Fluid Solutions

A Heat Transfer Fluid Magazine

Edition 2, December 2016

Molten salts: high temperature heat transfer fluid and thermal storage medium

ASTM corrosion testing for heat transfer fluids

Removing moisture from cryogenic heat transfer fluids

Chloride testing methods

Should glycol be stored outside?

learn the effect sunlight can have on your glycol

See the next page to know more about the cover

DYNALENE

Tomorrow's Solutions **Flow** Through Us

Commentary

“Fluid Solutions” Magazine in its Second Year!

Last year was the inaugural edition of our “Fluid Solutions” magazine dedicated to heat transfer fluids. The magazine was well received by our customers and we got a large amount of positive feedback. Many of our readers enjoyed going through some interesting facts about heat transfer fluids, refrigeration, corrosion and nano-technology. Some readers were fascinated by the tardigrade on our cover page. Even many of us were surprised to know the interesting facts about these tiny creatures; how they can survive under extreme conditions such as very low or high temperatures, the vacuum of space or the high pressure of the deepest part of the ocean, without food or water, and lethal radiation. These tardigrades teach us something: nature’s innovations sometimes far outweigh human accomplishments. Scientists and engineers should continue to learn from our surroundings to develop ground-breaking technologies and products.

This year you will find two review articles, three research papers, and a section called “Ask the Experts”, where we have covered several questions that are frequently asked by our customers. Similar to last year, we have incorporated a number of “Fun Facts” related to the topics covered. The articles in this edition cover a variety of topics ranging from contamination or degradation of heat transfer fluids to high temperature molten salt fluids. There is a review article discussing different types of corrosion measurement techniques commonly used for heat transfer fluids. A thorough research paper demonstrates the effect of sunlight, specifically UV light, on the degradation of glycols. This is important to many glycol users since they might be storing the product outside. Another research paper shows how moisture solubility in a hydrocarbon-based heat transfer fluid changes with temperature and how to desiccate a fluid at very low temperatures. Chloride ion measurement techniques are discussed in another research paper since these ions can negatively affect a heat transfer fluid system by initiating pitting corrosion.

Please enjoy reading the articles and have a wonderful holiday and a prosperous new year 2017.

Sincerely,

Dr. Satish Mohapatra, President & CEO, Dynalene

About The Cover

A fluorophore absorbs light energy of a specific wavelength and re-emits light at a longer wavelength, creating a fluorescent effect. Fluorescent dyes are widely used in heat transfer fluid applications to detect leaks. The cover picture depicts some of the dyes used in glycol based fluids. The photograph was taken by adding drops of three different dyes into a glycol-water mixture, lighted by a 3W LED UV lamp of 395nm wavelength. Fluorescence has many practical applications, including mineralogy, gemology, medicine, chemical sensors (fluorescence spectroscopy), fluorescent labelling, dyes, biological detectors, cosmic-ray detection, and, most commonly, fluorescent lamps. Fluorescence also occurs frequently in nature in some minerals and in various biological states in many branches of the animal kingdom.

Fluorescein is one of the compounds used as a fluorescent material. One of the first uses of fluorescein was in 1877 in a major ground-water tracing experiment in southern Germany. The results of this experiment showed that the River Danube and Rhine are connected by underground streams. Fluorescein was placed in the Danube and about 60 hours later it appeared in an affluent of the Rhine. 10 Kilograms of fluorescein were used!

Source: Barbieri.,Beniamino, "A Short History of Fluorescence", The Fluorescence Foundation, 2010.

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09. Ask The Experts

Numerous questions from our customers are answered by our experts everyday. Here are a few.

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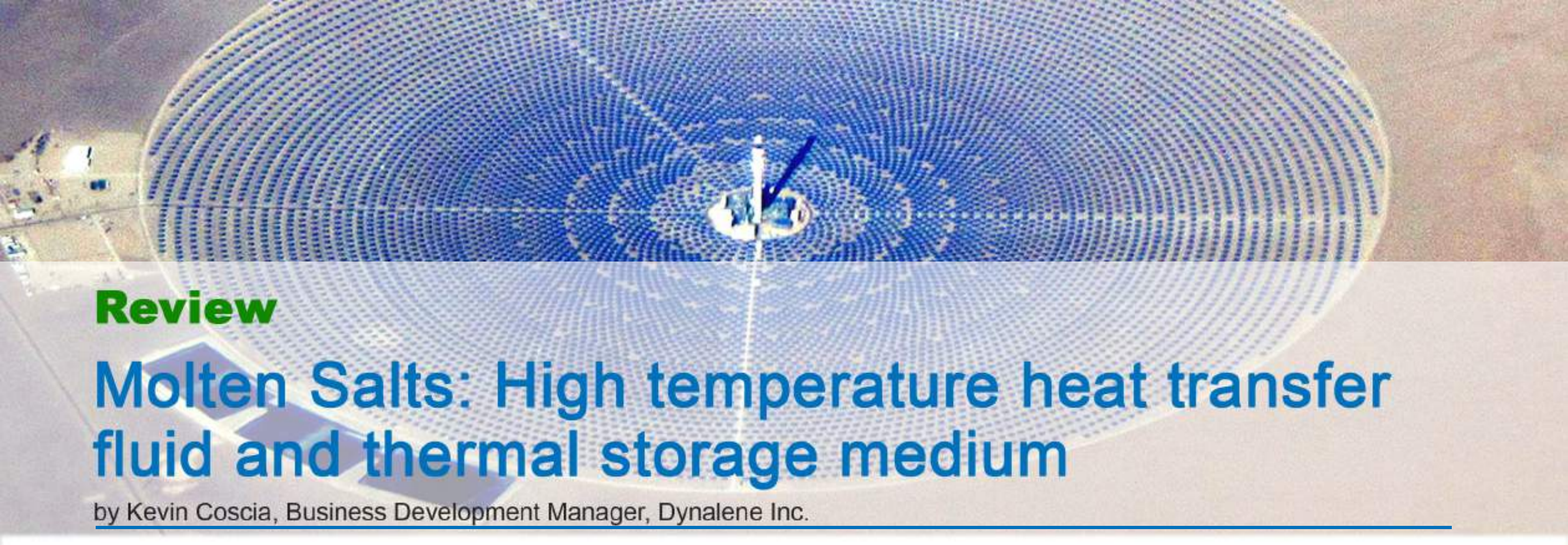

Review

Molten Salts: High temperature heat transfer fluid and thermal storage medium

by Kevin Coscia, Business Development Manager, Dynalene Inc.

Introduction

There are many varieties of heat transfer fluids (HTF's) that can be used in process heating applications. How can an engineer identify which type of heat transfer fluid would be best for their system? There are many design parameters that must be taken into account, such as heat transfer efficiency, pumping power, and operational life that will factor into operating expenses. There are also safety parameters that cannot be overlooked, such as pressure build up, flash point, flammability, and toxicity concerns. With these concerns in mind, an engineer can pick from a selection of water-based heat transfer fluids, oil based heat transfer fluids, and even high temperature molten salts.

Water-based fluids will generally perform the best due to their excellent thermophysical properties. Mineral oils and synthetic oils are generally used at slightly higher temperatures (100°C to 350°C), where the oil will not degrade as readily as a glycol and the system pressure will be kept low. However, above 350°C the option for using a hydrocarbon heat transfer fluid becomes limited. Natural oils or mineral oils will degrade near this temperature and need to be replaced after just a year or two of use, as depicted in Figure 1. Synthetic hydrocarbon heat transfer fluids, such as biphenyl/diphenyl oxide, can be used up to 390°C [1]. These synthetic fluids can be very expensive and will still undergo some degree of degradation which leads to fouling of heat exchanger equipment and loss of efficiency. In some processes, 10-20% of the fluid must be replaced every year, with complete replacement of the fluid every 5-10 years. For operating temperatures higher than this that require a liquid heat transfer media, a molten salt fluid must be used.

What is a Molten Salt?

Molten salts are just salts in the liquid state. The most common types of salts used in industry are usually mixtures of different alkali or alkaline nitrates, nitrites, chlorides, fluorides, and carbonates. Each salt has its own unique properties, which allows them to be used for a number of different processes, but the most important feature of these salts is their high-temperature thermal stability. The term 'molten salt,' especially if associated with reactive sodium or fluorine, generally carries a negative connotation, but in reality, molten salts are some of the most stable compounds on earth. Bonding an alkali metal, such as potassium, to a reactive halogen, such as chlorine, yields an incredibly stable and non-toxic potassium chloride salt that melts at 770°C with a boiling temperature near 1400°C [2].

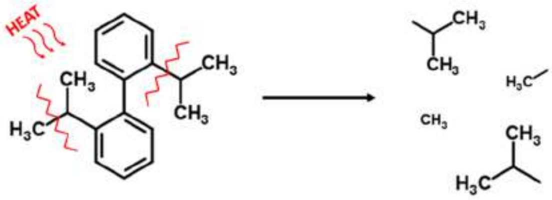

Figure 1: High temperature breakdown of a synthetic HTF can result in the formation of undesirable gases and polymers that hinder performance and damage systems.

Of all the salts that exist in the world, the most commonly used salts for heat transfer applications are the nitrate salts, due to their low melting point, high operating temperature, thermophysical properties, low cost, low toxicity, low vapor pressure, and corrosion performance.

A widely used high temperature salt is an eutectic blend of sodium nitrate and potassium nitrate. Individual salts can be used, but by mixing the two different salts together the melting point is reduced, allowing for lower minimum operating temperatures and thus reducing the chance of freeze up. Sodium nitrate has a melting point of 307°C and potassium nitrate has a melting point of 333°C, but by mixing the two together, you can create a salt blend with a melting point of 222°C, as shown in Figure 2 [3]. This significantly expands the operational flexibility of the salt in high temperature applications.

What is the temperature range of molten salt?

There are many different salt blends that can be created to achieve certain operating temperatures. The sodium nitrate/potassium nitrate salt is generally used from 285°C to 565°C to provide enough safety factor above the freezing point. When using a molten salt, it is recommended to be at least 20°C to 30°C above the melting point to reduce the risk of freeze ups where there may be cold spots. It is also recommended to keep the maximum operating temperature of the salt around 20°C below the degradation temperature and stainless steel compatibility for different types of salts.

The ternary lithium nitrate/sodium nitrate/potassium nitrate mixture is another option, with the eutectic melting temperature occurring at 130°C. The disadvantage of this mixture compared to the binary mixture is that it can only be used to a maximum temperature near 500°C before the lithium nitrate begins to degrade. And with the advent of lithium-ion batteries, lithium nitrate has become very expensive compared to sodium or potassium salts [5].

What are the best types of salts to use and why?

Nitrate salts provide the optimal balance of wide operating range, thermal stability, corrosion performance, and cost. For applications that need a liquid heat transfer media above 600°C, the options are limited to chloride and fluoride salts, as these salts are more stable than the nitrate salts. Chlorides and fluorides can operate slightly higher, near 900°C. These salts also have higher melting points (300°C to 500°C) which increases the risk of the salt freezing. Chlorides and fluorides are also more corrosive than nitrate salts, especially at high temperatures, and require more exotic alloy materials of construction [2].

What about corrosion?

One of the biggest advantages of the nitrate salts is their corrosion performance. Nitrate salts are compatible with stainless steels up to 565°C and even carbon steel if the operating temperature is kept below 400°C. This makes them advantageous to use with common materials of construction that keep construction costs low. It is not advised to use yellow metals, such as copper or brass, with molten salts as they are not as compatible as steel at the higher temperatures [6].

Chlorides and fluorides, on the other hand, pose significant material engineering problems. Mentioning the word 'chloride' alone to an engineer may cause them to squirm, as we are all familiar with the corrosive potential of chlorides, even in small amounts. Some high-nickel alloys fare well with chloride and fluoride coolants, but the longevity of these materials when used with these salts is yet to be fully understood. These alloys are also much more expensive than traditional stainless steels, so for large scale systems the initial capital and maintenance costs would be immense and economically infeasible.

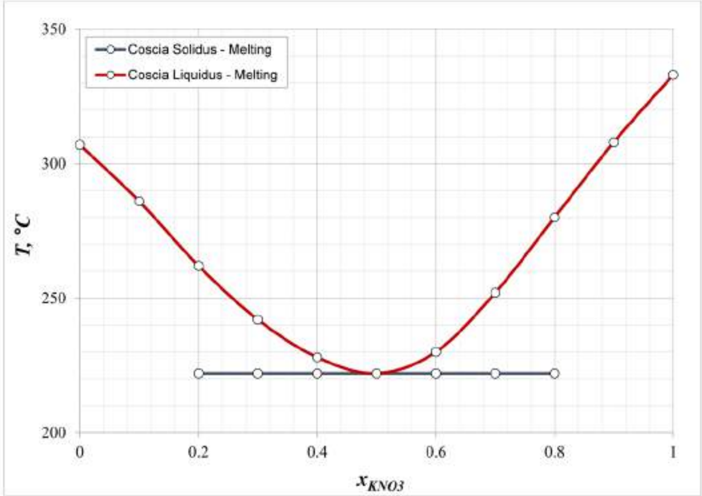

Figure 2: Phase diagram of sodium nitrate ($NaNO_3$) - potassium nitrate (KNO_3) mixture [3].

Table 1: Melting points, operating temperatures, toxicity, and stainless steel compatibility for different types of salts.

Criteria	Nitrates	Carbonates	Chlorides	Fluorides
Mixture melting range	120 – 220°C	400 – 600°C	350 – 700°C	500 – 900°C
Operating range	150 – 590°C	450 – 650°C	400 – 800°C	550 – 1000°C
Toxicity	Low	Low	Low	Low
Stainless steel compatibility	Good	Good	Poor	Poor

What are the advantages of molten salts?

Compared to synthetic oils or silicone fluids, molten salts can be used at higher temperatures, possess better heat transfer properties, and are less expensive, as depicted in Table 2 [1, 5, 6]. This allows the salt to absorb and store heat for long periods of time, longer than hydrocarbons can. Synthetic fluids also have maximum operating temperatures that limit them to 350°C before undergoing degradation. Even below this temperature, oils will degrade and need to be replaced after several years of use. An additional consequence of this degradation is that oil will eventually break down into carbon or polymerize, and this can lead to fouling of heat exchanger equipment or sensitive electronics.

Another big advantage molten salts have is that when used near peak operating temperatures they experience almost negligible vapor pressures, relinquishing the need for high pressure piping and equipment. Some nitrate salts, for example, will not degrade until about 600°C.

The thermophysical properties of molten salts also make them excellent heat transfer fluids. When used near their peak operating temperatures, the heat transfer coefficient of a molten salt is comparable to the heat transfer coefficient of water at room temperature. Water is the best heat transfer fluid there is, and from Figure 3, it is apparent that molten salts are only slightly less efficient.

What are the disadvantages of molten salt?

While molten salts can satisfy the thermal stability and efficient heat transfer parameters, their main deterrent is that they will freeze well above room temperature (120°C to 220°C) [8]. This can potentially create significant risks for systems that are not designed with this in mind. Oil heat transfer fluids are easy to use because they are liquid at room temperature, but applications that use molten salts require heat tracing in all piping and equipment to prevent the salt from freezing. Salts also expand when they re-melt, and if the salt is not uniformly heated during the re-melting process it can damage piping, valves, and other equipment. When using a molten salt, it is essential to take the necessary precautions to prevent the salt from freezing during operation.

Depending on the salt and the materials used, corrosion may also be an issue. If using nitrates with stainless steel, corrosion will be minimal, however with more aggressive salts the corrosion could be severe. Chlorides and fluorides must be used with higher alloys as they will corrode stainless steels at high temperatures. In many heat transfer fluid applications corrosion inhibitors are added to the fluid to reduce corrosion. Corrosion inhibitors are a common additive used to protect systems, as the inhibitors can easily coat the inside of piping and components without affecting the heat transfer performance of the fluid.

Table 2: Comparison of synthetic HTFs vs. molten nitrate salts

Property	Synthetic HTF	Molten Nitrate Salt
Melting/freezing point	< 25°C	130°C to 220°C
Operating temperature range	25°C to 390°C	150°C to 565°C
Volumetric heat capacity	~2,000 kJ/m^3	~3,000 kJ/m^3
Thermal conductivity	0.2 W/mK	0.6 W/mK
Viscosity	< 1 cP	< 2 cP
System pressure	Moderate to High	Low
Cost	$8-15/lb	$1-5/lb
Toxicity	Moderate	Low

How do you install molten salt?

Nitrate salts are usually manufactured in prill form (small beads) and may arrive in 2000 lb super sacks or smaller packages. For smaller systems, the salt can be added directly after opening the bag, ensuring the salt is spread out uniformly for better heat distribution. As the salt melts, its volume from crystalline prill form to liquid form will reduce by about 50% (due to the air spaces between the prills). If the melting tank is large enough, the salt can be added all at once, but if there are some space restrictions, the salt can be melted and added in several increments. For much larger systems, the salt may be emptied and loaded onto conveyor systems where it is deposited inside the large tank as the salt melts.

Are molten salts Hazardous?

Nitrate molten salts are very stable and have low toxicity, but they are oxidizers. In the presence of fire or open flames they can break down and liberate oxygen, providing a fuel source for the flame. Molten nitrate salts should be kept away from open flame, sparks, and other sources of ignition. If proper precautions are taken the salts should not pose any hazards.

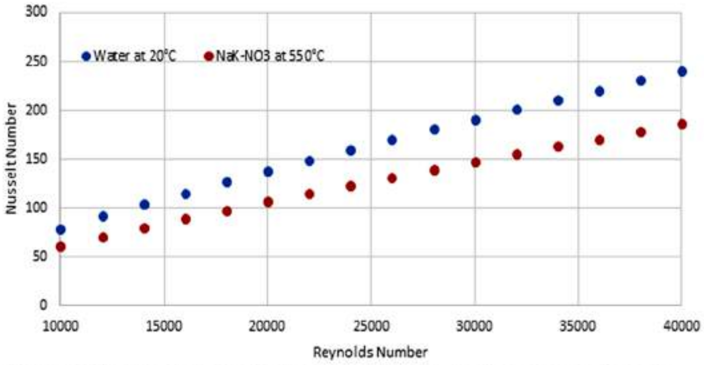

Figure 3: Heat transfer comparison between water at 20°C and eutectic NaK-NO$_3$ at 550°C.

Conclusion

Molten salts offer advantages and disadvantages compared to hydrocarbon-based heat transfer fluids. Some applications may find molten salts to be the preferred choice for thermal storage if the risk of salt freezing can be avoided. Other applications may not operate under such extreme temperatures, and thus hydrocarbon fluids can be used. Salts possess excellent thermophysical properties and be very efficient as a heat transfer fluid in the liquid state, however the biggest impediment to their widespread use is their high melting points. As long as proper precautions are taken to ensure the salt does not freeze, they offer an excellent high temperature HTF option that extends beyond the range of hydrocarbon fluids.

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DID YOU KNOW?

The Great Salt Lake is four to five times saltier than the ocean (or six to eight times saltier than the ocean in drought years!) and contains about 4.5 billion tons of salt. Utah makes about $1.3 billion every year from The Great Salt Lake ($1.1 billion in mineral extraction, $136 million from recreation and $57 million from harvest of brine shrimp.)

Research Spotlight

Should Glycol Be Stored Outside?

Learn the effect sunlight can have on your glycol

by Lauren Fotiades, Quality Control Supervisor, Dynalene Inc.

Introduction

We all know the UV radiation from the sun is powerful. It can burn us or give us cancer. It can bleach the colors out of objects. It can weaken cloth and plastics left outdoors. So what can it do to heat transfer fluids? If you, for example, store a container of propylene glycol outdoors until you need it, will that fluid remain unchanged?

Sunlight reaches the earth in three wavelength categories: infrared (700nm to 1mm), visible (380nm to 700nm), and ultraviolet (100nm to 400nm). Ultraviolet light is typically broken down into three further regions: UVC (100nm to 280nm), UVB (280nm to 315nm), and UVA (315nm to 400nm). UVC radiation is almost completely absorbed by earth's atmosphere, as is about 95% of UVB. About 30% of total solar radiation is absorbed or scattered by the earth's atmosphere, as shown in Figure 1.

It is well known that the UV radiation that does reach earth is capable of damaging materials both natural and artificial. Lignocellulosic compounds will yellow with UV exposure, and proteins in wool or biopolymers can undergo main-chain scission, altering their molecular weight. Plastics exposed to UV radiation may change color, become brittle, or suffer other degradation to physical properties – both scission and cross-linking are possible, depending on the material. [1] UV radiation can cause mutagenic and cytotoxic damage to DNA in everything from bacteria to humans. [2]

Issues of space can lead companies to store chemicals outdoors, and heat transfer fluids are no exception. While in the past storage tanks were often made of metal, the improvement in polymer technology over the last few decades has led to a proliferation of plastic tank options for outdoor storage. However, where metal will not allow any solar radiation to pass through to the fluid inside, plastic will not necessarily be proof against it. This makes it important to understand the potential for glycol to be affected by UV radiation.

There is not much scholarly research available on the UV degradation of heat transfer fluid glycols. In this article, we look at the results of an experiment testing the results of sun exposure on a number of different Dynalene glycols.

Experimental Description

For these experiments, we used ethylene glycol (EG), propylene glycol (PG) USP grade, propylene glycol technical grade, and 1,3-propanediol (bioglycol), a bio-derived propylene glycol isomer. We used each glycol both as received from the manufacturer (raw) and also blended with Dynalene's PE-1 inhibitor. The exception was bioglycol, which we only used in the uninhibited state. Distilled water, both inhibited and uninhibited, stood as the control fluid.

We used two different types of containers for this experiment. The first was translucent (not transparent) 500mL HDPE bottles, the same material that makes up our storage pails and totes. Thin PTFE film replaced the standard cap liners, and the threads were coated with DuPont Severe Service Grease with Teflon before closing the caps. The second type was 150mL quartz vessels with quartz lids from AdValue Technology. The rims of each lid were coated with the Teflon grease before closing, and electrical tape was wrapped around the rim to complete the seal.

Table 1 contains a summary of each glycol used, along with the container type and location. HDPE bottles were either left fully exposed to all sunlight, placed under a basic glass pet store aquarium coated with UV film by TechnologyLK (Figure 2), or completely covered with aluminum foil. The quartz vessels were left fully exposed. We kept control samples in a closed cabinet inside the laboratory. Each sample was run in triplicate for a total of 75 samples, not counting the controls.

A plywood sheet placed at an angle held the full exposure and foil-covered samples together, with wooden tracks to keep them in place (Figure 3). After placing the foil bottles, we spray painted their upper surfaces with matte grey paint. This setup remained outside for eighteen weeks, from late spring through early fall. The aquarium setup was placed right next to this plywood sheet and stayed outside for sixteen weeks.

At several- week intervals, we opened the bottles to measure the temperature of the fluid, check for the development of any odor, and pull a small sample on which to run gas chromatography (GC) headspace analysis. Our GC is an Agilent 7890A with an attached 5975C VL mass spectrometry detector (MSD).

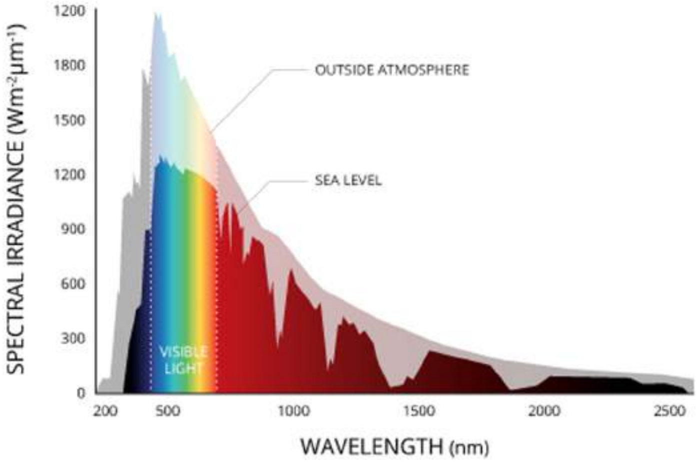


Figure 1: Solar radiation outside the atmosphere vs what reaches sea level [3].

Table 1: Glycol distribution throughout the experiment.

	HDPE Sun	HDPE Foil	HDPE Aquarium	Quartz Sun	Lab
Raw PG, USP grade	x	x			x
Raw PG, technical grade	x	x	x	x	x
Raw EG	x	x			x
Raw Bio	x	x			x
Distilled Water	x	x			x
Raw PG, tech, argon purge	x	x			
Inhibited PG, USP grade	x	x			x
Inhibited PG, technical grade	x	x	x		x
Inhibited EG	x	x			x
Inhibited Water	x	x			x
Inhibited PG tech, argon purge	x	x			

Results of Experiment

We tested the samples simply by having various members of the lab team open the bottles and see if they could smell anything. While "smell" is a rather subjective parameter, the compounds formed over the course of this experiment could be easily detected by anybody exposed to the

Figure 2: Aquarium sample setup

Figure 3: Fully exposed and foil-protected HDPE bottles on plywood rack.

Table 2: Odor test results

Location	Sample	1st check	2nd check	3rd check	4th check
Full Sun HDPE	Raw PG USP	Y	Y	Y	Y
	Raw PG Tech	Y	Y	Y	Y
	Raw PG Tech, Argon	Y	Y	n/a	n/a
	Raw EG	Y	Y	Y	Y
	Distilled Water	N	N	N	N
	Raw Bio	N	N	N	N
	Inhibited PG USP	N	Y	Y	Y
	Inhibited PG tech	mixed	Y	Y	Y
	Inhibited EG	N	N	N	N
	Inhibited Water	N	N	N	N
Foil HDPE	Raw PG USP	N	N	N	N
	Raw PG Tech	N	N	N	N
	Raw PG Tech, Argon	N	N	n/a	n/a
	Raw EG	N	N	N	N
	Distilled Water	N	N	N	N
	Raw Bio	N	N	N	N
	Inhibited PG USP	N	N	N	N
	Inhibited PG tech	N	N	N	N
	Inhibited EG	N	N	N	N
	Inhibited Water	N	N	N	N
Aquarium HDPE	Raw PG Tech	N	N	N	n/a
	Inhibited PG tech	N	N	N	n/a
Full Sun Quartz	Raw PG Tech	Y	Y	n/a	n/a

degraded samples. Indeed, by the end of the experiment, simply opening the raw PG sample bottles indoors would cause the smell to spread through the entire lab. Table 2 shows these results in a simple yes/no format.

If we look at these results further, we find that there were a limited number of samples that ended up with a significant odor by the end of the experiment. The raw PGs, both tech and USP grades, and raw EG all have a strong, solvent-like smell. The two PG types smell the same, while the EG is distinctly different. The inhibited PGs, again both tech and USP grades, also have a strong smell, but this is a sweeter, almost fruity or floral, smell, unlike the smell of any of the raw glycols.

We performed GC testing on each sample in two ways. First, each sample was run with the GC hooked up to a flame ionization detector (FID), for the best possible peak resolution and separation, in order to see as many compounds as we could get out of the headspace. Once any new, non-glycol peaks were uncovered this way, we switched to the MSD to try to determine the identity of the unknown compounds.

We chose to do headspace analysis in order to avoid dealing with a large glycol peak that could potentially swamp contaminant peaks. Since some glycols had already developed a strong odor by four weeks, we knew the breakdown products were volatiles and thus should be easy to capture in the sample headspace. Additionally, injecting only vapor rather than liquid onto the column helps to prevent carryover from run to run, which was beneficial considering the large number of samples tested.

The FID results of the UV vs non-UV samples are striking. The samples that were not exposed to UV rays (the foil-wrapped HDPE, the HDPE protected by the glass and UV film combination) give chromatograms that look very similar to the control samples (Figure 4). Some of the fully exposed glycols have several strong new peaks, increasing in size the longer the samples remained exposed.

Interestingly, not all of the fluids that were fully exposed to sunlight showed new compounds developing. The most affected samples were the raw propylene glycols. Both tech and USP grade glycols show several new peaks by the end of the experiment, with the most dramatic peaks at around 9 and 20 minutes, along with several other smaller peaks. Raw ethylene glycol has new peaks at 15 and 17 minutes. Raw bioglycol has only a few small peaks (about 9 minutes) that don't come close in height or area to the glycol peak itself. For both EG and PG the contaminant peaks dwarf the original glycol peak by an order of magnitude (Figure 5).

The inhibited glycols have less going on in their GC curves (Figure 6). Inhibited PG shows some of the same new peaks as raw PG, but not others. Inhibited EG does not show new peak development at all.

If we look at the GC/MS results we begin to see the source of the difference in smell noted previously. In all of the raw glycols with strong odors, the MS software identified (with > 80% probability) various dioxolane compounds, as shown

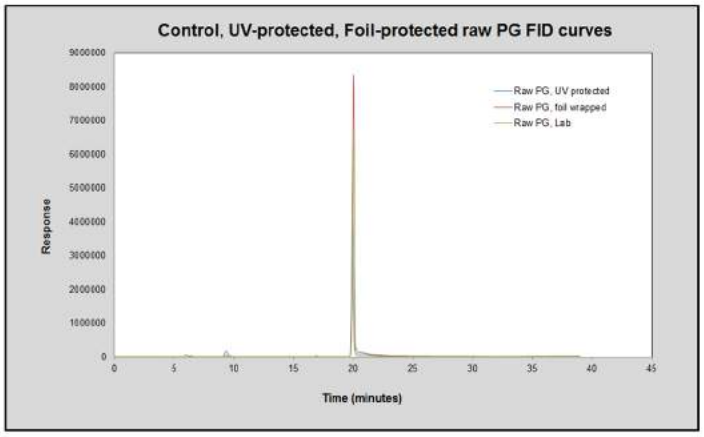


Figure 4: Control, UV-protected, Foil-protected raw PG FID curves

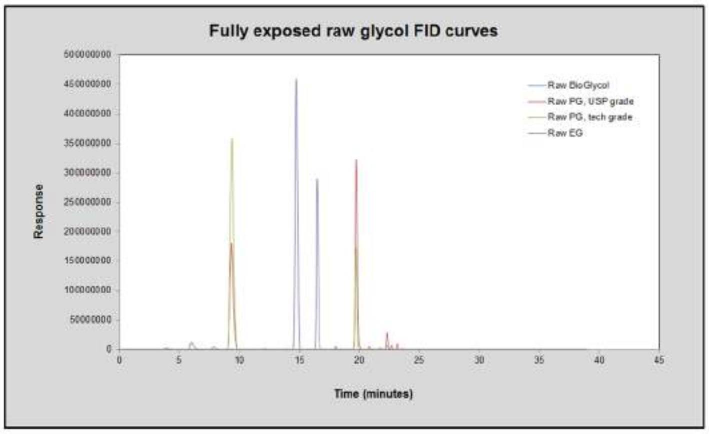


Figure 5: fully exposed raw glycol FID curves

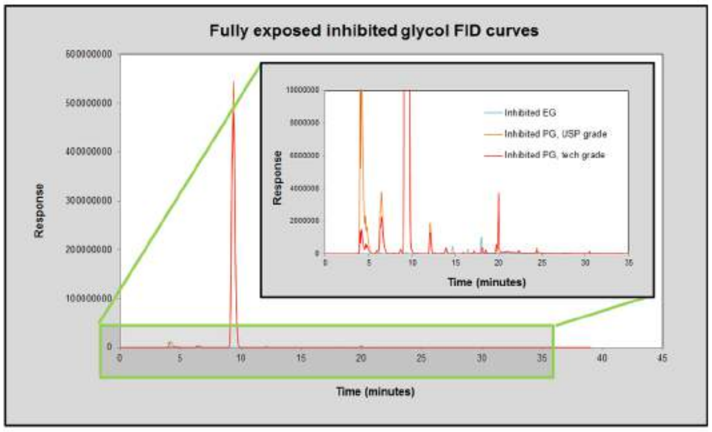
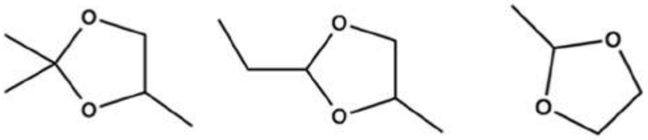


Figure 6: Fully exposed inhibited glycol FID curves



2,2,4-trimethyl-1,3-dioxolane

2-ethyl-4-methyl-1,3-dioxolane

2-methyl-1,3-dioxolane

Figure 7: Dioxolanes found in degraded glycols

in Figure 7. In tech and USP grade PG, the primary dioxolane formed is 2,2,4-trimethyl-1,3-dioxolane. 2-ethyl-4-methyl-1,3-dioxolane also appears, but in smaller quantities (based on MS peak area). In EG, we see 2-methyl-1,3-dioxolane. No dioxolanes of any kind were found in the MS results of the inhibited glycols. 1-propanol was found in those samples and could be a possibility for the source of the inhibited PG smell.

Discussion

Comparing the results of the fully exposed HDPE bottles with the foil-wrapped bottles shows that the formation of these compounds must be due to some sort of solar radiation. We ruled out heat as the cause of degradation because the measurements we took each month showed only a few degrees of variation across each glycol set, and the foil samples consistently had a higher average temperature than the non-foil samples (anywhere from 1 to 5°C difference). If heat by itself were capable of causing the breakdown, we would have expected to see it occur in the foil samples as well. Since the aluminum foil would have blocked any UV, visible, or IR radiation from reaching the glycol, we can infer that it must be one or more of those three that caused the breakdown in the non-foil samples.

The results of the aquarium experiment back this up. The average temperature of these samples was around 5 to 10°C hotter than the foil-covered samples, probably due to a greenhouse effect from the aquarium glass. Again, if heat alone were the culprit, we should have seen degradation occur in these samples as well as the fully-exposed ones, and it did not. This also narrows the field of possible culprits, as the aquarium did nothing to prevent visible light from passing through, but the combination of glass and UV-blocking film significantly decreased the amount of UV light that could reach the fluids.

It is true that we don't know what effect, if any, the glass and UV-film had on the passage of IR radiation, so that experiment does not officially rule it out as a possible cause of glycol breakdown. However, it seems intuitively unlikely. A Google search for "UV degradation" returned over 30 million hits, with the links on the first few pages discussing the breakdown of specific materials, especially plastics, and ways to prevent it. A search for "IR degradation" also returned over 30 million hits, but the first few pages return studies using IR spectroscopy to monitor degradation from other causes, and "insulation resistance" degradation. While we may at some point be able to design an experiment to isolate the effect of IR radiation on glycol, it does not seem as though that would be a particularly effective use of resources.

Conclusion

Uninhibited propylene or ethylene glycol can be quickly degraded by UV radiation. Some of the breakdown products have distinctive, unpleasant odors. Though we don't yet know if these compounds have any impact on the heat transfer performance of the glycols, they certainly become unpleasant to use due to the smell. Ideally, these fluids should be stored indoors. If they must be kept outside, they should be kept in containers that cannot be penetrated by UV rays.

We would like to do further research into this topic, looking at possible mechanisms for the dioxolane formations. It would of course be valuable to learn what impact, if any, these breakdown products have on the glycols' physical properties as heat transfer fluids. Analyzing the difference between inhibited and non-inhibited glycols is another topic of interest.

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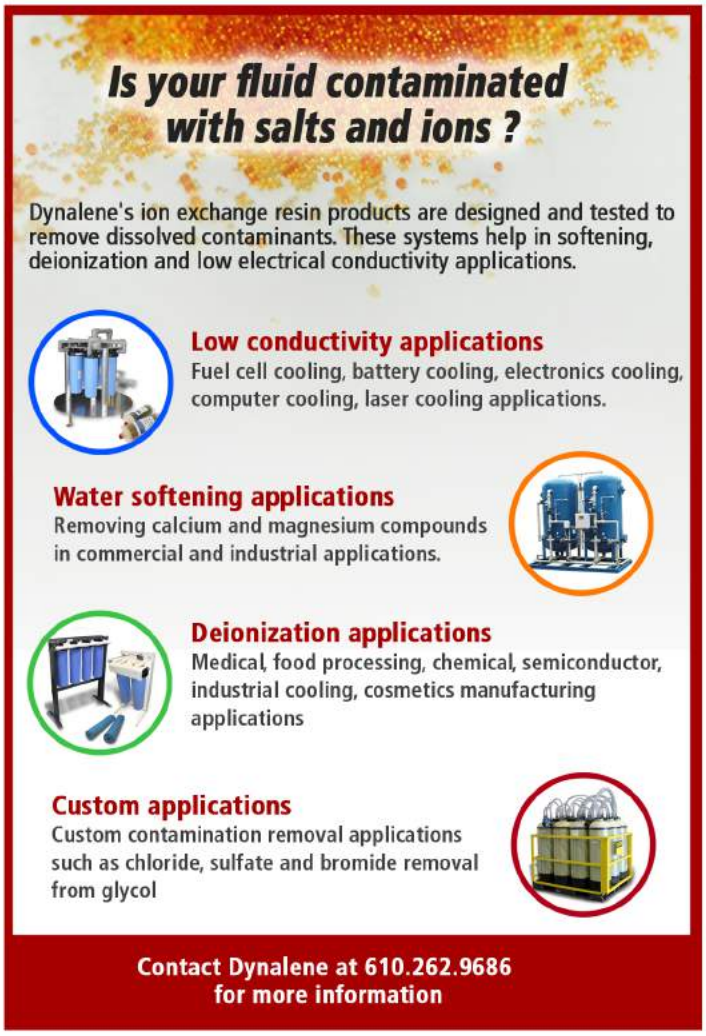
3. Photo credit: Fundamentals of Environmental Measurements/Fondriest Environmental

DID YOU KNOW?

Bees can see ultraviolet light, as they use it to direct themselves to pollen when the UV radiation is reflected off of flower petals. Bees play a critical role in the ecosystem by facilitating pollination. It has often been said that bees are responsible for one out of every three bites of food we eat.



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GLYCOL DO'S & DON'TS

- Use Deionized water when blending glycols to help maintain low corrosion rates.
- Check the pH and the concentration of the glycol regularly. This is a great indicator of the quality of the glycol and shows if something is degrading or diluting the glycol.
- Flush the system before replacing with new glycol to keep the system free of particles that could cause clogging.
- Use filtration when possible. Adding filtration to your system will help keep it clean and improve heat transfer.

- Do not use galvanized metals. These will actually have higher corrosion rates when used with corrosion inhibitor.
- Do not use aluminum unless consulting with Dynalene first. Glycol will work well with aluminum, but specific inhibitor must be used.
- Do not store glycol in sunlight. Sunlight will cause glycol to degrade, increasing odor and decreasing product quality.
- Do not mix with automotive antifreeze. The inhibitors are different and could interact with Dynalene inhibitors in a negative way.

ASK THE EXPERTS

Q. What chemistries are commonly used as heat transfer fluids?

A. Traditional HTFs can be made from several different base chemistries such as water, glycol/water solutions, hydrocarbons, silicones, fluorocarbons, salt /water solutions and pure salts. In the category of glycols, there are several types such as propylene glycol, ethylene glycol, triethylene glycol and 1, 3-propanediol. Similarly, hydrocarbon-based HTFs come in several categories such as aromatic, paraffinic/mineral oil and other synthetic compounds.

Q. How must I prepare my system before installing a heat transfer fluid?

A. For new/existing systems, the cleaning procedure is to flush the system with deionized (DI) water or a DI water/detergent mixture to remove all fluids, dirt and debris. Then drain, dry and fill with the desired fluid. Multiple flushes may be required before the drying step.

Q. Must I have a filter on my system?

A. It is always good to have a filtration system (in-line or slip-stream) in the heat transfer fluid circulating loop. Fluids over time accumulate debris, corrosion products and precipitates due to contamination, system corrosion or fluid degradation. If the fluid is not filtered then it may lead to system clogging, reduction in heat transfer rate, and excessive corrosion.

Q. What materials of construction should I use for my system?

A. When selecting materials of construction there are two factors to consider. The temperature range the fluid will be used in and the chemistry chosen for the fluid. With this information all compatible materials can be selected by contacting the fluid manufacturer.

Q. How do I know when to replace my fluid?

A. There are several factors to consider in determining the health of a fluid and if it needs to be replaced. The most effective tool is analyzing a sample. However, simple indicators are changes in color, pH, odor or efficiency. Any or all of these changes means the fluid needs to be replaced or adjusted.

Q. What is the difference between inhibited and un-inhibited glycol?

A. Inhibited glycol contains an additive package that prevents corrosion and stabilizes the pH of the fluid. Un-inhibited or raw glycol is pure glycol with no additives.

Q. I have a Glycol in a system but I am not sure if it is Propylene or Ethylene glycol. How can I check this in the field?

A. It is really difficult to determine the type of glycol in the field. The best way is to look at the density of the product; however this might not be accurate. When trying to determine the type of glycol it is always best to send a sample or to contact a manufacturer.

Q. Can I use water as a heat transfer fluid?

A. Water is the best heat transfer fluid available within the temperature range of 32°F to 212°F. If your system requires a broader temperature range you will need to either add a glycol to the water (to increase the range) or change to a non-water based fluid.

Q. Who can I call for disposal of a heat transfer fluid?

A. Disposal of heat transfer fluid depends on the chemistry of the coolant and the contaminants that might be in the fluid. Each fluid chemistry has its own disposal method. Some can be sent down the drain while others must be disposed of by a chemical treatment company. If you are unsure of how to dispose of a heat transfer fluid you should consult with a chemical disposal company or a manufacturer.

Q. What damage will happen to the system or the fluid if the system is not used for an extended period of time and the fluid sits stagnant?

A. In most cases, systems will be stable with heat transfer fluids for an extended period of time; however it is always good to maintain the system. Stagnant systems that contain air might tend to have corrosion in the air spaces. Also, stagnation can lead to leaking seals and pumps. Once again this will depend primarily on the system and the heat transfer fluid.

Q. How does viscosity affect the performance of a heat transfer fluid and are low viscosity fluids more likely to leak?

A. The performance of a heat transfer fluid is dictated by four basic thermophysical properties: viscosity, thermal conductivity, specific heat and density. When pumping a fluid it is important to maintain turbulent flow to create excellent heat transfer and this is achieved using low viscosity fluids. However, viscosity is not linked to the leak potential of a fluid; that issue is related to the surface tension of a fluid. The lower the surface tension the more difficult the fluid is to contain within a system.

Q. What determines if a system is considered open or closed?

A. A closed system is air tight and capable of building pressure thus protecting the fluid and system from oxygen and eliminating the risk of corrosion. An open system will expose the heat transfer fluid and the system to oxygen in the atmosphere and increase the potential for corrosion.

Q. Can I use a fluid above its flash point?

A. In closed air tight systems it is not uncommon to use a heat transfer fluid above its flash point. Unlike an open system, which allows the vapors to escape into the atmosphere and potentially be exposed to an ignition source, a closed system contains the vapors and eliminates the risk of exposure within the system.

DID YOU KNOW?

Propylene Glycol as a food ingredient?

Propylene glycol gives **dry cake mix** its soft, powdery texture. Without the additive, the mixture would crystallize.

Nearly all **carbonated beverages** contain propylene glycol, which inhibits bacteria growth, ensuring optimal flavor on consumption and to evenly distribute fatty acids for flavor consistency.

Propylene glycol is in alginate, the most common type of stabilizer used in **icing for cake** and cookies. Food stabilizers rely on propylene glycol to maintain uniformity, which is why the icing on the cake has a fluffy consistency.

Antifreeze and **ice cream**? Seems logical. Propylene glycol prevents ice crystals from ruining the dessert's creaminess and helps maintain its shape.

Some forms of **kibble and treats** benefit from humectants, which absorb moisture. Humectants contain propylene glycol, keeping dog food and treats crunchy.

Why freeze your food?

Frozen foods do not require any added preservatives to keep them safe and consumable, because microbes cannot grow on any food that is at a temperature less than 0°F. The microbes don't die at that temperature, but they stop multiplying. Be careful when you unfreeze food; microbes will instantly start growing as they do on unfrozen food, so it's best to handle thawing food as you would fresh food. Contrary to the popular belief, freezing food does not remove any nutrients. You don't need to be afraid of freezer burn or color changes in your properly frozen food. Freezer burn is just the result of air hitting frozen food and allowing the ice to sublimate; other color changes can be blamed on long freezing times or poor packaging. It might look gross, but if your frozen food has maintained a proper temperature, it's fine to eat.

Source: www.mentalfloss.com

Research Spotlight

Removing moisture from cryogenic heat transfer fluids

by Bojanna Shantheyanda, Research Engineer, Dynalene Inc.
Stephen N. Csernica, Ph.D, Research Asistant, Lehigh University

Background

Moisture or water is an integral part of the environment we live in. It exists in hydrocarbon, silicone and other non aqueous heat transfer fluids essentially the same way it exists in the atmosphere. It starts off in the dissolved phase, dispersed throughout the heat transfer fluid with no visual traces. However, once the saturation point is exceeded, moisture is typically present in the emulsified phase, creating a milkiness in the heat transfer fluid. As water is typically heavier than the heat transfer fluids, when sufficient water exists, free water will settle below, at the bottom of sumps and reservoirs.

The point at which a heat transfer fluid contains the maximum amount of dissolved water is termed the saturation point. The saturation point is dependent on the heat transfer fluid temperature, age and additive composition. The higher the temperature, the higher the saturation point and hence more water can be held in solution in the dissolved phase. Similarly, the older the oil, the higher the level of water that can be dissolved. This is due to polar by-products of oxidation in the heat transfer fluid, which can assist holding on to the water molecules and keeping them in solution.

Maintaining low moisture content in a heat transfer fluid for low and high temperature applications is crucial to attaining or maintaining designed system efficiency. Moisture, which can enter into a fluid system due to improper seals, system leaks, improper handling or malfunctioning driers can cause system failures. Moisture in hydrocarbons operated below the freezing point of water can form ice crystals that may clog valves and form a coating on heat exchanger surfaces, thereby affecting the operational efficiency of the refrigeration system. In high temperature applications, the presence of moisture can cause steam, which may increase internal pressure, therefore resulting in an eruption. Additionally, moisture in the hydrocarbon and silicone based heat transfer fluid causes formation of acids, resulting in metal corrosion and chemical damage to the gasket materials.

In addition to using desiccating equipment, improved seal technology as well as training of the maintenance and operations personnel about the correct practices to use a heat transfer fluid are essential. Other methods such as inexpensive gravity separation and complex vacuum dehydration are also utilized to remove moisture from hydrocarbons [1].

Different types of moisture adsorption media are used to remove water from heat transfer fluids. Molecular sieves, a material with pores with a diameter less than 2 nm (microporous) to 50 nm (macroporous) are commonly used desiccants to adsorb moisture [2, 3]. Various aluminosilicate-based molecular sieves are commonly used in commercial dehydration processes and are capable of adsorbing water at temperatures as high as 90°C [4]. In addition, salts such as calcium sulfate is used to dehydrate both liquids and gases.

In our present work, the moisture solubility characteristic of a heat transfer fluid will be studied. Later, microporous molecular sieves will be used to study the adsorption rate of the desiccant in the heat transfer fluid at various temperatures. The primary analytical instrument for water determination is a Karl Fischer coulometric titrator.

In order to study the moisture solubility and the moisture adsorption characteristics of the molecular sieves at various temperatures, Dynalene MV was selected as the model heat transfer fluid. Dynalene MV is an aromatic hydrocarbon based heat transfer fluid with a very low operating temperature range, down to -112°C (-170°F). The

Table 1: Critical diameter of water, benzene, and benzene-derivatives [4].

Molecular	Critical Diameter (Å)
Water	3.2
Benzene	6.7
Toluene	6.7
***p*-xylene**	6.7
***m*-xylene**	7.1

170°C. The freezing temperature of Dynalene MV is significantly lower than that of water; as a result, any residual water remaining in the fluid can form ice at low temperatures and reduce its performance.

Molecular sieves operate by excluding all molecules with diameters greater than the sieve pore diameter of their internal structure. Thus, the selection of an ideal molecular sieve is dependent on the size of the molecules in the liquid solution. The critical diameter of water, benzene, and some benzene-derivatives are shown in Table 1.

Experiment

Water Solubility Experiment:

Sample vials each containing the heat transfer fluid and ultra-pure water at a 5:1 volumetric ratio were prepared and vigorously mixed. The samples were allowed to equilibrate for 24 hours. At equilibrium, the two-component system separated into two distinct phases: the Dynalene MV rich phase existed as the top phase while the water existed as the bottom phase. An example of this separation is shown in Figure 1. The top phase of the mixture, considered to be moisture saturated Dynalene MV, was decanted and used for later experiments. 10 grams of saturated Dynalene MV was placed at various temperatures for 24 hours to determine the moisture solubility at those temperatures. The temperatures used for this testing were: -58°C, obtained in a low-temperature VWR freezer; -20°C, obtained in a So-low FDC-4000 standard freezer; 0°C, obtained in a VWR standard refrigerator; and 23°C, obtained at room temperature. For the lowest temperature samples the density of the ice and the Dynalene MV are very close. Therefore, as the sample cools, any water previously saturated in the Dynalene MV phase will precipitate out in the form of ice. However, since the densities are so close, there is very little driving force for the ice to settle to the bottom phase. Thus, when samples were withdrawn from the vials stored in the freezers and the refrigerator, a 0.45 µm PTFE filter was used to remove the largest of the ice crystals. Therefore, only the Dynalene MV with liquid water dissolved in it was withdrawn and tested to determine moisture saturation. The Dynalene MV sample was massed and injected into the Karl Fischer coulometric titrator for moisture content analysis.

Figure 1: Dynalene MV-water two-component system in equilibrium at T = 23°C.

Table 2: Properties and characteristics of molecular sieves [4].

Type	Bead Mesh Size	Pore Diameter (Å)	Bulk Density (kg m^{-3})	Equilibrium H_2O Capacity (wt. %)	Regeneration Temperature (°C)	Max ΔH_{ads} (kJ/kg)
4Å	4-8	4	720	= 22	200-315	4186

Sigma-Aldrich 4A metal aluminosilicate-based molecular sieves were used for the adsorption of water in saturated solution of Dynalene MV at various temperatures. The properties of the 4Å molecular sieves are listed in Table 2.

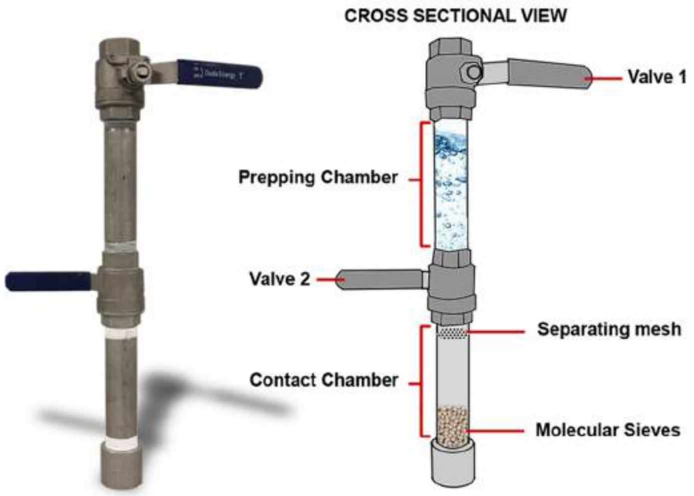

Figure 2: The experimental setup used for moisture adsorption experiment.

Figure 2 shows the experimental setup used to determine the moisture adsorption characteristics of the molecular sieves in Dynalene MV at various temperatures. The setup consists of the ‘Prepping Chamber’ and the ‘Contact Chamber’. 80 grams of Dynalene MV heat transfer fluid was secured in the Prepping Chamber by closing Valve 1 and Valve 2. 16 grams of molecular sieve (which is 20 wt.% of the fluid test sample) was placed in the Contact Chamber. The fluid and the molecular sieve were separated by Valve 2, which was in the closed position before the start of the experiment. The experimental setup was placed in the test temperature for 1 hour. The temperatures used for this testing were: -80°C, -58°C, obtained in a low-temperature VWR freezer; -20°C, obtained in a So-low FDC-4000 standard freezer; 0°C, obtained in a VWR standard refrigerator; and 23°C, obtained at room temperature. Later, Valve 2 was opened to initiate contact between the molecular sieve and Dynalene MV heat transfer fluid in the ‘Contact Chamber’. The allowed contact time of the fluid with the molecular sieve was 30 minutes. The setup was tilted so that fluid flows to the Prepping Chamber and Valve 2 was turned to closed position. The ‘Separating Mesh’ in the Contact Chamber prevented the molecular sieve from entering the Prepping Chamber. The setup was warmed to room temperature using a heat gun before the fluid sample was collected for analysis. Valve 1 was opened and syringes were used to carefully withdraw the heat transfer fluid sample. The sample was massed and injected into the Karl Fischer coulometric titrator for moisture content analysis.

Table 3. Solubility of water in Dynalene MV as a function of temperature.

Temperature	H2O (ppm)				
	1	2	3	4	Average
-58°C	14.6	34.5	23.8	19.4	23.08
-18°C	58.8	57.0	55.0	52.8	55.9
-1°C	164.2	170.7	172.9	175.0	170.7
23°C	239.0	222.0	231.8	240.7	233.4

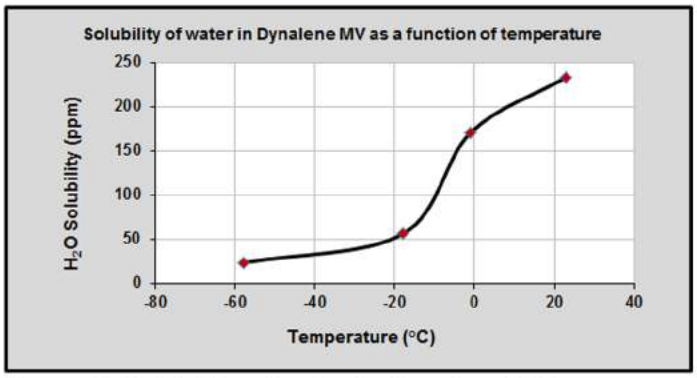

Figure 3: Solubility of water in Dynalene MV as a function of temperature.

Results and Discussion

Water Solubility Experiment

The results of the water solubility experiment are listed in Table 3 and Figure 3 shows the plotted results. The results from the water solubility experiment show that the solubility of water in Dynalene MV ranges from 35 ppm at T = -58°C up to 233 ppm at T = 23°C, indicating that the moisture saturation point of the Dynalene MV heat transfer fluid decreases at lower temperatures. With less water held in solution in the dissolved phase, the moisture in the fluid formed ice crystals which remained suspended in the fluid due to the similarity of the densities of ice and the heat transfer fluid at lower temperatures.

Moisture Adsorption Experiment

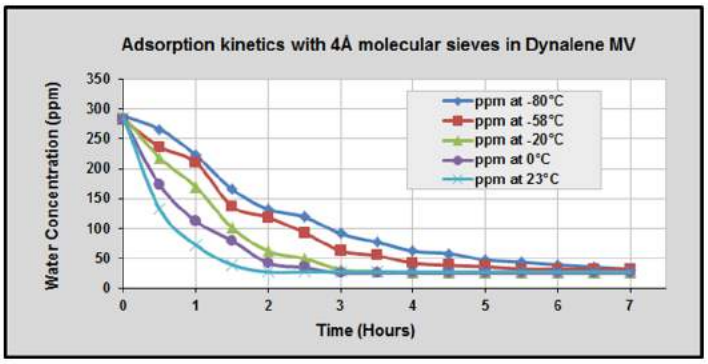

Figure 4: Effect of temperature on the adsorption kinetics with 4Å molecular sieves (20 wt.%).

Figure 4 shows the effect of temperature on the adsorption kinetics with the 4Å molecular sieves. A data point at t = 0 hour was taken at room temperature just before Dynalene MV was loaded to the moisture adsorption experimental setup. The result from the moisture adsorption experiment indicates that adsorption rates decrease significantly with the decrease in temperature. Though there is a change in adsorption kinetics with the decrease in fluid temperature, it is interesting to note that moisture content has been decreasing with time. Despite the slow adsorption kinetics, the equilibrium at T = -58°C was approximately equal to the equilibrium at T = 23°C. It should be noted, however, that the conditions inside the low temperature freezer are unfavorable for mixing as a stir plate cannot be inserted in the freezer. Adsorption is defined as the adhesion of atoms from a gas, liquid, or dissolved solid onto a surface. Thus, it is worth discussing the fact that the moisture adsorption experiments showed that all of the water was removed from the Dynalene MV solution, even the water existing as suspended ice particles. It is likely that the solid ice particles suspended in the Dynalene MV are not adsorbing on the adsorbent surface since adsorption can only occur for a dissolved solid species. Thus, when the soluble portion of the water in the low-temperature Dynalene MV is adsorbed, the concentration of soluble water in the MV decreases. This creates a concentration gradient between the solid ice particles suspended in the Dynalene MV (high water concentration) and the bulk of the Dynalene MV (low water concentration). This concentration gradient results in a driving force for the mass transfer of the solid ice to dissolve back into the bulk of the MV. The dissolved water now back in the bulk of the MV can then be adsorbed. This process continues until all of the water has been removed from the Dynalene MV heat transfer fluid.

Conclusion

The solubility of water in Dynalene MV heat transfer fluid ranges from 35 ppm at T = -58°C up to 233 ppm at T = 23°C. The moisture saturation level in the heat transfer fluid decreases with the decrease in temperature. The moistures adsorption kinetics of the 4A molecular sieve decreases significantly at low temperature. Despite the slow adsorption kinetics, it is determined that the equilibrium at T = -58°C was approximately equal to the equilibrium at T = 23°C. Therefore, molecular sieves can be used for moisture removal in low temperature heat transfer fluid applications.

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Review

ASTM corrosion testing for heat transfer fluids

by Sreya Dutta, Ph.D, Materials Scientist, Dynalene Inc.

Introduction:

Heat transfer fluids are used in various applications including HVAC, food and beverage, pharmaceuticals, wind tunnels, climactic chambers, cooling of reactors, etc. The desirable attributes of a heat transfer fluid are low toxicity, low viscosity, high specific heat, thermal conductivity, and excellent compatibility with the components of the system. For very high temperature applications, for example, solar power plants, steam turbines, etc., where most of the regular coolants will degrade, molten salt (e.g. nitrate, chloride) is generally an excellent candidate for such uses.

How corrosion affects your system

It is important that the heat transfer fluid is compatible with both metals and non-metals and provides long-term protection against corrosion. Degradation and corrosion of metal components can lead to pinholes, leakage in piping systems and reduction of strength in the pipes, joints, etc. Deposition of corrosion debris can cause blockage of the piping and tubes which would reduce the flow rate and cause pressure build-up in the system. Corrosion of the components can lead to loss of fluid, reduced efficiency, and breakdown of the system.

ASTM Corrosion Testing

The ASTM laboratory standard guide is very important for specifying standard design, dimension, equipment and methods used to conduct various experiments. These standards help the manufacturers, labs, and users identify good quality products. These test methods discuss all the factors that should be considered, calculated and reported. Various ASTM standard test methods discuss corrosion testing of metals and alloys in fluids and coolants on a lab scale. These tests provide a wealth of information about the compatibility of different metals with heat transfer fluids and different corrosion mechanisms that can affect the heat transfer systems.

Types of Corrosion Testing

The objective of any heat transfer testing is to simulate the conditions faced by metals and the fluid and to generate similar chemical and physical reactions. ASTM corrosion tests are designed to vary parameters that affect corrosion and measure values like weight loss, corrosion rate, etc. It is difficult to mimic the actual conditions in a heat transfer system and it is not possible for a single test to provide complete information: what is causing it, how serious is it, and if possible, how can it be prevented. Hence, a series of different tests is needed to provide a comprehensive understanding of the effect of corrosion parameters on the metals and their alloys. Table 1 shows the different corrosion types and the ASTM methods to study them. Some of the standard test methods are discussed below.

Table 1: Table showing different corrosion types and ASTM testing to evaluate them [1]

Type of Corrosion	ASTM Testing
General Corrosion	G31, D1384, G4, G5, G59
Pitting Corrosion	G46, G48, G150, G61, G100, F746
Crevice Corrosion	G48, G78, G61, G100, F746
Galvanic Corrosion	G71, G82, G116
Intergranular/Exfoliation Corrosion	G28, G34, G66, G67, G108, G110
Stress Corrosion Cracking	B858, G37, G41, G108
Cavitation and Erosion Corrosion	G32, D2809

Figure 1: ASTM D1384 glassware with coupons in glycol-water mixture.

General Corrosion

ASTM D 1384: Standard Test Method for Corrosion Test for Engine Coolants in Glassware [2].

Description: This method is a preliminary qualification of heat transfer fluids, to compare the corrosion rates of non-inhibited fluids to that of the inhibited ones. Six types of commonly used metal/alloy test coupons are immersed in a mixture of coolant and corrosive water. The test is run in triplicate at 88°C for 2 weeks with constant air purging. The samples are cleaned after testing. Sample weights are noted before and after testing. Mass loss and corrosion rate information are reported. Figure 1 shows glassware set-up with the metal coupons after a test was completed.

Advantages: Quick screening test to qualify heat transfer fluid for a system. Corrosion rate data can be obtained in a short period of time.

Disadvantages: Specific evaluation of localized corrosion, environmentally assisted cracking and effects of solution flow are not within the scope of this method. Operating temperatures can be higher than the test temperature [1]. Glassware testing is not adequate for aluminum alloys which are mostly attacked by pitting and crevice corrosion. Poor reproducibility of the solder corrosion data has been reported in various cases [3].

Pitting Corrosion / crevice corrosion

ASTM G48: Standard Test Methods for Pitting and Crevice Corrosion Resistance of Stainless Steels and Related Alloys by Use of Ferric Chloride Solution [4]

Description: G48 consists of six different types of testing for stainless steels and their alloys to evaluate their resistance against pitting/crevice corrosion. These tests rank the stainless steel and the alloys based on their resistance to initiation of pitting and crevice corrosion. Test methods A, C and E are used for pitting corrosion while B, D and F are used for crevice corrosion. Methods C to F are used to assess the critical temperature for pitting and crevice corrosion. A ferric chloride test solution is used to create a chloride-rich atmosphere. For crevice corrosion testing, fluorocarbon blocks are used as crevice formers. Figure 2a shows the pitting corrosion. Figure 2b shows the crevices corrosion [5].

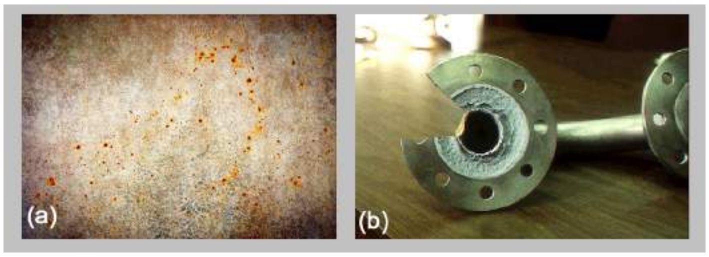

Figure 2: (a) pitting corrosion. (b) crevice corrosion [5].

Table 2: Table showing different corrosion types and ASTM testing to evaluate them [1]

Type of Test	Data
A, C, E	Visual examination of pits and measurement of pits using micrometer gage or microscope. Report maximum pit depth.
B, D, F	Visual examination of crevice attack and depth measurement of crevices. Report maximum and average depth of attack and number of attacked sites.
C, D, E, F	Report critical temperature for pitting/crevice corrosion.
A, B	Report specimen mass loss. Mass loss > 0.0001 g/cm^2 might indicate pitting or crevice corrosion.

ASTM G61: Standard Test Method for Conducting Cyclic Potentiodynamic Polarization Measurements for Localized Corrosion Susceptibility of Iron-, Nickel-, or Cobalt-Based Alloys [6].

Description: This method covers the cyclic potentiodynamic polarization test for determining localized corrosion attacks (pitting/crevice corrosion) in a chloride environment. In this corrosion testing, localized corrosion increases with an increase in anodic current. For metals with strong corrosion resistance, the potential at which the current increases is much higher. In cyclic polarization, a hysteresis loop is completed at a fixed scan rate. A more electropositive loop is an indicator that it is less likely for a localized attack to occur. This test method is followed for 304 stainless steel, iron, nickel and cobalt based alloys. This experiment is performed in sodium chloride solution at room temperature and the test coupons are polished before use. The test solution is purged to de-aerate it and the metal coupon is immersed at least for 1 hour to reach equilibrium. Open Circuit Potential is measured for about 1 hour followed by a positive scan at 0.6 V/h; the onset of corrosion is marked by a rapid increase in anodic current. When the current reaches 5 mA, reverse scan direction is initiated and continued until the hysteresis loop is closed or corrosion potential is reached. The results are plotted as potential vs. log (current), as shown in Figure 3 [6].

Advantages: This method provides rapid, qualitative assessment of localized corrosion attack on steel and related alloys. Along with critical pitting potential, this test method gives information regarding the corrosion rate, passive current and the transpassive region.

Disadvantages: Crevice corrosion under gaskets can lead to erroneous corrosion data. Due to the small coupon size, minor defects, scratches, etc. on the surface can cause problems with the reproducibility of the data. This method is good for general corrosion rates, but not so much for localized corrosion rates.

Galvanic Corrosion

ASTM G71: Standard Guide for Conducting and Evaluating Galvanic Corrosion Tests in Electrolytes [7].

Description: In this test, two dissimilar metals are placed in

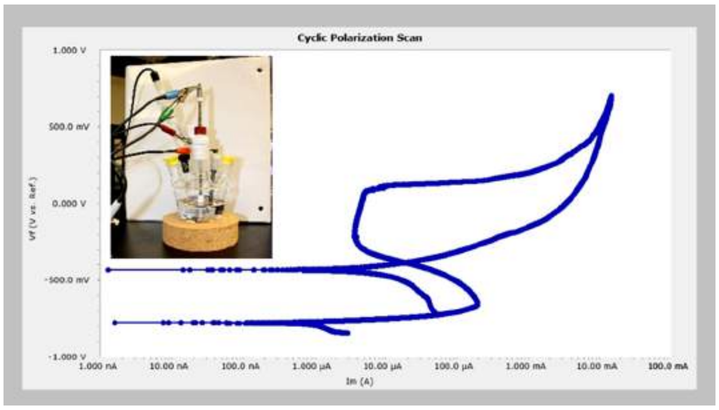

Figure 3: Cyclic polarization curve (hysteresis loop) Inset showing the electrochemical cell [8].

a fluid and the metals are connected by an electrical connection using a zero ammeter. This study helps to understand the behavior of different metals in a galvanic couple. Electrical connections are brazed/soldered to the metals. The galvanic metal couple is immersed in the electrolyte with no or limited flow. Galvanic current and potential are measured while exposed to the fluid and the corrosion rate is calculated from that. The direction of the current flow determines the anode material. After exposure to the fluid, the corrosion products are physically and chemically analyzed. Along with visual observations, galvanic mass loss studies are performed on the cleaned samples from which corrosion rate is calculated. Further analysis on the areas with localized corrosion attack, for example, pitting and crevice corrosion, should be performed and reported. A current increase of more than an order of magnitude is considered to be significant for corrosion to occur.

Advantages: This test method can be used both in the lab and in the field. Galvanic testing is an inexpensive method to screen coolants and inhibitors in a short period of time before using them in real systems. The testing helps to study the effect of various environmental parameters and how anode/cathode surface area can accelerate or impede corrosion.

Disadvantages: Short term testing such as galvanic electrochemical tests will not show the corrosion problems that might arise after an incubation period in a real system. Any contamination in the heat transfer system can become corrosive over time.

Cavitation and Erosion corrosion

ASTM D2809: Standard Test Method for Cavitation Corrosion and Erosion-Corrosion Characteristics of Aluminum Pumps with Engine Coolants [9]

Description: This method covers evaluation of cavitation and erosion corrosion of aluminum alloy-based pumps used for coolants and heat transfer fluids. An aqueous coolant solution is pumped at 113°C (or the maximum operating temperature) at 103 kPa pressure with a flow rate of 4600 r/min. The test coolant consists of the coolant mixture with corrosive water. The fluid and cleaning solutions are circulated using the Al pumps at various temperatures and flow rates for several hours. After testing, the extent of erosion corrosion and cavitation corrosion are rated based on a rating table available in the guide. An example of erosion corrosion of a pump impeller is shown in Figure 4.

Advantages: This is a simulated screening test to qualify a glycol-based heat transfer fluid. Proper control of the test variables helps to determine the effect of pump design, materials of construction, pump operating conditions, etc.

Disadvantages: Components present in the aluminum pump can also affect the corrosion behavior of the pump. The presence of copper can cause galvanic accelerated erosive attack. This test method is not recommended for two-phase coolant mixtures.


Figure 4: Pump erosion corrosion

Conclusions

The benefits of ASTM testing of heat transfer fluids are that they are accelerated and conducted on a smaller scale to accommodate them in a lab. Though these results cannot be directly applied to actual working systems, they provide important guidance on what materials to choose under what conditions. Testing consists of mostly glassware/immersion tests, electrochemical tests and simulated service testing. The tests help the users to screen the metals that are compatible with the heat transfer system and identify the inhibitor/additive package that provide sufficient corrosion protection. ASTM testing also provides valuable knowledge on how operating conditions, along with metal and fluid properties, can affect corrosion in a system.

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Research Spotlight

Chloride testing methods

by Tony Rivera, Research Associate, Dynalene Inc.

Background

Water is one of the best Heat Transfer Fluids (HTF) available, because it has excellent heat transfer properties, is nontoxic, and inexpensive. However, this HTF has its limits which is why a variety of HTFs have been designed. Water freezes at 0°C (32°F) and boils at 100°C (212°F), so using water alone as a HTF will confine a system to have to work in this temperature range. For this reason, science has expanded the product line of HTFs to work within the operating temperatures of individual systems (working below the freezing point and above the boiling point of water). The choice of HTFs available are brines (salt-water based), alcohols, glycols, and oils. Brines, alcohols, and glycols are usually mixed with water, which is why water quality plays an important role in an HTF performance. Low quality water can have a negative effect on the performance of HTFs and the system, such as scale build-up and corrosion.

Water with a high concentration of chloride ions can cause corrosion in systems. Even against high quality steel, which is alloyed to exhibit a greater resistance to corrosion, exposure to common oxidizing agents such as chlorides and oxygen will eventually lead to localized pitting (Figure 1). Aggressive pitting is an attribute of high chloride content. Chloride ions act as a catalyst, accelerating the oxidation of metal atoms. The film layer degrades ($Fe = Fe^{2+} + 2e$-) and a new electric potential develops. The new positive charges invite Cl^- ions to aggregate into a pit, which induces acidic conditions mechanistically favoring the rate of the reaction towards corrosion [1]. Iron exposed under these conditions will eventually corrode.

Due to the importance of chloride ions in water, tests have been developed and employed in the field to provide instant chloride measurements. A myriad of chloride tests exists such as strip tests [2], ion chromatography (IC) [3] and titrations. While some of these tests require little technical know-how and are easy to use, certain tests can only be done in a lab by trained professionals.

In this research, three tests are used with seven different water samples for chloride measurement: 1) Silver Nitrate titration; 2) Chloride test strips; and 3) Ion chromatography. This article presents the effectiveness of each method, and how the results may be interpreted.

Experimental Methods

Samples (S1-S7) from separate water sources were studied. City tap water was collected from multiple areas of Pennsylvania within the Lehigh valley for testing. In addition, deionized water (DI) and samples from a well and creek were included in the study. The experimental water samples are: S1) well water from Emaus, PA, S2) Monocacy Creek, Bethlehem, PA, S3) Deionized water from Dynalene's Whitehall, PA manufacturing facility, S4) tap water from city of Bethlehem, PA, S5) tap water from city of Allentown, PA, S6) tap water from Whitehall, PA, S7) tap water from Macungie, PA.

In this study, the chloride concentration in all samples (S1-S7) was measured using METTLER TOLEDO DM 141-SC conductivity probe and SympHony VWR meter (See Figure 2). Rather than observing color or pH change as an indicator of reaching the equivalence point, this test

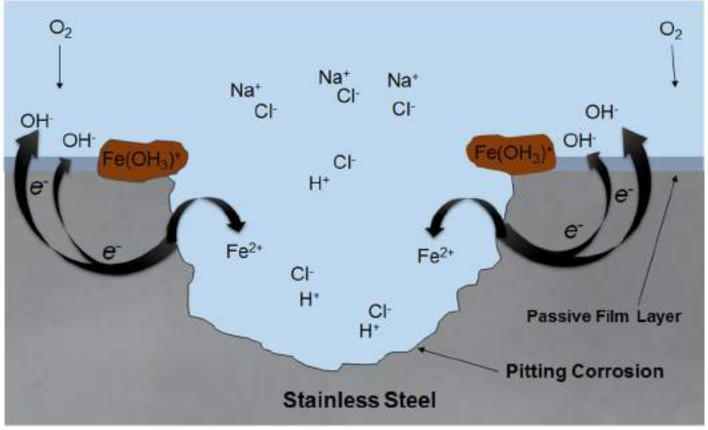

Figure 1: Redox reaction of chloride induced pitting corrosion

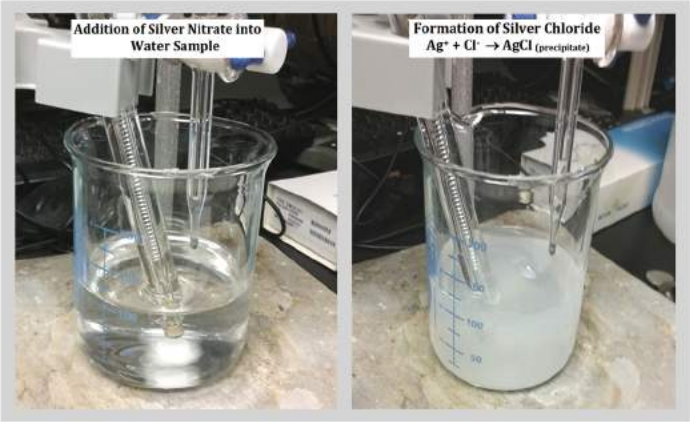

Figure 2: Silver nitrate titration test set-up

measures the electrical potential in millivolts (mV) until the concentration of both ions are at equilibrium. This creates a breakdown voltage where a jump in mV can be observed, indicating the equivalence point.

A 0.01 M $AgNO_3$ (aq) standard solution was used to titrate the test sample solution. 10 grams of sample water was added to 80 mL of a 1:1:1 (Acetone: Methanol: H2O) mole ratio mix. 5 mL of a 0.002 M NaCl (aq) solution was used to spike up the unknown. The unknown solution was very basic; therefore, a few aliquots of nitric acid (HNO_3 (aq)) were added to lower the pH level to near 3. A conductivity probe was placed into the unknown solution, and the electrical potential was measured over the period of titration. The volume of standard ($V_{standard}$) added to reach the equivalence point depends on the chloride content of the unknown. Higher chloride content requires longer titration periods in order to reach equilibrium. We can calculate the concentration of the unknown, using the equation below:

$$[Cl\text{-}] \text{ ppm} = \frac{[(V_{standard} - V_{blank}) \text{ x } 35.5 \text{ a.m.u of Cl x } 0.01 \text{ M } AgNO_3 \text{ x } 1000]}{10\text{g mass of sample}}$$

The second test method for Cl^- involved individual test strips (HACH® chloride strip, Loveland, Colorado) inserted in small glass vials containing 1-2 mL of water sample (Figure 3). Measurement units were recorded after the top mark on the strip turned black. The unit covered by the apex of the solvent front was converted into an approximate Cl- concentration based on the conversion chart provided by the manufacturer.

Chloride analysis of all samples were also conducted using a Metrohm 761 Compact Ion Chromatograph (Metrohm, Herisau, Switzerland). Sodium chloride solution was used to build a standard curve (5 to 100 ppm).

Results and Discussion

The chloride contents for all the seven water samples are presented in Table 1. Titrations yielded better results at concentrations ≥ 37 ppm. In the first column of Table 1, Allentown tap water yielded the highest results, with a chloride content of 66 ppm.

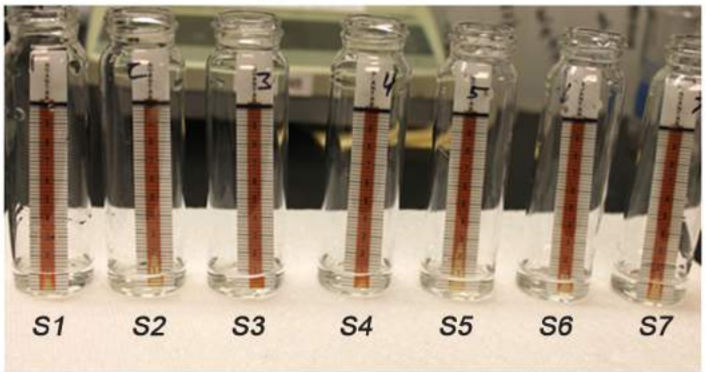

Figure 3: Chloride strip test set-up.

Table 1: Chloride test results for various water samples

		METHODS		
No.	**Water Samples**	**Titration-AgNo$_3$ (ppm)**	**Chloride Strips (ppm)**	**IC (ppm)**
S1	Emmaus Well Water	NA	NA	19
S2	Bethlehem, Monacacy Creek	37	49	42
S3	Deionized Water, Dynalene	NA	NA	NA
S4	Bethlehem Tap	NA	NA	10
S5	Allentown Tap	66	81	54
S6	Whitehall Tap	NA	35	33
S7	Macungie Tap	37	43	42

(NA) Not applicable to test. Measurement too low for testing.
The values reported are rounded to the tenth decimal place.

Strip testing was able to measure samples having a chloride content of ≥ 35 ppm. S1-Well, S3-DI, and S4-Bethlehem water exhibited very low levels of chloride that could not be measured using this method. Allentown tap water yielded the highest level of chloride, having a concentration of 81 ppm. In all results, strip measurements read higher values than measurements recorded by titration and IC.

S3-DI water was the only sample with chloride content too low (NA) for strip method to detect. The lowest level the IC was able to measure was in S4-Bethlehem tap water, measuring 10 ppm. The IC detected S5-Allentown to have the highest concentration of chloride. Measurements for IC were all lower than the values found in strip testing.

Which of the three test methods is the best? The answer is there is none. Choosing a method depends on the application of your aqueous mixture. There are pros and cons to each of these tests.

Chloride strips are the most user friendly, but at concentrations lower than 35 ppm this type of test will not give you an accurate measurement. As shown in Table 1, S1, S3, and S4, chloride levels are too low to detect. DI samples barely show any trace of chlorides because DI systems remove chloride ions from water by ion exchange. If you are extremely concerned about having the lowest level of chloride, DI is the best water source. Assuming that you have an idea of the chloride content of your water, use test strips. In this situation strip testing is the best choice, because in these lower limits the water is safe to use as a solvent. Strips are compact and can be easily stored, which is useful when traveling or in the field away from a laboratory. Moreover, they are easily disposable, and neither application nor clean up consumes much time. When measurements get ≥ 35 ppm, caution should be taken when using this method. Strips give you an approximate range; the measurement can be several ppm under or over the actual value. This is especially important for aqueous HTF mixtures with higher percentages of water such as glycol fluids. The glycol:H2O ratio mix varies in different applications, but for Dynalene glycol products engineers recommend that the final mixture be ≤ 25 ppm chloride to maintain the effectiveness of the inhibitors [4]. In a 50:50 mixture, the chloride content is diluted to half that of the source of water. Under 40 ppm, the water is safe to use, because in this 50:50 mix, the chloride content in the final solution is around 20 ppm. But for solutions that have a higher percentage of water in the final volume, the chloride content is less diluted and can reach unfavorable levels. Under these conditions, strips may not provide an accurate enough chloride concentration, and the water should be sent out for further testing.

For other applications that require higher degrees of accuracy, titration and IC are better choices. When working with HTF mixtures, with larger volume of water, knowing the exact level of chloride in that final solution will make a significant difference in how well the fluids will work. For example, if a strip test showed a chloride content of 35 ppm, a 50:50 mixture is safe to work with as the final solution would contain half the chloride content. The exact chloride content in this case is irrelevant, because the final solution would not be above the threshold chloride level of 25 ppm. But for other mixtures, strip testing is not sensitive enough to detect the maximum allowable chloride in the fluid. Take a look at Table 1, chloride levels for S5 are lower using the titration and IC method. An engineer wanting to dilute Dynalene glycol products, would not use Allentown tap water because the strip testing measures 81 ppm. For mixtures of a 50:50 ratio, 81 ppm becomes 40.5 ppm in the final solution. This would make the final solution well above the 25 ppm threshold. However, IC data shows a different picture. It tells us that the chloride content for S5 is actually closer to 54 ppm, which is slightly above 25 ppm after a 50:50 dilution.

Unlike IC, titrations have a limit. Titration is best at measuring higher chloride concentrations. In this experiment the test range threshold was ≥35 ppm. Results for samples having a concentration lower than 35ppm were not applicable (values were negative intergers). Best results are obtained when titrating solutions that initially start with a conductivity of 70 mV or higher. If samples are expected to have levels lower than 35 ppm, modification must be made to the test procedure so that initial conductivities are higher. In this experiment, the 0.002 M NaCl standard used was not strong enough. Four samples (S1, S3, S4, and S6) measured to be under 36 ppm by the IC method. Results for these samples collected after titration were not applicable, and did not match up to measurements found in the other two methods. These samples were too low to titrate, because the initial conductivity started below 50 mV. Although IC and titration testing get you closer to the amount of chloride in your solution, both tests are very technical and require a science background. Compared to the strip test, these test methods need to be done in a lab. Thereafter, results need to be configured, calculated, and quantified. Moreover, the time for each test will be much longer than strip testing; in some cases, titrating highly concentrated solutions can take up to an hour. Generally, the time is much longer to run titrations compared to both strip testing and IC.

Conclusion

In conclusion, chloride test strips should be the first measure of testing. If levels test high, don't risk operating the system or machinery under these conditions. Seek further testing via titration or IC, especially if you require chloride levels to be lower than 35 ppm. Don't assume that the HTF is free of chlorides. A more accurate measurement method is needed for very low levels and help keep your HTFs working efficiently.

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DID YOU KNOW?

- Some frogs have a chlorine compound in their skin that is a very strong painkiller, according to Los Alamos National Laboratory. The compound, called epibatidine, has no side effects in small doses, but in large doses, it is fatal to humans.
- Chlorine destroys ozone, contributing to the process of ozone depletion. In fact, one chlorine atom can destroy as many as 100,000 ozone molecules before it is removed from the stratosphere, according to the U.S. Environmental Protection Agency.
- Due to its toxic properties, chlorine was used as a chemical weapon during World War I, according to the Royal Society of Chemistry.

Source: www.livescience.com

Dynalene News

Dynalene receives STTR Phase I Award from the National Science Foundation

Dynalene is pleased to announce that it has been awarded a Phase I STTR grant from the National Science Foundation (NSF). This grant will help Dynalene to collaborate with Lehigh University researchers to develop a cost effective, high temperature molten salt heat transfer fluid and thermal storage medium for concentrated solar power (CSP) plants.

With the worldwide growing need for energy, alternative sources of energy have been the primary focus of research over the past few decades. Molten salt heat transfer fluid used in concentrated solar power plants is one sub-area of such research. The current NSF grant will help Dynalene to further advance its technology by development of high temperature (>650°C) molten chloride salts. These molten chloride salts display excellent high temperature properties compared to nitrate salts, but their extreme corrosive nature towards stainless steel has been a deterrent for their CSP applications.

With the current grant, Dynalene is developing inhibited molten chloride salts with excellent high temperature stability which would minimize corrosion by forming an in-situ, corrosion resistant, ceramic layer on the stainless steel surface. Use of the inhibited molten chloride salt would increase the efficiency of energy generation in solar power plants and provide potential cost savings by utilizing ubiquitous economical metals such as stainless steel in place of expensive superalloys.

Satish Mohapatra, Ph.D, President and CEO said, “We are excited to receive the support from the National Science Foundation for the development of inhibited, molten chloride salt. With the NSF Phase I funding, Dynalene looks forward to demonstrating the feasibility of a high temperature, inhibited molten salt, which can be used as a thermal storage and heat transfer media in the CSP plants, built with low cost stainless steel.”

Dynalene PG now has an NSF HT-1 rating

Dynalene’s propylene glycol chemistry, one of the leading products in the Dynalene fluid lineup, is now available with all food grade ingredients. We are proud to present two formulations, Dynalene PG-FG with corrosion inhibitors and raw PG-FG without inhibitors. These formulations have NSF HT-1 rating for incidental food contact while maintaining the same heat transfer efficiency as the original formula. With several formulations of propylene glycol heat transfer fluid (Dynalene PG, PG-XT, PG-V1, PG-V2, PG-FG and Raw PG) to choose from, process engineers and designers of heat transfer systems around the world now have a “go to” fluid for any food or non-food application. Visit Dynalene’s website to know more about the Dynalene PG-FG product or contact us today at 1.877.244.5525.

Dynalene increases its manufacturing capabilities

Whitehall, PA manufacturing facility: Dynalene recently added an additional building of 5000 sq.ft to its existing two buildings located at the Whitehall, PA location. This building is being used for storage, production and additional laboratory space. With more than 50 heat transfer fluids in Dynalene’s product offering, it is now able to manufacture and store up to 50,000 gallons of products in the expanded facility.

Chicago, IL manufacturing facility: Dynalene recently has expanded its manufacturing at the Chicago location to 20,000 gallons. This facility produces most of Dynalene’s glycol products and caters to the customers in the midwestern states.

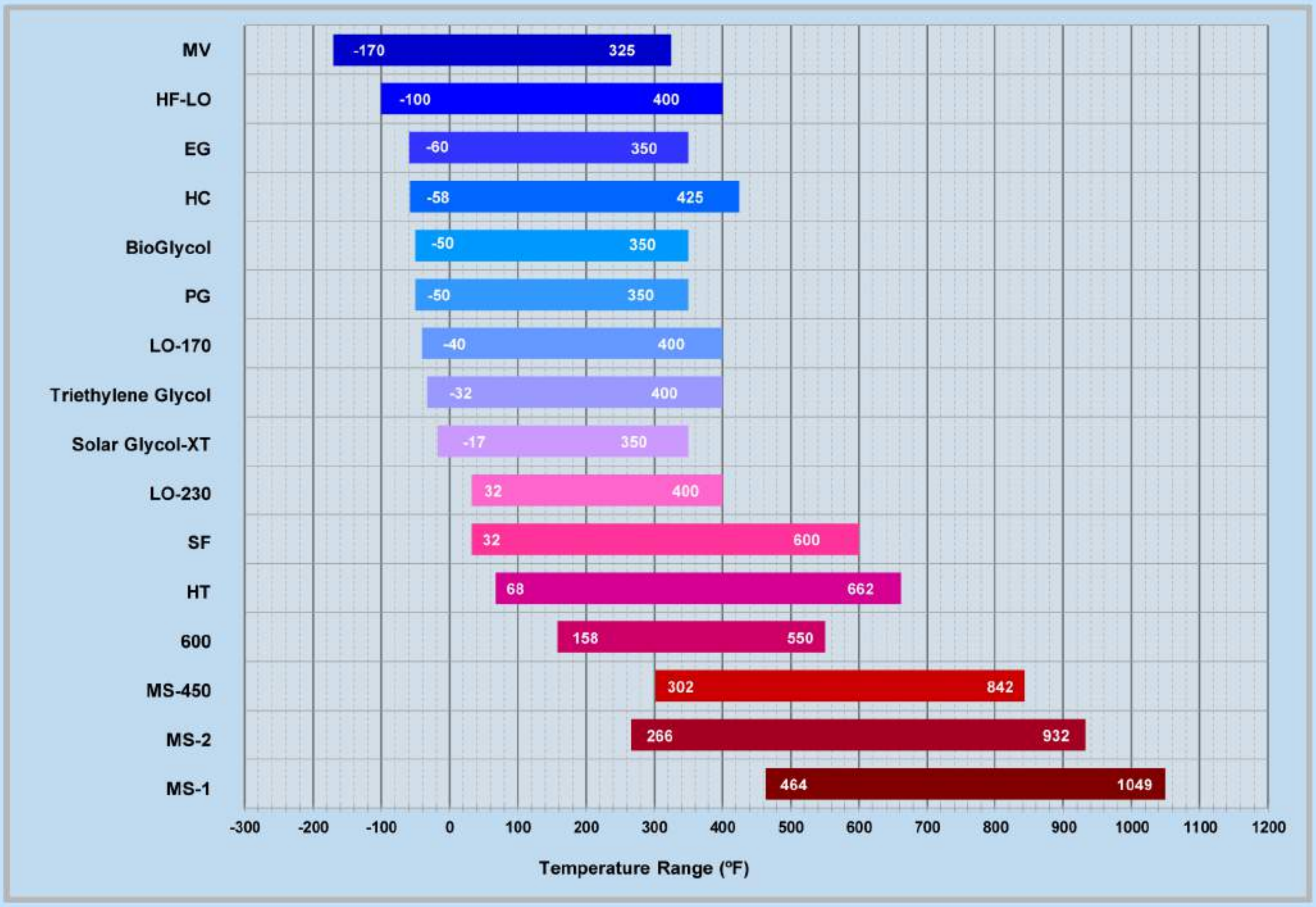
Salt Lake City, UT manufacturing facility: Established late 2015, this facility has completed one year of its operation and has produced in excess of 300,000 gallons of heat transfer fluids for customers in the western US and countries in Asia and Australia.

Dynalene launches chloride removal system

Dynalene’s new chloride removal system was recently commissioned at a customer site in California to remove a very high concentration of salts from its triethylene glycol (TEG) loop. It was able to reduce the chloride concentration from 2500 ppm to below 25 ppm in a system containing about 7500 gallons of fluid in less than 72 hours. This chloride removal system can be used in a variety of applications, such as natural gas storage, natural gas extraction, cooling loops in power plants and applications where heat transfer fluid is contaminated with an ionic compound (i.e. salts).

High chloride concentration in the heat transfer fluid can cause corrosion, system clogging with salts, reduction in heat transfer efficiency and can sometimes lead to system shutdowns. In addition to prolonging the life of the heat transfer fluid and protecting the capital equipments from corrosion, this system also helps to maintain a very low chloride level while the existing system is still in operation so that costly shutdowns and maintenance are avoided.

Temperature range reference chart for Dynalene heat transfer fluids



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Dynalene is an ISO-9001 certified leading manufacturer and supplier of heat transfer fluids. For the past 20 years, Dynalene's quality products have been used in a variety of applications by thousands of customers worldwide.