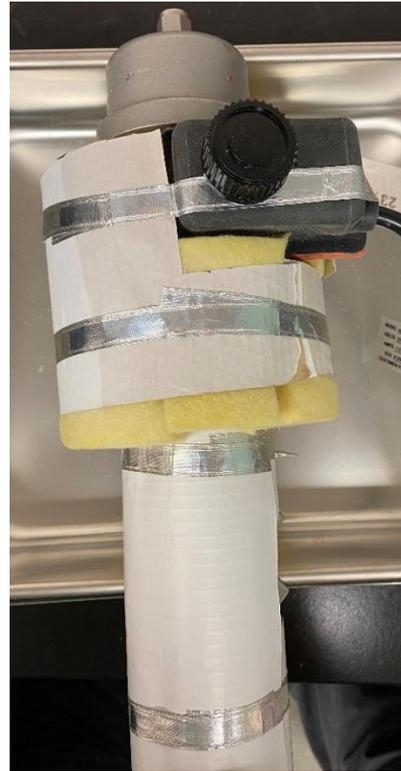
The 50.8 mm (2 in.) piping insulation was attached next. As with the 12.7 mm ( $\frac{1}{2}$  in.) insulation, the 50.8 mm (2 in.) insulation was not able to fit around the entire vessel due to excess

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 12.7 mm (½ in.) insulation was also small enough to allow the 50.8 mm (2 in.) insulation to fit snugly over top and eliminate any possible gaps. Two criteria had to be met when putting this insulation on. First, as mentioned before the heat tape temperature control box has to be physically touching and grounded to the assembly. Second, the control box handle has 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 as seen in Figure 28.

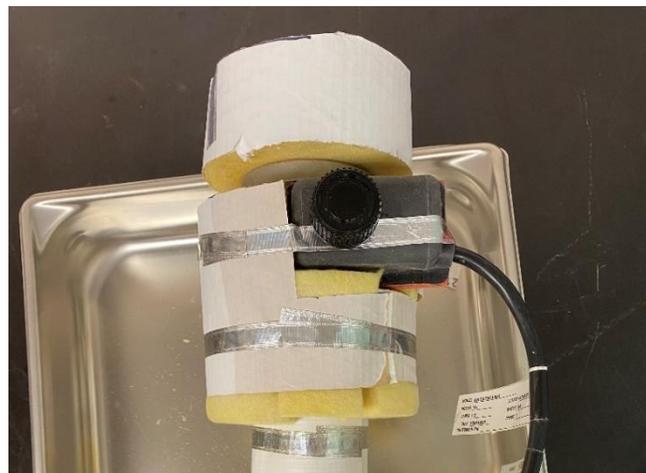
The vessel cap insulation was formed from both the 50.8 mm (2 in.) and 12.7 mm (½ in.) insulation. The larger insulation was cut to length where it would just cover the cap body and leave the cap flange exposed. The top of the cap was covered by a cut piece of 12.7 mm (½ in.) insulation which not only fit inside the outer 50.8 mm (2 in.) insulation but also had enough room for the cap fitting to poke through.



a) Temperature controller is hanging loosely with only support from aluminum tape.



b) Insulation is supporting temperature controller and keeping good contact with the vessel surface.



c) Cap insulation after installation. The cap flange is exposed as it is larger than the cap body.

FIGURE 28. INSTALLING THE 2-INCH PIPE INSULATION USED FOR THE VESSEL AND CAP.

## 2.2 Glovebox Setup

With the fabrication of the drip assembly complete, the components of the experimental apparatus were transferred into the glovebox environment. An inert environment filled glovebox at the Center for Advanced Energy Studies (CAES) was identified for the main experimental tasks. The reactive metal fire suppressant Met-L-X was transferred into the glovebox first, followed by the sodium metal. Lastly, the drip assembly was transferred into the glovebox.

### 2.2.1 Met-L-X

Before the sodium could be placed into the glove box in the Radiochemistry Lab (RCL) at the CAES facility, 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 argon gas with maintained oxygen and moisture levels below 10 ppm. This inert atmosphere reduces the risk of a sodium fire, but there still needed to be some form of fire suppression as a safety precaution.

The fire suppressant was achieved by filling small plastic bags with Met-L-X powder, a safety data sheet is provided in Appendix B. Met-L-X is a Class D fire extinguishing agent used for putting out metal fires, such as sodium. Each small bag contained 20 grams of powder and was made by first weighing the powder out 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 29.



a) Transferring the Met-L-X powder into the bags.



b) Weighing the Met-L-X powder.



c) Completed 20-gram bags placed inside of two larger bags.

FIGURE 29. PREPARING THE MET-L-X BAGS BEFORE BEING PLACED INSIDE THE GLOVEBOX.

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. 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 six and seven 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 process can be seen in Figure 30. 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 will 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.



a) Met-L-X bags being transferred into the glovebox via the antechamber tray.



b) Opening the Met-L-X bags for outgassing with the help of tweezers



c) Arraying the open bags for long term outgassing in the glovebox.

FIGURE 30. TRANSFERRING OF MET-L-X BAGS INTO THE GLOVEBOX VIA THE ANTECHAMBER.

### 2.2.2 Sodium Metal Transfer

Once the Met-L-X was present in the glovebox, the sodium metal was transferred in. The sodium metal was purchased through Sigma-Aldrich and came in a sealed bag and container. The sealed bag was removed to reveal a cardboard box container. The cardboard box container was opened and a metal cylindrical container was kept inside. The metal container was opened to show

packing material inside as shown in Figure 31. The sealed bag, cardboard box, metal container, and packing material were removed from the glovebox to decrease the introduction of new oxygen and water content into the argon atmosphere of the glovebox. The final container was opened and the sodium metal was inspected for purity. The pieces of sodium metal within the container were covered in a white powder, indicating that a sodium oxide layer had formed on the sodium metal as seen in Figure 32.



a) Removal of sealed bag from sodium metal packaging



b) Metal canister contained within cardboard box



c) Opening of metal canister to reveal packing material

**FIGURE 31 UNPACKING OF THE SODIUM METAL BY REMOVING THE SEALED BAG, CARDBOARD BOX, AND OPENING OF THE METAL CONTAINER.**



a) Final container held in a plastic bag



b) Removal of seal from final container



c) Cap removed from final container to view inside



d) Sodium chunk removed for observation revealing oxide layer

FIGURE 32 SODIUM METAL CONTAINER SEAL REMOVED AND OPENED TO SHOW THE SODIUM METAL HAD OXIDIZED.

### 2.2.3 Assembly Transfer and Setup

The drip assembly was placed in the RCL fume hood to provide a forced flow of air to wick away any excess moisture. After sitting in the fume hood for a day, the assembly was placed in the glovebox antechamber to out-gas under vacuum of 85 millipascals (mPa) for approximately 72 hours. The antechamber was purged with dry argon gas several times prior to the drip assembly entering the glovebox environment to reduce the amount of remnant oxygen and water vapor that could be introduced to the glovebox environment. The drip assembly was then attached to a lab

stand within the glovebox via a chain clamp to hold it vertically up as seen in Figure 33. Once attached and secured to the lab stand, quartz wool was used to fill the gaps between the bulk fiberglass insulation to decrease the thermal losses, specifically at locations near the ball valve handle, cap lip, and top of the cap, as shown in Figure 34.



a) Fabricated assembly in fume hood.



b) Fabricated assembly transferred to glovebox



b) Fabricated assembly set up on lab stand

FIGURE 33 DRIP ASSEMBLY PLACED IN A FUME HOOD TO THEN BE TRANSFERRED INTO THE GLOVEBOX AND SETUP ON A LAB STAND USING A CHAIN CLAMP.



a) Insulation material inserted into gaps around the cap



b) Insulation material inserted around ball valve handle

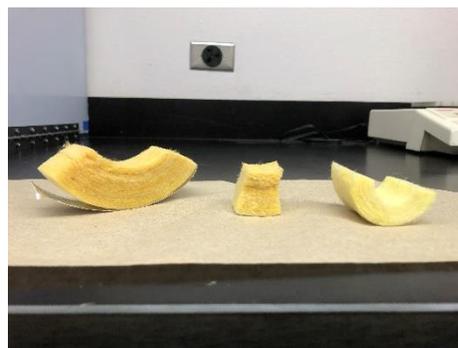
FIGURE 34 MINERAL WOOL INSERTED INTO EXPOSED AREAS TO DECREASE THERMAL LOSSES.

## 2.3 Drip Assembly Experiments

With the drip assembly, sodium metal, and Met-L-X set up in the glovebox, experiments to drip the sodium metal onto the fiberglass insulation were performed. While still empty, the drip assembly was first heated to around 100°C using the heat tape to remove any moisture within the assembly components and insulation that was not removed in the fume hood or antechamber outgassing. Once the assembly heating was complete, an insulation sample was prepared from the same insulation material used around the drip assembly. The sample was cut into a 1-inch cube and placed in the antechamber to outgas before the sample was brought in as shown in Figure 35.



a) Top view of insulation sample compared to 2 - inch and ½ - inch insulation



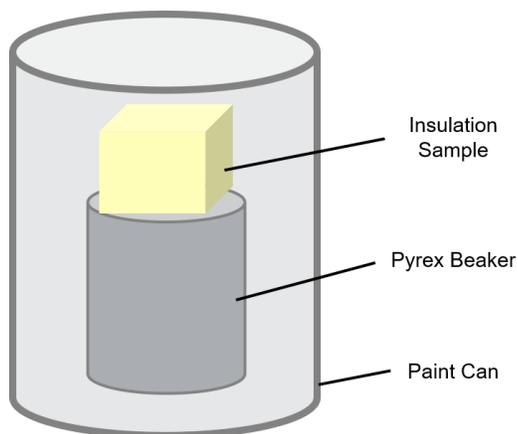
b) Side view of insulation sample compared to 2 - inch and ½ - inch insulation

FIGURE 35 FIBERGLASS INSULATION 1-INCH CUBE SAMPLE FOR THE DRIP ASSEMBLY TEST.

The insulation sample was placed inside of a clean metal paint can on top of a Pyrex glass beaker to elevate it. The paint can was used to contain any sodium that would leak from the sample and prevent contamination of the glovebox floor. The paint can, with the sample inside, was placed upon a scissor lab stand that was used to manipulate the position of the paint can, and therefore, the insulation, during testing as shown in Figure 36.



a) Assembly set with paint can on lab stand



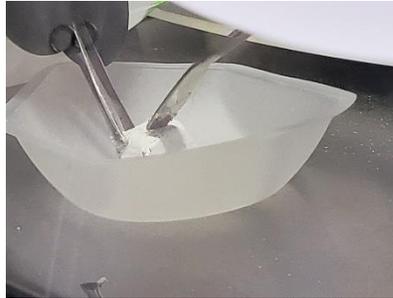
b) Depiction of the inside of the paint can

FIGURE 36 DRIP ASSEMBLY WITH LAB STAND HOLDING THE PAINT CAN BENEATH CONTAINING INSULATION SAMPLE INSIDE.

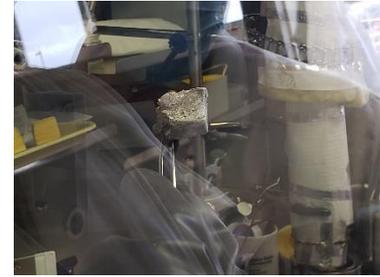
The sodium to be placed in the vessel of the drip assembly was extracted from the larger pieces held in the Sigma-Aldrich container. The use of a plastic weighing dish was utilized to hold the oxidized piece, which was then divided into multiple pieces using stainless steel tweezers and scraper. The oxide layer that had formed on the large piece was manually removed as seen in Figure 37. The sodium piece was massed (5.751 g) and placed inside the vessel. A handheld retractable mirror was used to verify the status of the sodium piece inside of the vessel, as shown in Figure 38; thereafter, the vessel cap and thermocouple were secured on the assembly.



a) Sodium chunk split in half to reduce total mass



b) Sodium metal scraped to remove oxide layer



a) Sodium metal oxide removed from piece

FIGURE 37 OXIDIZED SODIUM CHUNK SPLIT AND OXIDE REMOVED FROM SODIUM PIECE TO BE PLACED IN THE DRIP ASSEMBLY.



FIGURE 38 SODIUM PIECE PLACED INTO THE DRIP ASSEMBLY VESSEL AND VISUALLY CONFIRMED USING A MIRROR.

The heat tape was turned on and then incrementally increasing the temperature of the drip assembly by rotating the knob of the heat tape controller to a marking made for 150°C. The thermocouples (TCs) that were placed in the vessel and at the ball valve handle were plugged into a TC reader to output their temperature readings. The insulation sample was lifted up to the nozzle of the drip assembly so that droplets could be observed. Droplets were not observed at 150°C resulting in the temperature on the heat tape to be raised to 180°C with droplets still not observed. A spot check of the nozzle indicated that the nozzle was not reaching the melting point of sodium. The drip assembly was taken down and the insulation removed to allow for the heating tape to be reapplied to focus more heat towards the nozzle.

After adjustments were made to the drip assembly, the temperature of the heat tape was raised to 200°C and the insulation sample was slightly compressed against the nozzle to help increase the temperature at the nozzle. After one hour at a stable temperature of 200°C, the insulation sample was lowered slightly to observe what had happened. The insulation indicated no sodium metal had interacted with the sample. Shortly afterwards, sodium droplets were observed sitting on the surface of the insulation as seen in Figure 39. A total of five droplets were observed coming out of the drip assembly. The droplets rolled off the surface of the insulation shortly after solidifying into the paint can.



FIGURE 39 SODIUM METAL DROPLETS OBSERVED SITTING ON INSULATION SAMPLE.

Once the drip assembly had cooled, the lab stand was lowered to observe the results of the dripped sodium into the paint can. Within the paint can, it was observed that there were more than just the sodium droplets present. As seen in Figure 40, a larger mass of sodium had pooled in the

bottom of the paint can and indicated that the sodium metal was not interacting as expected with the fiberglass insulation.



FIGURE 40 INSIDE VIEW OF THE PAINT CAN AFTER SODIUM DROPLETS FORMED.

## 2.4 Jeweler's Furnace Experiments

With the difficulty of the assembly to both produce molten sodium droplets and maintain contact of the sodium on the fiberglass insulation, experiments were paused while the research team considered options to develop an experiment that would produce more consistent interactions between the insulation and molten sodium. It was decided that a jeweler's furnace would be used to maintain a higher constant temperature for both the insulation and the sodium, allowing the insulation and sodium metal to be kept in contact while the temperature was increased. The process and developments of the experiments with the jeweler's furnace were iterative and learned along the way.

The jeweler's furnace used was a KerrLab Auto Electro Melt Maxi, comprised of a graphite mantle that is heated via electrodes and insulated radially with a lid that can be closed. The operating temperature of the jeweler's furnace ranged from room temperature up to 1120°C. Thus, the jeweler's furnace was able to provide the necessary temperatures that the drip assembly could not achieve.

The apparatus for the jeweler's furnace experiments comprised of using stainless steel foil, an alumina crucible, insulation sample, and sodium metal. An alumina beaker was procured that was able to be inserted into the graphite mantle of the jeweler's furnace. The alumina beaker would be lined with stainless steel foil to prevent the alumina beaker and the graphite mantle from being contaminated by direct contact with sodium metal. The insulation and sodium metal were then placed inside the stainless steel foil so that contact was maintained while the temperature was increased.

The stainless steel foil was measured out into six-inch squares and cut from the roll that was procured. Once the stainless steel foil was cut, the stainless steel foil was shaped and molded to fit inside the alumina beaker. Insulation samples were cut from fiberglass insulation and shaped to fit inside the stainless steel foil. Figure 41 shows the process of fabricating the insulation samples, stainless steel foil, and alumina crucible for the jeweler's furnace. With the fabrication of the stainless steel foils to hold the insulation sample and sodium metal complete, the stainless steel foils with its insulation sample were transferred into the glovebox with an outgassing period prior to entry into the glovebox environment.



FIGURE 41 INSULATION SAMPLE, STAINLESS STEEL FOIL, AND ALUMINA CRUCIBLE FABRICATION FOR THE JEWELER'S FURNACE.

Experiments were performed with insulation that had not been pre-heated, just as had been done with the drip assembly. A piece of sodium metal was placed on top of the insulation sample that was inside the stainless steel foil as shown in Figure 42. The jeweler's furnace was set to 150°C just as the drip assembly and after stabilizing was left to sit for an hour.



FIGURE 42 SODIUM METAL PIECE SET ATOP INSULATION SAMPLE AND PLACED INSIDE GRAPHITE MANTLE OF THE JEWELER'S FURNACE.

After an hour at 150°C, the retractable mirror was used to view the sodium inside the furnace using a hole lid of the jeweler's furnace; during operation, a chunk of insulation was placed over the hole to restrict heat loss. No visible signs of the sodium metal or insulation could be seen via the mirror. The furnace was deactivated and allowed to cool off; once cooled, the alumina

crucible with the sodium metal and insulation was removed. Using a pair of stainless steel tweezers, the sodium metal piece was investigated along with the surface of the insulation sample as shown in Figure 43.



FIGURE 43 RETRACTABLE MIRROR USED TO OBSERVE SODIUM METAL INSIDE JEWELER'S FURNACE. STAINLESS STEEL TWEEZERS USED TO INVESTIGATE SODIUM METAL AFTER COOLDOWN.

The sodium metal showed no signs of interaction with the insulation sample. The sodium metal did however to appear to have a white coating. This sodium chunk was split to confirm the outer layer was only affected and displayed a white coating. The experiment was performed again at 200°C for a longer time frame - five hours. The same piece of sodium that was split from the first experimental run was used again to verify whether the sodium metal was actually molten. Throughout the five-hour time frame, the sodium metal was checked via the retractable mirror and was observed that the two pieces melded together confirming that the sodium metal pieces had indeed melted inside the jeweler's furnace. The same observations were made as in the first experimental run once the jeweler's furnace had cooled down. A white coating had formed over the surface of the sodium metal during the time it was inside the jeweler's furnace. This indicated that some source of oxygen was oxidizing the sodium metal during the experiment.

A third experimental run was setup with a new sodium metal piece, insulation sample, and stainless steel sleeve. The sodium metal was extracted from the sodium metal deposited from the drip assembly in an attempt to remove the possibility of oxide layers to the sodium metal provided by Sigma-Aldrich. The furnace was set to 200°C and operated again for a five-hour period; no changes were observed during the experiment or once cooled. A new configuration was implemented in an attempt for some results in the sodium metal interacting with the insulation sample.

The new configuration utilized was to split the insulation sample in half and place the sodium metal sample in between the two pieces of insulation. A second alumina crucible was placed on top to provide pressure to the entire configuration. This second alumina crucible was covered in stainless steel foil to prevent contact with sodium metal directly as shown below in Figure 44. The alumina crucible was placed through the hole in the lid and insulation was utilized to fill the gaps and cover the alumina crucible. The jeweler's furnace was set to 200°C and operated for a five-hour period once again. The alumina crucibles were removed after cooldown of the jeweler's furnace. Removal of the crucible that was providing pressure to the insulation sodium sandwich revealed a nodule of sodium metal appearing on the stainless steel sleeve above the insulation. Further investigation revealed that the sodium metal had spread out along the surface of the halved sections and rose up the side. The sodium metal easily fell off the insulation as shown in Figure 45.



FIGURE 44 NEW CONFIGURATION EXPERIMENT WHERE THE INSULATION SAMPLE IS HALVED TO PLACE SODIUM METAL INSIDE WITH ALUMINA BEAKER PLACED ON TOP TO ADD PRESSURE.



FIGURE 45 NEW CONFIGURATION RESULTS AFTER COOLDOWN SHOWING THE SODIUM METAL NOT INTERACTING WITH THE INSULATION AND SPREADING OUT INSTEAD.

With the sodium metal and insulation not interacting as anticipated, an adjustment was made to the experiment. Insulation material can degrade over time due to the temperatures that it experiences. The experiments previously performed used insulation material straight from the manufacturer/distributor and was therefore not aged or pre-heated in any manner. The team decided to thermally age the insulation and repeat the experiments already performed to compare the results of the sodium-insulation interaction.

To achieve an aged state for the insulation, samples were placed into an industrial oven to bake. The oven was set to a temperature of 300°C to ensure that the organic binder of phenolic resin would degrade in the insulation samples. Samples were baked for six hours in the industrial oven. The samples placed in the oven had a yellow color and displayed rigidity of their shape. The samples after being baked had the yellow color fade to grayish tones with the rigidity of the shape being easier to manipulate. Figure 46 shows the contrast between the insulation samples before and after aging in the industrial oven.



FIGURE 46 INSULATION SAMPLES BEING PLACED INSIDE THE INDUSTRIAL OVEN AND LATER REMOVED ONCE DONE BAKING.

The same set of experiments were performed with an aged sample of insulation though the time left at temperature was significantly increased. The sodium metal was first set atop the baked insulation samples and heated to 200°C for eight hours. The results of this experiment were the same as with the insulation sample not being baked. The baked insulation was then split in half with the sodium metal placed in the middle with the secondary alumina beaker atop. The insulation sandwich was heated to 200°C to stay at temperature for eight hours. These results were also the same as tests performed with the insulation sample not baked.

A variation to the initial experiments was tested, whereby a hole was cut into the insulation sample to allow the sodium metal to enter and allow access to the other layers of insulation. The prevailing thought was that the sodium metal was not interacting with the insulation sample due to the sodium metal not being able to penetrate the top layer of insulation. With the introduction of the hole, the sodium metal could bypass the initial layer of insulation and propagate through the insulation as shown in Figure 47. The setup was operated at 200°C for a duration of seven hours. Observations afterwards showed the same result as previously observed, with the sodium metal not propagating through the insulation.

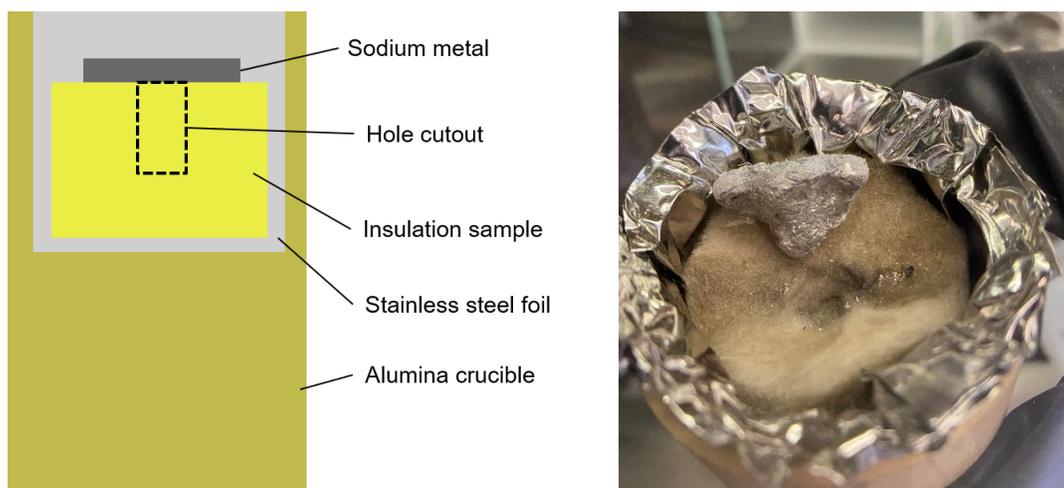


FIGURE 47 INSULATION HOLE EXPERIMENT.

With the results from experiments currently ran a step back was taken to analyze how to proceed. The conclusion was to utilize the insulation hole method and add pressure to the sodium metal directly as shown in Figure 48. If no propagation occurred, the temperature would be increased 50°C starting at 250°C up to a maximum temperature of 400°C. The secondary alumina crucible wrapped in stainless steel foil was utilized to provide the pressure to the sodium metal. Tests were performed in intervals from 250-400°C with the time duration around six hours per test

run. No signs of the sodium metal propagating into the insulation sample were observed though the insulation blackened around the sodium metal.

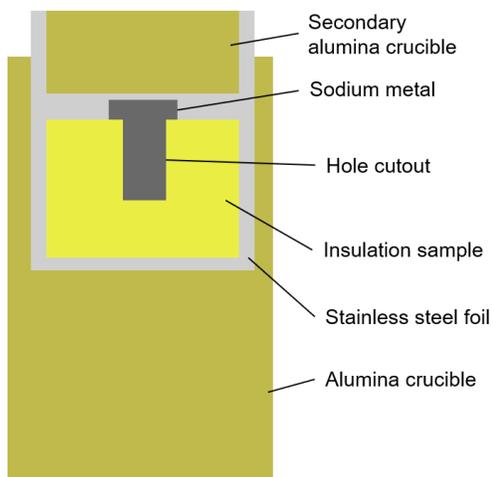


FIGURE 48 INSULATION HOLE CONFIGURATION WITH ALUMINA BEAKER APPLYING PRESSURE.

With the results being the same for all experiments currently performed, another set of experiments with the sandwich method were conducted. The alumina beaker wrapped in stainless steel was utilized to apply pressure while the sodium metal was placed in the middle of the baked insulation sample as shown in Figure 49. The experiments ran in intervals from 250-400°C, the same duration of around six hours for each experimental run. Propagation of the sodium metal into the baked insulation was not observed, though it was again observed that the insulation had blackened in areas that were in direct contact with and around the sodium metal.

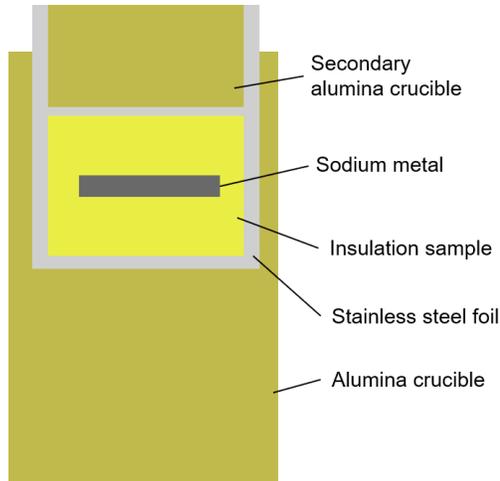


FIGURE 49 SANDWICH METHOD WITH BAKED INSULATION.

Two addition experiments were performed with the sodium metal at the bottom of the stainless steel foil while having the baked insulation atop of the sodium metal as seen in Figure 50. The alumina beaker applied pressure to the baked insulation. The experiments were performed at 450 and 500°C for six hours each. It was observed that the insulation darkened and became significantly brittle near the sodium metal.

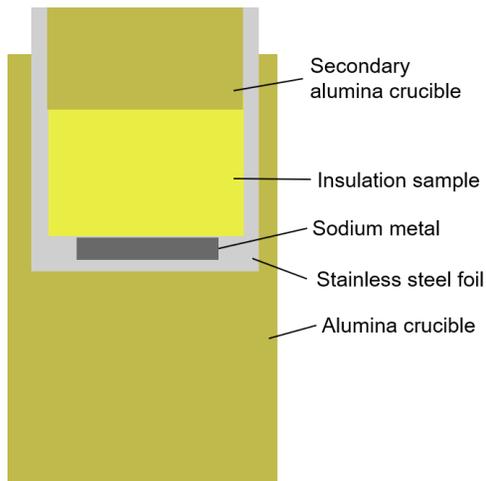


FIGURE 50 VARIATION OF THE SANDWICH METHOD WHERE SODIUM METAL IS BELOW THE INSULATION.

## 2.5 Sample Extraction

Samples were needed for chemical analysis from the experimental runs performed. Samples from experiments that displayed darkening of the insulation around the sodium metal were taken. The experiments that experienced darkening around the sodium metal were: 1) the hole in the baked insulation ran at 400°C which will be referred to as ‘piston’; 2) the baked insulation split in half with the alumina beaker acting as a weight ran at 400°C which will be referred to as ‘sandwich’; 3) the baked insulation atop of the sodium metal with the alumina beaker as a weight ran at both 450 and 500°C which will be referred to as ‘sandwich 2.0’ with the temperature following to identify specific experiments.

The samples were procured inside the glovebox environment and required transfer out of the glovebox for analysis. Due to the uncertainty of the sodium content present in each sample, the samples had to undergo an oxidation process to ensure no pure sodium metal was present outside the glovebox environment. A small portion of fibers were removed from each sample and massed as shown in Figure 51. The fibers from the samples ranged from 0.001-0.012 grams. To transfer the fibers out of the glovebox environment, they were placed inside of a small metal paint can with the cap was placed on. The paint can was wiped with Kimwipes prior to entry into the glovebox to remove any unwanted materials clinging to the surfaces. The paint can containing the fibrous sample was transferred to the antechamber of the glovebox in preparation for removal.

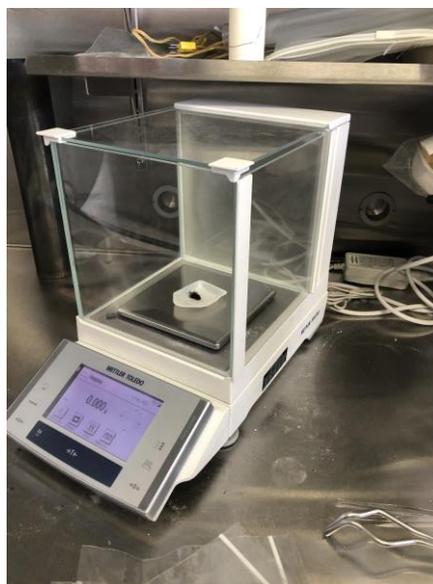


FIGURE 51 LARGER SAMPLES ON THE LEFT WHERE FIBERS FROM EACH SAMPLE ARE MASSED ON THE RIGHT.

The oxidation of the samples occurred at a fume hood located approximately 10 feet from the glovebox antechamber access point. Within the glovebox was an argon bottle with attached regulator and hose, retractable mirror, stainless steel tray and tweezers, Met-L-X baggies, and glass vials as shown in Figure 52. The paint can was opened via the stainless steel tweezers with argon released into the can by the hose attached to the argon bottle. Leaving the can in the fume hood, the flow of argon was turned off and the sample visually checked with the retractable mirror as shown in Figure 53. The temperature of the sample was measured using an infrared sensor to ensure that no localized exothermic reactions were taking place. Once verified, the sample was removed from the can and placed in a glass vial for labelling.



FIGURE 52 FUME HOOD OXIDATION SETUP.



FIGURE 53 OPENING OF THE PAINT CAN FOR THE ARGON FEED TO BE INSERTED. THE SAMPLE IS VISUALLY CHECKED UTILIZING A RETRACTABLE MIRROR.

After the samples completed oxidizing in the fume hood, they were taken out of the RCL for chemical analysis. Three samples were taken each from the piston, sandwich, and sandwich 2.0 experiment runs, a total of 12 samples in total. Control samples of both the normal insulation and aged insulation were procured for comparison to the samples that had interacted with the sodium metal in the furnace experiments.

## Chapter 3: Results

### 3.1 Sample Analysis

The analysis of the samples was performed by a Focused Ion Beam (FIB) equipped with an Energy Dispersive X-ray Spectroscopy (EDS) unit. The samples were attached to carbon tape atop pedestals that fit a mounting bracket for the FIB as shown in Figure 54 before being inserted for analysis. The FIB uses a beam of gallium ions to ionize a sample that produces ions, neutral atoms, and electrons. The ions, neutral atoms, and electrons are used to create an image of the sample. The ions, neutral atoms, and electrons are used to create an image of the sample. An example of a FIB setup and the process of imaging is shown in Figure 55.

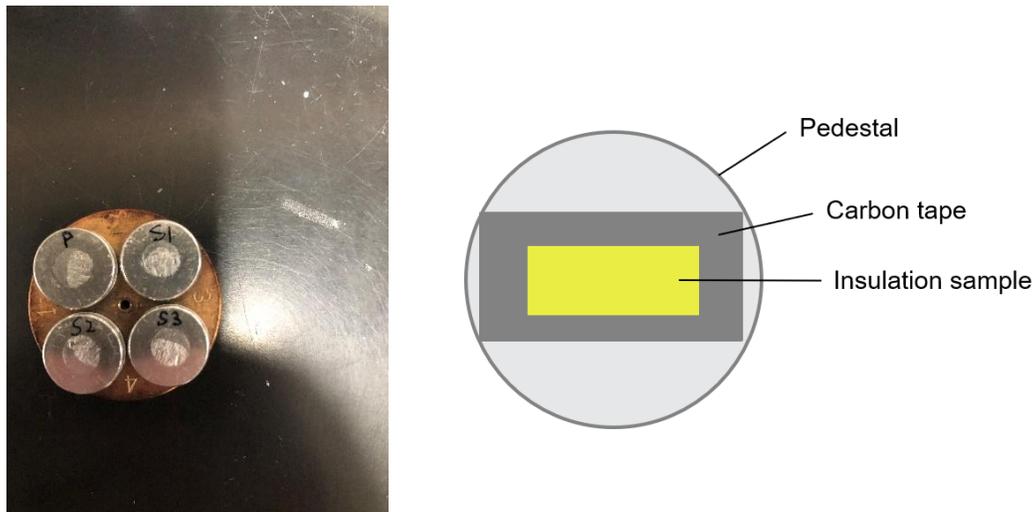


FIGURE 54 FIB PEDESTAL AND PLATFORM USED FOR SAMPLE MOUNTING FOR ANALYSIS.

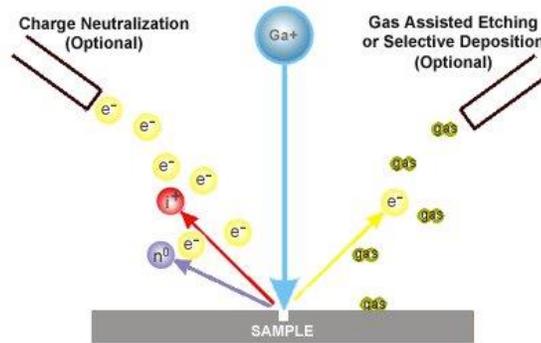
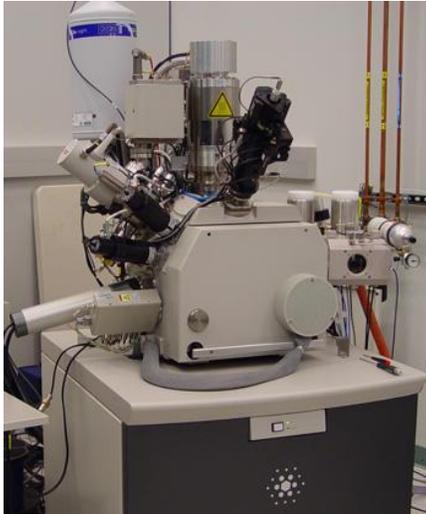


FIGURE 55 FIB EXAMPLE SETUP SHOWN ON THE LEFT WITH THE PROCESS OF IMAGING SHOWN ON THE RIGHT.

The EDS device utilizes an electron beam to eject the ground state electrons from the atoms in the sample. When the ground state electron is ejected, a higher energy electron will fill its place. When the electron fills the gap, an X-ray is emitted allowing for the electromagnetic emission spectrum to create a mapping of the elements present as shown in Figure 56. The EDS was used for elemental analysis of all samples to obtain a baseline elemental map for the controls and any changes from control for the experiment samples. The EDS reports for the control and experiment samples can be found in Appendixes A and B. A total of 20 samples were analyzed with 12 experimental samples and 8 controls samples. Each sample had an image taken at magnifications of 100x and 500x. The elemental analysis was performed twice per sample in separate areas to increase the amount of data.

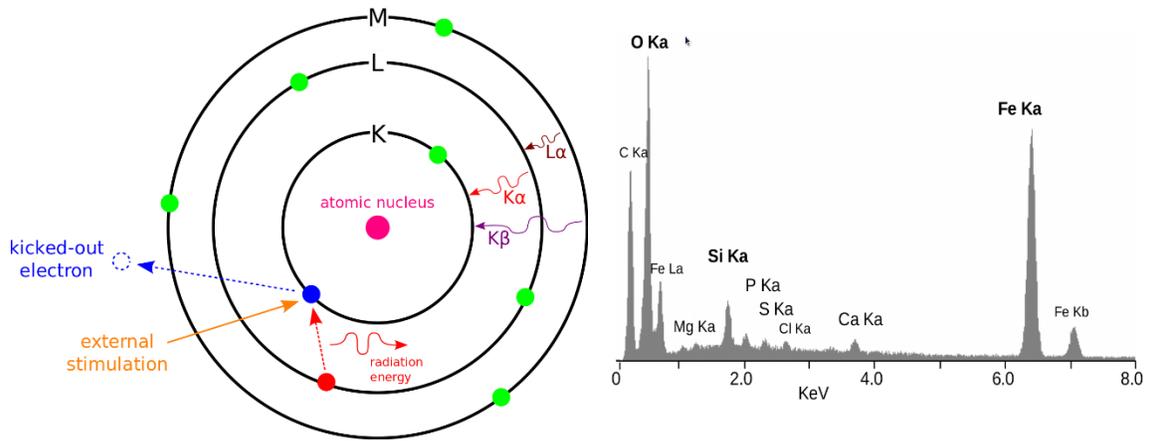
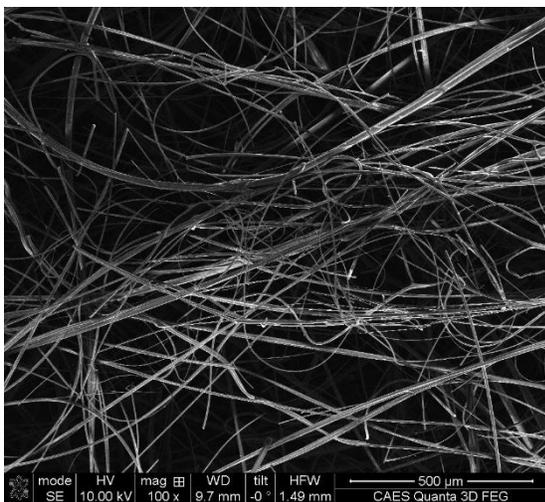


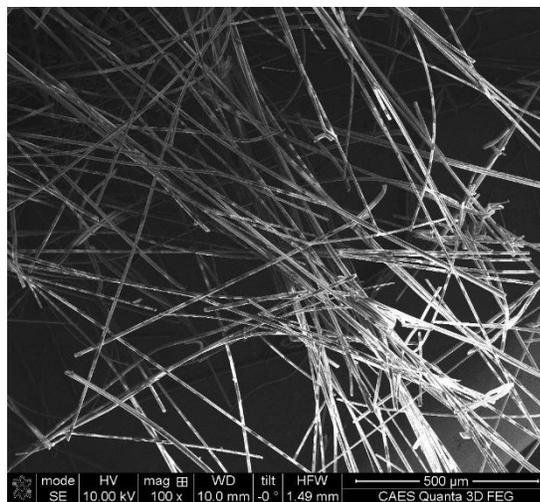
FIGURE 56 THE PROCESS OF EDS WITH SUBSEQUENT ELEMENTAL SPECTRUM.

### 3.2 Electron Microscopy

Samples from the control insulation and from each of the experimented insulation were collected and compared using electron microscopy. The control samples of unbaked and baked insulation showed long strands of fibers as part of the structure as shown in Figure 57. The fibers displayed lengths longer than 500  $\mu\text{m}$ . The control sample fibers ranged in thickness from 1-10  $\mu\text{m}$ . The fibers of the baked control samples displayed patchiness in the outmost layer compared to the controls as shown in Figure 58.



a) 100x image of unbaked insulation sample



b) 100x image of baked insulation sample

FIGURE 57 100X IMAGES OF BOTH CONTROL SAMPLES DISPLAYING LONG FIBERS AS PART OF THE STRUCTURE.

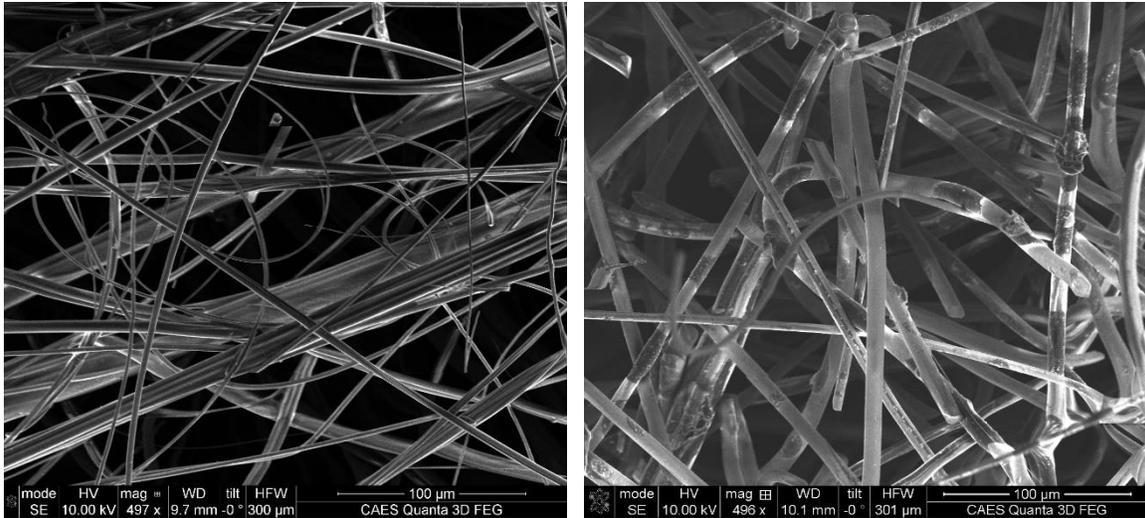


FIGURE 58 CONTROL FIBERS ON THE LEFT SHOW LITTLE TO NO PATCHINESS ON THE OUTER LAYER. THE BAKED CONTROL FIBERS ON THE RIGHT DISPLAY PATCHINESS IN THE OUTERMOST LAYER.

The experiment samples displayed no patchiness compared to the baked controls. The fibers of the experiment samples were most similar to the control fibers in appearance. One experiment sample displayed a deviation from the control fibers themselves. The experiment sample came from the piston experiment where the sample was taken from inside of the insulation hole that was made. Since the sample came from a portion of the fiberglass that was in direct contact with the sodium, it is proposed that the sodium metal itself is the leading cause for the fiber deviation shown in Figure 59.



FIGURE 59 IMAGES OF A CONTROL, BAKED CONTROL, AND SODIUM EXPOSED SAMPLE. SODIUM EXPOSED SAMPLE SHOWS DEVIATIONS COMPARED TO THE OTHERS.

### 3.3 EDS Analysis

Fiberglass insulation is made from molten glass, slag, and rock consisting of silicon dioxide ( $\text{SiO}_2$ ), calcium oxide ( $\text{CaO}$ ), calcium carbonate ( $\text{CaCO}_3$ ), sodium carbonate ( $\text{Na}_2\text{CO}_3$ ), aluminum oxide ( $\text{Al}_2\text{O}_3$ ), and magnesium oxide ( $\text{MgO}$ ). The control and baked control samples displayed levels of oxygen (O), sodium (Na), magnesium (Mg), aluminum (Al), silicon (Si), and calcium (Ca); carbon (C) was not counted in the analysis as the true signal provided by the EDS was altered due to the use of carbon tape to secure the fibrous samples to the pedestal. The elemental constituents were found to be consistent with the compounds found in fiberglass insulation. The percentages of the elements present in the control and baked control samples were approximately the same, with an increase in one element percentage compared to the other indicating an increase in that element and vice versa. The average atom percent along with the standard deviation ( $\sigma$ ) of each element from control and baked control samples are shown in Table 7.

TABLE 7 AVERAGE ATOM PERCENT AND STANDARD DEVIATION FOR CONTROL AND BAKED CONTROL SAMPLES.

<b>Element</b>	<b>Control average at%</b>	<b>Control <math>\sigma</math></b>	<b>Baked control average at %</b>	<b>Baked control <math>\sigma</math></b>
O	56.83	1.33	54.92	0.45
Na	12.30	0.36	10.28	0.14
Mg	1.00	0.04	1.32	0.07
Al	1.07	0.15	1.02	0.19
Si	26.08	1.35	28.17	0.49
Ca	2.73	0.30	4.30	0.25

The elements of oxygen, sodium, magnesium, aluminum, silicon, and calcium were all present in samples collected from the sodium-insulation experiments. The amount of sodium (by

atom percent) increased throughout the experiment samples when compared with the baked controls. The changes in average atom percent along with the standard deviation of the experiment samples from the baked control are shown in

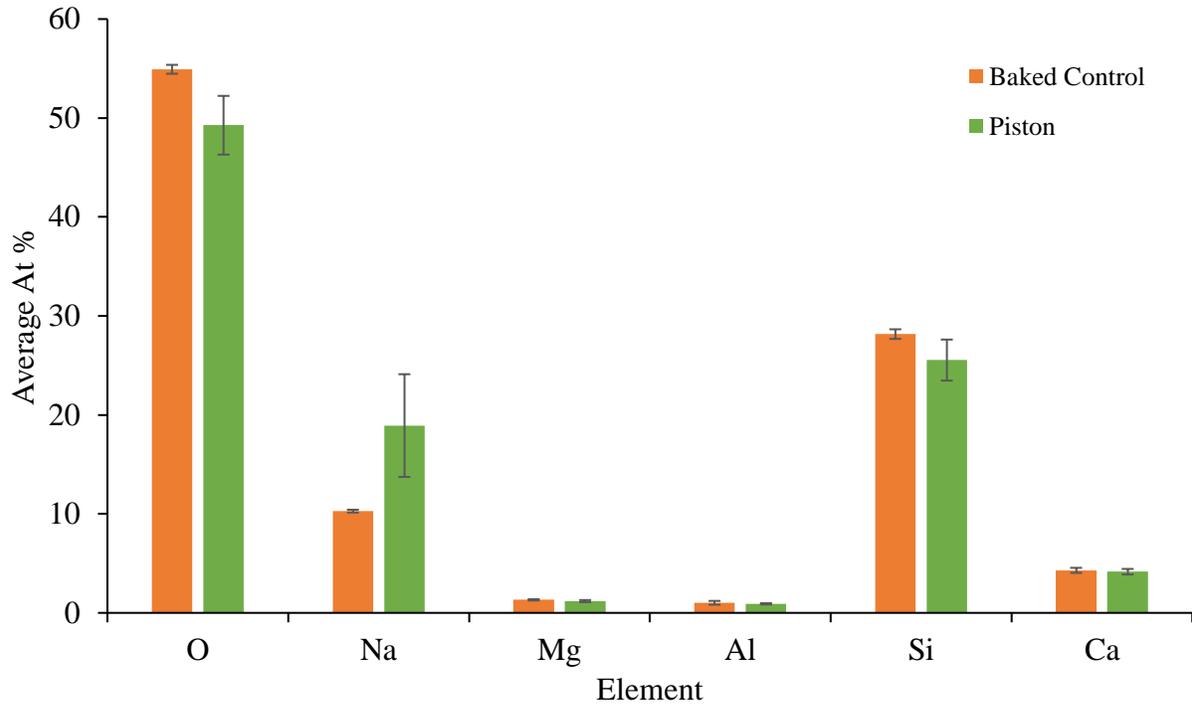


Figure 60-63.

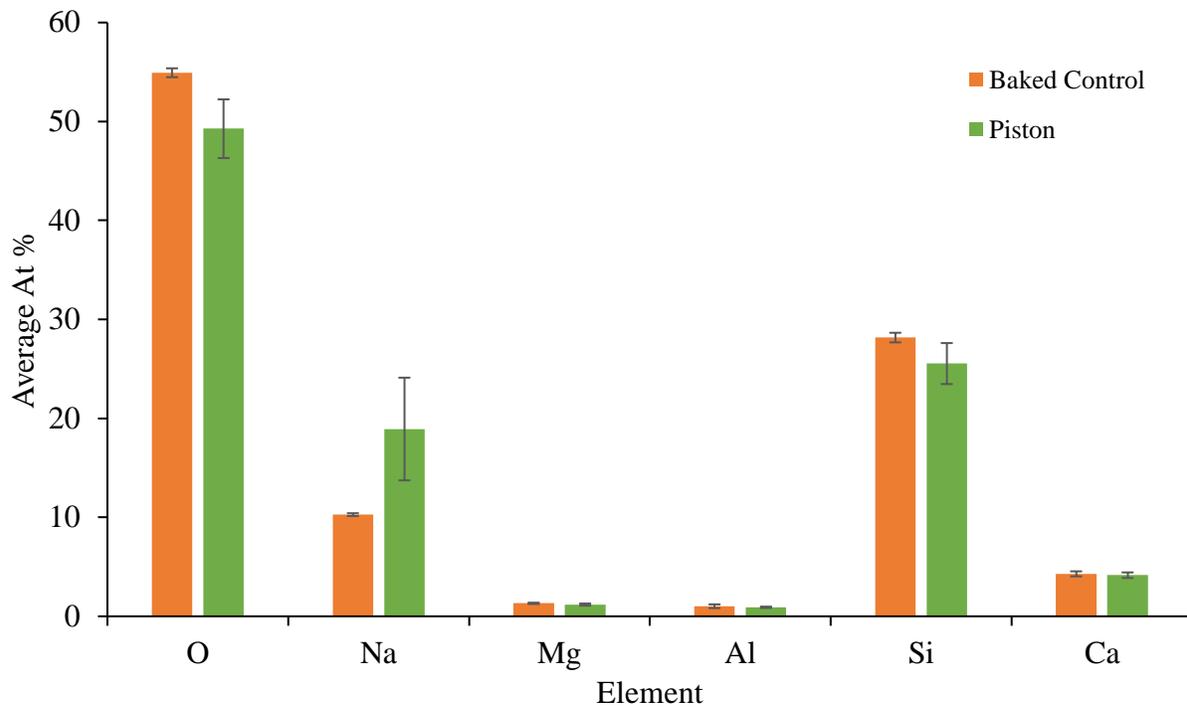


FIGURE 60 AVERAGE WEIGHT PERCENT OF EACH ELEMENT IN THE PISTON EXPERIMENT SAMPLES COMPARED TO THE BAKED CONTROL.

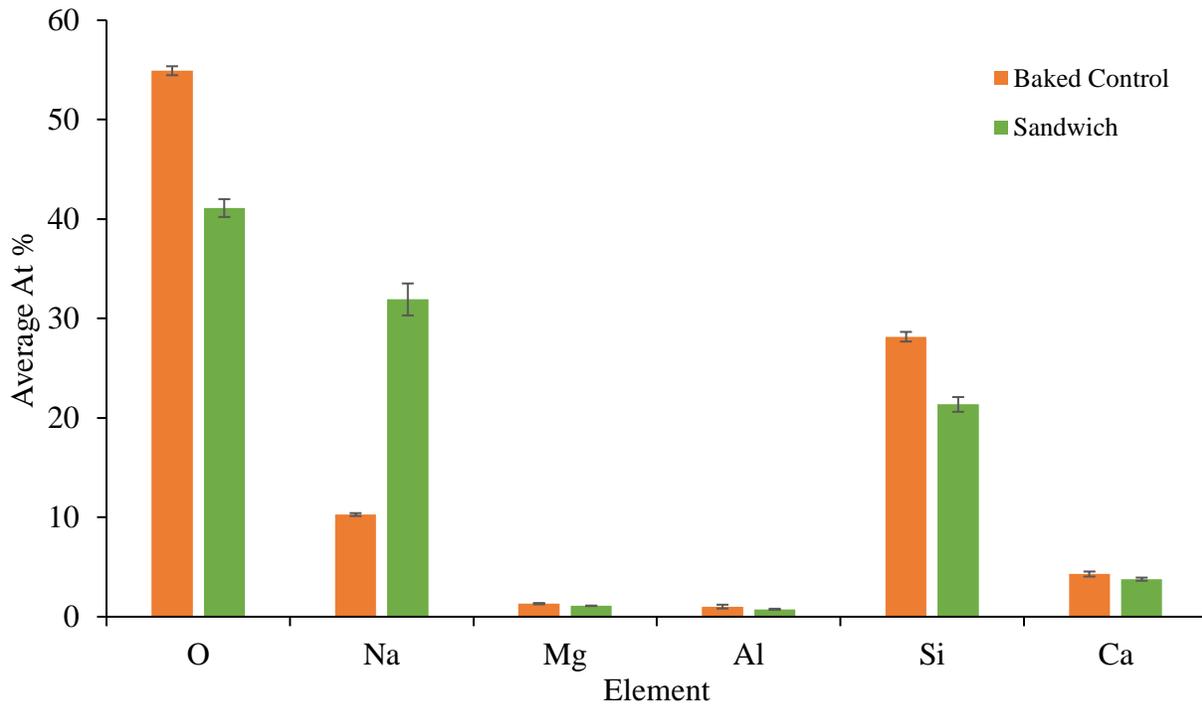


FIGURE 61 AVERAGE ATOM PERCENT OF EACH ELEMENT IN THE SANDWICH EXPERIMENT SAMPLES COMPARED TO THE BAKED CONTROL.

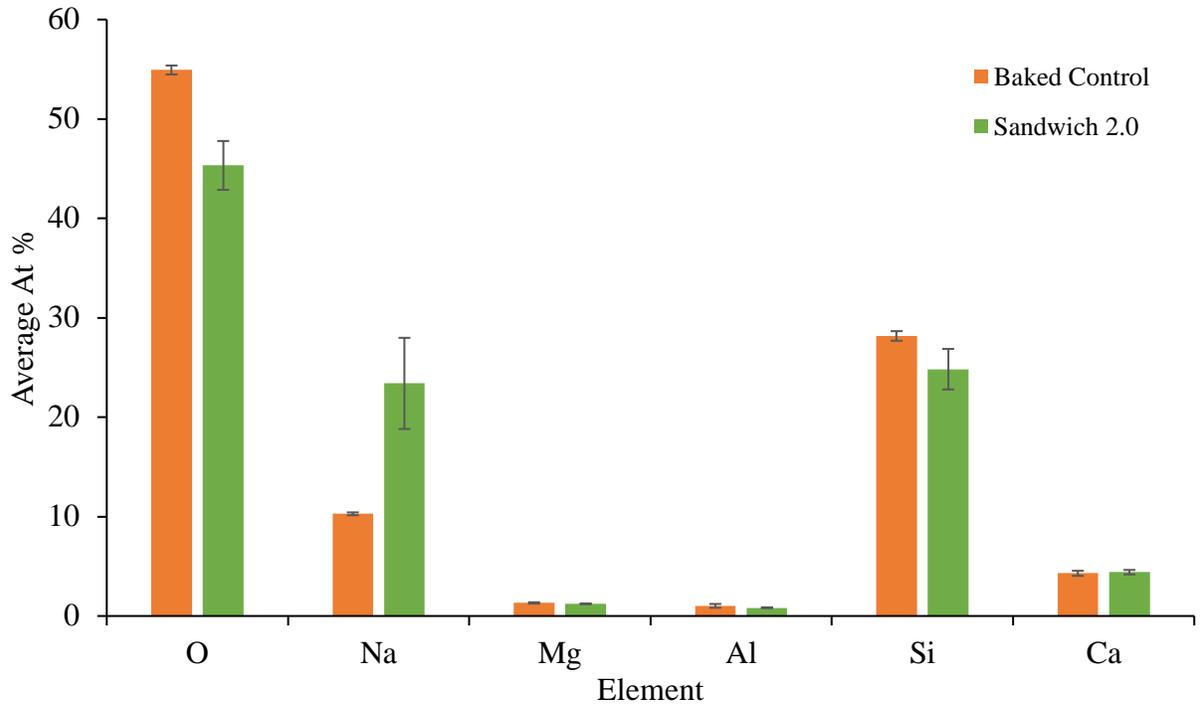


FIGURE 62 AVERAGE ATOM PERCENT OF EACH ELEMENT IN THE SANDWICH 2.0 450°C EXPERIMENT SAMPLES COMPARED TO THE BAKED CONTROL.

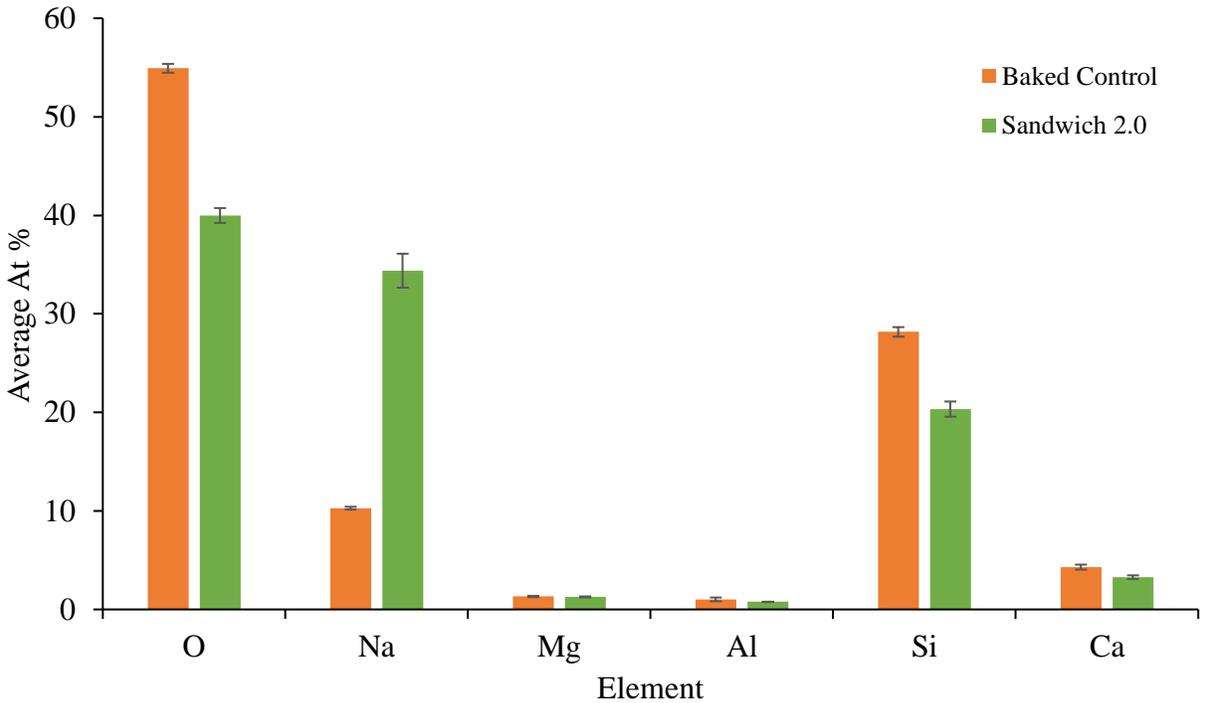


FIGURE 63 AVERAGE ATOM PERCENT OF EACH ELEMENT IN THE SANDWICH 2.0 500°C EXPERIMENT SAMPLES COMPARED TO THE BAKED CONTROL.

The melting points of the compounds present in fiberglass insulation range from 851-2852°C. The temperatures the experiment samples that were analyzed ranged from 400-500°C,

well below the lowest melting point of the insulation compounds present from manufacturing. The sodium increase experienced in the experiment samples indicates that sodium compounds beyond that of sodium carbonate could be present. The sodium compounds that could result from the environment the insulation samples experienced are shown in Table 8.

TABLE 8 SODIUM COMPOUNDS WITH ASSOCIATED CHEMICAL FORMULAS

<b>Compound name</b>	<b>Chemical formula</b>
Sodium oxide	Na <sub>2</sub> O
Sodium peroxide	Na <sub>2</sub> O <sub>2</sub>
Sodium hydroxide	NaOH
Sodium carbonate	Na <sub>2</sub> CO <sub>3</sub>
Sodium bicarbonate	2NaHCO <sub>3</sub>

The presence of sodium hydroxide and sodium bicarbonate are possible from an EDS analysis view since the EDS unit cannot detect hydrogen. Sodium hydroxide forms from the reactions of sodium metal with water and sodium oxide with water. Sodium hydroxide reacts with carbon dioxide (CO<sub>2</sub>) to create sodium carbonate due to the glovebox environment unable to maintain pure argon and the oxidation process for sample extraction. Sodium bicarbonate forms from the reaction of sodium carbonate, carbon dioxide, and water. However, sodium bicarbonate decays to sodium carbonate at temperatures above 50°C which were exceeded. Sodium hydroxide cannot be present in the experiment samples due to exposure to CO<sub>2</sub> in the oxidation process while sodium bicarbonate cannot be present due to chemical decay caused by the experiments operating above 50°C.

The presence of sodium carbonate not from the insulation fibers is possible due to the reaction of sodium hydroxide with carbon dioxide and the decomposition of sodium bicarbonate.

Sodium peroxide is formed from the reaction of sodium metal and oxygen (O<sub>2</sub>) with a further reaction with carbon dioxide to form sodium carbonate. Sodium peroxide is not possible as a compound to be found in the experiment samples due to the interaction with carbon dioxide during the oxidation process. Sodium oxide forms from the reactions of sodium hydroxide with sodium metal and sodium metal with oxygen. Sodium oxide is possible to be present in the experiment samples from the oxidation process of the samples, as shown in Table 9.

TABLE 9 SODIUM COMPOUND POSSIBILITY TO BE FOUND

<b>Compound name</b>	<b>Chemical formula</b>	<b>Presence</b>
Sodium oxide	Na <sub>2</sub> O	Possible
Sodium peroxide	Na <sub>2</sub> O <sub>2</sub>	Not possible
Sodium hydroxide	NaOH	Not possible
Sodium carbonate	Na <sub>2</sub> CO <sub>3</sub>	Possible
Sodium bicarbonate	2NaHCO <sub>3</sub>	Not possible

## Chapter 4: Conclusion

Sodium metal was successfully melted and allowed to interact with the standard insulation material of fiberglass insulation. The initial experimental setup of the drip assembly provided necessary insights into temperature control and sodium metal behavior. The evolution to the use of the jeweler's furnace provided a way to have the sodium metal remain in a molten state when placed in contact with the fiberglass insulation. A total of 17 experiments were performed with the jeweler's furnace in five configurations. It was observed that the insulation material darkened around the sodium sample at temperatures above 400°C. Fibrous samples were extracted from the darkened areas of insulation and removed from the glovebox environment for analysis. The analysis included imaging and elemental mapping of the fibrous samples. A total of 12 experiment samples were taken with 8 control samples for comparison.

The control samples consisted of insulation that was new and pre-heated. The images of the new insulation compared to the pre-heated insulation showed patchiness on the pre-heated fibers. The experiment insulation fibers resembled the new insulation fibers with a sample taken from an area in direct contact with the sodium sample showing deviations from the control fibers. The element profile for the new and pre-heated insulation revealed the presence of oxygen, sodium, magnesium, aluminum, silicon, and calcium being consistent with the compounds found in fiberglass insulation. The experiment insulation samples contained the same elements with an increase in the amount of sodium observed, as shown in Figure 64.

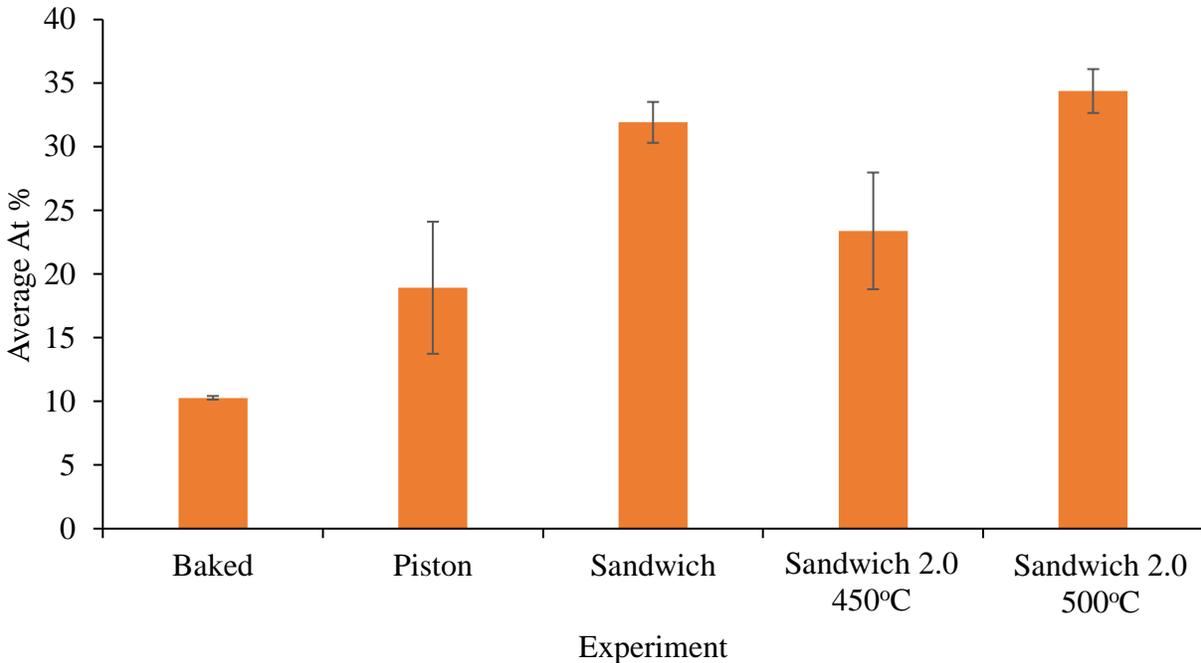


FIGURE 64 SODIUM AVERAGE ATOM PERCENT OF EXPERIMENTAL SAMPLES COMPARED TO BAKED CONTROLS.

The increased sodium existing in the experimental samples indicates that sodium compounds beyond what constitutes fiberglass insulation was present. The compounds of sodium oxide, sodium peroxide, sodium hydroxide, sodium carbonate, and sodium bicarbonate were considered for possibility to be present in the experiment samples. Sodium oxide is considered possible due to the exposure of sodium metal with oxygen in the glovebox environment and oxidation process as well as sodium hydroxide reacting with sodium metal. Sodium carbonate is considered possible from the exposure of sodium hydroxide to carbon dioxide during the oxidation process and the decomposition of sodium bicarbonate to sodium carbonate at temperatures above 50°C experienced during testing. Sodium peroxide is considered not possible due to exposure with carbon dioxide during the oxidation process to form sodium carbonate. Sodium hydroxide is considered not possible due to exposure with carbon dioxide during the oxidation process to formulate sodium carbonate. Sodium bicarbonate is not considered possible due to exposure with temperatures above 50°C during the testing of the samples resulting in decay to sodium carbonate.

and sodium carbonate were considered possible within the environmental conditions experienced by the experiment samples. Sodium peroxide, sodium hydroxide, and sodium bicarbonate were considered not possible due to chemical reactions that could happen during the environmental conditions experienced.

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# Appendix A: Drip Assembly Information

## A.1 Pipe Tape

TABLE 10 LAYERS OF PIPE TAPE NEEDED FOR EACH THREAD

<b>Part</b>	<b>Layers of pipe tape</b>
Cap Fitting	3 ½
Vessel (top)	5
Vessel (bottom)	5
Ball Valve	4 ½
Valve Fitting	3 ½
Nozzle	3 ½

## A.2 Air Test Procedures

- 1 Charge the air tank to 30 psi. (This was accomplished by using a compressor at a local gas station)
- 2 Fill the five-gallon bucket with water, about ¾ full
- 3 Wrap all threads with sufficient pipe tape (in accordance with Table 10)
- 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 needle valve (the valve should still be attached to the bushing)
- 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)
- 25

\* Bubbles may appear either at the threads or at the valve opening. Depending on where the bubbles appear, see below

#### Threads

- Take the assembly 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.3 Water Test Procedures**

- 1 Wrap all threads with sufficient pipe tape (in accordance with Table 10)
- 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 assembly 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 assembly
- 14 Attach the ¼-inch male, ½-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 threads or the valve opening. Depending on where the leak appears, see below

#### Threads

- Drain the assembly 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

## Appendix B: Met-L-X Safety Data Sheet



## Safety Data Sheet

This safety data sheet complies with the requirements of: 2012 OSHA Hazard Communication Standard ( 29CFR 1910.1200)

Product name MET-L-X® Dry Powder

### 1. Identification

#### 1.1. Product Identifier

Product name MET-L-X® Dry Powder

#### 1.2. Other means of identification

Product code 418288  
Synonyms None  
Chemical Family No information available

#### 1.3. Recommended use of the chemical and restrictions on use

Recommended use Fire extinguishing agent.  
Uses advised against Consumer use.

#### 1.4. Details of the Supplier of the Safety Data Sheet

Company Name Tyco Fire Protection Products  
One Stanton Street  
Marinette, WI 54143-2542  
Telephone: 715-735-7411  
Product Stewardship at 1-715-735-7411  
Contact point  
E-mail address psra@tycofp.com

#### 1.5. Emergency Telephone Number

Emergency telephone CHEMTREC 001-800-424-9300 or 001-703-527-3887

### 2. Hazards Identification

#### Classification

This chemical is considered hazardous by the 2012 OSHA Hazard Communication Standard (29 CFR 1910.1200)

#### 2.2. Label Elements

##### Hazard Statements

The product contains no substances which at their given concentration, are considered to be hazardous to health

##### Precautionary Statements

#### 2.3. Hazards Not Otherwise Classified (HNOC)

Not Applicable.

#### 2.4. Other Information

May be harmful if swallowed.

Unknown Acute Toxicity 99.5015% of the mixture consists of ingredient(s) of unknown toxicity

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### 3. Composition/information on Ingredients

#### 3.1. Mixture

The following component(s) in this product are considered hazardous under applicable OSHA(USA)

Chemical name	CAS No.	weight-%
Sodium chloride	7647-14-5	80 - 100
Attapulgit	12174-11-7	1 - 5
Magnesium stearate	557-04-0	1 - 5

### 4. First aid measures

#### 4.1. Description of first aid measures

**Eye Contact** Rinse immediately with plenty of water, also under the eyelids, for at least 15 minutes. If eye irritation persists: Get medical advice/attention.

**Skin contact** Wash skin with soap and water. Get medical attention if irritation develops and persists.

**Inhalation** If experiencing respiratory symptoms: Call a POISON CENTER or doctor.

**Ingestion** If swallowed. Call a POISON CENTER or doctor/physician if you feel unwell.

#### 4.2. Most Important Symptoms and Effects, Both Acute and Delayed

**Symptoms** None known.

#### 4.3. Indication of Any Immediate Medical Attention and Special Treatment Needed

**Note to physicians** Treat symptomatically.

### 5. Fire-fighting measures

#### 5.1. Suitable Extinguishing Media

Product is extinguishing agent. Use extinguishing measures that are appropriate to local circumstances and the surrounding environment.

#### 5.2. Unsuitable Extinguishing Media

None.

#### 5.3. Specific Hazards Arising from the Chemical

None in particular.

#### 5.4. Explosion Data

**Sensitivity to Mechanical Impact** None.

**Sensitivity to Static Discharge** None.

#### 5.5. Protective Equipment and Precautions for Firefighters

As in any fire, wear self-contained breathing apparatus pressure-demand, MSHA/NIOSH (approved or equivalent) and full protective gear.

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## 6. Accidental release measures

### 6.1. Personal precautions, protective equipment and emergency procedures

Personal Precautions	Provide adequate ventilation. Avoid creating dust. Avoid breathing dust/fume/gas/mist/vapors/spray.
For emergency responders	Use personal protection recommended in Section 8.

### 6.2. Environmental Precautions

Environmental Precautions	Prevent entry into waterways, sewers, basements or confined areas. Do not flush into surface water or sanitary sewer system. Prevent further leakage or spillage if safe to do so. Prevent product from entering drains. See Section 12 for additional Ecological Information.
---------------------------	--

### 6.3. Methods and material for containment and cleaning up

Methods for Containment	Stop leak if you can do it without risk. If sweeping of a contaminated area is necessary use a dust suppressing agent which does not react with product. Dike far ahead of spill; use dry sand to contain the flow of material. Absorb with earth, sand or other non-combustible material and transfer to containers for later disposal.
Methods for Cleaning Up	Following product recovery, flush area with water.

## 7. Handling and Storage

### 7.1. Precautions for Safe Handling

Advice on safe handling	Avoid generation of dust. Do not breathe dust/fume/gas/mist/vapors/spray. Use with local exhaust ventilation. Use personal protective equipment as required. Wash thoroughly after handling.
-------------------------	--

### 7.2. Conditions for safe storage, including any incompatibilities

Storage Conditions	Store in a well-ventilated place. Keep cool. Keep container tightly closed. Guard against dust accumulation of material. Use care in handling/storage.
Incompatible Materials	Strong acids.

## 8. Exposure Controls/Personal Protection

### 8.1. Control Parameters

#### Exposure guidelines

Chemical name	ACGIH TLV	OSHA PEL	NIOSH IDLH	Mexico OEL
Attapulgite 12174-11-7	TWA: 1 mg/m <sup>3</sup> respirable particulate matter	-	-	-
Magnesium stearate 557-04-0	TWA: 10 mg/m <sup>3</sup> inhalable particulate matter TWA: 3 mg/m <sup>3</sup> respirable particulate matter TWA: 10 mg/m <sup>3</sup> inhalable particulate matter except stearates of toxic metals TWA: 3 mg/mg <sup>3</sup> respirable particulate matter except stearates of	-	-	-

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	toxic metals		
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ACGIH (American Conference of Governmental Industrial Hygienists). OSHA (Occupational Safety and Health Administration of the US Department of Labor). NIOSH IDLH: Immediately Dangerous to Life or Health

**8.2. Appropriate Engineering Controls**

Engineering controls                      Ensure adequate ventilation, especially in confined areas.

**8.3. Individual protection measures, such as personal protective equipment**

Eye/Face Protection                      Avoid contact with eyes. Tight sealing safety goggles.

Skin and Body Protection                      No special precautions are needed in handling this material.

Respiratory Protection                      In case of insufficient ventilation, wear suitable respiratory equipment.

Ventilation                                      Use local exhaust or general dilution ventilation to control exposure with applicable limits

**8.4. General hygiene considerations**

Do not eat, drink or smoke when using this product. Handle in accordance with good industrial hygiene and safety practice.

**9. Physical and Chemical Properties**

**9.1. Information on basic physical and chemical properties**

Physical State	powder		
Odor	None	Color	Light brown
Odor Threshold	No data available		

Property	Values	Remarks - Method
pH	No data available	
Melting point/freezing point	No data available	
Boiling point / boiling range	No data available	
Flash Point	No data available	
Evaporation Rate	No data available	
Flammability (solid, gas)	No data available	
Flammability limit in air		
Upper flammability limit:	No data available	
Lower flammability limit:	No data available	
Vapor Pressure	No data available	
Vapor Density	No data available	
Specific gravity	No data available	
Water Solubility	No data available	
Solubility in Other Solvents	No data available	
Partition coefficient	No data available	
Autoignition Temperature	No data available	
Decomposition Temperature	No data available	
Kinematic viscosity	No data available	

**10. Stability and Reactivity**

**10.1. Chemical Stability**

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Stable under recommended storage conditions.

#### 10.2. Reactivity

No data available

#### 10.3. Possibility of hazardous reactions

None under normal processing.

**Hazardous Polymerization** Hazardous polymerization does not occur.

#### 10.4. Conditions to Avoid

Extremes of temperature and direct sunlight.

#### 10.5. Incompatible Materials

Strong acids.

#### 10.6. Hazardous decomposition products

Carbon oxides. Nitrogen oxides (NOx).

### 11. Toxicological Information

#### 11.1. Information on Likely Routes of Exposure

##### Product information

<b>Inhalation</b>	May cause irritation of respiratory tract.
<b>Eye Contact</b>	May cause irritation.
<b>Skin contact</b>	May cause irritation.
<b>Ingestion</b>	Ingestion may cause irritation to mucous membranes. May be harmful if swallowed.

##### Acute Toxicity

Chemical name	Oral LD50	Dermal LD50	Inhalation LC50
Sodium chloride 7847-14-5	= 3 g/kg ( Rat )	> 10 g/kg ( Rabbit )	> 42 g/m <sup>3</sup> ( Rat ) 1 h

#### 11.2. Information on Toxicological Effects

**Symptoms** No information available.

#### 11.3. Delayed and immediate effects as well as chronic effects from short and long-term exposure

<b>Skin Corrosion/Irritation</b>	No information available.
<b>Serious eye damage/eye irritation</b>	No information available.
<b>Sensitization</b>	No information available.
<b>Germ Cell Mutagenicity</b>	No information available.
<b>Carcinogenicity</b>	Attapulgit (palygorskite fibers) is a hydrated magnesium aluminum silicate. Long palygorskite (attapulgit) fibers (>5 micrometers) are possibly carcinogenic to humans (Group 2B). Short palygorskite (attapulgit) fibers (<5 micrometers) cannot be classified as to their carcinogenicity to humans (Group 3). The attapulgit present in this product contains fibers 0.5-2.5 um range, so would be considered by IARC as Group 3.

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Chemical name	ACGIH	IARC	NTP	OSHA
Attapulgit 12174-11-7	-	Group 3	-	X

*ACGIH (American Conference of Governmental Industrial Hygienists)*  
*A2 - Suspected Human Carcinogen*  
*IARC (International Agency for Research on Cancer)*  
*Not classifiable as a human carcinogen*  
*Group 1 - Carcinogenic to Humans*  
*NTP (National Toxicology Program)*  
*Known - Known Carcinogen*  
*OSHA (Occupational Safety and Health Administration of the US Department of Labor)*  
 X - Present

Reproductive Toxicity No information available.  
 STOT - Single Exposure No information available.  
 STOT - Repeated Exposure No information available.  
 Aspiration Hazard No information available.

**11.4. Numerical Measures of Toxicity - Product information**

The following values are calculated based on chapter 3.1 of the GHS document  
 ATEmix (oral) 3481 mg/kg

**12. Ecological Information**

**12.1. Ecotoxicity**

Not classified.

7.02% of the mixture consists of component(s) of unknown hazards to the aquatic environment

Chemical name	Algae/aquatic plants	Fish	Crustacea
Sodium chloride 7647-14-5	-	LC50 (96h) static = 12946 mg/L Lepomis macrochirus LC50 (96h) static 6020 - 7070 mg/L Pimephales promelas LC50 (96h) flow-through 5560 - 8080 mg/L Lepomis macrochirus LC50 (96h) static 8420 - 8700 mg/L Pimephales promelas LC50 (96h) semi-static = 7050 mg/L Pimephales promelas LC50 (96h) flow-through 4747 - 7824 mg/L Oncorhynchus mykiss	EC50 (48h) Static 340.7 - 469.2 mg/L Daphnia magna EC50 (48h) = 1000 mg/L Daphnia magna

**12.2. Persistence and Degradability**  
 No information available.

**12.3. Bioaccumulation**  
 No information available.

**12.4. Other Adverse Effects**  
 No information available.

**13. Disposal Considerations**

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**13.1. Waste Treatment Methods**

**Disposal of wastes**

Disposal should be in accordance with applicable regional, national and local laws and regulations.

**Contaminated Packaging**

Do not reuse container.

**14. Transport Information**

<u>DOT</u>	NOT REGULATED
<u>TDG</u>	NOT REGULATED
<u>MEX</u>	NOT REGULATED
<u>ICAO (air)</u>	NOT REGULATED
<u>IATA</u>	NOT REGULATED
<u>IMDG</u>	NOT REGULATED

**15. Regulatory Information**

**15.1. International Inventories**

TSCA	Complies
DSL/NDSL	Complies
ENCS	Does not comply
IECSC	Complies
KECL	Does not comply
PICCS	Complies
AICS	Complies

**Legend:**

TSCA - United States Toxic Substances Control Act Section 8(b) Inventory  
DSL/NDSL - Canadian Domestic Substances List/Non-Domestic Substances List  
ENCS - Japan Existing and New Chemical Substances  
IECSC - China Inventory of Existing Chemical Substances  
KECL - Korean Existing and Evaluated Chemical Substances  
PICCS - Philippines Inventory of Chemicals and Chemical Substances  
AICS - Australian Inventory of Chemical Substances

**15.2. US Federal Regulations**

**SARA 313**

Section 313 of Title III of the Superfund Amendments and Reauthorization Act of 1986 (SARA). This product does not contain any chemicals which are subject to the reporting requirements of the Act and Title 40 of the Code of Federal Regulations, Part 372

**SARA 311/312 Hazard Categories**

Acute Health Hazard	No
Chronic health hazard	No
Fire Hazard	No
Sudden Release of Pressure Hazard	No
Reactive Hazard	No

**CWA (Clean Water Act)**

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This product does not contain any substances regulated as pollutants pursuant to the Clean Water Act (40 CFR 122.21 and 40 CFR 122.42)

**CERCLA**

This material, as supplied, does not contain any substances regulated as hazardous substances under the Comprehensive Environmental Response Compensation and Liability Act (CERCLA) (40 CFR 302) or the Superfund Amendments and Reauthorization Act (SARA) (40 CFR 355). There may be specific reporting requirements at the local, regional, or state level pertaining to releases of this material

**15.3. US State Regulations**

**California Proposition 65**

This product contains the following Proposition 65 chemicals

Chemical name	California Proposition 65
Attapulgite - 12174-11-7	Carcinogen
Quartz - 14808-80-7	Carcinogen

**U.S. State Right-to-Know Regulations**

Chemical name	New Jersey	Massachusetts	Pennsylvania
Quartz 14808-80-7	X	X	X

**16. Other information, including date of preparation of the last revision**

<b>NFPA</b>	Health Hazards 0	Flammability 0	Instability 0	Physical and chemical properties -
<b>HMIS</b>	Health Hazards 0	Flammability 0	Physical Hazards 0	Personal Protection X

Revision date 27-Jul-2017

Revision note No information available.

**Disclaimer**

The information provided in this Safety Data Sheet is correct to the best of our knowledge, information and belief at the date of its publication. The information given is designed only as a guidance for safe handling, use, processing, storage, transportation, disposal and release and is not to be considered a warranty or quality specification. The information relates only to the specific material designated and may not be valid for such material used in combination with any other materials or in any process, unless specified in the text.

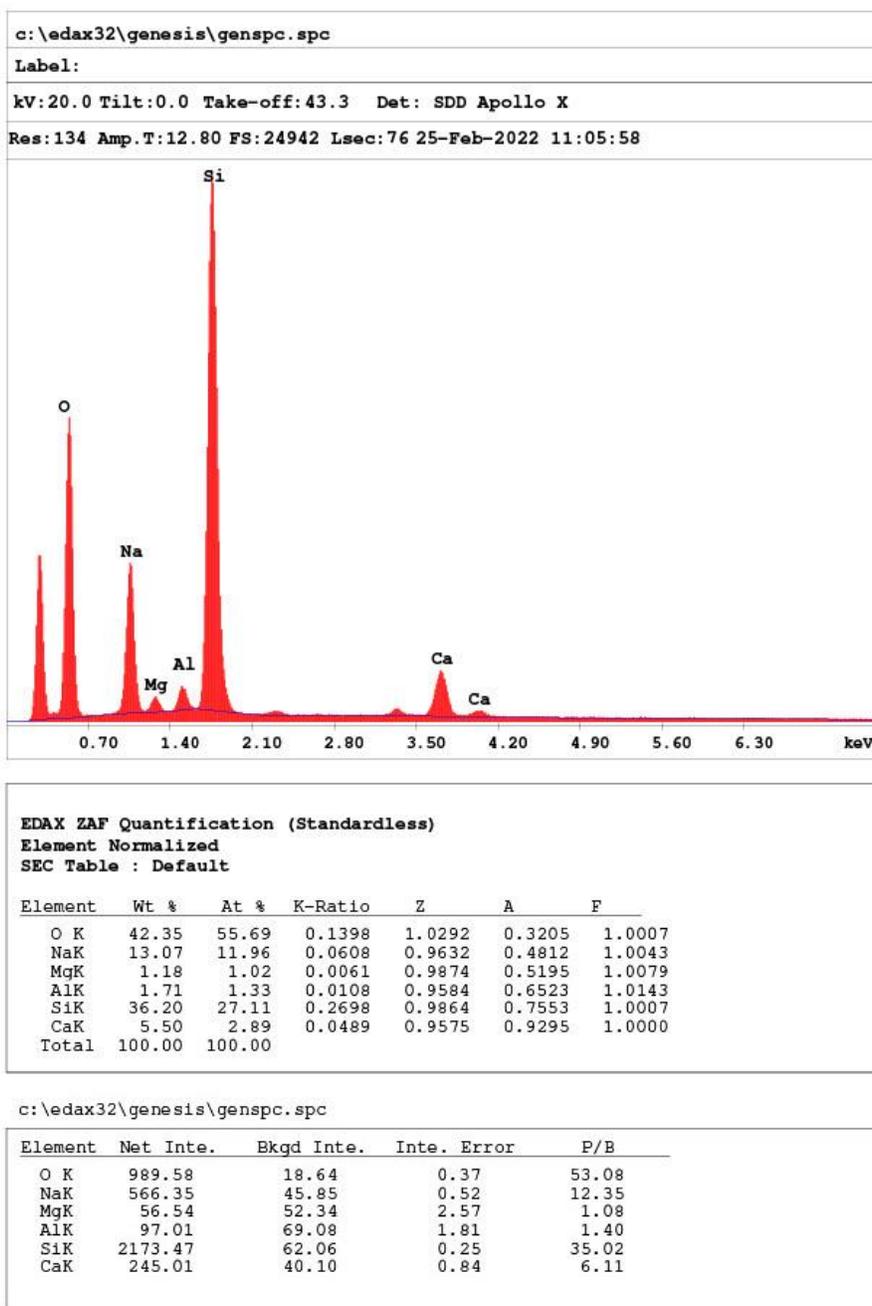
End of Safety Data Sheet

Revision date 27-Jul-2017

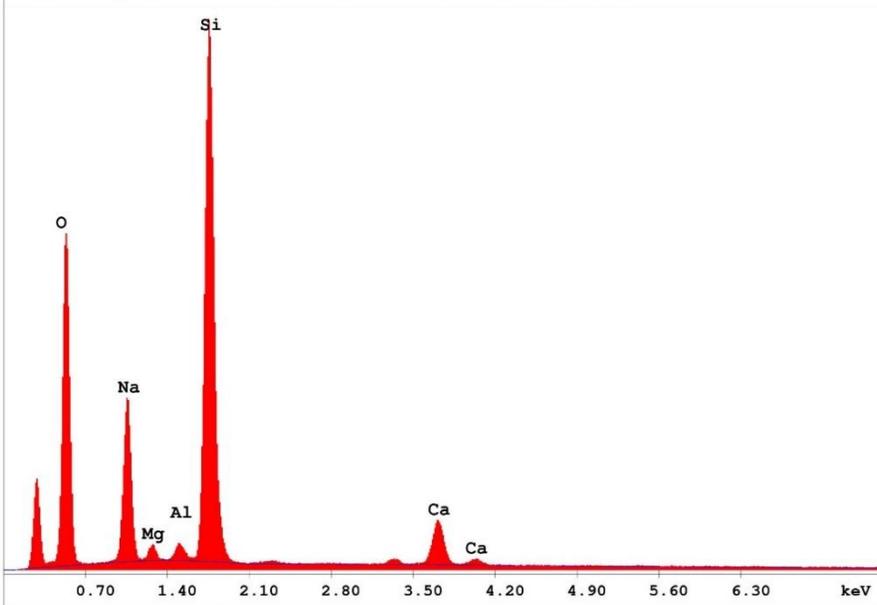
Version 32

# Appendix C: EDS Reports of Control and Baked Control Samples

## C.1 Control EDS Reports



c:\edax32\genesis\genspc.spc  
 Label:  
 kV:20.0 Tilt:0.0 Take-off:43.4 Det: SDD Apollo X  
 Res:134 Amp.T:12.80 FS:25392 Lsec:76 25-Feb-2022 11:09:29

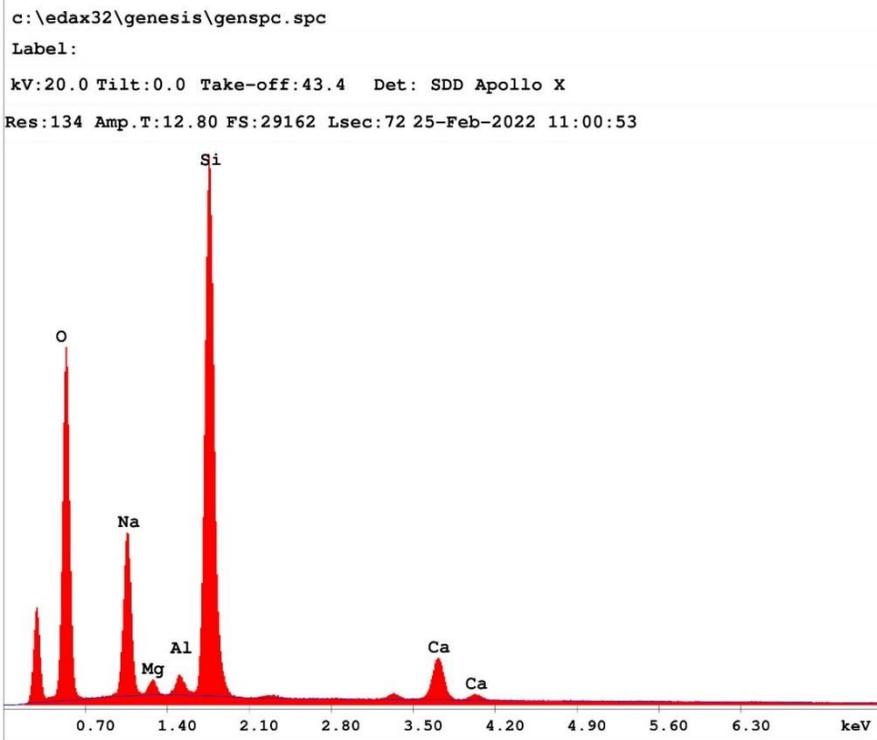


**EDAX ZAF Quantification (Standardless)**  
**Element Normalized**  
**SEC Table : Default**

Element	Wt %	At %	K-Ratio	Z	A	F
O K	43.81	57.03	0.1493	1.0285	0.3312	1.0007
NaK	13.81	12.51	0.0638	0.9626	0.4777	1.0041
MgK	1.20	1.03	0.0061	0.9868	0.5112	1.0074
AlK	1.24	0.96	0.0077	0.9578	0.6443	1.0136
SiK	34.81	25.82	0.2585	0.9857	0.7529	1.0006
CaK	5.13	2.67	0.0458	0.9568	0.9321	1.0000
Total	100.00	100.00				

c:\edax32\genesis\genspc.spc

Element	Net Inte.	Bkgd Inte.	Inte. Error	P/B
O K	1111.26	18.18	0.35	61.11
NaK	623.92	43.45	0.49	14.36
MgK	59.15	49.72	2.43	1.19
AlK	73.03	56.62	2.13	1.29
SiK	2188.03	44.39	0.25	49.29
CaK	240.74	35.89	0.84	6.71



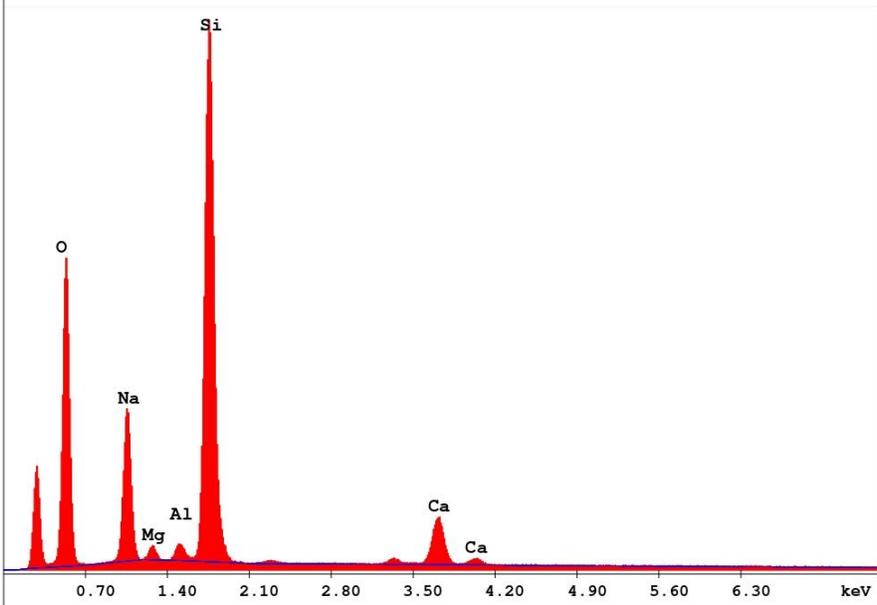
EDAX ZAF Quantification (Standardless)  
 Element Normalized  
 SEC Table : Default

Element	Wt %	At %	K-Ratio	Z	A	F
O K	44.51	57.67	0.1544	1.0281	0.3373	1.0006
NaK	13.81	12.45	0.0636	0.9622	0.4764	1.0040
MgK	1.16	0.99	0.0059	0.9864	0.5102	1.0073
AlK	1.35	1.03	0.0084	0.9574	0.6438	1.0134
SiK	34.37	25.37	0.2547	0.9854	0.7515	1.0006
CaK	4.81	2.49	0.0429	0.9564	0.9328	1.0000
Total	100.00	100.00				

c:\edax32\genesis\genspc.spc

Element	Net Inte.	Bkgd Inte.	Inte. Error	P/B
O K	1425.58	23.87	0.32	59.73
NaK	771.48	57.26	0.45	13.47
MgK	70.69	64.46	2.35	1.10
AlK	98.41	73.07	1.86	1.35
SiK	2673.49	58.00	0.23	46.09
CaK	280.02	44.37	0.80	6.31

c:\edax32\genesis\genspc.spc  
 Label:  
 kV:20.0 Tilt:0.0 Take-off:43.4 Det: SDD Apollo X  
 Res:134 Amp.T:12.80 FS:24998 Lsec:76 25-Feb-2022 11:03:13

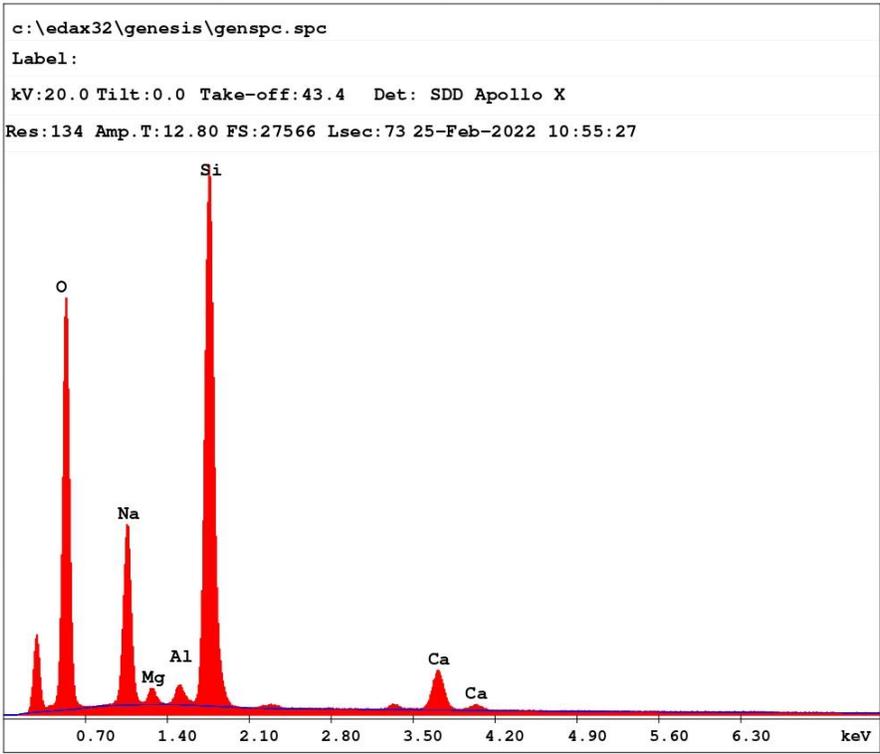


**EDAX ZAF Quantification (Standardless)**  
 Element Normalized  
 SEC Table : Default

Element	Wt %	At %	K-Ratio	Z	A	F
O K	42.68	56.04	0.1406	1.0290	0.3200	1.0006
NaK	13.35	12.20	0.0619	0.9631	0.4791	1.0042
MgK	1.04	0.90	0.0054	0.9873	0.5158	1.0077
AlK	1.25	0.97	0.0079	0.9582	0.6501	1.0143
SiK	35.96	26.90	0.2689	0.9862	0.7577	1.0007
CaK	5.72	3.00	0.0509	0.9573	0.9305	1.0000
Total	100.00	100.00				

c:\edax32\genesis\genspc.spc

Element	Net Inte.	Bkqd Inte.	Inte. Error	P/B
O K	1005.14	15.95	0.37	63.03
NaK	581.42	45.99	0.51	12.64
MgK	49.92	53.56	2.88	0.93
AlK	71.33	55.45	2.17	1.29
SiK	2185.77	43.99	0.25	49.69
CaK	257.47	38.01	0.81	6.77



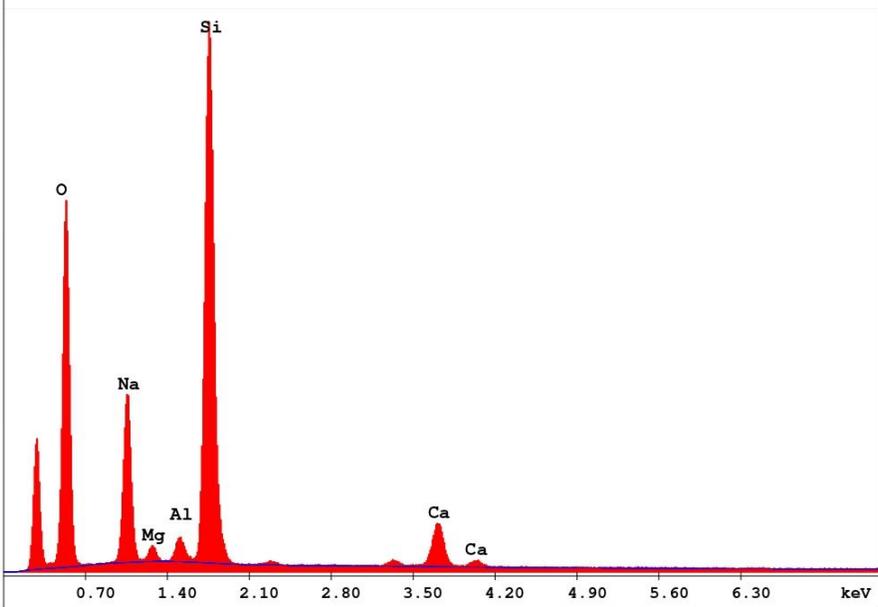
EDAX ZAF Quantification (Standardless)  
 Element Normalized  
 SEC Table : Default

Element	Wt %	At %	K-Ratio	Z	A	F
O K	46.50	59.45	0.1694	1.0272	0.3544	1.0006
NaK	14.38	12.80	0.0655	0.9614	0.4719	1.0037
MgK	1.20	1.01	0.0060	0.9856	0.5026	1.0068
AlK	1.35	1.02	0.0083	0.9566	0.6362	1.0124
SiK	32.39	23.59	0.2378	0.9845	0.7452	1.0005
CaK	4.17	2.13	0.0373	0.9555	0.9358	1.0000
Total	100.00	100.00				

c:\edax32\genesis\genspc.spc

Element	Net Inte.	Bkqd Inte.	Inte. Error	P/B
O K	1562.87	26.07	0.30	59.96
NaK	794.58	59.13	0.44	13.44
MgK	72.07	62.46	2.27	1.15
AlK	97.19	68.18	1.83	1.43
SiK	2494.94	54.10	0.24	46.12
CaK	243.51	39.51	0.86	6.16

c:\edax32\genesis\genspc.spc  
 Label:  
 kV:20.0 Tilt:0.0 Take-off:43.4 Det: SDD Apollo X  
 Res:134 Amp.T:12.80 FS:21564 Lsec:77 25-Feb-2022 10:58:07



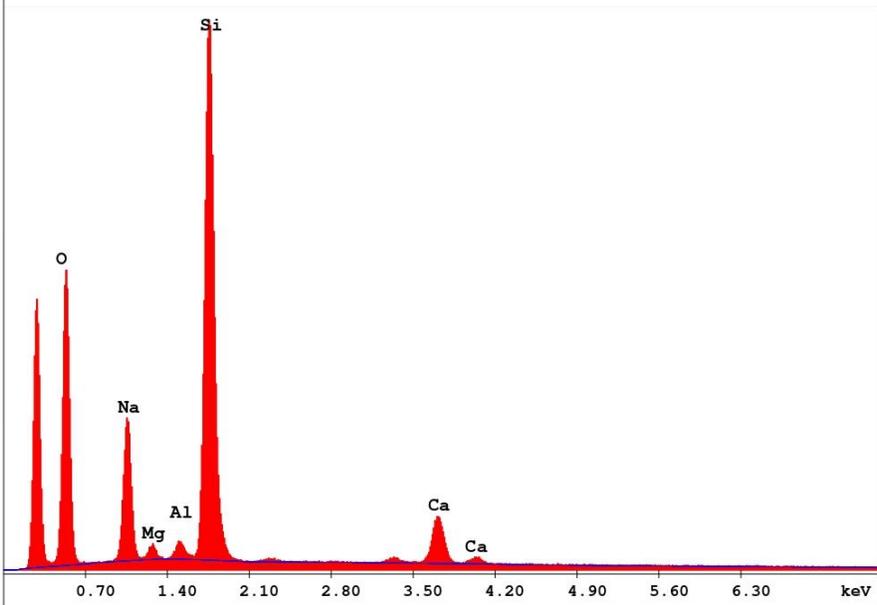
**EDAX ZAF Quantification (Standardless)**  
 Element Normalized  
 SEC Table : Default

Element	Wt %	At %	K-Ratio	Z	A	F
O K	44.76	57.92	0.1558	1.0281	0.3383	1.0006
NaK	13.86	12.48	0.0636	0.9623	0.4753	1.0039
MgK	1.16	0.99	0.0059	0.9864	0.5090	1.0072
AlK	1.69	1.29	0.0105	0.9575	0.6427	1.0130
SiK	33.66	24.81	0.2481	0.9854	0.7476	1.0006
CaK	4.87	2.51	0.0435	0.9564	0.9335	1.0000
Total	100.00	100.00				

c:\edax32\genesis\genspc.spc

Element	Net Inte.	Bkqd Inte.	Inte. Error	P/B
O K	1020.85	18.16	0.36	56.20
NaK	548.33	41.91	0.52	13.08
MgK	50.16	46.64	2.71	1.08
AlK	87.34	51.28	1.79	1.70
SiK	1848.74	40.36	0.27	45.80
CaK	201.40	31.31	0.91	6.43

c:\edax32\genesis\genspc.spc  
 Label:  
 kV:20.0 Tilt:0.0 Take-off:43.4 Det: SDD Apollo X  
 Res:134 Amp.T:12.80 FS:25009 Lsec:75 25-Feb-2022 10:50:02

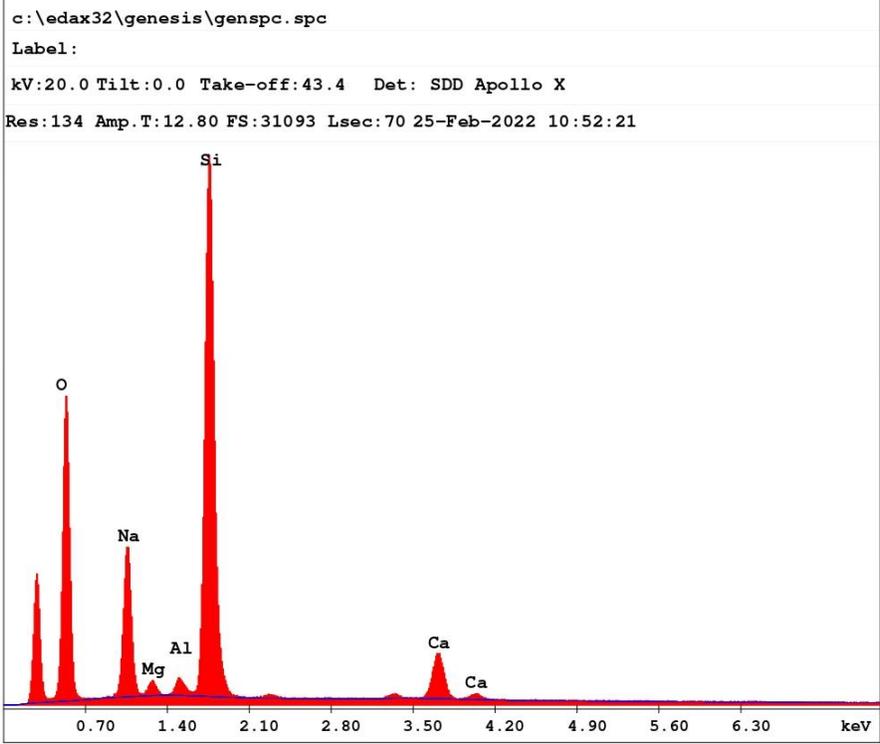


**EDAX ZAF Quantification (Standardless)**  
 Element Normalized  
 SEC Table : Default

Element	Wt %	At %	K-Ratio	Z	A	F
O K	42.41	55.82	0.1385	1.0290	0.3171	1.0006
NaK	12.75	11.68	0.0591	0.9631	0.4794	1.0043
MgK	1.17	1.01	0.0060	0.9873	0.5208	1.0079
AlK	1.31	1.02	0.0083	0.9583	0.6537	1.0146
SiK	36.57	27.42	0.2743	0.9862	0.7601	1.0007
CaK	5.80	3.05	0.0516	0.9574	0.9297	1.0000
Total	100.00	100.00				

c:\edax32\genesis\genspc.spc

Element	Net Inte.	Bkgd Inte.	Inte. Error	P/B
O K	991.57	20.34	0.37	48.74
NaK	556.69	48.76	0.53	11.42
MgK	56.43	55.96	2.65	1.01
AlK	75.60	64.77	2.18	1.17
SiK	2234.29	53.05	0.25	42.12
CaK	261.38	46.42	0.83	5.63



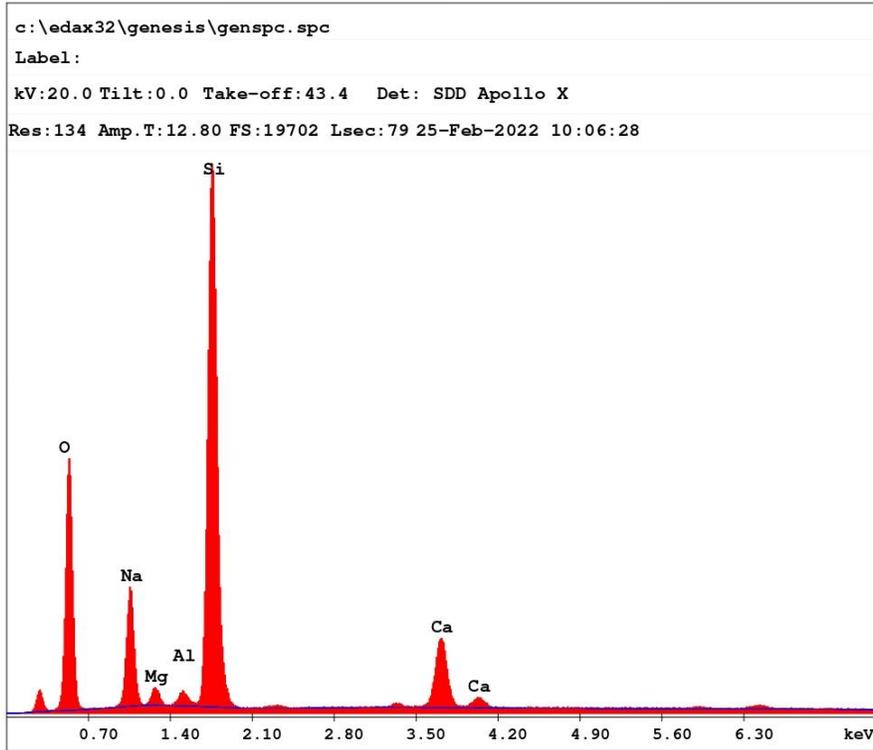
EDAX ZAF Quantification (Standardless)  
 Element Normalized  
 SEC Table : Default

Element	Wt %	At %	K-Ratio	Z	A	F
O K	42.38	55.69	0.1401	1.0291	0.3210	1.0007
NaK	13.36	12.22	0.0622	0.9632	0.4814	1.0043
MgK	1.20	1.04	0.0062	0.9874	0.5174	1.0078
AlK	1.26	0.98	0.0080	0.9583	0.6501	1.0144
SiK	36.31	27.18	0.2714	0.9863	0.7575	1.0007
CaK	5.50	2.88	0.0489	0.9574	0.9297	1.0000
Total	100.00	100.00				

c:\vedax32\genesis\genspc.spc

Element	Net Inte.	Bkgd Inte.	Inte. Error	P/B
O K	1342.03	23.08	0.33	58.14
NaK	783.80	56.87	0.46	13.78
MgK	77.41	66.10	2.23	1.17
AlK	96.58	75.91	1.94	1.27
SiK	2957.82	60.93	0.22	48.54
CaK	331.47	58.97	0.76	5.62

## C.2 Baked Control EDS Reports



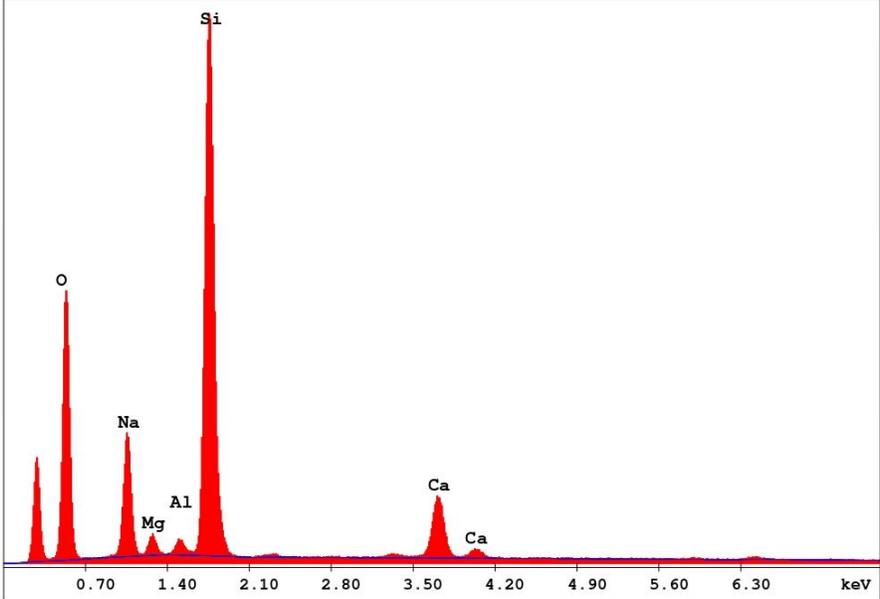
**EDAX ZAF Quantification (Standardless)**  
 Element Normalized  
 SEC Table : Default

Element	Wt %	At %	K-Ratio	Z	A	F
O K	40.80	54.74	0.1200	1.0299	0.2853	1.0006
NaK	10.74	10.03	0.0490	0.9639	0.4716	1.0045
MgK	1.38	1.22	0.0073	0.9881	0.5314	1.0083
AlK	1.09	0.87	0.0070	0.9590	0.6617	1.0153
SiK	37.23	28.45	0.2828	0.9870	0.7689	1.0011
CaK	8.75	4.69	0.0780	0.9583	0.9298	1.0000
Total	100.00	100.00				

c:\edax32\genesis\genspc.spc

Element	Net Inte.	Bkqd Inte.	Inte. Error	P/B
O K	628.05	9.54	0.46	65.85
NaK	337.53	27.25	0.66	12.39
MgK	49.98	31.90	2.40	1.57
AlK	46.73	32.47	2.54	1.44
SiK	1683.95	25.17	0.28	66.89
CaK	288.67	30.17	0.73	9.57

c:\edax32\genesis\genspc.spc  
 Label:  
 kV:20.0 Tilt:0.0 Take-off:43.4 Det: SDD Apollo X  
 Res:134 Amp.T:12.80 FS:22866 Lsec:76 25-Feb-2022 10:15:37



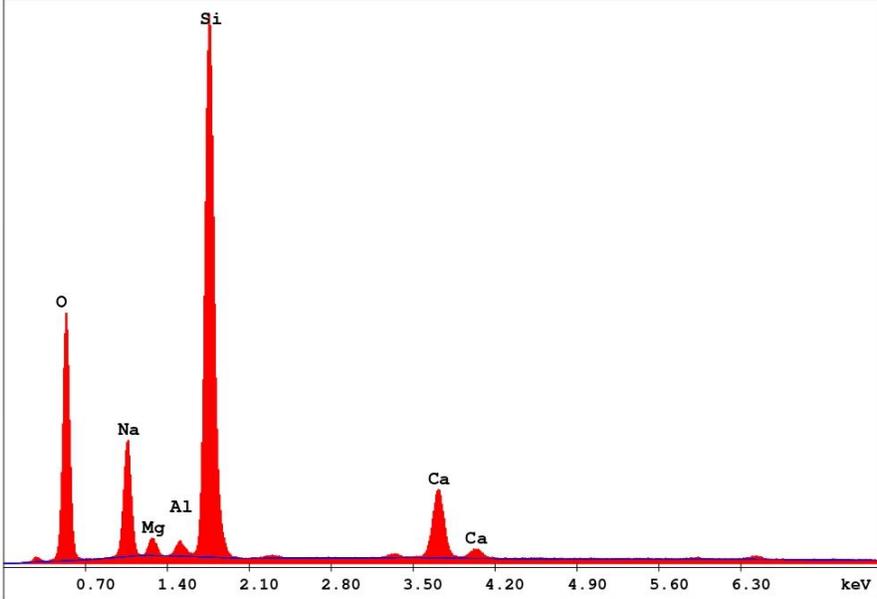
**EDAX ZAF Quantification (Standardless)**  
 Element Normalized  
 SEC Table : Default

Element	Wt %	At %	K-Ratio	Z	A	F
O K	41.78	55.55	0.1280	1.0293	0.2975	1.0006
NaK	11.10	10.27	0.0508	0.9633	0.4730	1.0044
MgK	1.58	1.38	0.0083	0.9875	0.5295	1.0081
AlK	1.15	0.91	0.0074	0.9585	0.6581	1.0150
SiK	36.77	27.85	0.2779	0.9864	0.7653	1.0009
CaK	7.62	4.04	0.0679	0.9577	0.9302	1.0000
Total	100.00	100.00				

c:\edax32\genesis\genspc.spc

Element	Net Inte.	Bkqd Inte.	Inte. Error	P/B
O K	814.92	12.72	0.41	64.07
NaK	425.09	32.12	0.59	13.23
MgK	69.03	38.22	1.99	1.81
AlK	59.46	44.89	2.34	1.32
SiK	2011.55	36.24	0.26	55.50
CaK	305.55	34.33	0.72	8.90

c:\edax32\genesis\genspc.spc  
 Label:  
 kV:20.0 Tilt:0.0 Take-off:43.3 Det: SDD Apollo X  
 Res:134 Amp.T:12.80 FS:27258 Lsec:74 25-Feb-2022 10:19:57



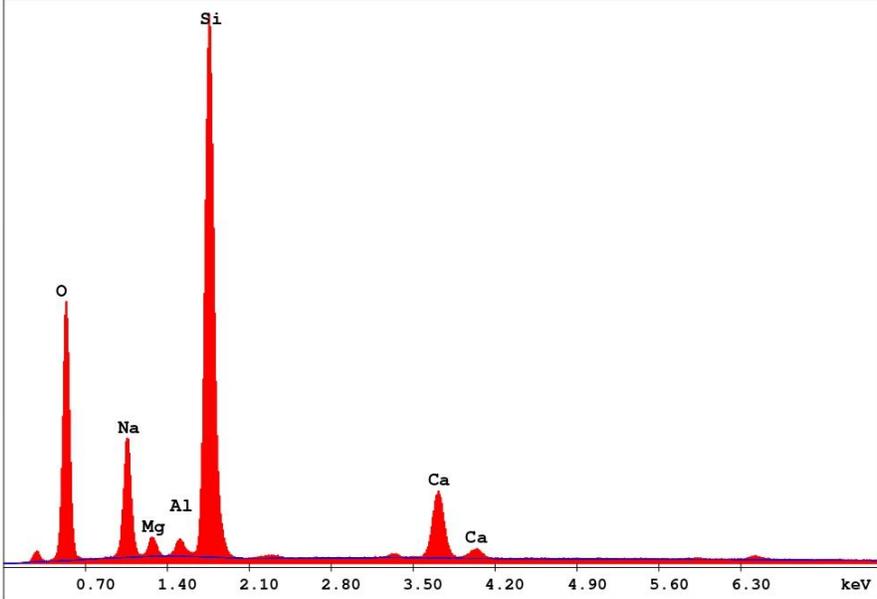
**EDAX ZAF Quantification (Standardless)**  
 Element Normalized  
 SEC Table : Default

Element	Wt %	At %	K-Ratio	Z	A	F
O K	40.50	54.40	0.1192	1.0300	0.2856	1.0006
NaK	10.81	10.11	0.0496	0.9640	0.4734	1.0045
MgK	1.39	1.22	0.0073	0.9882	0.5321	1.0084
AlK	1.11	0.88	0.0071	0.9591	0.6623	1.0155
SiK	37.64	28.80	0.2861	0.9871	0.7692	1.0011
CaK	8.56	4.59	0.0763	0.9584	0.9290	1.0000
Total	100.00	100.00				

c:\edax32\genesis\genspc.spc

Element	Net Inte.	Bkqd Inte.	Inte. Error	P/B
O K	900.84	13.21	0.39	68.22
NaK	492.57	39.31	0.56	12.53
MgK	72.47	46.20	2.05	1.57
AlK	68.37	47.26	2.16	1.45
SiK	2459.60	36.92	0.24	66.62
CaK	407.68	43.40	0.63	9.39

c:\edax32\genesis\genspc.spc  
 Label:  
 kV:20.0 Tilt:0.0 Take-off:43.4 Det: SDD Apollo X  
 Res:134 Amp.T:12.80 FS:27211 Lsec:73 25-Feb-2022 10:23:25



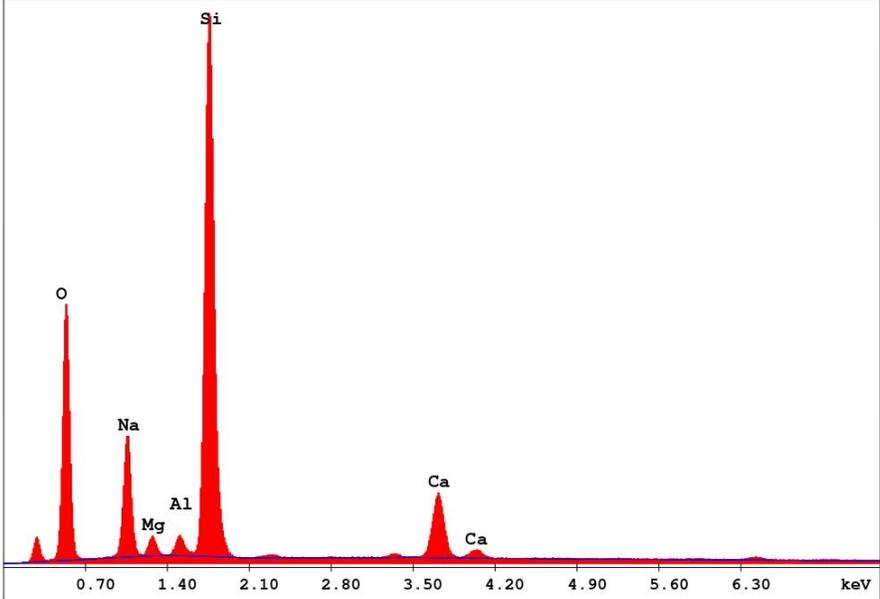
**EDAX ZAF Quantification (Standardless)**  
 Element Normalized  
 SEC Table : Default

Element	Wt %	At %	K-Ratio	Z	A	F
O K	41.07	54.92	0.1228	1.0298	0.2901	1.0006
NaK	11.02	10.26	0.0504	0.9638	0.4728	1.0044
MgK	1.54	1.35	0.0081	0.9880	0.5300	1.0081
AlK	1.29	1.02	0.0083	0.9589	0.6589	1.0150
SiK	36.78	28.01	0.2778	0.9869	0.7647	1.0010
CaK	8.30	4.43	0.0740	0.9582	0.9300	1.0000
Total	100.00	100.00				

c:\edax32\genesis\genspc.spc

Element	Net Inte.	Bkqd Inte.	Inte. Error	P/B
O K	965.18	14.32	0.38	67.40
NaK	521.49	35.38	0.55	14.74
MgK	83.23	41.14	1.81	2.02
AlK	82.46	47.53	1.89	1.73
SiK	2483.77	37.83	0.24	65.66
CaK	411.24	42.92	0.63	9.58

c:\edax32\genesis\genspc.spc  
 Label:  
 kV:20.0 Tilt:0.0 Take-off:43.4 Det: SDD Apollo X  
 Res:134 Amp.T:12.80 FS:29619 Lsec:73 25-Feb-2022 10:29:46

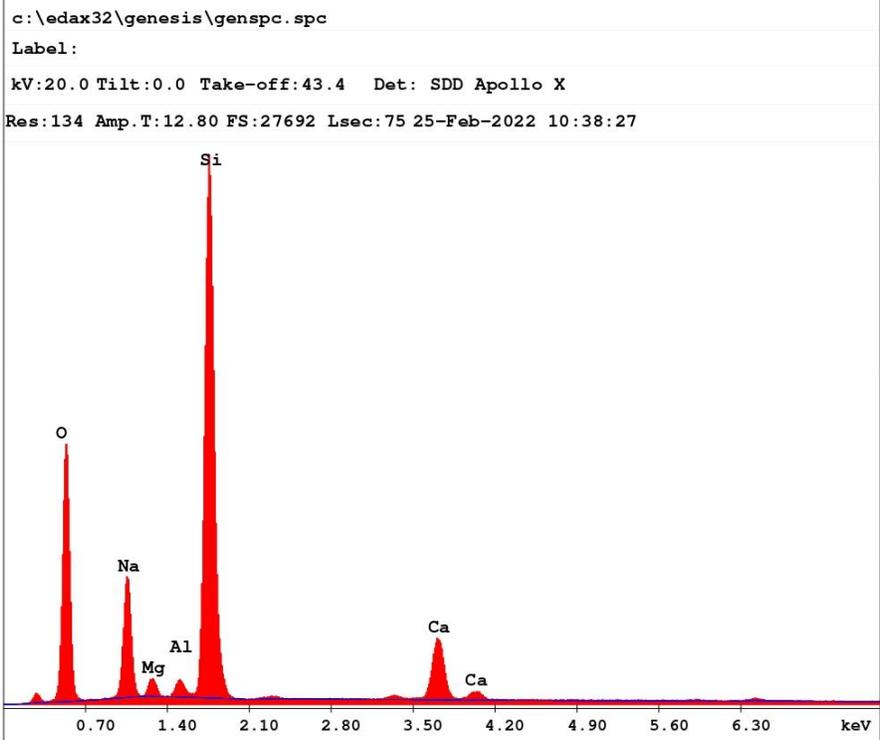


**EDAX ZAF Quantification (Standardless)**  
 Element Normalized  
 SEC Table : Default

Element	Wt %	At %	K-Ratio	Z	A	F
O K	40.68	54.46	0.1225	1.0299	0.2921	1.0006
NaK	11.09	10.34	0.0512	0.9639	0.4762	1.0045
MgK	1.53	1.35	0.0081	0.9881	0.5320	1.0083
AlK	1.46	1.16	0.0094	0.9591	0.6607	1.0152
SiK	37.37	28.49	0.2823	0.9870	0.7646	1.0010
CaK	7.87	4.21	0.0701	0.9583	0.9287	1.0000
Total	100.00	100.00				

c:\edax32\genesis\genspc.spc

Element	Net Inte.	Bkqd Inte.	Inte. Error	P/B
O K	1040.77	15.72	0.37	66.19
NaK	571.78	40.49	0.52	14.12
MgK	89.96	47.91	1.77	1.88
AlK	101.24	55.94	1.68	1.81
SiK	2728.66	44.55	0.23	61.25
CaK	421.08	49.45	0.63	8.52



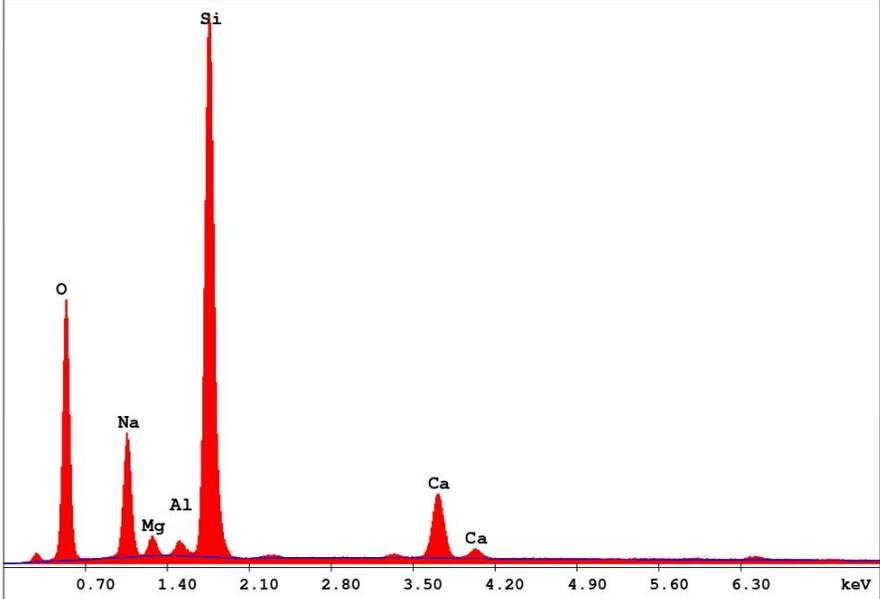
EDAX ZAF Quantification (Standardless)  
 Element Normalized  
 SEC Table : Default

Element	Wt %	At %	K-Ratio	Z	A	F
O K	41.20	54.99	0.1249	1.0297	0.2941	1.0006
NaK	11.20	10.40	0.0514	0.9637	0.4743	1.0045
MgK	1.45	1.28	0.0077	0.9879	0.5297	1.0082
AlK	1.29	1.02	0.0083	0.9588	0.6594	1.0151
SiK	37.02	28.14	0.2797	0.9868	0.7651	1.0010
CaK	7.83	4.17	0.0698	0.9581	0.9296	1.0000
Total	100.00	100.00				

c:\edax32\genesis\genspc.spc

Element	Net Inte.	Bkgd Inte.	Inte. Error	P/B
O K	954.32	14.15	0.38	67.44
NaK	516.78	40.40	0.54	12.79
MgK	76.42	46.83	1.96	1.63
AlK	80.44	48.25	1.90	1.67
SiK	2431.79	38.05	0.24	63.92
CaK	377.00	35.96	0.65	10.49

c:\edax32\genesis\genspc.spc  
 Label:  
 kV:20.0 Tilt:0.0 Take-off:43.4 Det: SDD Apollo X  
 Res:134 Amp.T:12.80 FS:26126 Lsec:75 25-Feb-2022 10:41:11



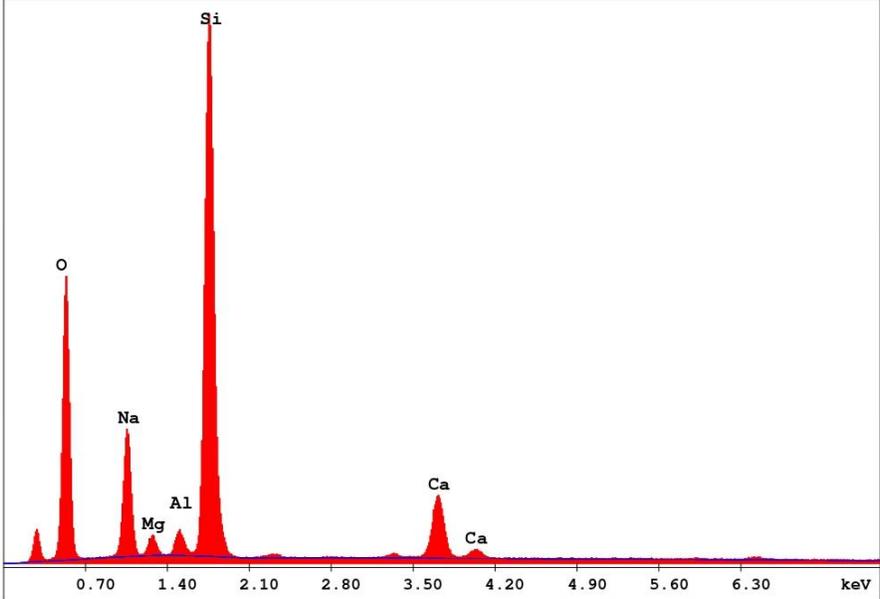
**EDAX ZAF Quantification (Standardless)**  
 Element Normalized  
 SEC Table : Default

Element	Wt %	At %	K-Ratio	Z	A	F
O K	40.94	54.73	0.1233	1.0298	0.2923	1.0006
NaK	11.19	10.41	0.0514	0.9637	0.4748	1.0045
MgK	1.54	1.35	0.0081	0.9879	0.5302	1.0082
AlK	1.11	0.88	0.0071	0.9589	0.6590	1.0152
SiK	37.27	28.38	0.2821	0.9869	0.7664	1.0010
CaK	7.96	4.25	0.0708	0.9582	0.9293	1.0000
Total	100.00	100.00				

c:\edax32\genesis\genspc.spc

Element	Net Inte.	Bkqd Inte.	Inte. Error	P/B
O K	896.72	12.69	0.39	70.66
NaK	491.70	33.34	0.55	14.75
MgK	76.99	40.38	1.87	1.91
AlK	65.77	46.13	2.20	1.43
SiK	2333.56	35.56	0.24	65.61
CaK	364.27	39.97	0.66	9.11

c:\edax32\genesis\genspc.spc  
 Label:  
 kV:20.0 Tilt:0.0 Take-off:43.4 Det: SDD Apollo X  
 Res:134 Amp.T:12.80 FS:22216 Lsec:77 25-Feb-2022 10:47:15



**EDAX ZAF Quantification (Standardless)**  
 Element Normalized  
 SEC Table : Default

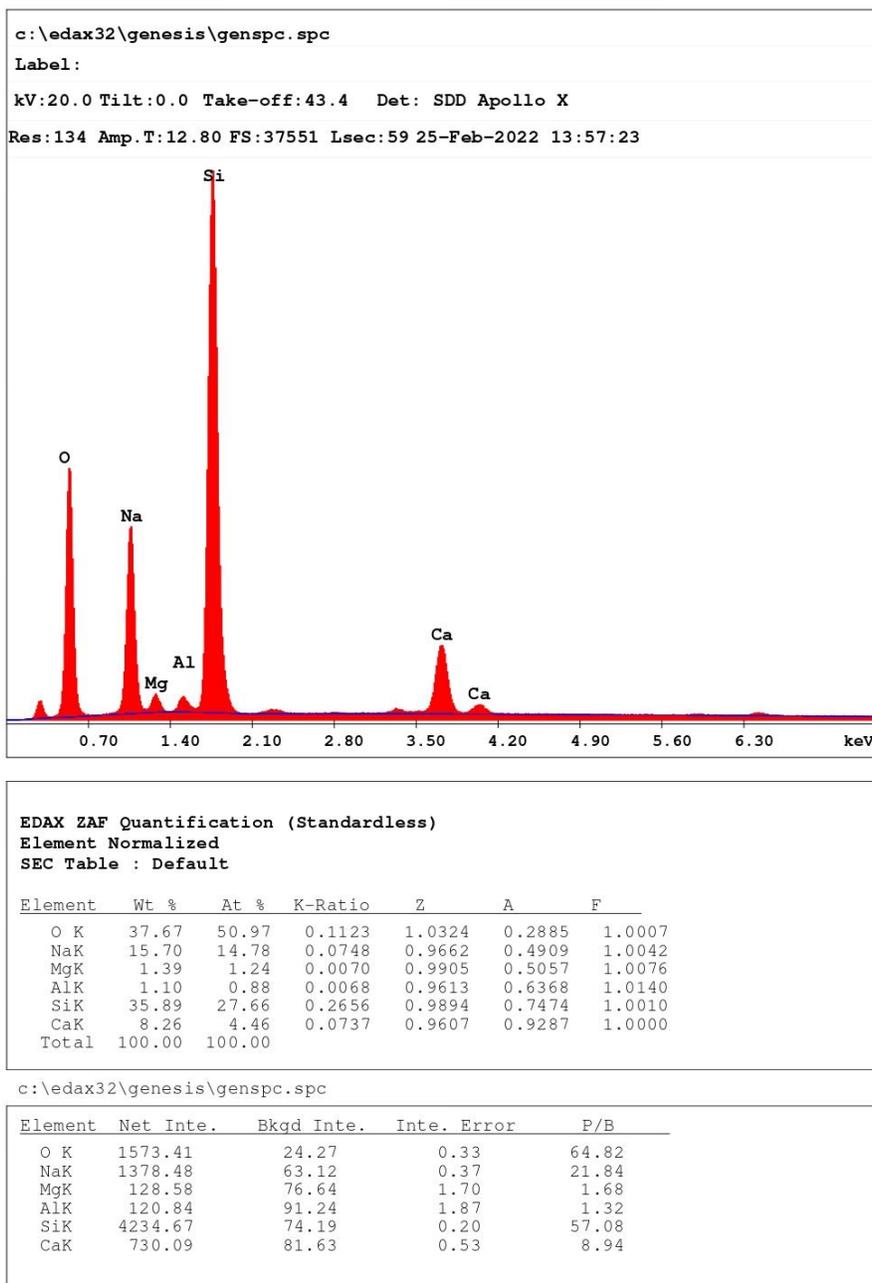
Element	Wt %	At %	K-Ratio	Z	A	F
O K	41.87	55.60	0.1292	1.0295	0.2995	1.0006
NaK	11.26	10.41	0.0516	0.9635	0.4734	1.0044
MgK	1.57	1.37	0.0083	0.9877	0.5285	1.0080
AlK	1.81	1.42	0.0116	0.9586	0.6572	1.0145
SiK	35.98	27.22	0.2695	0.9866	0.7585	1.0009
CaK	7.51	3.98	0.0669	0.9578	0.9306	1.0000
Total	100.00	100.00				

c:\edax32\genesis\genspc.spc

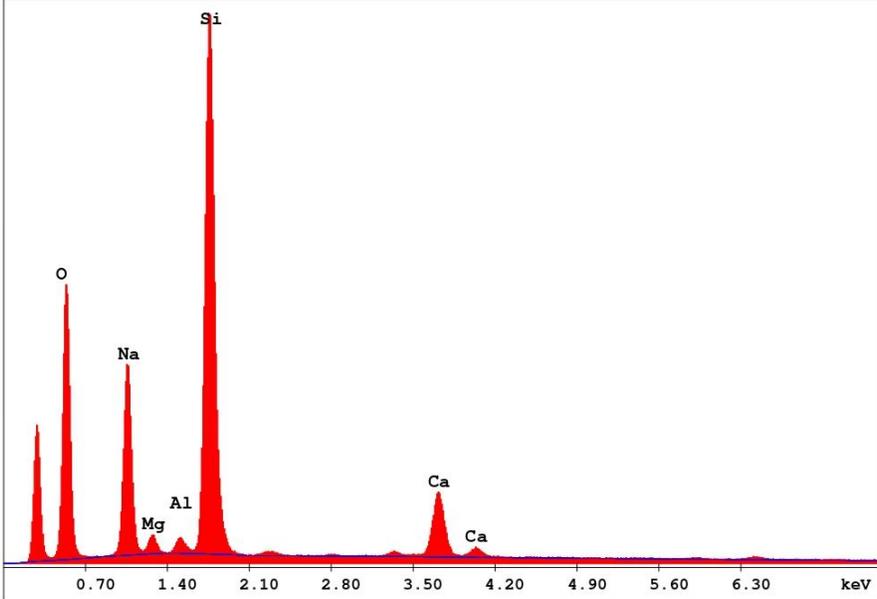
Element	Net Inte.	Bkgd Inte.	Inte. Error	P/B
O K	810.89	12.55	0.40	64.64
NaK	425.99	30.58	0.59	13.93
MgK	67.75	35.51	1.97	1.91
AlK	91.98	40.87	1.63	2.25
SiK	1924.39	32.26	0.26	59.65
CaK	297.08	31.60	0.73	9.40

# Appendix D: EDS Reports of Experiment Samples

## D.1 Piston EDS Reports



c:\edax32\genesis\genspc.spc  
 Label:  
 kV:20.0 Tilt:0.0 Take-off:43.4 Det: SDD Apollo X  
 Res:134 Amp.T:12.80 FS:34878 Lsec:60 25-Feb-2022 13:59:53



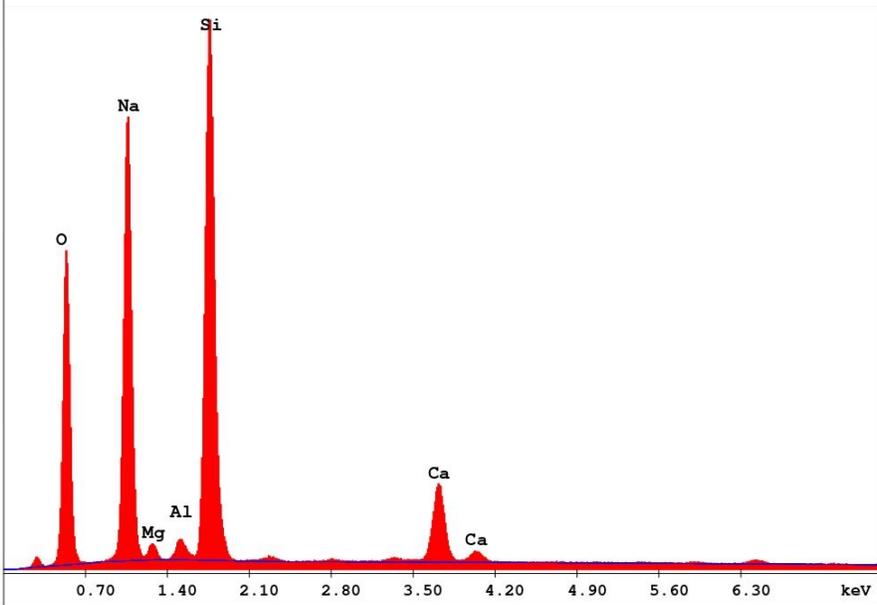
EDAX ZAF Quantification (Standardless)  
 Element Normalized  
 SEC Table : Default

Element	Wt %	At %	K-Ratio	Z	A	F
O K	39.17	52.43	0.1214	1.0315	0.3001	1.0007
NaK	15.73	14.66	0.0746	0.9654	0.4888	1.0041
MgK	1.50	1.32	0.0075	0.9896	0.5039	1.0074
AlK	1.11	0.88	0.0068	0.9605	0.6343	1.0136
SiK	35.12	26.78	0.2590	0.9885	0.7453	1.0009
CaK	7.37	3.94	0.0658	0.9598	0.9299	1.0000
Total	100.00	100.00				

c:\edax32\genesis\genspc.spc

Element	Net Inte.	Bkqd Inte.	Inte. Error	P/B
O K	1585.81	29.71	0.33	53.38
NaK	1281.13	75.08	0.38	17.06
MgK	128.46	88.42	1.75	1.45
AlK	113.14	105.24	2.05	1.08
SiK	3849.05	87.61	0.21	43.93
CaK	607.60	80.40	0.59	7.56

c:\edax32\genesis\genspc.spc  
 Label:  
 kV:20.0 Tilt:0.0 Take-off:43.4 Det: SDD Apollo X  
 Res:134 Amp.T:12.80 FS:29202 Lsec:56 25-Feb-2022 13:52:36

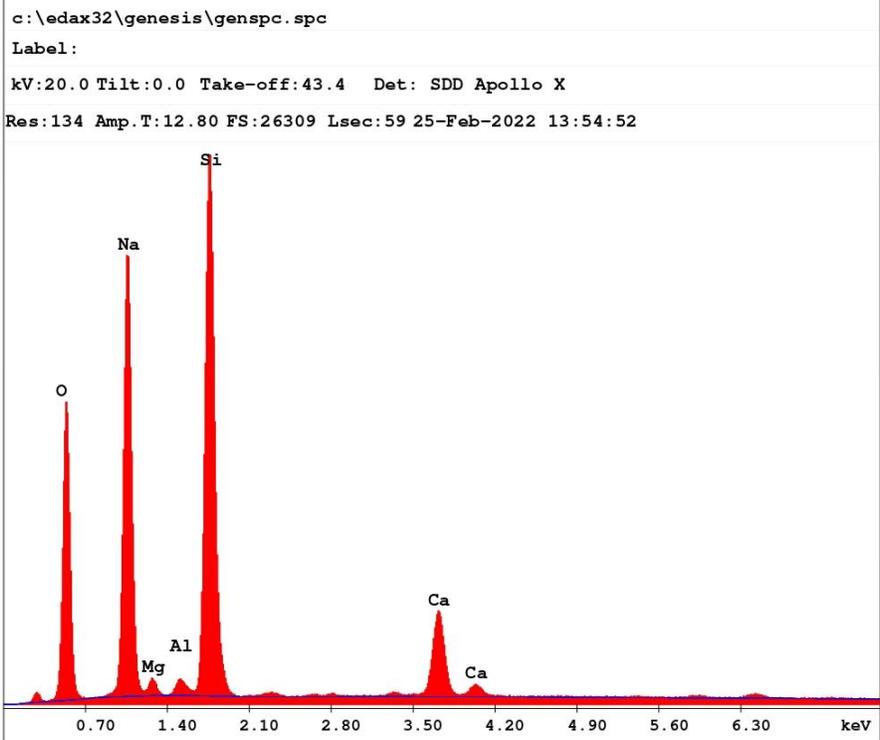


**EDAX ZAF Quantification (Standardless)**  
 Element Normalized  
 SEC Table : Default

Element	Wt %	At %	K-Ratio	Z	A	F
O K	34.00	46.00	0.1087	1.0365	0.3083	1.0009
NaK	26.83	25.26	0.1369	0.9700	0.5244	1.0033
MgK	1.17	1.04	0.0052	0.9944	0.4468	1.0056
AlK	1.28	1.02	0.0072	0.9652	0.5802	1.0103
SiK	29.65	22.85	0.2052	0.9933	0.6961	1.0008
CaK	7.08	3.82	0.0636	0.9644	0.9317	1.0000
Total	100.00	100.00				

c:\edax32\genesis\genspc.spc

Element	Net Inte.	Bkgd Inte.	Inte. Error	P/B
O K	1592.26	30.80	0.34	51.70
NaK	2636.78	70.29	0.27	37.52
MgK	99.85	77.67	2.12	1.29
AlK	134.11	88.56	1.75	1.51
SiK	3417.61	75.48	0.23	45.28
CaK	658.47	79.24	0.58	8.31

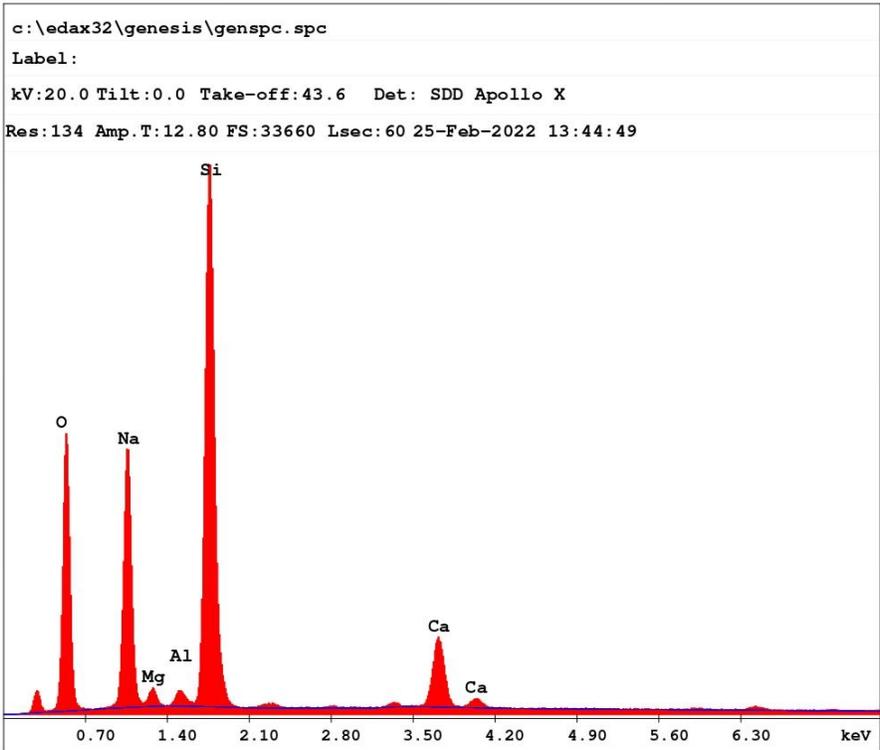


EDAX ZAF Quantification (Standardless)  
 Element Normalized  
 SEC Table : Default

Element	Wt %	At %	K-Ratio	Z	A	F
O K	33.27	45.27	0.1037	1.0370	0.3005	1.0009
NaK	26.95	25.52	0.1376	0.9705	0.5243	1.0033
MgK	1.21	1.08	0.0054	0.9948	0.4461	1.0056
AlK	1.04	0.84	0.0059	0.9656	0.5792	1.0104
SiK	29.76	23.07	0.2063	0.9937	0.6970	1.0009
CaK	7.77	4.22	0.0699	0.9649	0.9317	1.0000
Total	100.00	100.00				

c:\edax32\genesis\genspc.spc

Element	Net Inte.	Bkqd Inte.	Inte. Error	P/B
O K	1295.30	24.77	0.37	52.29
NaK	2259.17	56.42	0.28	40.04
MgK	87.73	61.41	2.14	1.43
AlK	92.84	70.42	2.13	1.32
SiK	2929.95	60.50	0.24	48.43
CaK	616.80	73.56	0.58	8.38



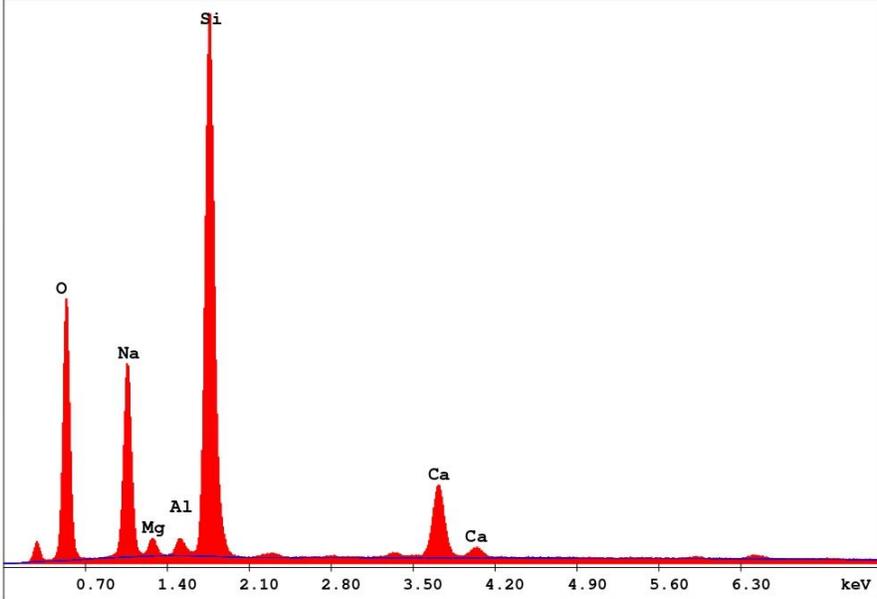
EDAX ZAF Quantification (Standardless)  
 Element Normalized  
 SEC Table : Default

Element	Wt %	At %	K-Ratio	Z	A	F
O K	36.76	49.59	0.1139	1.0335	0.2997	1.0008
NaK	19.51	18.32	0.0954	0.9673	0.5034	1.0039
MgK	1.38	1.23	0.0067	0.9916	0.4856	1.0068
AlK	1.11	0.89	0.0067	0.9624	0.6173	1.0126
SiK	33.78	25.96	0.2446	0.9905	0.7303	1.0009
CaK	7.46	4.02	0.0667	0.9617	0.9300	1.0000
Total	100.00	100.00				

c:\edax32\genesis\genspc.spc

Element	Net Inte.	Bkqd Inte.	Inte. Error	P/B
O K	1529.26	24.73	0.33	61.84
NaK	1681.33	60.72	0.32	27.69
MgK	117.32	70.93	1.76	1.65
AlK	113.37	83.18	1.90	1.36
SiK	3728.01	68.82	0.21	54.17
CaK	631.93	87.85	0.58	7.19

c:\edax32\genesis\genspc.spc  
 Label:  
 kV:20.0 Tilt:0.0 Take-off:43.4 Det: SDD Apollo X  
 Res:134 Amp.T:12.80 FS:31562 Lsec:63 25-Feb-2022 13:49:49



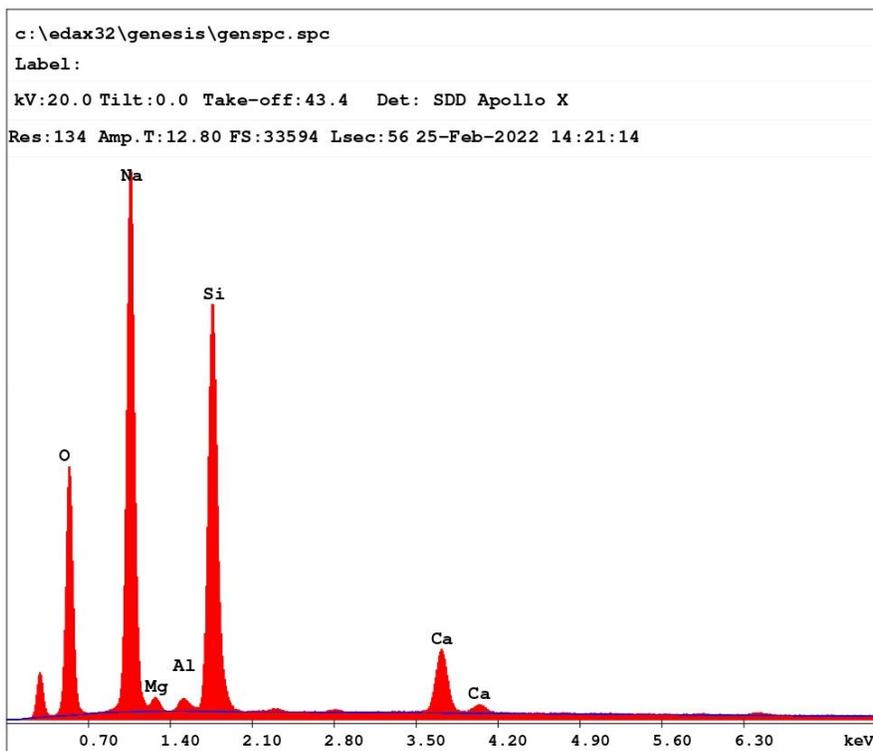
**EDAX ZAF Quantification (Standardless)**  
 Element Normalized  
 SEC Table : Default

Element	Wt %	At %	K-Ratio	Z	A	F
O K	38.05	51.36	0.1138	1.0323	0.2896	1.0007
NaK	15.99	15.02	0.0759	0.9661	0.4892	1.0041
MgK	1.37	1.22	0.0069	0.9904	0.5023	1.0074
AlK	1.22	0.97	0.0075	0.9613	0.6339	1.0136
SiK	35.02	26.93	0.2580	0.9893	0.7439	1.0010
CaK	8.36	4.50	0.0747	0.9606	0.9298	1.0000
Total	100.00	100.00				

c:\edax32\genesis\genspc.spc

Element	Net Inte.	Bkgd Inte.	Inte. Error	P/B
O K	1283.18	20.49	0.36	62.63
NaK	1124.78	50.15	0.39	22.43
MgK	100.92	58.47	1.84	1.73
AlK	107.25	68.78	1.83	1.56
SiK	3307.78	56.26	0.22	58.79
CaK	595.16	60.61	0.56	9.82

## D.2 Sandwich EDS Reports



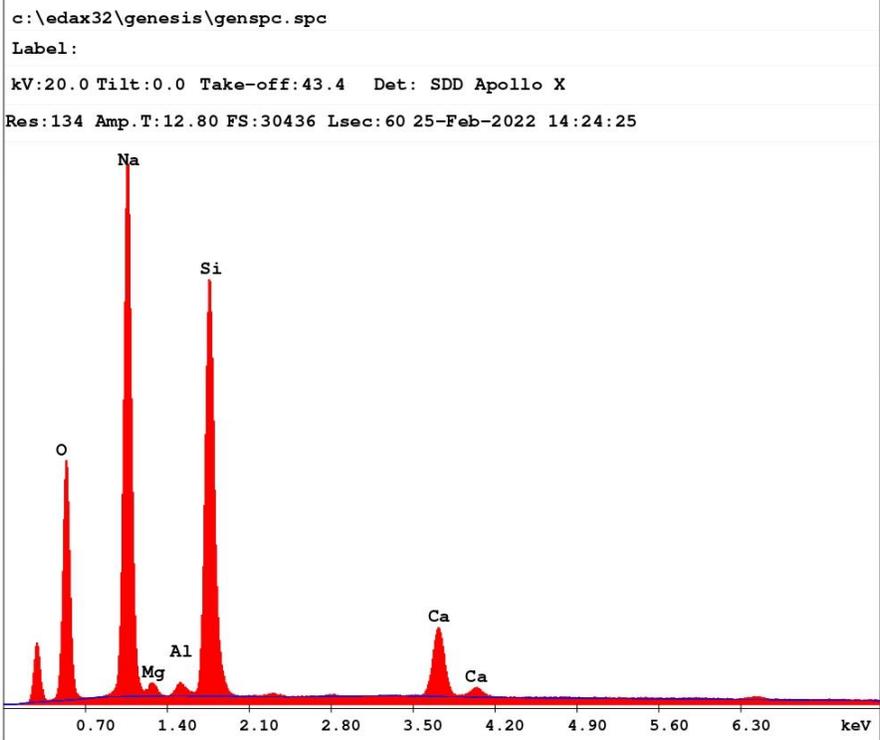
### EDAX ZAF Quantification (Standardless)

Element Normalized  
 SEC Table : Default

Element	Wt %	At %	K-Ratio	Z	A	F
O K	29.82	40.72	0.0983	1.0402	0.3164	1.0011
NaK	35.00	33.25	0.1905	0.9735	0.5576	1.0029
MgK	1.25	1.13	0.0052	0.9979	0.4125	1.0047
AlK	0.96	0.78	0.0051	0.9686	0.5437	1.0086
SiK	26.47	20.59	0.1756	0.9968	0.6651	1.0007
CaK	6.49	3.54	0.0585	0.9678	0.9318	1.0000
Total	100.00	100.00				

c:\edax32\genesis\genspc.spc

Element	Net Inte.	Bkgd Inte.	Inte. Error	P/B
O K	1464.43	33.50	0.36	43.71
NaK	3733.77	74.03	0.22	50.44
MgK	100.80	79.61	2.13	1.27
AlK	96.71	90.32	2.29	1.07
SiK	2976.79	78.48	0.25	37.93
CaK	616.99	84.48	0.60	7.30

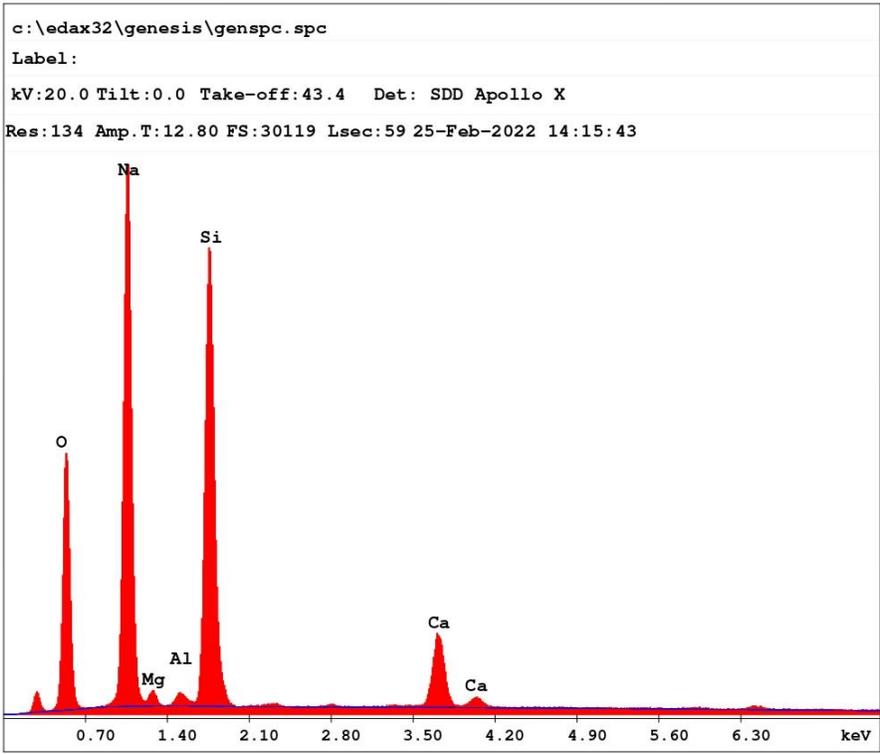


EDAX ZAF Quantification (Standardless)  
 Element Normalized  
 SEC Table : Default

Element	Wt %	At %	K-Ratio	Z	A	F
O K	29.42	40.34	0.0948	1.0404	0.3094	1.0011
NaK	34.51	32.94	0.1874	0.9737	0.5561	1.0029
MgK	1.22	1.10	0.0051	0.9981	0.4147	1.0048
AlK	0.97	0.79	0.0052	0.9688	0.5463	1.0088
SiK	26.87	20.99	0.1790	0.9970	0.6674	1.0008
CaK	7.01	3.84	0.0632	0.9680	0.9315	1.0000
Total	100.00	100.00				

c:\vedax32\genesis\genspc.spc

Element	Net Inte.	Bkgd Inte.	Inte. Error	P/B
O K	1193.71	28.47	0.38	41.92
NaK	3103.53	63.60	0.23	48.80
MgK	83.27	67.12	2.27	1.24
AlK	82.59	76.05	2.38	1.09
SiK	2563.17	67.12	0.26	38.19
CaK	562.78	85.17	0.62	6.61



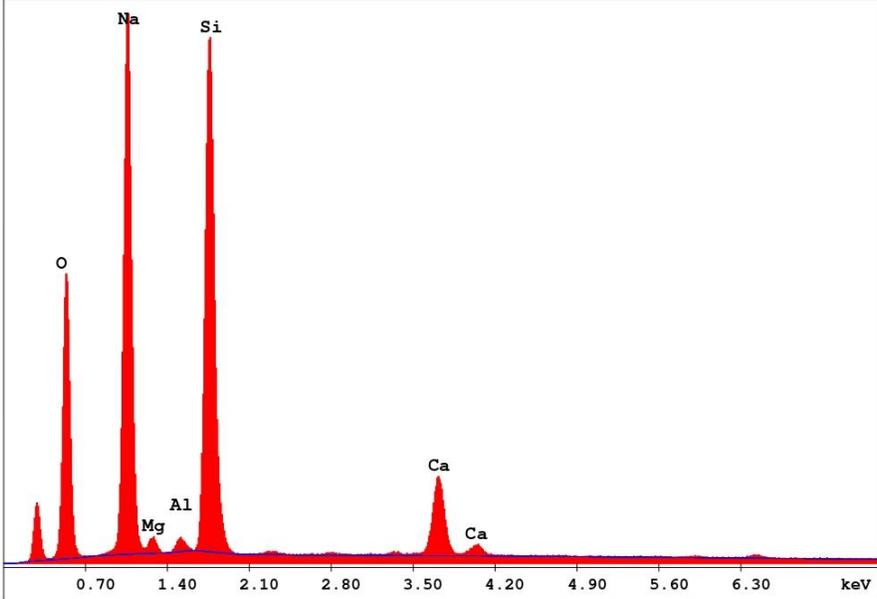
EDAX ZAF Quantification (Standardless)  
 Element Normalized  
 SEC Table : Default

Element	Wt %	At %	K-Ratio	Z	A	F
O K	29.89	40.97	0.0958	1.0399	0.3077	1.0011
NaK	33.32	31.78	0.1795	0.9732	0.5518	1.0030
MgK	1.19	1.08	0.0050	0.9976	0.4198	1.0049
AlK	0.93	0.76	0.0050	0.9683	0.5518	1.0091
SiK	27.58	21.53	0.1850	0.9965	0.6728	1.0008
CaK	7.09	3.88	0.0638	0.9676	0.9312	1.0000
Total	100.00	100.00				

c:\edax32\genesis\genspc.spc

Element	Net Inte.	Bkqd Inte.	Inte. Error	P/B
O K	1284.81	29.29	0.37	43.86
NaK	3166.37	64.51	0.23	49.08
MgK	88.05	68.24	2.20	1.29
AlK	85.34	76.92	2.34	1.11
SiK	2823.61	66.79	0.25	42.27
CaK	605.65	74.17	0.59	8.17

c:\edax32\genesis\genspc.spc  
 Label:  
 kV:20.0 Tilt:0.0 Take-off:43.4 Det: SDD Apollo X  
 Res:134 Amp.T:12.80 FS:27393 Lsec:60 25-Feb-2022 14:18:05



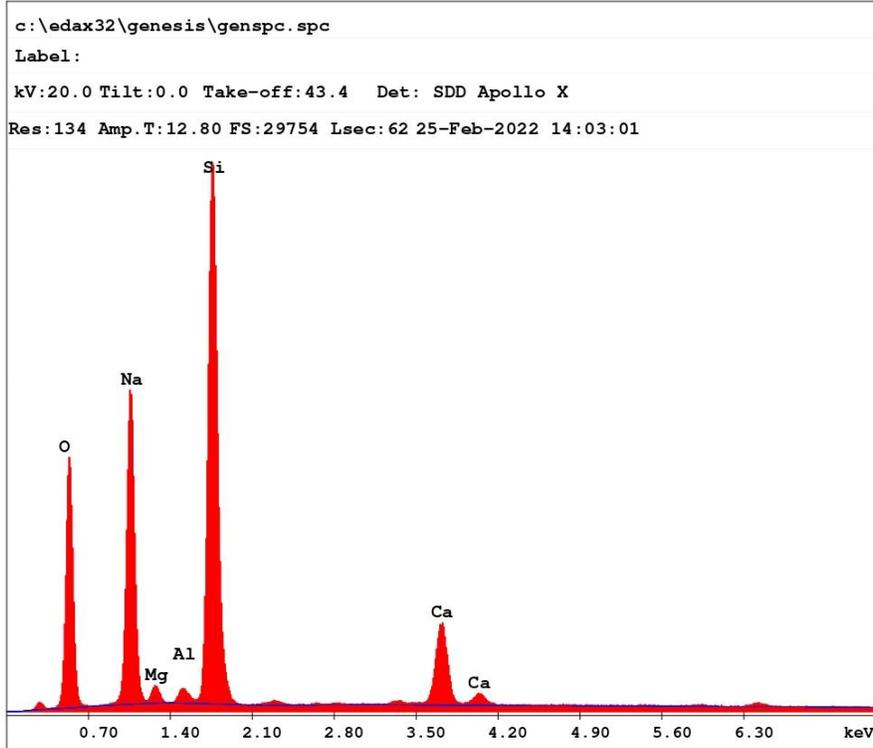
**EDAX ZAF Quantification (Standardless)**  
 Element Normalized  
 SEC Table : Default

Element	Wt %	At %	K-Ratio	Z	A	F
O K	31.03	42.40	0.0992	1.0388	0.3073	1.0010
NaK	31.24	29.70	0.1656	0.9722	0.5437	1.0031
MgK	1.20	1.08	0.0051	0.9966	0.4286	1.0052
AlK	0.82	0.67	0.0045	0.9673	0.5611	1.0096
SiK	28.66	22.31	0.1948	0.9955	0.6822	1.0008
CaK	7.05	3.85	0.0634	0.9666	0.9309	1.0000
Total	100.00	100.00				

c:\edax32\genesis\genspc.spc

Element	Net Inte.	Bkgd Inte.	Inte. Error	P/B
O K	1276.09	28.60	0.37	44.61
NaK	2802.95	65.43	0.25	42.84
MgK	86.30	72.35	2.26	1.19
AlK	73.51	94.05	2.82	0.78
SiK	2851.07	85.66	0.25	33.28
CaK	577.33	74.66	0.60	7.73

### D.3 Sandwich 2.0 450°C EDS Reports

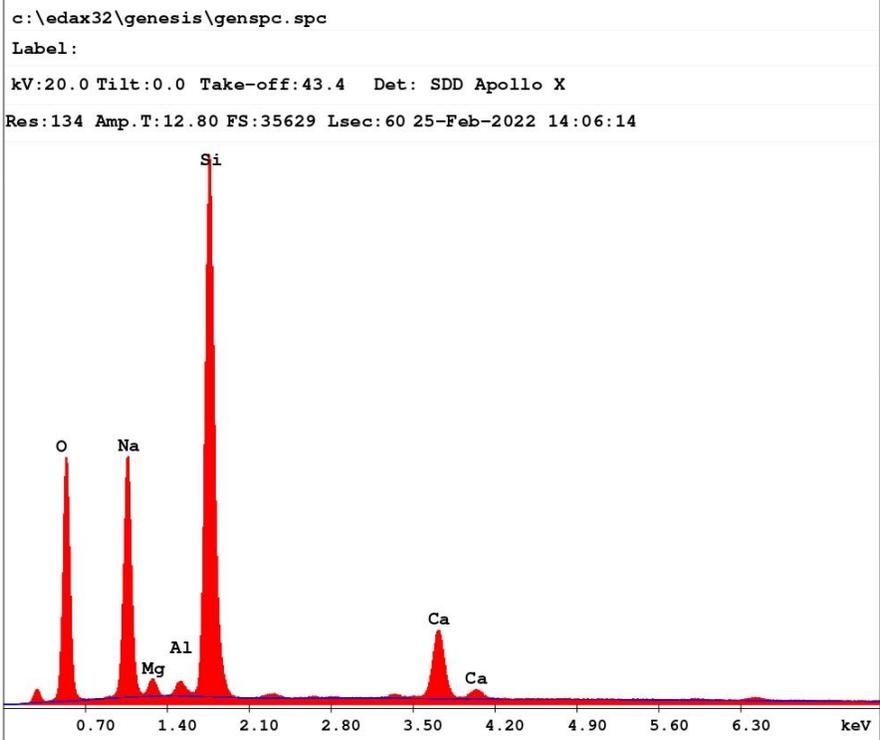


EDAX ZAF Quantification (Standardless)  
 Element Normalized  
 SEC Table : Default

Element	Wt %	At %	K-Ratio	Z	A	F
O K	33.67	46.16	0.0999	1.0359	0.2861	1.0008
NaK	22.25	21.23	0.1111	0.9694	0.5132	1.0038
MgK	1.35	1.22	0.0064	0.9938	0.4715	1.0066
AlK	1.04	0.85	0.0062	0.9645	0.6036	1.0121
SiK	33.08	25.83	0.2362	0.9927	0.7187	1.0010
CaK	8.61	4.71	0.0771	0.9639	0.9290	1.0000
Total	100.00	100.00				

c:\vedax32\genesis\genspc.spc

Element	Net Inte.	Bkqd Inte.	Inte. Error	P/B
O K	1173.44	22.82	0.38	51.43
NaK	1716.64	55.20	0.31	31.10
MgK	97.73	63.24	1.93	1.55
AlK	91.55	72.23	2.12	1.27
SiK	3156.62	59.27	0.23	53.26
CaK	640.86	65.40	0.55	9.80



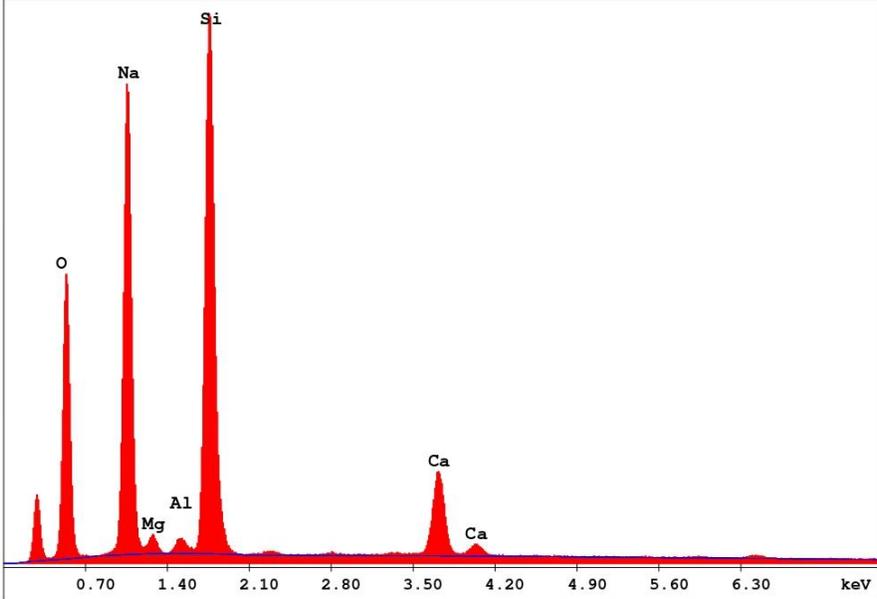
EDAX ZAF Quantification (Standardless)  
 Element Normalized  
 SEC Table : Default

Element	Wt %	At %	K-Ratio	Z	A	F
O K	35.56	48.40	0.1070	1.0340	0.2909	1.0008
NaK	18.98	17.98	0.0931	0.9677	0.5049	1.0041
MgK	1.41	1.26	0.0069	0.9920	0.4899	1.0072
AlK	1.08	0.87	0.0065	0.9629	0.6213	1.0133
SiK	35.10	27.22	0.2555	0.9909	0.7340	1.0009
CaK	7.88	4.28	0.0703	0.9622	0.9277	1.0000
Total	100.00	100.00				

c:\edax32\genesis\genspc.spc

Element	Net Inte.	Bkqd Inte.	Inte. Error	P/B
O K	1439.57	26.09	0.34	55.18
NaK	1647.17	64.93	0.33	25.37
MgK	120.74	76.03	1.76	1.59
AlK	111.49	89.40	1.96	1.25
SiK	3909.41	72.40	0.21	54.00
CaK	668.63	71.94	0.55	9.29

c:\edax32\genesis\genspc.spc  
 Label:  
 kV:20.0 Tilt:0.0 Take-off:43.3 Det: SDD Apollo X  
 Res:134 Amp.T:12.80 FS:28245 Lsec:60 25-Feb-2022 14:10:23

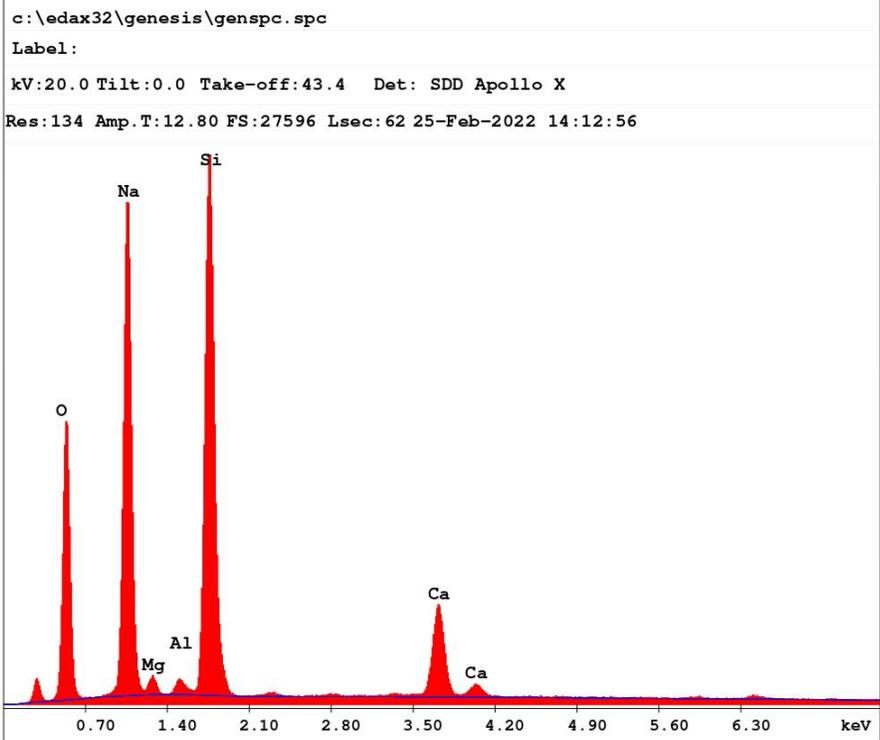


**EDAX ZAF Quantification (Standardless)**  
 Element Normalized  
 SEC Table : Default

Element	Wt %	At %	K-Ratio	Z	A	F
O K	31.98	43.73	0.0994	1.0378	0.2994	1.0009
NaK	28.13	26.77	0.1459	0.9713	0.5322	1.0033
MgK	1.38	1.24	0.0061	0.9956	0.4424	1.0056
AlK	0.99	0.80	0.0055	0.9664	0.5739	1.0103
SiK	29.86	23.26	0.2059	0.9945	0.6926	1.0009
CaK	7.67	4.19	0.0689	0.9657	0.9305	1.0000
Total	100.00	100.00				

c:\edax32\genesis\genspc.spc

Element	Net Inte.	Bkqd Inte.	Inte. Error	P/B
O K	1323.33	28.60	0.36	46.27
NaK	2553.45	65.65	0.26	38.89
MgK	106.19	72.94	1.93	1.46
AlK	93.37	84.34	2.23	1.11
SiK	3116.81	72.45	0.24	43.02
CaK	648.93	75.22	0.56	8.63



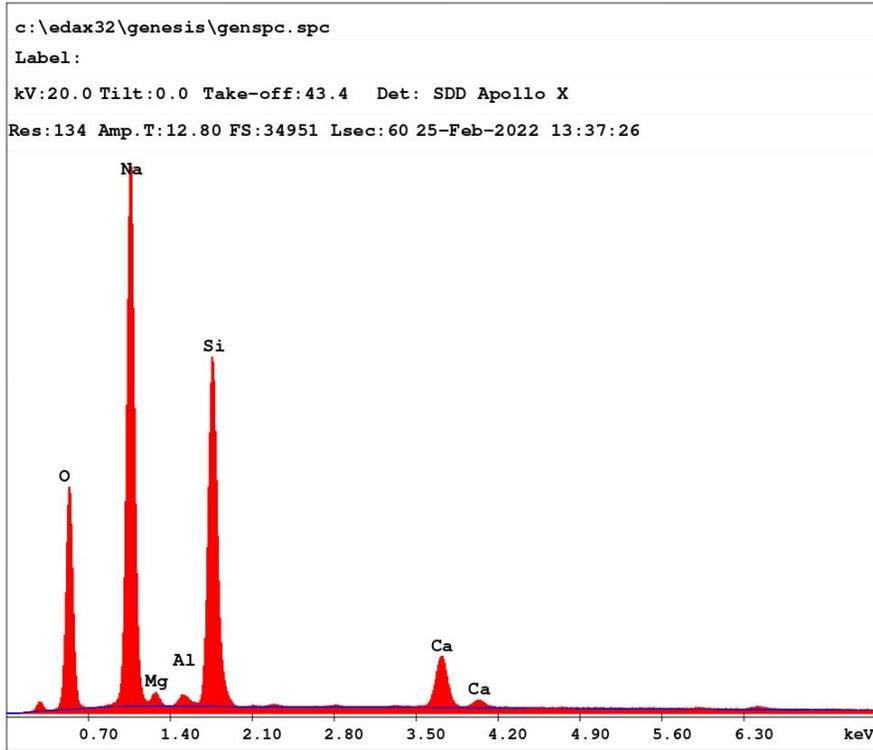
EDAX ZAF Quantification (Standardless)  
 Element Normalized  
 SEC Table : Default

Element	Wt %	At %	K-Ratio	Z	A	F
O K	31.32	43.00	0.0962	1.0384	0.2954	1.0010
NaK	28.89	27.60	0.1504	0.9718	0.5340	1.0033
MgK	1.29	1.16	0.0057	0.9962	0.4386	1.0055
AlK	0.96	0.78	0.0053	0.9669	0.5708	1.0101
SiK	29.43	23.01	0.2023	0.9951	0.6900	1.0009
CaK	8.11	4.45	0.0730	0.9662	0.9309	1.0000
Total	100.00	100.00				

c:\edax32\genesis\genspc.spc

Element	Net Inte.	Bkqd Inte.	Inte. Error	P/B
O K	1233.83	25.93	0.37	47.59
NaK	2537.51	61.53	0.26	41.24
MgK	94.84	68.74	2.04	1.38
AlK	86.87	77.88	2.27	1.12
SiK	2951.07	64.58	0.24	45.69
CaK	661.84	70.09	0.54	9.44

## D.4 Sandwich 2.0 500°C EDS Reports



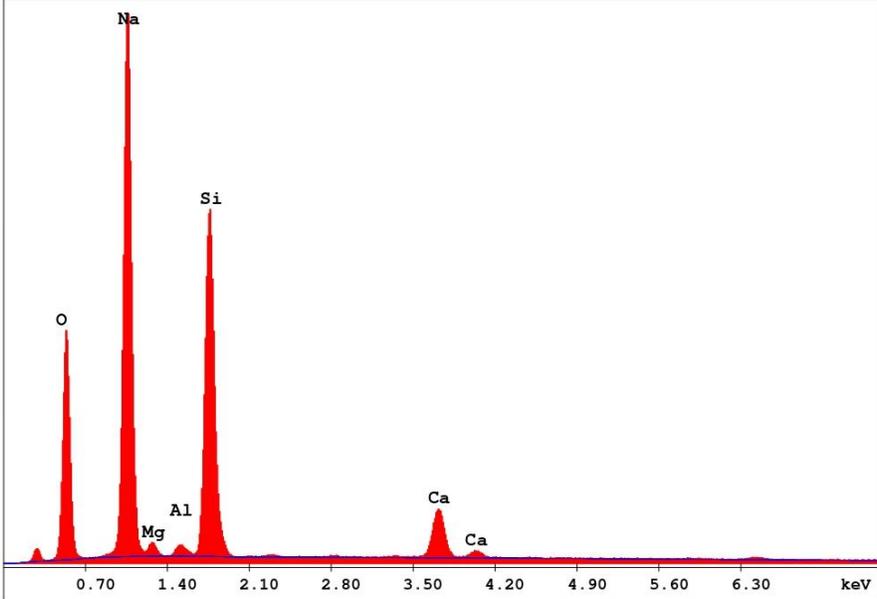
EDAX ZAF Quantification (Standardless)  
 Element Normalized  
 SEC Table : Default

Element	Wt %	At %	K-Ratio	Z	A	F
O K	28.78	39.29	0.0974	1.0412	0.3247	1.0012
NaK	37.76	35.87	0.2104	0.9744	0.5703	1.0027
MgK	1.33	1.19	0.0054	0.9989	0.4020	1.0044
AlK	0.96	0.77	0.0050	0.9695	0.5320	1.0080
SiK	25.31	19.68	0.1654	0.9978	0.6542	1.0007
CaK	5.86	3.19	0.0529	0.9687	0.9319	1.0000
Total	100.00	100.00				

c:\edax32\genesis\genspc.spc

Element	Net Inte.	Bkqd Inte.	Inte. Error	P/B
O K	1288.53	29.95	0.37	43.02
NaK	3659.06	64.39	0.22	56.82
MgK	92.53	66.59	2.09	1.39
AlK	83.38	73.86	2.35	1.13
SiK	2487.17	64.00	0.27	38.86
CaK	494.41	71.43	0.66	6.92

c:\edax32\genesis\genspc.spc  
 Label:  
 kV:20.0 Tilt:0.0 Take-off:43.4 Det: SDD Apollo X  
 Res:134 Amp.T:12.80 FS:35721 Lsec:61 25-Feb-2022 13:41:34



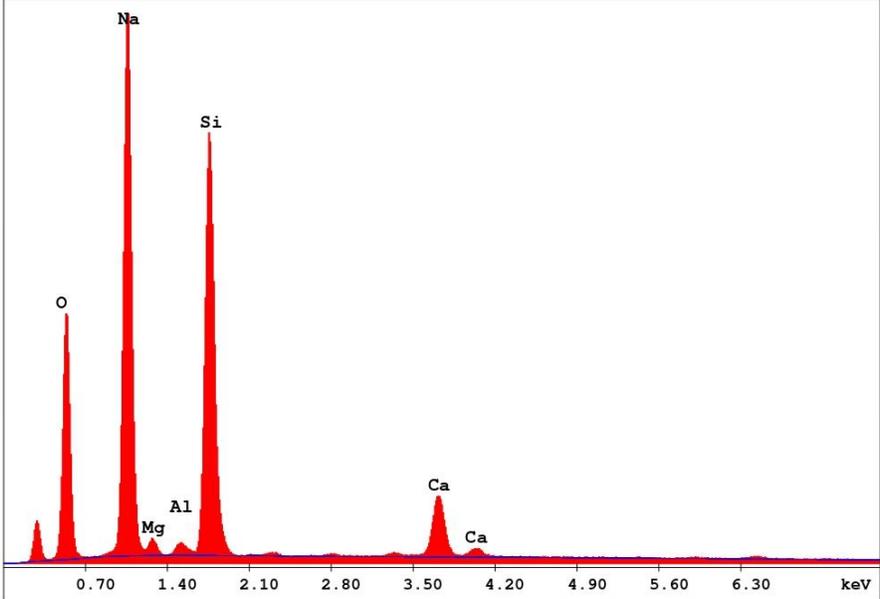
**EDAX ZAF Quantification (Standardless)**  
 Element Normalized  
 SEC Table : Default

Element	Wt %	At %	K-Ratio	Z	A	F
O K	28.90	39.39	0.0988	1.0411	0.3279	1.0012
NaK	37.81	35.86	0.2111	0.9743	0.5714	1.0027
MgK	1.40	1.26	0.0057	0.9988	0.4021	1.0043
AlK	0.97	0.79	0.0050	0.9694	0.5316	1.0079
SiK	25.33	19.66	0.1653	0.9977	0.6537	1.0006
CaK	5.59	3.04	0.0504	0.9686	0.9317	1.0000
Total	100.00	100.00				

c:\edax32\genesis\genspc.spc

Element	Net Inte.	Bkqd Inte.	Inte. Error	P/B
O K	1313.31	28.74	0.36	45.69
NaK	3690.60	62.99	0.21	58.59
MgK	98.22	67.18	1.99	1.46
AlK	85.04	75.87	2.32	1.12
SiK	2499.58	64.77	0.26	38.59
CaK	473.98	70.70	0.67	6.70

c:\edax32\genesis\genspc.spc  
 Label:  
 kV:20.0 Tilt:0.0 Take-off:43.4 Det: SDD Apollo X  
 Res:134 Amp.T:12.80 FS:31763 Lsec:59 25-Feb-2022 14:27:15



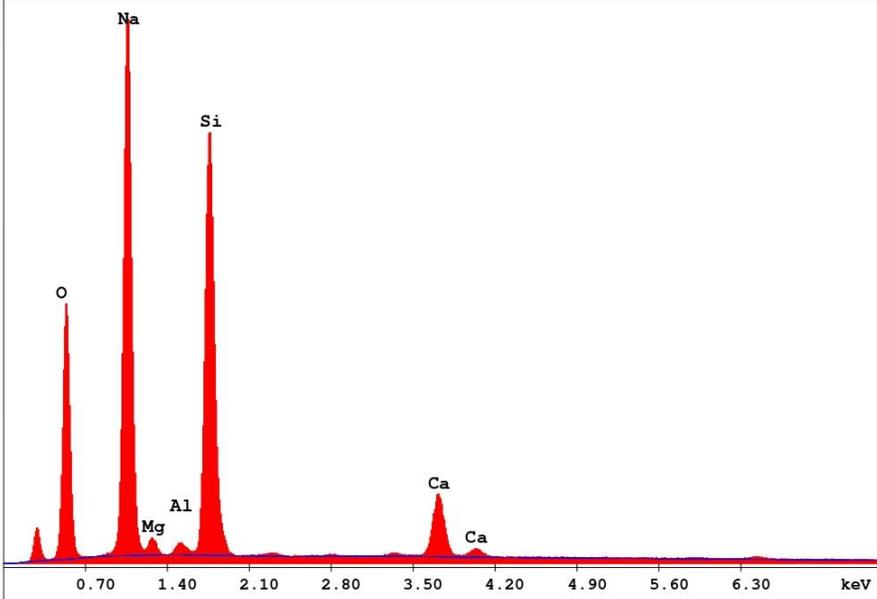
**EDAX ZAF Quantification (Standardless)**  
 Element Normalized  
 SEC Table : Default

Element	Wt %	At %	K-Ratio	Z	A	F
O K	29.60	40.46	0.0976	1.0402	0.3167	1.0011
NaK	34.82	33.12	0.1902	0.9735	0.5595	1.0029
MgK	1.43	1.29	0.0060	0.9979	0.4143	1.0047
AlK	0.93	0.76	0.0050	0.9686	0.5443	1.0087
SiK	26.88	20.93	0.1785	0.9968	0.6658	1.0007
CaK	6.33	3.45	0.0571	0.9678	0.9311	1.0000
Total	100.00	100.00				

c:\edax32\genesis\genspc.spc

Element	Net Inte.	Bkqd Inte.	Inte. Error	P/B
O K	1302.72	27.92	0.37	46.66
NaK	3337.84	62.69	0.23	53.24
MgK	103.65	68.14	1.93	1.52
AlK	84.01	78.11	2.38	1.08
SiK	2709.73	67.78	0.25	39.98
CaK	538.37	73.86	0.63	7.29

c:\edax32\genesis\genspc.spc  
 Label:  
 kV:20.0 Tilt:0.0 Take-off:43.4 Det: SDD Apollo X  
 Res:134 Amp.T:12.80 FS:32636 Lsec:58 25-Feb-2022 14:29:43



**EDAX ZAF Quantification (Standardless)**  
 Element Normalized  
 SEC Table : Default

Element	Wt %	At %	K-Ratio	Z	A	F
O K	29.88	40.79	0.0988	1.0399	0.3175	1.0011
NaK	34.39	32.67	0.1873	0.9732	0.5581	1.0030
MgK	1.47	1.32	0.0061	0.9977	0.4161	1.0048
AlK	0.92	0.75	0.0049	0.9684	0.5460	1.0088
SiK	27.10	21.07	0.1804	0.9966	0.6675	1.0007
CaK	6.24	3.40	0.0563	0.9676	0.9310	1.0000
Total	100.00	100.00				

c:\edax32\genesis\genspc.spc

Element	Net Inte.	Bkqd Inte.	Inte. Error	P/B
O K	1387.05	31.70	0.36	43.76
NaK	3459.94	69.58	0.23	49.72
MgK	112.52	74.26	1.87	1.52
AlK	87.38	84.19	2.38	1.04
SiK	2880.97	73.07	0.25	39.43
CaK	558.64	78.55	0.62	7.11