

MITIGATION OF CORROSION UNDER INSULATION (CUI) OF CARBON STEEL OF DIFFERENT INSULATING MATERIALS

A Literature Review and Testing Analysis

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Abstract

Equipment often found in refineries may be enveloped in insulation and weathering jackets to maintain internal processing temperatures. In many cases moisture from the environment will penetrate the weathering jacket and infiltrate the insulation, leaching corrosive ions to the surface of metal equipment, effectively creating a corrosion cell. The goal of this project is to investigate different insulating materials for their ability to inhibit corrosion under insulation (CUI). The inhibiting mechanism utilizes water ingress that leaches ions from the insulator to bond with the surface metal and create a passive layer, inhibiting any further corrosion. This study will follow ASTM C1617-15 in order to examine the corrosion mitigating effects of three insulators. Insulators are ground down and boiled to extract leachable ions. The leachant is filtered to remove solids and then introduced to heated carbon steel coupons in a drip wise fashion over a span of four days. Insulators are ranked quantitatively on their ability to prevent corrosion based on gravimetric analysis. The mass loss corrosion rate of the insulators were: Sproule WR-1200® (4.595 mils/yr), Thermo-12® Gold (5.523 mils/yr), and Pyrogel® XT-E (24.767 mils/yr). Sproule and Thermo-12 both exhibit corrosion inhibiting ions whereas Pyrogel XT-E does not, and these results conclude the effectiveness of these insulators to mitigate corrosion.

Key terms: corrosion under insulation (CUI), insulator, weatherproof jacketing, passive layer, hydrophobic, calcium silicate, expanded perlite, aerogel, corrosion mitigating ions, mass loss corrosion rate (MLCR).

Introduction

Corrosion:

Corrosion is the deterioration of a material and its properties due to a physical, chemical or electrochemical process implemented by that material's environment. Materials subjected to corrosion in engineering applications are commonly metals. Examples of physical corrosion include erosion corrosion and fretting corrosion. Chemical corrosion is simply one material reacting with its environment, such as oxidation or rusting of iron. Electrochemical corrosion implies there is an electric pathway aiding the corrosion mechanism. The electrochemical process requires an anode, a cathode, an electronic path, and an ionic current path to complete a corrosion cell.¹ Figure 1 illustrates the relationship of these four requirements.

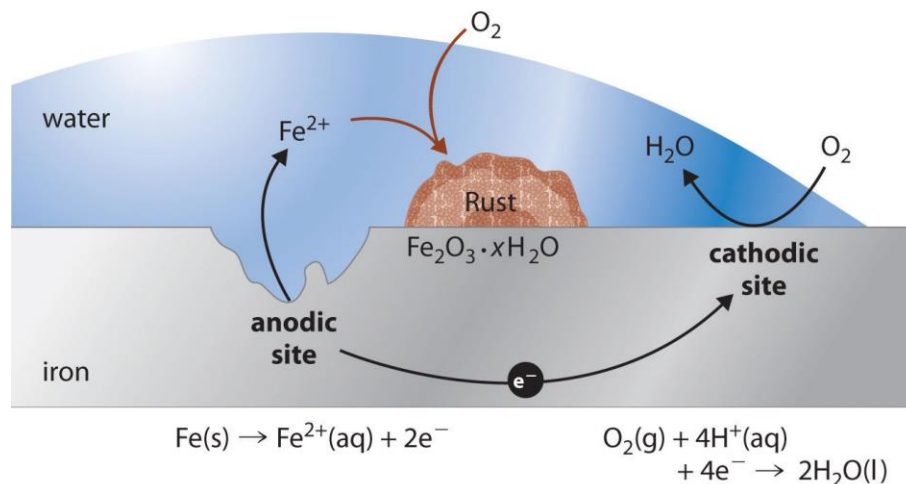


Figure 1. Schematic of the relationship between the four requirements of a corrosion cell.²

Corrosion is dictated by two half-cells that make up a redox reaction within the system. Net oxidation is occurring at the anode, whereas net reduction occurs at the cathode. In the case where a metal is corroding locally in its own system as shown in Figure 1, anodic and cathodic regions are formed on the surface which produce the electric potential driving force for corrosion to occur. Oxidation of the anode takes place on the

metal with a lower electrode potential, and will lose electrons to the cathode yielding a higher electrode potential. The ionic current pathway is generally a salt bridge induced by moisture where ions are able to move freely.¹ An electronic pathway is any conductive or metallic connection between the anode and cathode that allows electron transfer.¹

Corrosion is an ever-present problem that is intensely considered in many industries, including the oil and gas processing industry.

Corrosion Under Insulation (CUI)

In the oil and gas processing industry, insulators are commonly used to cover certain equipment in order to maintain a necessary operating temperature. Other reasons equipment may be insulated include; fire protection, personnel protection, condensation control, sound control, and process control.³ Any equipment under insulation has potential to undergo corrosion under insulation (CUI). CUI is the external damage of metallic components and equipment that are insulated.⁴ This process is caused by water ingress through the insulation material.

The most commonly affected materials are carbon steels, low-alloy steels, and austenitic stainless steels.⁴ The general mechanism associated with CUI is localized corrosion at the interface of the insulation material and metal surface.⁴ Pitting is also observed in features of equipment that promote stagnant and highly acidic environments.⁴ Chloride stress corrosion cracking (SCC) is common for austenitic and duplex stainless steels, but for the sake of this report will not be concerned.

As mentioned above, localized corrosion at the interface of the insulation material and a metallic surface is a common mechanism behind CUI. Localized corrosion is initiated by

the oxidation of a metal and can produce anodic and cathodic regions on the metal's surface that will pass electrons and ions induced by electric potentials between the two regions.¹ This implies an electrochemical process. Even though an insulated system is not as exposed to the atmosphere as greatly as other components, mediums like fog, humidity, and moisture from air ducts and vents lead to necessary water ingress in an insulated system for CUI to occur.

Pitting corrosion may be initiated by the damaging of a protective oxide film or passive coating as a result of localized chemical corrosion or mechanical damage.⁵ Here, a stagnant solution with a high concentration of ions and high acidity may collect and stimulate metal loss. Pitting may also be initiated by local abnormal anodic regions in a normal surface forcing the normal surface to act as a cathode, or by local abnormal cathodic regions surrounded by otherwise normal surface metal forcing the normal surface to act as an anode.⁵

Factors Effecting CUI:

Water, chemical content of water, the type of insulator, and service temperature are four major factors that promote CUI.⁶ Water is necessary at the interface between the insulation and the surface of the equipment to initialize corrosion. Instances where water infiltration occurs include:

- (a) During insulation storage and/or installation.
- (b) Through system leakages.
- (c) Vapor/condensation due to temperature differentials.
- (d) Insulation absorbing moisture from the environment/atmosphere.

During storage and installation of an insulator it is important to keep components dry to avoid any initial presence of water. Weathering jackets are commonly used as an external layer to the insulator to prevent moisture absorption from atmospheric or environmental factors.⁶ However, there are still many opportunities for water to infiltrate insulators and reach the surface metal. The equipment and insulation design plays a role in water infiltration.

Insulated equipment may have joint attachments such as small-bore connections that bring about complications in insulator design. At such connections an insulator might be discontinuous due to the shape, geometry, and orientation of the attachments.³ Having a discontinuous insulator will allow for moisture or rainwater to leak down to the surface metal, where the water will concentrate and induce corrosion. Flanges and end caps are examples on pipelines or equipment that provide an easy access for water.⁴

A solution to avoiding breaks and gaps within insulators or weathering jackets is caulking the gap shut in order to properly seal any entry point, as illustrated in Figure 2. Caulk will deteriorate over time and inspection of sealants should be implemented throughout service. Sealants should be reapplied where and when necessary. Other maintenance inspections of in-service insulators are important in order to discover any unexpected penetrations on the weathering jackets or on the outer insulation surface that will lead to water ingress. Inspection intervals and procedures are adhered to API 570.⁴

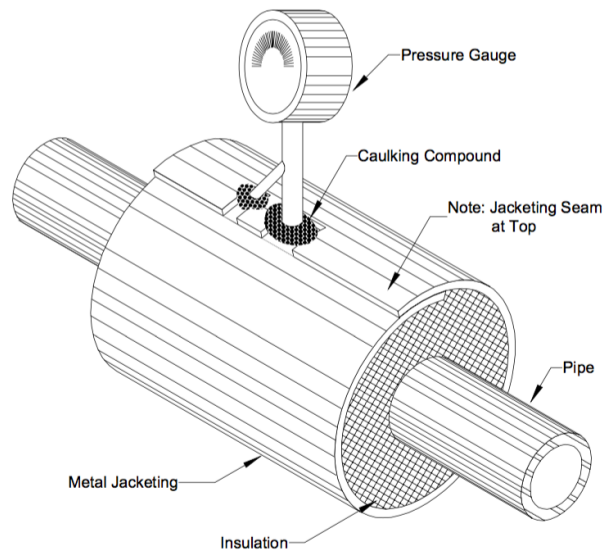


Figure 2. Caulking is used to effectively seal water infiltration sites.³

Frequently these attachments that protrude the insulation are operating in a temperature range that promotes CUI, whereas the main process equipment is not.⁴ In this case the equipment is likely to be exposed to condensation due to temperature differentials. Condensation will also occur on equipment operating below the dew point.⁴ The resulting moisture will promote corrosion mechanisms to initiate. This is common for equipment in cold service, which is also known as sweating service.⁴

Insulation Systems:

An important step to preventing CUI is choosing the proper insulator for the scenario. Cost along with functionality is considered. One of three common types of insulation that API RP 583 identifies is granular insulation.⁴ The three basic forms of granular fill include perlite, vermiculite, and polystyrene beads. Granular insulation is also known as granular fill insulation as it has many large grains of material that fill a space in need of insulation. Since the insulator fill is discontinuous it does not fair well in impeding moisture from absorbing and wicking to the surface of insulated equipment.

Manufacturers have implemented waterproofing agents, such as silicone to increase the moisture resistance of some granular fill insulators.⁷

Fibrous insulation is another common type of insulation identified by API RP 583 that is used in industrial and domestic applications. The material is composed of a matrix of fibers that limit convection as a form of heat transmission due to the trapping of air molecules within the fibers.⁸ This ultimately inhibits motion of molecules, decreasing collisions between the particles.⁸ Fibrous insulators have also been used in soundproofing applications as the porous structure dampens any sound waves that may pass through.⁸ Some examples of fibrous insulation include fiberglass (aerogels), mineral wool, and cellulose.⁸ It is common in industrial applications to use a combination of granular and fibrous insulators in one, to achieve a composite insulation that yields desired properties.

The last insulation material API RP 583 identifies is cellular.⁴ Cellular insulators are porous and absorbent in structure. A common material classified as a cellular insulator is calcium silicate. Calcium silicate works similarly to granular insulators and fibrous insulators in that they trap air molecules between their structures, effectively insulating a system.

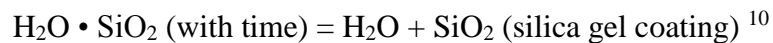
Hydrophobic Insulators:

Insulators are commonly termed “hydrophobic” when treated during manufacturing with organic hydrophobic additives. Often these “hydrophobic” insulators are materials that will readily absorb water if not for the organic hydrophobic additives.¹⁰ At certain operating temperatures organic hydrophobic additives will oxidize and burn off, rendering the “hydrophobic” insulator to readily absorb water.¹⁰ Temperatures greater

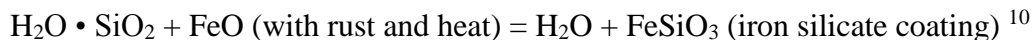
than 450°F tend to cause these organic hydrophobic additives to oxidize, which are common temperatures that insulators are exposed to.¹¹ These are important considerations when determining an appropriate insulator.

Chemistry of Insulators:

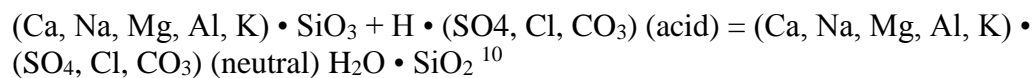
Water ingress is not only the factor that completes a corrosion cell for CUI to occur, but it will also leach ions that may be corrosive from insulators to the surface of the metals. Commonly mineral wools are composed of chlorides or sulfides that promote an acidic corrosion cell. A corrosion inhibiting chemistry has been incorporated in two industrial insulations to effectively mitigate CUI.¹⁰ These insulators depend on water ingress to dissolve different silicate anions within the insulators chemistry and bring the silicates to the surface of the metal.¹⁰ These silicate anions form a “silica gel” coating as shown below:



As the silica gel is introduced to heat and iron compounds it forms an inorganic silicate coating that further prevents corrosion as shown below:



The silicates also act as a pH buffer to the absorbed water due to the alkaline components to neutralize any acidic components. This can be shown below:



Incorporating corrosion mitigating ions in the chemistry of insulators is another way to defend CUI along with treating insulators with hydrophobic additives and the use of weatherproof jacketing. The silicate anions make up 95% of the two industrial insulators

that utilize this mechanism, and are claimed to remain present for the majority of the insulators lifetime. ¹¹

Operating Conditions:

When considering carbon steels for piping or equipment, operating with surface temperatures in the temperature range of -4°C (25°F) to 175°C (350°F) promotes a greater likeliness of CUI occurring.³ At temperatures below -4°C carbon steel equipment that is under insulation commonly remains standing without falling victim to CUI.³

Operating temperatures of carbon steel equipment above 175°C tend to exhibit surfaces warm enough to burn away moisture that may initiate corrosion.³ It is important to note that higher temperatures can be beneficial as they stay warm enough to evaporate moisture, yet can be detrimental since the corrosion rate of a material is generally increased as service temperatures rise.

When considering an open system, aerated water has a decrease in oxygen content as temperature increases. At temperatures above 80°C, the corrosion rate of carbon steels exposed to aerated water decreases as temperature rises. However in a closed system, when carbon steel is subject to water it is shown that an increase in the water temperature results in an increase in corrosion rate.³ This is due to the oxygen-saturated environment that the closed system exhibits. It is found that field measurements of carbon steels subjected to CUI are closely related to carbon steels corroding in a closed system, and can be shown in Figure 3.³ An increase in temperature correlated to a greater corrosion rate for the carbons steels under insulation.³ The insulator surrounding the carbon steels acts as a barrier, trapping a surface layer of aerated, oxygen-saturated water. Figure 3 compares the relationship between water temperature and corrosion rate of an open

system, a closed system, and various field measurements of carbon steel CUI rates at a chemical plant.³

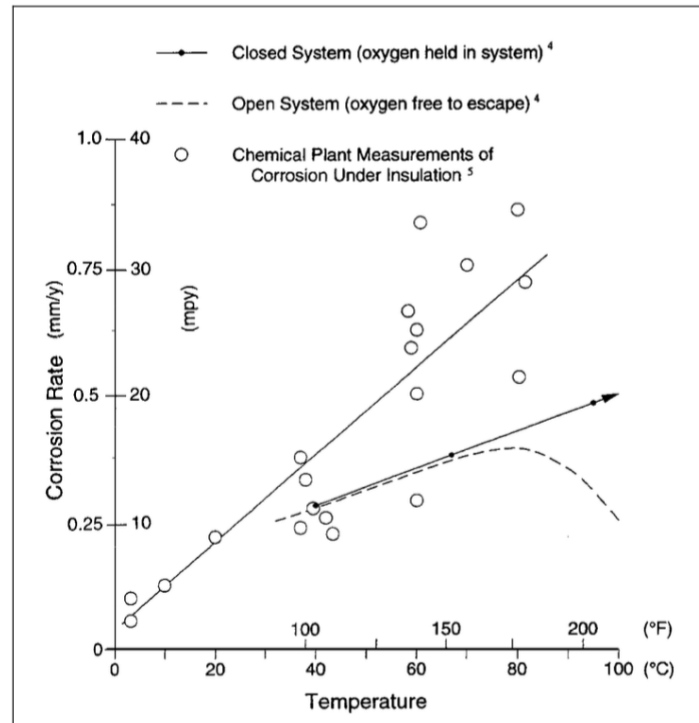
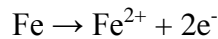


Figure 3. Effect of temperature on steel corrosion in water.³

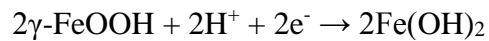
Generally an increase in service temperature will increase the corrosion rate of carbon steels. It is also detrimental for the operating temperature to undergo cyclic service, or intermittent periods of hot and cold service. Carbon steels operating under -4°C are considered in a safe operating temperature region in the presence of water, yet due to operating services steels may heat up and cool off again. During these hot periods water on the surface may dry up to the atmosphere or insulation, and these wet-dry cycles will promote atmospheric corrosion.

Stratmann's Wet-Dry Cycle Model:

The wet-dry cyclic mechanism of corrosion of plain carbon steel is illustrated by Stratmann's model as proposed in 1987.⁹ The initial wetting of the steel surface is the first stage, and causes the anodic dissolution of iron as shown below:



The iron is yielding a high dissolution rate, and in comparison the reduction reaction of O_2 is slow. Due to this, the anodic dissolution of iron is balanced by the cathodic reduction of the iron oxide-hydroxide surface layer:

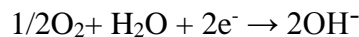


The formation of the cathodic iron oxide-hydroxide $\gamma\text{-FeOOH}$, also known as lepidocrocite, is shown as follows:



This initiates a corrosion cell. Although the dissolution rate of iron is high, the amount of reducible $\gamma\text{-FeOOH}$ in the rust layer is limiting the amount of iron able to be dissolved.⁹

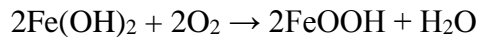
The second stage of Stratmann's model regards the wet surface of the metal, and commences once the cathodic reduction of $\gamma\text{-FeOOH}$ is used up.⁹ At this point the reduction reaction of O_2 takes over as the cathodic reaction:



The dissolution of iron is now limited by the reduction reaction of O_2 . Stratmann and Müller found that oxygen is reduced within the oxide scale (rust layer) and not at the metal/electrolyte interface.⁹ The O_2 must diffuse through the electrolyte saturated pores in the oxide rust layer, therefore the current density of the O_2 reduction reaction is diffusion limited.⁹ This implies that the corrosion rate and reduction of O_2 is influenced by the

electronic/conductive properties of the oxides. Good conductive properties lead to more reduction of O₂ in oxides, causing further dissolution of iron, which in turn leads to an increase in corrosion rate.

Drying out of the wet surface as the third stage of Stratmann's model causes a sharp increase of the diffusion-limited cathodic O₂ reduction reaction, which is connected to the electrolyte film on the inner surface of the rust layer decreasing in thickness and saturation.⁹ As the cathodic O₂ reduction reaction is increased, both the dissolution of iron and the corrosion rate are increased. The O₂ is also able to reoxidize the reduced layer of γ -FeOOH formed in stage 1 as follows, restarting the cycle:



These factors leading to increased corrosion rate in the drying mechanism of stage 3 causes the most metal loss throughout the wet-dry cycle. The regeneration of lepidocrocite (γ -FeOOH) and production of goethite (α -FeOOH) are the main corrosion products and are a result of reoxidizing the γ -FeOOH and other ferrous species. This alters the composition of the rust layer and directly affects the intensity of the next wet-dry cycle. The corrosion process is fully stopped once the electrolyte film is completely used up, as shown in Figure 4.⁹

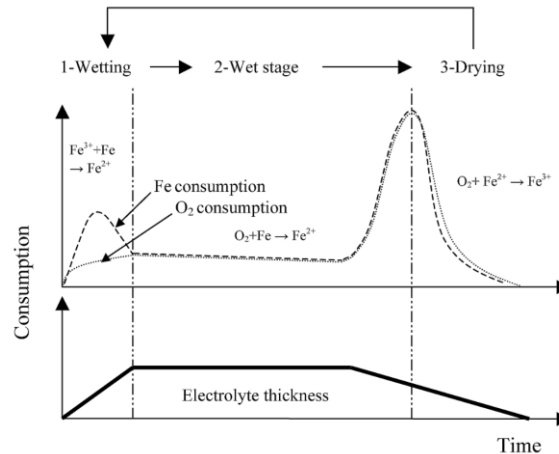


Figure 4. Schematic of the atmospheric corrosion wet-dry cycle.⁹

Overview:

Corrosion under insulation is a critical problem that is present in the oil and gas processing industry. Considering factors that contribute to CUI and an understanding of how the mechanisms occur are the first steps to preventing the mechanism. Avoiding water infiltration eliminates the potential for CUI, however this can be a challenging solution. Design of the insulator along with proper sealing between the insulator and weathering jacket interface are steps taken to ensure no water ingress may occur. The goal in choosing an insulator for servicing equipment is to consider one that will hinder water infiltration, leaching of ions from the insulator, and efficiently and successfully serve its purpose in insulating service equipment. The goal of this project is to investigate different insulating materials for their ability to inhibit corrosion under insulation (CUI). The inhibiting mechanism under study utilizes water ingress that leaches ions from the insulator to bond with the surface metal and create a passive layer, inhibiting any further corrosion.

Methods and Materials

ASTM C1617-15:

In order to test industrial insulators for their ability to mitigate CUI, an experiment is ran in accordance to the standard ASTM C1617-15. This standard is titled “Standard Practice for Quantitative Accelerated Laboratory Evaluation of Extraction Solutions Containing Ions Leached from Thermal Insulation on Aqueous Corrosion of Metals.”¹² ASTM C1617-15 is comprised of two procedures, Procedure A and Procedure B.

Procedure A:

Procedure A is specified for insulator materials that are not classified as hydrophobic. Insulators are ground down to a dust and mixed with de-ionized (DI) water and boiled to extract any leachable ions that may be produced in a CUI mechanism.¹² Once the solution is produced it is delivered in a drip wise fashion over four days to the surface of three carbon steel coupons of .13wt% C. Three coupons is the minimum sample size for accurate results. The coupons are heated to 250°F on a hot plate for the entirety of testing to evaporate water producing a wet-dry cycle, to provide an accelerated corrosion test for significant results, and to also mimic temperature levels that CUI is likely to occur in a real-life scenario. Samples are weighed for their initial and final weight in order to calculate the mass loss corrosion rate (MLCR) of each solution. Reference standards are ran alongside the insulator solutions in order to compare the corrosiveness of different solutions. The reference standards are DI water, 1 ppm chloride solution, and 5 ppm chloride solution.¹² A blank test cell is produced to observe any mass loss due to the set up and cleaning of a test cell without being exposed to any corrosive solution.

Procedure B:

Procedure B is specified for insulator materials that are classified as hydrophobic.¹²

Insulators are cut into cross-sectional pieces and layered to produce a stack of the cross-sectional insulator layers. This stack is then placed in the bottom of a DI water bath and heated to a specific temperature recommended by the manufacturer.¹² The resulting solution represents any leachable ions that may be found in a CUI mechanism. This solution is then introduced to heated carbon steel coupons in a drip-wise fashion over a span of four days in the same manner as Procedure A. The reference standards are also ran alongside insulator solutions in Procedure B. Because of the previously stated observation that hydrophobic insulators will become non-hydrophobic throughout their lifetime, Procedure A is the only testing conducted for this experiment.

Apparatus:

A Ninja® blender was used to properly grind down insulators. The carbon steel coupons are heated on a Wolfgang Puck® hot plate that is monitored by a thermocouple to ensure consistent temperature levels. To deliver the solutions to the carbon steel coupons a 24-Channel Ismatec IPC® peristaltic pump is used. The full set up of the testing apparatus is shown in Figure 5.



Figure 5. Testing apparatus of ASTM C1617-15.

Each test cell is composed of a PVC tube sealed to a carbon steel coupon. An O-ring is put in place with a silicone sealant in order produce a sealed and defined area to analyze corrosion. Tubing from the peristaltic pump is placed in a small hole drilled at the top of the PVC cell, which delivers the tested solutions. An example of one test cell is shown in Figure 6 from ASTM C1617-15.¹²

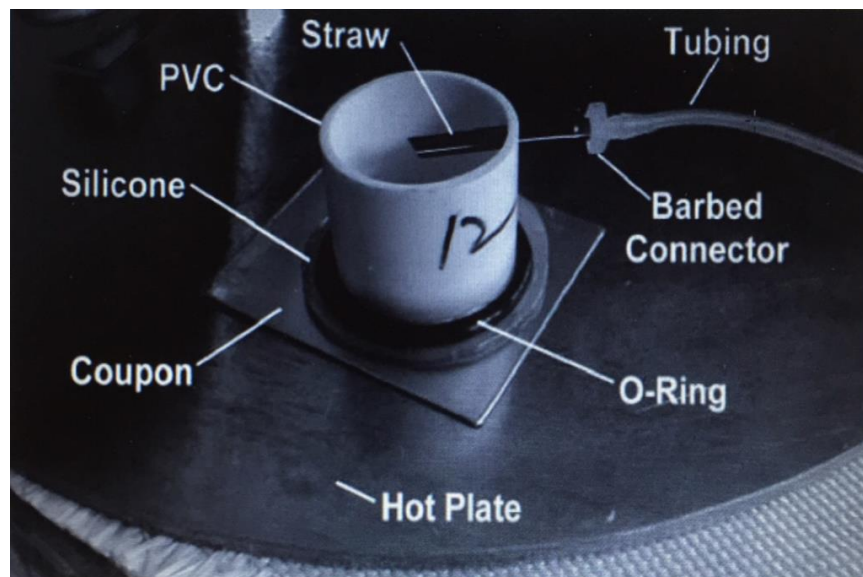


Figure 6. Test cell in accordance to ASTM C1617-15.¹²

Insulators Tested:

Three insulators are tested in accordance to ASTM C1617-15 Procedure A in order to compare the corrosion mitigating properties of each insulator. The insulators consist of two Johns Manville products that exhibit their XOX® corrosion inhibitor ions, Thermo-12® Gold and Sproule WR-1200®, and one Aspen Aerogel product, Pyrogel® XT-E, that does not exhibit corrosion inhibiting ions within its chemistry. Thermo-12® Gold is a calcium silicate insulator material and is qualified to operate up to temperatures of 1200°F.¹³ Sproule WR-1200® is an expanded perlite insulator material classified as hydrophobic, and is also qualified to operate up to temperatures of 1200°F.¹⁴ Pyrogel® XT-E is a silica aerogel insulator material that is also classified as hydrophobic, and is too qualified to operate up to temperatures of 1200°F.¹⁵ All insulators are tested in accordance of Procedure A. The three insulators are shown in Figure 7.



Figure 7. Insulators tested in accordance to ASTM C1617-15

Results

Upon completion of the four day trial samples are cleaned from any corrosion product and weighed to produce a MLCR. MLCR is calculated using the following equation¹²:

$$\text{MLCR} = \frac{K \times W}{A \times T \times D} \text{ mils/year}$$

Where:

K = 3.45e6 (a constant to convert cm/h to mils/year)

T = time of exposure in h

A = area of exposure (cm²), based on inner radius of PVC cell.

W = mass loss from the coupon (g)

D = bulk density of the test metal (g/cm³)

It is important to properly clean the samples of corrosion product formed without stripping the coupon of any metal that was not lost to corrosion product. Proper cleaning techniques followed that of ASTM C1617-15, however the use of a dental pick to scrap the metal was avoided so as not to lose metal material not lost to corrosion product. The final MLCR in mils/year of each sample with the respective solution can be shown in Table I.

Table I. MLCR (mils/year) of each solution tested.

	DI Water	1 ppm Cl ⁻ Solution	5 ppm Cl ⁻ Solution	Sproule WR-1200®	Thermo-12® Gold	Pyrogel® XT-E
Sample 1	11.0025	35.9900	98.1606	2.6512	4.6396	31.3504
Sample 2	8.6827	37.1168	53.9519	5.5012	6.0978	19.4200
Sample 3	-	36.7191	54.4158	5.6338	5.8326	23.5294
AVG	9.843	36.609	68.843	4.595	5.523	24.767

The average mass loss corrosion rate of the insulators were: Sproule WR-1200® at 4.595 mils/yr, Thermo-12® Gold at 5.523 mils/yr, and Pyrogel® XT-E at 24.767 mils/yr.

Graphically, the MLCR results are illustrated in Figure 8. The red line is depicting the average MLCR of DI water.

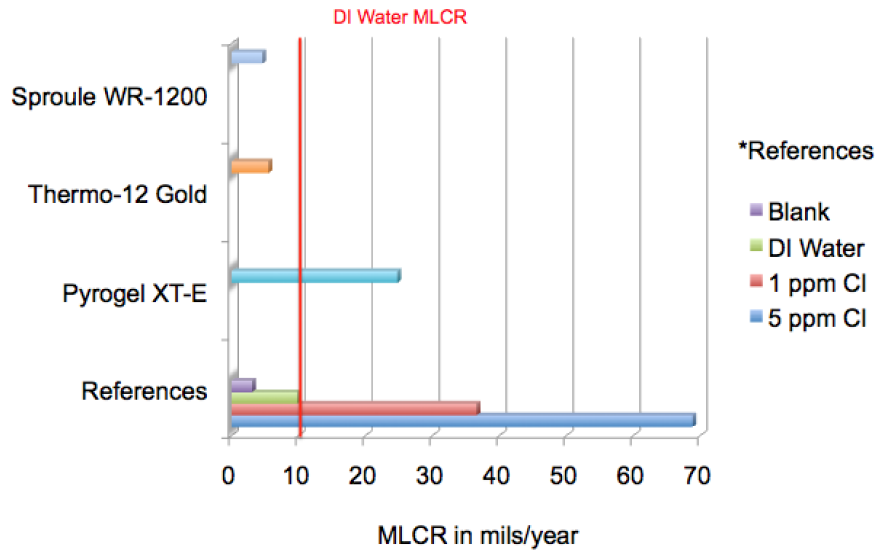


Figure 8. MLCR (mils/year) of each solution tested

Discussion

Results determine that Sproule WR-1200® yielded the lowest average MLCR at 4.595 mils/yr. Thermo-12® Gold yielded the second lowest average MLCR at 5.523 mils/yr. Pyrogel® XT-E had the highest average MLCR of the three tested insulators at 24.767 mils/yr. The blank cell coupon exhibited a MLCR of 3.0489 mils/yr, only 1.5462 mils/yr less than Sproule WR-1200®'s average MLCR.

When considering these results it is important to note that the only thing added to the insulator solution upon being ground up is DI water. Since this is the case, any insulator solutions yielding a MLCR greater than DI water is composed of corrosive ions that promote an environment causing corrosion to occur. This is a safe claim, as DI water is not composed of any ions whatsoever. Alternatively, any insulator solutions yielding a MLCR lower than DI water is composed of corrosion mitigating ions that deter corrosive environments. Therefore Thermo-12® Gold and Sproule WR-1200® are confirmed to incorporate corrosion mitigating ions, and Pyrogel® XT-E is confirmed to be composed

of corrosive ions. A passive layer on both corrosion mitigating insulator solution coupons is shown in Figure 9. Heavy layers of corrosion product can be observed on the samples exposed to Pyrogel® XT-E's solution.

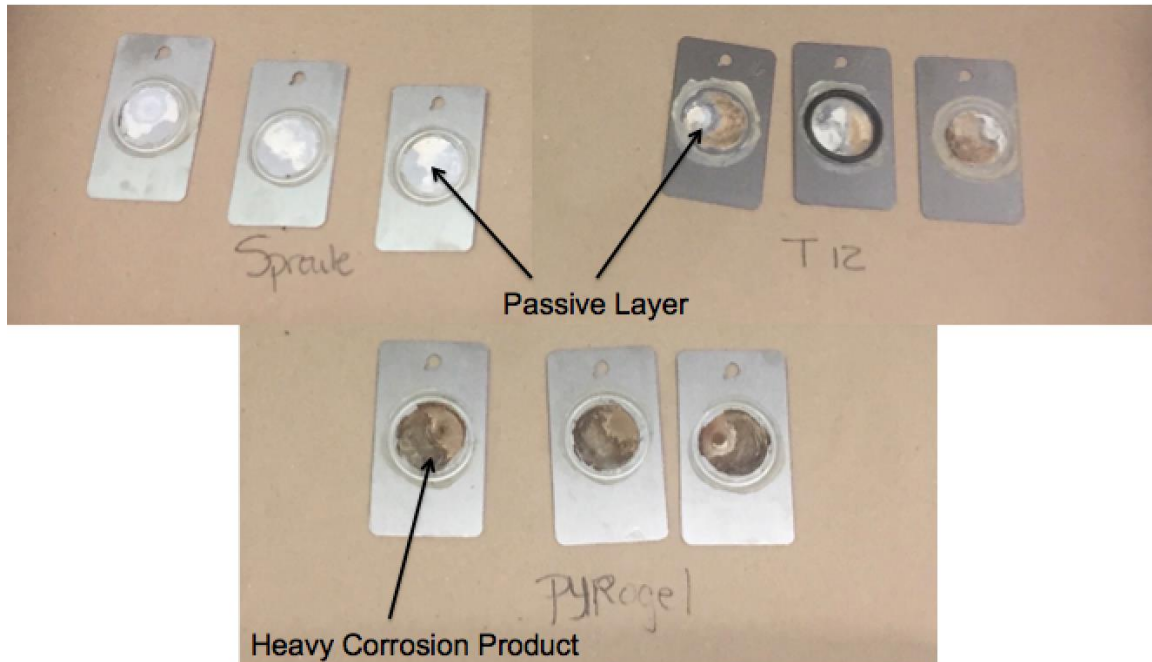


Figure 9. Corrosion product formed on carbon steel samples after the four-day trial.

ASTM standards call for different “PASS” levels of each insulator. Table II depicts these different “PASS” levels of each the insulator types tested.

Table II. MLCR Thresholds of different insulation materials.

Material Standard or Test Method	ASTM C533 Calcium Silicate Pipe & Block Thermal Insulation ⁴	ASTM C1728 Flexible Aerogel Insulation ⁵	ASTM C612 Mineral Fiber Block and Board Thermal Insulation ⁶
C1617 Corrosion	PASS	PASS	PASS
Maximum Mass Loss Corrosion Rate to Pass	Less than Deionized (DI) Water	Less than 5 ppm Chloride Solution	Less than 5 ppm Chloride Solution

From this table it is shown that calcium silicate insulators (Thermo-12® Gold) are the only insulator material required to exhibit a lower MLCR than that of DI water in order to “PASS” as a usable insulator. Insulators that are flexible aerogels (Pyrogel® XT-E) and mineral fibers are only required to exhibit a lower MLCR than that of 5 ppm chloride

solution in order to “PASS” as a usable insulator. DI water is never seen in environments where systems need to be insulated, so it seems ridiculous to use this as the point at which calcium silicate insulators must pass. However, the Thermo-12® Gold still did pass. Pyrogel® XT-E did in fact exhibit a lower MLCR than that of 5 ppm chloride solution, however it is appropriate to analyze the results in comparison to DI water to see what corrosive environment the insulators produce at the chemical level. Therefore, all insulators should be held against the MLCR of DI water, as done in this analysis, to properly observe the corrosiveness of each insulator’s chemistry.

Conclusions

1. Sproule WR-1200® and Thermo-12® Gold solutions both yielded a lower MLCR than DI water, confirming these insulators leached corrosion mitigating ions.
2. Pyrogel® XT-E’s solution yielded a greater MLCR than DI water, therefore this insulator leached corrosion promoting ions.
3. Incorporating ions in thermal insulators to produce a passive layer on metal equipment effectively mitigates CUI.

Future Work

Some manufacturers of insulator materials classified as “hydrophobic” may find the claim to use only Procedure A inaccurate. For these reasons it would be appropriate to run all of the insulators again in accordance to Procedure B. Results should be compared to that of the results from Procedure A to justify the claim, and to provide further evidence that Sproule WR-1200® and Thermo-12® Gold produced corrosion mitigating ions, while Pyrogel® XT-E produced corrosion promoting ions.

This particular study was conducted on an off-campus site and time was limited. Due to this, cleaning and weighing of the samples was done before any characterization of the passive layer formed could be conducted. In future studies SEM imaging of the corrosion product would serve as investigative measures to further understand the properties of the silicate layer. Chemical analysis of the solutions would be an option for future testing in a scenario where time is not a limitation, and should follow in accordance to ASTM C871.

As mentioned earlier, it is uncommon to see DI water in an environment that requires insulator materials. Testing in accordance to ASTM C1617-15 with reference standards of rain water or a mimic solution of ocean fog in marine applications may prove to have interesting results, and would show a more accurate corrosion rate of a real life scenario of CUI.

Another mode of testing insulators properties to mimic CUI is ASTM C692. This tests for chloride stress corrosion cracking (SCC) of stainless steels. Running ASTM C692 in a modified test for carbon steel coupons to instead analyze general corrosion and not SCC would more appropriately serve as a physical representation of a CUI attack. ASTM C692 is shown in Figure 10.¹⁶



Figure 10. ASTM C692.¹⁶

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Appendix I

Standard Operating Procedure for Corrosion Under Insulation (CUI) Investigation Procedure

Procedure: ASTM C1617-15 PROCEDURE A

Description: Standard practice for quantitative accelerated laboratory evaluation of extraction solutions containing ions leached from thermal insulation on aqueous corrosion of metals.

1. Scope

1.1 This practice uses controlled amounts of test solutions delivered in a drip wise fashion onto a defined area of carbon steel coupons at elevated temperatures for the purpose of producing, comparing, and measuring the corrosion that occurs on the metals due to the exposure of test solutions.

2. Hazards

2.1 Use of hydrochloric acid (35%)

3. Standards Reference Tests

3.1 DI water

3.2 1 ppm chloride solution

3.3 5 ppm chloride solution

4. Reagents and Materials

4.1 Carbon steel coupons: ASTM D609-Type 1, Temper = ¼ hard, Carbon = 0.13 wt%; size = 0.032 x 2 x 3.5 in.

4.2 Sproule WR-1200®

4.3 Thermo-12® Gold

4.4 Pyrogel® XT-E

5. Testing Equipment

5.1 Wolfgang Puck® hot plate

5.2 24-Channel Ismatec IPC® peristaltic pump

5.3 Ninja® Blender

5.4 Silicone rubber tubing to deliver solutions

5.5 Mettler Toledo Mass Balance Scale (capable of 0.0001 (+/- 0.0002) mass determination

5.6 Drying oven

- 5.7 Nominal 1-in. Thin-wall PVC Pipe
- 5.8 Thermal cover for consistent heating
- 5.9 High temperature grease or oil for consistent heating
- 5.10 Thermocouple to ensure consistent heating
- 5.11 Silicone sealant
- 5.12 Rubber O-rings
- 5.13 Filter paper of 0.45 micron filter
- 5.14 DI water
- 5.15 Chloride Standard, 1000 ppm
- 5.16 VWR Signature™ Ergonomic High-Performance Pipettor
- 5.17 Fiberglass-felt clothe abrasive

6. Test Cell Preparation

- 6.1 Mark each coupon for identification.
- 6.2 Heat coupons in 180°F drying oven to drive off surface moisture. Weigh for initial mass.
- 6.3 Cut PVC pipe into 1.25 in lengths, smoothing ragged edges. Drill a 1/8 in hole in the side of the pipe 1/8 in from the top end of the PVC pipe. Clean pipe with DI water and dry.
- 6.4 Position O-ring on PVC cell 0.5 in from bottom end of the PVC pipe. Place the PVC pipe on a metal coupon. Apply silicone sealant on PVC pipe between the metal coupon interface and the O-ring. Push firmly into the metal coupon while sliding the O-ring down to the interface forming a continuous, watertight seal. Avoid silicone sealant on the inside of the pipe and metal. Allow silicone to cure overnight prior to testing. Insert silicone tubing in 1/8 in hole for testing. Produce three test cells for each solution tested. Produce one blank cell for control.

7. Standard Reference Preparation

- 7.1 *DI water* 3000 mL for testing of four-day span (250mL/day on 3 coupons).
- 7.2 *1 ppm chloride solution* Using the VWR Signature™ Ergonomic High-Performance Pipettor measure out 0.001 mL of Chloride Standard and add to 1000 mL of DI water. Do this three times to total 3000 mL.

7.3 *5 ppm chloride solution* Using the VWR Signature™ Ergonomic High-Performance Pipettor measure out 0.005 mL of Chloride Standard and add to 1000 mL of DI water. Do this three times to total 3000 mL.

8. Procedure A Insulator Solution Preparation

- 8.1 Weigh out 20.0 g of insulator to be tested.
- 8.2 Grind down each insulator in Ninja® Blender until fine dust. If unable to produce a fine dust, add 450 mL of DI water prematurely to help mix. This proved necessary when producing the silica aerogel solution.
- 8.3 Add previously stated 450 mL of DI water to ground insulator.
- 8.4 Boil this mixture for 30 min.
- 8.5 Adjust solution to total 500 mL by adding more DI water.
- 8.6 Filter this solution to remove solids.
- 8.7 Adjust this solution to total 3000 mL by adding more DI water. This provides for enough for a four-day trial on 3 coupons (250mL/day each coupon).

9. Dripping Solutions on Hot Plate

- 9.1 Ensure hot plate stays at constant 250°F (+/- 10°F) for 1 h to calibrate for testing.
- 9.2 Place test cells in a manner that allows each respective silicone tube to properly deliver test solutions without error.
- 9.3 Connect silicone tubing to the peristaltic pump and place opposite end in each respective test solution to be delivered.
- 9.4 Set peristaltic pump rate to deliver 250 mL/day (+/- 50 mL).
- 9.5 Run test over a four-day span

10. Evaluation

10.1 Cleaning coupons

10.1.1 Gently remove the PVC cell from the metal coupons, leaving the silicone sealant and O-ring in tact when possible. This aids to prevent stripping the coupons of any metal not lost to corrosion.

10.1.2 Using proper PPE (eyewear, nitrile gloves, apron, closed-toe shoes), place each metal coupon in a Pyrex bowl and introduce HCl to the coupon. Lightly agitate the bowl. After a minute grab the coupons and rub with fiberglass-felt clothe abrasive to remove corrosion product.

10.1.3 Repeat these steps if necessary to properly remove as much corrosion product as possible. Record the amount of HCl baths for each coupon.

10.2 *Weighing the Samples*

10.2.1 Rinse coupons with DI water upon completion of cleaning.

10.2.2 Place coupons in a 180°F drying oven to drive off moisture.

10.2.3 Record final mass

10.3 *MLCR Calculation*

10.3.1 Using equation 1 below calculate MLCR:

$$\text{MLCR} = \frac{K \times W}{A \times T \times D} \text{ mils/year}$$

Where:

K = 3.45e6 (a constant to convert cm/h to mils/year)

T = time of exposure in h

A = area of exposure (cm²), based on inner radius of PVC cell.

W = mass loss from the coupon (g)

D = bulk density of the test metal (g/cm³)

11. Report

11.1 Report on MLCR of all tested cells and solutions including DI water, 1 ppm chloride solution, 5 ppm chloride solution, and the blank test cell.

12. Uncontrolled Variables

12.1 Atmosphere within laboratory. A fan is helpful in circulating the air above the cells and reducing localized humidity from the water evaporating from the cells.

12.2 Rate of evaporation from the coupon.

12.3 Distribution of localized and general corrosion.

12.4 Change in the corrosion rate. Longer testing may provide information on the change in corrosion rate over time.

12.5 Different metal coupon qualities. All coupons meet ASTM specification however possible localized variances with the coupon are probable.

13. Controlled Variables

13.1 Temperature of the hot plate during testing.

13.2 Duration of the test.

13.3 Rate and volume of testing solution delivered.

13.4 Solution concentrations.

13.5 Cell tubes and defined corrosion area.

13.6 Cleaning of the coupons.

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Documentation of Training for SOP of CUI Investigation ASTM C1617-15

Primary Investigator and Supervisor

Primary Investigator:

Michael Lowes

Date:

Supervisor:

Dr. Trevor Harding

Date:

Reference

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Appendix II

Materials/Insulators Tested Safety Analysis for Corrosion Under Insulation (CUI) Investigation Procedure

Thermo-12® Gold Calcium Silicate Insulation

HAZARDS IDENTIFICATION

- **GHS Classification:** Not a hazardous substance or mixture
- **GHS Label element:** Not a hazardous substance or mixture
- **Other hazards:** Temporary mechanical abrasion (itching) of skin, eyes and respiratory may occur upon exposure to fibers or dust during handling of this product.

COMPOSITION

Chemical Name	CAS-No.	Concentration (%)
Synthetic calcium silicate	1344-95-2	>=90
Cellulose fiber	9004-34-6	>= 0 - <= 4

FIRST AID MEASURES

General advice: No hazards, which require special first aid measures.

If inhaled: If symptoms persist, call a physician.

In case of skin contact: If symptoms persist, call a physician.

In case of eye contact: Remove contact lenses. Protect unharmed eye. Rinse thoroughly with plenty of water, also under the eyelids. If eye irritation persists, consult a specialist.

If swallowed: Keep respiratory tract clear. Never give anything by mouth to an unconscious person. If symptoms persist, call a physician.

FIREFIGHTING MEASURES

Suitable extinguishing media: Use extinguishing measures that are appropriate to local circumstances and the surrounding environment.

Hazardous combustion products: No hazardous combustion products are known

Specific extinguishing methods: Use extinguishing measures that are appropriate to local circumstances and the surrounding environment.

Further information: Standard procedure for chemical fires.

Special protective equipment for firefighters: Wear self-contained breathing apparatus for firefighting if necessary.

ACCIDENTAL RELEASE MEASURES

Personal precautions, protective equipment and emergency procedures: Avoid dust formation.

Methods and materials for containment and cleaning up: Pick up and arrange disposal without creating dust. Sweep up and shovel. Keep in suitable, closed containers for disposal.

HANDLING AND STORAGE

Advice on safe handling: For personal protection see section 8.

Smoking, eating and drinking should be prohibited in the application area.

Conditions for safe storage: Keep in a dry, cool place.

Materials to avoid: No materials to be especially mentioned.

PERSONAL PROTECTION

Respiratory protection: When workers are facing concentrations above the exposure limit they must use appropriate certified respirators. No personal respiratory protective equipment normally required.

Hand protection Remarks: For prolonged or repeated contact use protective gloves.

Eye protection: Safety glasses

Skin and body protection: If used and stored as directed, no special protective equipment is necessary.

Hygiene measures: Handle in accordance with good industrial hygiene and safety practice.

DISPOSAL CONSIDERATIONS

Contaminated packaging: Empty containers should be taken to an approved waste-handling site for recycling or disposal. Packaging that cannot be reused after cleaning must be disposed or recycled in accordance with all federal, national and local regulations.

Sproule WR-1200® Bonded Perlite Thermal Insulation

HAZARDS IDENTIFICATION

- **GHS Classification:** Not a hazardous substance or mixture
- **GHS Label element:** Not a hazardous substance or mixture
- **Other hazards:** None known.

COMPOSITION

Chemical Name	CAS-No.	Concentration (%)
Perlite	93763-70-3	$\geq 90 - \leq 100$

FIRST AID MEASURES

General advice: Do not leave victim unattended.

If inhaled: If unconscious place in recovery position and seek medical advice. If symptoms persist, call a physician

In case of skin contact: If symptoms persist, call a physician.

In case of eye contact: Remove contact lenses. Protect unharmed eye. Rinse thoroughly with plenty of water, also under the eyelids. If eye irritation persists, consult a specialist.

If swallowed: Keep respiratory tract clear. Never give anything by mouth to an unconscious person. If symptoms persist, call a physician.

FIREFIGHTING MEASURES

Specific extinguishing methods: Use extinguishing measures that are appropriate to local circumstances and the surrounding environment.

Further information: Standard procedure for chemical fires.

Special protective equipment for firefighters: Wear self-contained breathing apparatus for firefighting if necessary.

Hazardous combustion products: No hazardous combustion products are known.

ACCIDENTAL RELEASE MEASURES

Personal precautions, protective equipment and emergency procedures: Avoid dust formation.

Methods and materials for containment and cleaning up: Pick up and arrange disposal without creating dust. Sweep up and shovel. Keep in suitable, closed containers for disposal.

HANDLING AND STORAGE

Advice on safe handling: For personal protection see section 8.

Smoking, eating and drinking should be prohibited in the application area.

Conditions for safe storage: Keep in a dry, cool place.

Materials to avoid: No materials to be especially mentioned.

PERSONAL PROTECTION

Respiratory protection: When workers are facing concentrations above the exposure limit they must use appropriate certified respirators. No personal respiratory protective equipment normally required.

Hand protection Remarks: For prolonged or repeated contact use protective gloves. Barrier creams may help to protect the exposed areas of skin, they should however not be applied once exposure has occurred.

Eye protection: Safety glasses

Skin and body protection: If used and stored as directed, no special protective equipment is necessary.

Hygiene measures: Handle in accordance with good industrial hygiene and safety practice. Written instructions for handling must be available at the work place.

DISPOSAL CONSIDERATIONS

Contaminated packaging: Empty containers should be taken to an approved waste-handling site for recycling or disposal. Packaging that cannot be reused after cleaning must be disposed or recycled in accordance with all federal, national and local regulations.

Pyrogel® XT-E Silica Aerogel Insulation

HAZARDS IDENTIFICATION

- **GHS Classification:** Not a hazardous substance or mixture
- **GHS Label element:** Not a hazardous substance or mixture
- **Other hazards:** None known.

COMPOSITION

Chemical Name	CAS-No.	Concentration (%)
Synthetic Amorphous Silica	7631-86-9	30-40%
Methylsilylated Silica	68909-20-6	10-20%
Fibrous Glass (textile grade)	Not Applicable	40-50%
Iron Oxide (iron (III) oxide)	1309-37-1	1-10%
Aluminum Trihydrate (aluminum hydroxide)	21645-51-2	1-5%
Other components are either non-hazardous ingredients or are below the concentration limit for classification	Mixture	Balance

FIRST AID MEASURES

Inhalation: If dust is inhaled, remove to fresh air. Drink water to clear throat, and blow nose. If irritation occurs or symptoms develop, seek medical attention.

Skin contact: Wash skin with soap and water. If irritation develops, get medical attention, launder clothing before reuse.

Eye contact: Do not rub eyes. Dust particles may cause abrasive injury. Immediately flush eyes with water while lifting the upper and lower lids. Seek medical attention if irritation persists.

Ingestion: No first aid is generally required. No adverse effects are expected from incidental ingestion.

Most important symptoms/effects, acute and delayed: Dust may cause eye irritation. Silica aerogels are hydrophobic (repel water) and may cause temporary drying and irritation of the skin, eyes, and mucous membranes. Inhalation of dust from handling may cause temporary upper respiratory tract irritation. Handling may cause dryness and irritation of the skin.

Indication of immediate medical attention and special treatment, if necessary: Immediate medical attention is generally not required.

FIREFIGHTING MEASURES

Extinguishing media: Use any media that is suitable for the surrounding fire.

Specific hazards arising from the substance or mixture: Product is a super-insulator. Rolls of material will retain heat within internal layers that may be a source of ignition after the fire is extinguished. Keep hot material away from combustible materials and cool hot insulation with water.

Special protective equipment and precautions for fire-fighters: Normal firefighting procedures should be followed to avoid inhalation of smoke and gases produced by a fire.

ACCIDENTAL RELEASE MEASURES

Personal precautions, protective equipment, and emergency procedures: Wear appropriate protective clothing and equipment as described in Section 8. Avoid generating airborne dust during cleanup. Ensure adequate ventilation.

Environmental Precautions: Material is not water-soluble. Report spills as required under federal, state and local regulations.

Methods and materials for containment and cleaning up: Collect using methods that avoid the generation of dust (pick up or vacuum dust) and place in appropriate container for disposal.

HANDLING AND STORAGE

Precautions for safe handling: Aerogel blankets may generate dust when handled. Workplace exposures to all dusts should be controlled with standard industrial hygiene practices. Local exhaust should be the primary dust control method. Dust generated when handling this product should be cleaned up promptly. Dry vacuuming is the preferred method for cleaning up dust. Because aerogel dust is hydrophobic, water is not an effective dust control agent. Unpack material in the work area. This will help to minimize the area where dust exposure may occur. Trimmed material should be promptly packed in disposal bags. Trims and off-cuts may be reused in secondary applications; otherwise scrap material should be packed for disposal. Avoid dust contact with eyes, skin and clothing and avoid breathing dust. Wash hands with soap and water after handling.

Conditions for safe storage, including any incompatibilities: Keep tightly closed in the packaging until ready for use. Store in a dry location.

PERSONAL PROTECTION

Respiratory protection: If exposures exceed the occupational exposure limits or if inhalation of dust results in experiencing irritation, an appropriate certified particulate respirator is recommended. Selection of respiratory protection depends on the contaminant type, form and concentration. Select and use in accordance with OSHA 1910.134 and good Industrial Hygiene practice, e.g. a NIOSH approved P100 or N100 particulate filtering facepiece respirator.

Skin protection: Impervious gloves recommended for handling product. Long-sleeved and long-legged work clothing are also advised.

Eye protection: Safety glasses with side shields or dust goggles recommended.

Documentation of Training for Materials Safety

Primary Investigator and Supervisor

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