# FluidSolutions

JANUARY 2018

A HEAT TRANSFER FLUID MAGAZINE



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DYNALENE



#### Research in Heat Transfer: Why?

We have finally arrived at that time of the year when our "Fluid Solutions" magazine rolls out of the print shop and is sent to customers all around the globe. After two successful launches in the last two years, we are looking forward to the 3rd edition of this yearly magazine dedicated to heat transfer fluids, laboratory testing, and related equipment.

Research in heat transfer fluids or any related topic is rare. Government funding in this area is limited only to nano-fluids and molten salts for concentrated solar power (CSP) applications. Even the industrial sector does not invest an appreciable amount of money in developing new technologies in this field. Here are some of the reasons why the funding might be so limited. The two most widely used chemistries in heat transfer fluids, i.e. mineral oils and glycols (ethylene and propylene glycol), were developed several decades ago and cover a large temperature range. They perform well in their respective temperature ranges, and are inexpensive and abundantly available. Their thermo-physical properties are well established. When we get to the temperatures at which mineral oil will degrade or to a low-end temperature at which a glycol will be very viscous and inefficient to use, there are other chemistries such as aromatics, silicone oils, and brines available. Because of this, government funding is limited to developing molten salts for extremely high temperature (> 600°C) applications and cooling high-heat-flux electronics using fluids that are much more efficient (i.e. high thermal conductivity) than regular glycol/water solutions. The research work from private industry is limited to making subtle changes to existing heat transfer fluid formulations to match specific customer applications. This magazine was created to report some of the new technologies coming out of our labs in the field of heat transfer fluids and related topics.

In this 3rd edition of the "Fluid Solutions" magazine, you will find four research articles and some "Fun-Facts", including an article on caffeine content in various national coffee brands. Like in previous years, the research articles cover a variety of topics, ranging from corrosion prevention in molten salt fluids to developing a method to determine the freezing/melting point of high-concentration glycols. There is a research article continued from last year discussing the effect of sunlight on glycol stored outside in plastic containers. Another article demonstrates the partitioning effect of glycol/water mixtures in ion-exchange resins. This is a very important topic since ion-exchange columns are used to de-ionize coolants to create low electrical conductivity solutions.

Please enjoy reading the articles and have a wonderful holiday and prosperous new year in 2018.

Sincerely,

S. Mohapels



#### On The Cover

We have seen water or coffee bead up on a fabric that has been made non-wetting by chemical treatment. This is sometimes called the "Lotus Effect", which refers to the self-cleaning properties of the petals of the lotus flower. Dirt particles are picked up by water droplets due to the micro- and nanoscopic architecture on the surface of the petal, which minimizes the droplet's adhesion to that surface. This effect is also described as "wettability" of a surface, and in this case the wettability is very low or non-existent.

In heat transfer applications, the wettability of a heat exchanger surface for the heat transfer fluid is very important. The lower the wettability, the lower would be the heat transfer coefficient and vice versa. Fluids with high surface tension (i.e. water-based fluids) reduce wettability. Fluids with low surface tension (i.e. silicone and fluorocarbon fluids) improve wettability, but at the same time are prone to leaks through micro-capillaries present within joints.

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# CORROSION PREVENTION OF STAINLESS STEEL ALLOYS IN HIGH TEMPERATURE CHLORIDE SALTS FOR CONCENTRATED SOLAR POWER APPLICATIONS

by Dr. Sreya Dutta, and Tony Rivera, Dynalene Inc. and Dr. Animesh Kundu, Lehigh University

#### Introduction

With increasing demand for electricity and dwindling sources of fossil fuels, focus on renewable energy sources like wind and solar is going up. Sources of non-renewable energy, like coal, oil and gas, are becoming more and more expensive and adversely affecting the environment. Concentrating Solar Power (CSP) provides an efficient and green way of converting solar energy to electricity and heat. In a CSP plant, solar energy is focused to warm up a heat transfer fluid. That heat is then used to create super-heated steam, driving a steam turbine to generate electricity. Figure 1 shows a type of a CSP plant known as a Power Tower, where the solar energy is focused on the heat transfer fluid in the

tower using the surrounding mirrors.

In power tower operations, molten nitrate salts are used as the heat transfer media and also as a thermal storage media, to provide electricity at off-peak hours. Current temperature of operation is limited to around 550°C as the salts degrade at temperatures above 565°C. Degradation of nitrate salts causes breakdown to gaseous nitrous oxides, which changes the properties of the molten salt – the melting point, thermal capacity, etc.[1-3] A degraded salt mixture has to be replaced more often by the CSP owner, involving increased labor and materials costs.

Molten chloride salts, which are cheaper and have excellent thermal stability > 1000°C, would be an excellent replacement for the molten nitrate salts, pushing the possible operating temperature from 565°C to greater than 700°C. A higher operating temperature increases the Rankine cycle efficiency of the CSP plant (from 40% at around 565°C to > 48% at around 750°C) which would help to bring down the cost of electricity generation. [4] But molten chloride salts are extremely corrosive at high temperatures and attack stainless steel alloys, causing depletion of the protective chromium oxide and making the steel vulnerable to failure.[5]

Dynalene has developed an additive package that would enable the use of corrosive molten chloride salts for CSP. This inhibitor package would prevent the corrosion of stainless



Figure 1: Molten Salt Power Tower with heliostats

steel in molten chloride salt, allowing the use of cheaper (compared to the currently used superalloys) materials of construction in CSP plants. Dynalene and its research partner Lehigh University were granted a National Science Foundation Phase I STTR award to develop this inhibitor package for molten chloride salts. The low-cost molten salt thermal storage, cheaper materials of construction, and higher efficiency would decrease the cost of construction and maintenance of a CSP plant.

A number of researchers are trying to optimize the various components of a CSP plant in order to reduce the overall cost. So far, none of these efforts have specifically focused on the development of an additive package that can lower corrosion of stainless steel in presence of chloride based salts in the CSP plants. Development of a high temperature non-corrosive salt mixture could help in lowering the levelized cost of energy (LCOE) from 12 ¢/kWh to 6 ¢/kWh by 2020.

#### Technical Approach

Studies have shown that in extreme conditions (450-900°C), chloride salts attack stainless steel alloys, leading to intergranular corrosion and intergranular stress corrosion cracking.[6] No reports were found on the development of a corrosion inhibitor that can protect steel surfaces under these conditions. Though thermal spray, sol-gel coatings, chemical vapor deposition [7] have been studied in the literature, these methods would be difficult to apply to the several miles of piping and large molten salt tanks used in CSP systems. The addition of rare earth and alkaline earth metal oxides has shown to prevent chromium ion leaching when added to a chloride salt melt, but no coating formation was observed. Use of the Dynalene designed inhibitor package would lead to an insitu formation of a protective coating on the steel surface.

For this current study, various binary and ternary chloride salt mixtures containing NaCl, KCl, MgCl<sub>2</sub>, LiCl, CaCl<sub>2</sub>, and ZnCl<sub>2</sub> were formulated and tested for melting point. Of the salt mixtures with melting points <400°C, one ternary molten chloride (TMC) and one binary molten chloride (BMC) were selected for testing the inhibitor package. The inhibitor package consisted of rare earth metal and alkaline metal oxides. High-temperature corrosion testing was performed on the stainless steel alloys using the salt mixtures both with and without the inhibitor package.

#### **Experimental Procedure**

Technical Grade chloride salts – NaCl, KCl, MgCl<sub>2</sub>, LiCl, CaCl<sub>2</sub>, and  $ZnCl_2$  – were obtained from Sigma-Aldrich. The individual salts were oven dried to prevent any



reaction due to the presence of moisture. The dried salts were combined to form the binary and ternary salt mixtures, and the melting point of each blends was measured using a Mel-Temp Apparatus. The alloy makeups of the 316 and 316L stainless steel rods and tubes purchased from McMaster-Carr are shown in Table 1.

Table 1: Stainless Steel Alloy Composition										
Grade		C	Mn	Si	P	S	Cr	Мо	Ni	N
316	Min	**	381		0	58.00	16.0	2.00	10.0	
	Max	0.08	2.0	0.75	0.045	0.03	18.0	3.00	14.0	0.10
316L	Min	*	317.1			15.5	16.0	2.00	10.0	
	Max	0.03	2.0	0.75	0.045	0.03	18.0	3.00	14.0	0.10

A dynamic corrosion set-up (shown in Figure 2) was put together to mimic the conditions when salt flows inside the stainless steel piping in an actual solar plant. Prior to testing, 316L stainless steel rods were polished, first with 400, then with 600, 800 and 1200 SiC grit paper. A rod was inserted into the molten salt mixture inside an alumina tube. The tube with the salt mixture was heated by an upright MTI GSL-1100X tube furnace, and the steel rod rotated at 10 rpm within the salt. This setup was run for 100 hours at 650°C and 750°C. Following the heat-treatment, the metal rods were extracted from the salt mixture and cooled down under an inert gas blanket to prevent any air-driven corrosion. After cleaning, small pieces of the rod that had been immersed in the salt were sawed off. These metal pieces were characterized using Tescan Vega SBU Scanning Electron Microscopy (SEM) with Bruker Energy Dispersive Spectroscopy (EDS).

In an actual CSP plant, the molten salt will be around 720°C during the day. At night, the temperature is maintained slightly above the melting point of the salt. To mimic these conditions, the coated stainless steel coupons were immersed in the inhibited molten salts and cycled between 720°C and 450°C for 4 cycles, with a dwell time of 8hrs at each temperature. A FEI Scios DualBeam Focused Ion Beam (FIB) system was utilized to reveal a cross-section, an area just over  $100\mu m \times 50 \mu m$  ion-milled along one of the edges of the sample. Imaging was performed on the exposed cross-sections to examine the coherency of the coating.



Figure 2: Molten Salt Experiment Setup 1.Thermocouple 2.Vacuum Fitting 3.Alumina Tube (with the sample rod) 4.Chloride Trap 5.Tube Furnace 6.Vacuum Pump

# 750°C 650°C (A) BMC (B) BMC

Figure 3: SEM at two different magnifications of the SS surface with corrosion scales and products after 100hrs exposure to (A) BMC and (C) TMC at 750°C and (B) BMC and (D) TMC at 650°C

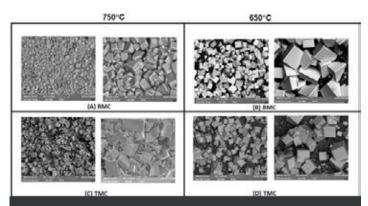


Figure 4: SEM at two different magnifications of the SS surface with protective crystal coating after 100hrs exposure to inhibited (A) BMC and (C) TMC at 750°C and (B) BMC and (D) TMC at 650°C

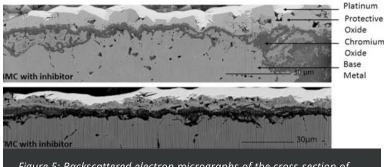


Figure 5: Backscattered electron micrographs of the cross-section of stainless steel samples that were exposed to both the chloride salt mixtures. The various corrosion layers are indicated in the images.

#### Results

After multiple tests, both uninhibited salt blends had melting points around 355°C. The surface morphology of the corroded stainless steel in each salt blend at 650°C and 750°C are shown in Figure 3 (A-D). The corrosion scale formed on the stainless steel surface in the presence of the binary salt at both temperatures consisted of a granular iron oxide layer with nickel segregation at the grain boundaries. The oxide layer was brittle and it spalled at various places to expose the steel surface, with leaching of chromium to the surface. For the ternary salt, scattered corrosion films were observed. Leaching of protective Cr ion left pores within the iron oxide matrix. At 750°C, Mo segregated to the surface, while at 650°C Mn segregation was observed. All these observations are consistent with the hot chloride corrosion studies reported by other researchers.[8,9]

When both of these salt mixtures were mixed with the inhibitor package, representative SEM micrographs of the treated steel (Figure 4, A-D) showed bimodal crystal coating on the steel surface. Almost full coverage of these inert ceramic crystals was observed on the steel surface for both inhibited salts at 750°C. Based on the underlying Cr<sub>2</sub>O<sub>3</sub> seen in the EDS, the crystals at 750°C showed 98.7% coverage for BMC, and 97% coverage for TMC. For similar inhibitor concentrations, the coverage at 650°C dropped to 93%

for both salt mixtures. Other corrosion testing (not discussed here) showed that though the extent of corrosion increased with the increase in temperature, the depletion of the Cr layer was within an order of magnitude compared to the lower temperature. On the other hand, experiments suggested the higher temperature (750°C) promoted growth and coverage of the protective coating better than at 650°C.

FIB cross-sections of the steel coupons were imaged after thermal cycling in both the inhibited salts as shown in Figure 5. A platinum layer was

deposited on the top surface as a protective cover before the cross-section was exposed in the FIB; it serves as a demarcation marker between the surface and the crosssection. The parts of the images beneath the platinum are representative of the cross-section. Larger crystals were seen in the BMC blend as compared to the TMC blend, indicating the growth of the coating was favored more in the BMC blend. Nevertheless, it can be clearly observed that the surface in both these salt blends is covered with cuboid-shaped crystals that impinge on one another, forming a continuous coating without any signs of spalling. There is a chromium-oxide-rich layer underneath the protective oxide layer, followed by a layer of stainless steel grains. There are a few voids between the crystals, which are completely isolated from the external surface. The adhesion between the chromium-rich layer and the top layer appeared to be strong in both the samples.

#### Conclusions

Molten chloride salts were investigated for their applications in high temperature CSP plants. The inhibited salts would help to increase their solar-to-electric conversion efficiency by allowing a higher operating temperature, and keep the capital and maintenance cost down by using cheaper steel alloys as materials of construction.

A binary and a ternary chloride salt mixture with low melting point were chosen for the corrosion experiments. At higher temperature, the corrosion of the steel surface was widespread and aggressive, though the chromium layer depletion was within one order of magnitude when the temperature was increased from 700 to 750°C. It was promising to observe that the higher temperature aided in the growth of the crystals and that almost continuous coating coverage was noted for both the inhibited salt mixtures. This would be beneficial in CSP plants operating at > 700°C, as the inhibitor package would be more effective in limiting the corrosion.

Thermal cycling results showed that in the presence of the inhibitor package, the coating adhered well to the steel surface. Continuous coverage with the protective crystals on top of the chromium oxide layer was observed on the steel surfaces for both these salts. This observation indicates that protective coating would be able to withstand the temperature fluctuations and prevent corrosion in a real CSP system.

After the successful preliminary lab testing, Dynalene plans to conduct long-term large-scale testing

of the corrosion inhibitors in various chloride salt blends. Validation of these inhibitor packages would be of great value to the CSP industry.

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# SHOULD GLYCOL BE STORED OUTSIDE? PART2

by Lauren Fotiades and Nick Arcury, Dynalene Inc

#### Introduction

Here at Dynalene, we have seen evidence that glycols stored outdoors sometimes developed strange odors. Last year, we began a long-term experiment designed to determine whether or not storing glycols outdoors would have an adverse effect on the product, since we were unable to find much scholarly research on UV degradation of heat transfer fluids. The ultimate goal will be to determine what compounds are forming and the mechanism by which it happens.

In the summer of 2016, our scientists performed some experiments in which a set of Dynalene glycols were exposed to sunlight throughout the summer. [1] The fluids were in containers made of HDPE, a material commonly used in storage totes, and placed on a setup designed to maximize the surface area exposed to the sun. Over the course of the experiment, we noted the appearance of several breakdown products, whose presence was confirmed both by gas chromatography analysis and by the increasing development of a strong disagreeable odor in many of the samples. Using mass spectroscopy, we were able to identify certain dioxolanes forming in the fluid that seemed to be contributing to the bad smell.

We wanted to follow up on these experiments by determining the rate of production of these compounds. Also, since we sell glycol-water blends as well as full strength glycols, we were interested in the stability of those blends under UV exposure. Our own experience has shown us that outdoor exposure in winter is not a problem, most likely due to the decreased daylight hours and subsequent lower UV intensity. As a result, we waited until summer came around again in order to begin our next set of experiments.

#### **Experimental Description**

For this year's experiments, we again used ethylene glycol (EG), propylene glycol technical (PG) grade, and 1,3-propanediol (bioglycol), a bioderived propylene glycol isomer. Each glycol was used either in an uninhibited (or raw) state, or blended with Dynalene's standard glycol inhibitor. For the kinetics experiment, we used EG and PG, raw and inhibited, at 100% concentration. For the glycol-water experiment, we used EG and PG, both raw and inhibited, and raw bioglycol, all diluted to 50 weight percent with distilled water. Table 1 contains a brief summary of the materials used.

Table 1: Materials used for outdoor exposure experiments

Glycol	Kinetics Experiment	Glycol-water experiment
Raw PG	100%	50%
Inhibited PG	100%	50%
Raw EG	100%	50%
Inhibited EG	100%	50%
Raw bioglycol	Not used	50%

The containers we used for this experiment were translucent 500mL HDPE bottles of the same type used in the 2016 experiment. The caps and liners were left as they came from the manufacturer. We placed all samples (in triplicate) outside on a plywood sheet held at an angle, with wooden tracks attached to keep the bottles in place. The plywood setup has been outside in all weathers since summer 2016, so the wood pieces were slightly warped, but everything was still straight enough to hold the bottles in the same position they were in last year.

We analyzed changes in the fluids via headspace analysis, using our Agilent 7890A GC with flame ionization detector. We pulled 5g samples from each bottle at each sampling period. For the kinetics experiment, we pulled samples every Monday-Wednesday-Friday of each week after the start of the experiment, for a total of 8 weeks. The glycol-water bottles were sampled at intervals of several weeks over a total period of 12 weeks.

#### Results

In GC analysis, the amount of a given compound in the original sample is proportional to the area under the curve of its representative peak. We performed this analysis on headspace samples, which concentrates the volatile breakdown components into a vapor before injecting it into the instrument. This means two things: the breakdown components will have much stronger chromatogram peaks than the less volatile glycols, in spite of the fact that there is still more glycol in the original fluid than any other compound, and those same components can be found by the GC long before they've built up in enough quantity to be detected by the human nose.

The GC results showed new peaks appearing in concentration of Ithe raw PG samples at 2.5 weeks, and the first faint odor was recorded at 5 weeks. One compound in particular continued to increase in concentration until the experiment ended (Fig 1). Raw EG, inhibited EG, and inhibited PG all saw no odor development or new GC chromatogram peaks throughout the experiment.

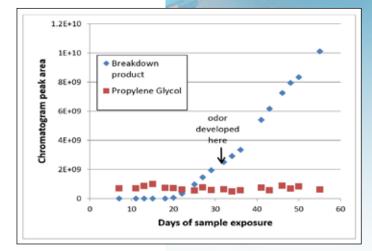


Figure 1: GC peak areas showing an increase in concentration of breakdown product over time.

#### Glycol/water experiment

These samples were kept outside for a total of 12 weeks. After about a week, we found that the inhibited EG used for this experiment had been made with EG that already had some degradation; GC peaks were coming out extremely strong for only a few days of exposure. When the source fluid was re-assessed, we found it already had a faint odor, so slight it was undetectable when we were not looking for it. As a result, the inhibited EG 50% results are not included in this write-up.

In just under two weeks, a brown discoloration began to form in the PG samples. At first the inhibited samples were more discolored than the raw samples. By three weeks of exposure, all PG samples, whether inhibited or raw, were the same shade of brown. No other samples were discolored.

By 11 weeks, the PG samples were still the only ones with any discoloration, and the color strength had actually decreased until the PG samples weren't much darker than any of the others (Fig 2). The PG also had the strongest smell, although it was not the same as the dioxolane smell found in the full-strength glycols. The raw EG containers just smelled like hot plastic. The raw bio containers smelled like hot wax

Strong degradation peaks appeared in the inhibited and raw PG GC chromatograms. When overlaid, the



Figure 2: 50% glycol solutions with > 11 weeks of exposure. PG samples are the top six bottles on the left side.

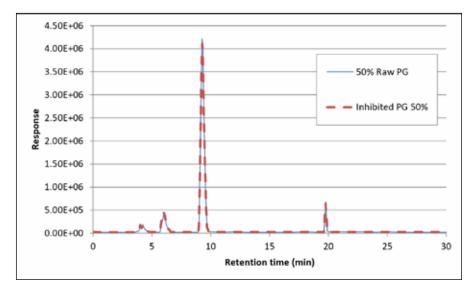


Figure 3: FID of Inhibited and raw 50% propylene glycol samples after 8 weeks exposure

chromatograms for the PG samples are identical, regardless of inhibitor content (Fig 3). There is only one significant peak in the final raw EG chromatogram and it appears at the retention time for the glycol itself. The bioglycol has one extra small but distinct peak at the 8 minute mark in the week 7 chromatogram.

#### Discussion

The most noticeable thing about this year's experiment results is that we simply did not get the same level of degradation that we saw in 2016. At the four-week point, the raw PG from 2017 has many of the same peaks present as the raw technical grade PG from 2016, but all the peak areas are much larger in the earlier sample (except for the propylene glycol peak at 20 minutes). The USP grade PG from 2016 not only has taller peaks than the new 2017 sample, but also a greater number of peaks (Table 2). In fact, even the chromatogram from eight weeks in 2017 does not match up to 4 weeks in 2016. All samples were run on the same instrument, using the same method and sampling.

In 2017, raw PG was the only full-strength glycol that suffered any degradation. As noted in the results, there were no breakdown products shown by GC analysis in the inhibited PG, the raw EG, or the inhibited EG. In 2016, both raw EG and inhibited PG developed distinct odors and strong new chromatogram peaks.

Table 2: Comparison of peak areas (listed by retention time in minutes) between 2016 and 2017 raw PG samples. Where no entry is made, there was no peak at that retention time for that sample.

Data at law times	2016	2016	2017	2017
Retention time (min)	USP PG 4 weeks	Tech PG 4 weeks	PG 4 weeks	PG 8 weeks
3.8	1.05E+08	1.06E+07	1.55E+07	5.02E+07
4.0	1.14E+09	7.66E+08	5.69E+07	5.05E+08
4.2	2.47E+09	1.85E+09	8.22E+07	1.21E+09
9.0	1.17E+10	3.33E+10	9.78E+08	1.01E+10
12.1	3.28E+07	5.44E+07		
16.9	1.40E+07			
18.0	3.95E+07			
19.6	7.80E+09			1.73E+08
20.0	1.20E+09	6.42E+08	5.48E+08	6.14E+08
22.3	3.19E+08			
22.7	3.79E+07			

There is not a clear reason why this should be. Our first thought was that the glycols used last year might have started with some degradation, as we found this year with the inhibited EG in the glycol/water experiment. However, the baseline runs for all glycols used in 2016 are clear and show no peaks other than the glycol itself, eliminating that possibility.

The next obvious possibility is weather conditions. We learned from last year's work that exposure to UV radiation is necessary for these breakdown reactions to take place, so a significant increase in cloud cover from last year to this year could have affected the results this way. We also have not fully determined what impact temperature has on the process, so a big difference in temperature over the course of the experiments is another potential factor.

While we did not gather environmental data ourselves during either 2016 or 2017, we can get an estimate of what we would have seen from the site weatherunderground.com [2], which reports historical data from the weather station at Lehigh Valley International Airport. The airport is less than four miles from Dynalene, so while there will almost certainly have been some variation, the data collected there can be considered a reasonable approximation of what we would have seen at our site.

Comparing the first 29 days of each experiment, we can see that the average temperature was actually higher in 2017 for more days than it was the same or lower (Fig 4). Both experiments had similar maximum temperatures,

ranging from the high 60s to the low 90s, with most of the recorded highs falling between 75 and 90°F. This seems to indicate that temperature was not the deciding factor. Cloud cover is a more interesting possibility. The LVIA weather station data states that in 2016 there were 12 days with rain, fog, thunderstorms, or a combination of the three. In 2017, there were 19 such days reported. An extra full week of partial to full cloud cover. It is conceivable that this extra cloud cover could have lowered the UV exposure enough to prevent the degradation reactions from initiating.

One more potential factor is the time of year when the samples were placed outside. In 2016, all samples were started outside in late May, before the summer solstice in the Northern Hemisphere.

In 2017, we started in mid-July, after the solstice. In addition to the hours of daylight increasing as solstice approaches, the intensity of solar radiation also increases, as the light from the sun hits earth's surface at a lower angle of incidence [3]. When we began this experiment, we didn't think that timing would have an effect – that as long as we were still working in the summer months, the amount of daylight would be roughly similar. In light of the results, however, it seems possible that the switch from lengthening days (2016) to shrinking days (2017) may have made a difference after all.

The other thing we can see in the 2017 results is that it appears that water impacts the reactions

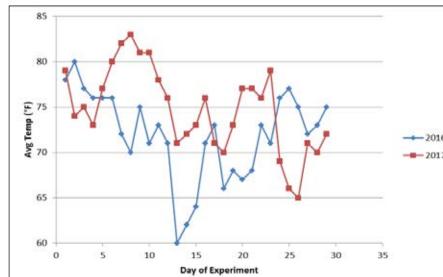


Figure 4: Average temperatures over the first 29 days of the 2016 and 2017 experiments

that occur in the glycol. Figure 3 above compares the GC results for raw and uninhibited 50% PG. The chromatograms are identical, showing that the presence or absence of inhibitor doesn't have an effect on the breakdown products formed in a glycol-water blend.

The primary peak (~19.5 min) is also the primary peak seen growing in the full strength raw PG samples. Other peaks begin to develop in the raw PG towards the end of the experiment and may have grown more prominent if it had been left outside longer. These compounds are likely the dioxolanes found via the MS testing in 2016. It is possible their formation is somehow prevented by the presence of water in the 50% samples.

Interestingly, the inhibited PG tech results from 2016 look nearly identical to these 50% PG curves (Figure 5). This hints that it is the small amount of water from the inhibitor package that caused its degradation behavior to differ from that of the full-strength raw PG, rather than the inhibitor itself as we thought last year.

While one might expect to see similar behavior in other glycols, that fact that we did not find breakdown products in EG in either 2016 or 2017, added to the failure

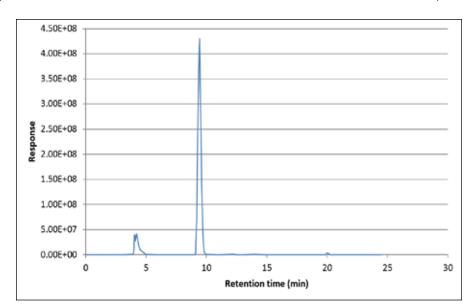


Figure 5: 2016 FID results for inhibited tech grade PG after 12 weeks exposure.

to find breakdown products even in raw EG in 2017, means we do not have enough evidence to draw this conclusion. As noted above, this year's bioglycol had one small but distinct new peak visible in the September 8 chromatogram. Now, this peak did appear in the bio runs from 2016, but it was not considered significant since it was so much smaller than the primary glycol peak. In all the 50% samples from 2017, the glycol peaks are very small compared to the ones seen in full strength runs (most likely their relative vapor pressures mean that there

is more water than glycol in the headspace), so this new peak in the bioglycol run seems more significant. On the other hand, the peak appears with both 50% and full-strength samples, arguing that in this case the water did not have an effect.

#### Conclusion

Depending on the exterior conditions, glycol breakdown during outdoor storage is not necessarily as rapid as we found in our initial experiment. Whether this is due to weather conditions such as cloud cover blocking solar UV radiation or seasonal variation due to the time of year is currently unclear. While our intent was to measure the rate of growth of the breakdown products, the glycols didn't form enough of them this year to be able to do that well. We did find that it is water, rather than inhibitor, that changes the degradation behavior of propylene glycol.

Further research we would like to perform on this topic includes another attempt at a kinetics run, starting earlier in the year to try for the maximum possible glycol breakdown. Shorter runs may also be done, to determine if there's a specific window where outdoor exposure

would be the most dangerous to glycols, especially propylene glycol. We would also like to uncover more details about the difference between exposure of raw glycols and exposure of glycols with water present.

Weather and UV radiation monitoring should be done to see if their effects can be pinpointed. If we can determine exactly what triggers the dioxolane formation and duplicate it in the lab, then we can continue our research yearround instead of needing to wait for the sun to come back after the winter. This would allow us greater repeatability of experiments, making it easier to accomplish things such as evaluating the reaction mechanism or mechanisms of the glycol breakdown.

Acknowledgements: Tara White and Allison Zolnowsky, Dynalene Inc.

#### References

[1] Fotiades, Lauren, "Should Glycols be Stored Outside?",

[2] Weather Underground. Weather History for KABE – October 2017. https://www.wunderground.com/history/airport/KABE/2017/10/4/DailyHistory.html (Accessed Oct 4, 2017)

[3] Jeffrey Brownson, EME810, Lesson 2: "Earth's Tilted Axis and the Seasons", (The Pennsylvania State University College of Earth and Mineral Sciences: OER Initiative), https://www.e-education.psu.edu/eme810/node/474 (Accessed Oct 4, 2017). License: Creative Commons BY-NC-SA 3.0

## Is your fluid contaminated with salts and ions?

Dynalene's ion exchange resin products are designed and tested to remove dissolved contaminants. These systems help in softening, deioninzation and low electrical conductivity applications.



Low conductivity applications

Fuel cell cooling, battery cooling, electronic cooling, computer cooling, laser cooling applications.

#### Water softening applications

Removing calcium and magnesium compounds in commercial and industrial applications.





#### **Deionization applications**

Medical, food processing, chemical, semiconductor, industrial cooling, cosmetics manufacturing applications

#### **Custom applications**

Custom contamination removal applications such as, chloride, sulfate and bromide removal from glycol



Contact Dynalene at 610.262.9686 for more information



Note: FP\* = Freezing Point BP\*=Burst Point

	Propyler	ne Glycol	Ethylene Glycol		
Vol%	FP* (°F)	BP* (°F)	FP* (°F)	BP* (°F)	
5	29	27	29	27	
10	26	22	26	23	
15	23	18	22	17	
20	19	11	16	8	
25	14	-1	10	-2	
30	8	-18	4	-14	
35	1	-46	-3	-45	
40	-8	-60	-13	-60	
45	-18	-60	-24	-60	
50	-31	-60	-36	-60	
55	-46	-60	-50	-60	
60	<-60	-60	< -60	-60	
65	<-60	-60	< -60	-60	
70	<-60	-60	< -60	-60	
75	<-60	-60	< -60	-60	

#### **FLUID FOCUS**

# Do you check chloride ion concentration in your heat transfer fluid?



Pitting corrosion due to excessive chloride ion

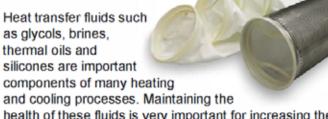
Water-based heat transfer fluids (i.e. glycols) can be

contaminated with aggressive chloride ions if tap/city water is used for on-site dilution. A chloride concentration of higher than 25 ppm in the fluid can cause pitting corrosion of metal components. For dilution purposes, deionized (DI) or distilled water should be used.

Chloride ions can be monitored in a glycol fluid by:

- Sending a sample to Dynalene
- Sending a sample to an outside lab
- On-site testing using a chloride strip such as the one shown in the picture

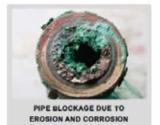
If your fluid is contaminated with chloride ion in excess of 25 ppm then contact the glycol experts at Dynalene for an action plan.

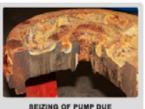


DO YOU FILTER YOUR

**HEAT TRANSFER FLUID?** 

health of these fluids is very important for increasing the service life of the system. Particulates in the fluid can cause severe system problems as discussed below. Filters are recommended to be used in heat transfer fluid loops to mitigate these issues.





SEIZING OF PUMP DUE TO RUST BUILD UP

- · Clogging of the heat exchangers
- Erosion of the piping and other components
- Seizing of pumps
- Increase of viscosity, therefore requiring higher pumping power
- Localized corrosion due to debris deposit
- Foaming

### Glycol Do's & Don'ts



Use deionized water when blending glycols to help maintain low levels of aggressive chloride ions, thus reducing corrosion.

Check the pH and the concentration of the glycol regularly. This 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.

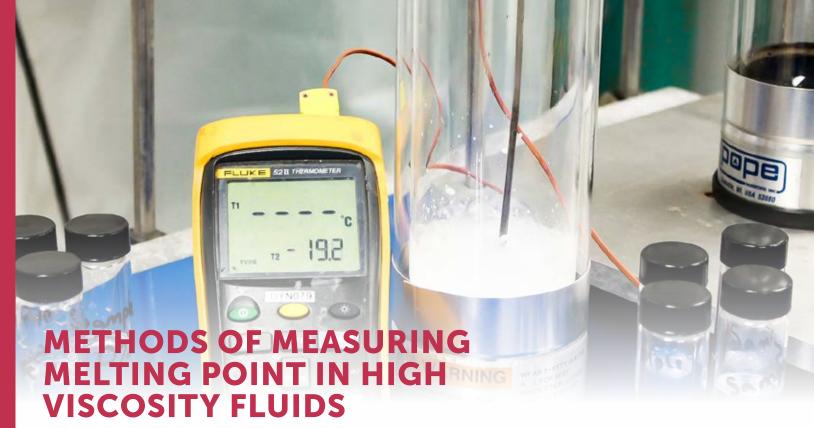
Use filtration when possible. Adding filtration to your system will help keep it clean and improve heat transfer.

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.



by Allison Zolnowsky, Dynalene Inc.

#### Introduction

In low-temperature systems that require circulating heat transfer fluids, the melting and freezing points of the circulating fluid are a must-know. With fluids that have low viscosity at low temperatures, their freezing and melting ranges can be determined in a cryostat or using a differential scanning calorimeter (DSC). Dynalene's TA DSC Q200 with Refrigerated Cooling System (RCS40) can be used to measure fluids with freezing and melting points higher than -62°C [1], due to the limits of the cooling system. It records freezing and melting data as it occurs.

The cryostat can perform freezing/melting point tests on fluids down to -196°C, because liquid nitrogen (LN2) is used to bring the temperature down. LN2 is circulated through a cage made of copper piping that is held inside a Dewar flask containing the fluid, while the fluid is mechanically stirred to improve the temperature distribution. Like the DSC, this method can be used to determine freezing and melting temperatures, and can present the freezing and melting points over time if a data logger is connected to the thermocouple system, or if it is recorded manually.

However, these methods are only usable on low-viscosity fluids, or fluids that don't thicken as temperature decreases. This is an issue in some fluids, such as propylene glycol (PG) at high concentrations. In an aqueous solution over 50% glycol, the fluid becomes

too viscous to freeze in both the cryostat and the DSC, because the viscosity is too high to allow proper freezing under the static conditions of the DSC or for stirring to be possible in the cryostat.

In order to determine melting and freezing data on higher concentrations of PG solutions, we designed an experiment with the goal of measuring the melting points of PG samples ranging from 0 to 100%. We chose to focus on melting point because the effect of supercooling, or taking fluids below their actual freezing point without changing phase, can happen regardless of measurement technique, whereas the change from solid to liquid will always occur at consistent temperature. With the lower sample concentrations, we can determine melting point in the DSC and compare to determine the accuracy of this experimental method.

#### Experimental Procedure

The experiment was run with 30 gram samples of raw propylene glycol solutions, ranging in 10% increments from PG100% to pure distilled water. Each sample was poured into a tall Dewar flask with a thermocouple probe held in place in the center of the vessel, the probe tip approximately in the center of the solution to avoid touching the warmer sides or colder base of the Dewar. LN2 was collected in a separate Dewar so it could be poured over the PG samples to freeze them. LN2 was used because of its extremely low temperature of -196°C. The thermocouple read the

fluid temperature from contact with the LN2 down to the ultimate low temperature reached (which varied depending on the amount of LN2 poured), then back up to the melting point. Each sample, upon melting completely—no crystals present in the fluid—was then frozen again to look for repeatability and accuracy in the observed melting temperature.

Afterwards, the same PG50%-PG10% samples used in the LN2 pourover method were used to run melting temperature in the DSC. The melting temperatures determined in the DSC was used to determine the accuracy of the pourover method.

#### Results

Our results show that the LN2 pourover method yields melting temperatures within 1 to 2  $^{\circ}$ C of those determined by

Figure 1: LN2 Pourover experimental setup. 1. Vacuum dewar 2. Temperature display 3. Propylene glycol with LN2 4. Thermocouple 5. LN2 dewar

the DSC, and in the PG100% solution, the melting point recorded was close to the published value [2]. As illustrated in Figure 1, the DSC determined melting points of PG50% to PG0% closely correlate with the melting points determined by the LN2 pour-over method.

The LN2 pouring method freezes the fluid rapidly when otherwise, under typical freezing conditions, it could remain in the liquid phase indefinitely due to its extreme viscosity. Our method ensures complete freezing in the fluid, as well as offering visualization of the melting range from the very beginning of the phase change until the fluid has completely melted. The reported values are temperature-adjusted to offset the error on the thermocouple probe,

which was determined with both LN2 and in a dry ice and methanol bath.

#### Conclusion

Because of the close correlation seen between the DSC and LN2 pour-over melting temperatures, we can conclude that the LN2 pour-over method is viable for determining melting points in high viscosity fluids that cannot be frozen in either DSC or cryostat.

#### References

1. "TA Instruments Thermal Analysis: Differential Scanning Calorimetry", TA Instruments, 2012.

2. "A Guide to Glycols", The DOW Chemical Company, 2003.

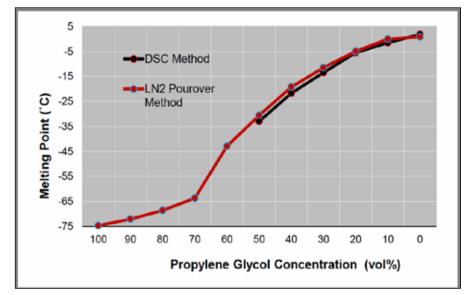


Figure 2: Change in melting point as a function of propylene glycol concentration using DSC and LN2 Pourover methods.

# WILL ION EXCHANGE RESIN AFFECT THE CONCENTRATION OF GLYCOL?

by Allison Zolnowsky, Dynalene Inc.

Ions exist within all fluids, either as a critical part of the solution makeup, or as a cause of negative effects to either the solution itself or to the system components that contain and transport the fluid. An example of the negative effects of some ions is the buildup and scales created by "hard" ions such as calcium (Ca2+) and magnesium (Mg2+) in water systems. These ions can leave deposits that block and corrode pipes and metal, causing damage to a system. To avoid this issue, these hard ions are exchanged for "softer", more water-soluble ions such as sodium (Na+) and chloride (Cl-) through ion exchange [1]. Using ion-specific resins, the hard ions are drawn out of the water and more watersoluble ions are pushed from the resin into the water to replace them. Ion-exchange resin is also used in creating low-conductivity (LC) heat transfer fluids [2]. The resin lowers a fluid's conductivity by exchanging strongly charged ions with more electrically neutral ions. This is especially important in systems that require LC fluids to avoid issues when a current is being passed through the fluid, such as fuel cell cooling applications.

Ion-exchange resin only takes up ions that it has an affinity for; e.g., if you have a resin that will readily take in Ca2+ ions and it's in a fluid without Ca2+ ions, it will not ion exchange.

When LC fluids are circulated with ion-exchange resin, the concentration and density of the fluids should remain unchanged, while any ionic molecules present are removed. But what happens if you take a de-ionized glycol that is electrically neutral and allow fresh resin to soak in it? What will happen to the resin? What will happen to the glycol?

Since glycol is not ionic and there should be no ions present to be exchanged, theoretically, nothing should happen except possibly a slight change in conductivity if there were any remaining ionic stragglers. However, when soaking 1X-10 ion exchange resin in uninhibited

(raw) propylene and ethylene glycol solutions, it was found that the solutions' concentrations were significantly affected. So if the resin is absorbing glycol, how much is actually going in? If a glycol-based coolant is de-ionized using ion-exchange resin, will the glycol concentration remain the same throughout the process?

To determine this, 150-gram samples of glycol-water solutions were prepared, ranging from 50% to 20% glycol content, and including a 100% sample. 100 grams of fresh 1X-10 resin was added to each fluid, the solution stirred to ensure complete saturation of all resin beads, and the container capped and labelled.

The final concentrations of the sample fluids were determined with a refractometer after the resin was allowed to soak over a period of 2 days. Next, small samples of the resin were scooped and placed in a vacuum filter to remove any extra fluid, and the samples were weighed and allowed to air dry before being placed in an Ohaus MB90 Moisture Analyzer (Figure 1). Unsoaked resin samples were tested with the moisture analyzer to get a baseline moisture content that naturally resides in the resin. Approximately 2-3 grams was tested for each



Figure 1: Ohaus MB90 Moisture Analyzer for solids.

sample, and each sample was dried to completion at 135°C.

Using this method, we were able to determine the amount of liquid present inside the resin after soaking in glycol solutions (Figure 2). Commercially avaiable ion exchange resins usually contain some amount of moisture in them. The solid moisture analyzer was used to determine the moisture content in the resin used and it was calculated to be at 49.5 wt%. As it can be seen in Figure 2, when the resin was soaked in different vol% of glycols, the liquid percent inside the resin decreased with decrease in glycol concentration.

The final concentration of the glycol solution after soaking for 2 days was determined using a refractometer

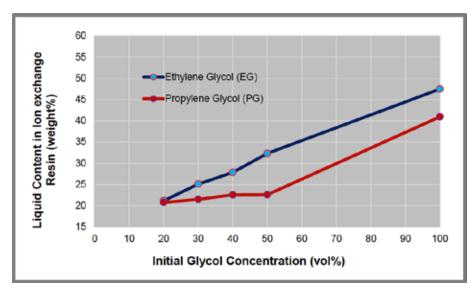


Figure 2: This graph illustrates the amount of absorbed liquid present in ion exchange resin soaked in different concentrations of EG and PG.

and is plotted against the concentration of the initial glycol solution as shown in Figure 3. It can be seen that there is a drop in the glycol vol% in the final solution after 2 days of soaking. For example, 50% propylene glycol solution decreased to 38% after the experiment. This is an important consideration for a cooling loop with a small volume of glycol solution with an ion exchange resin cartridge where the the concentration of glycol can decrease over a period of time causing system freeze up at low temperatures.

With this data we can determine that initial concentration is a deciding factor in how much glycol will be taken up by the resin, and how much water will be displaced into the solution. The more glycol available in a solution, the more the resin will absorb, and the more water will be displaced into the solution. It can also be noted that the

amount of resin present in a sample fluid directly correlates to the amount of glycol absorption. If there is a near 1:1 ratio of glycol-water to resin, the concentration will be affected on a large scale. If, however, there is a larger amount of fluid to resin, say 5:1, the concentration will be affected on a much smaller scale. Where the concentration drops upwards of 18% in a 50% ethylene glycol solution, if the fluid amount is much greater than the resin amount, the concentration will only drop 1-3%.

### References

1. "Ion Exchange for Dummies", www.lenntech.com, 2008.

2. Bojanna Shantheyanda, "Low **Electrical Conductivity Liquid Coolants** for Electronics Cooling", Electronics Cooling, Number 4, Volume 21, 2015.

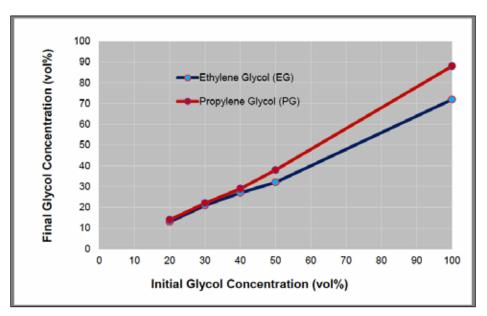


Figure 3: This graph illustrates the change in glycol concentration when soaked with ion exchange resin for 2 days...



by George Giannaras, Dynalene Inc.

Americans are working longer than ever before. When taking into account hours worked per week and the number of weeks per year, Americans are working an additional month per year compared to 35 years ago, with the largest increase in jobs in high skill occupations that also require extensive training and experience. So it's understandable that people are looking for a pick-me-up to get the day started. One of the most common ways to do that is with a cup of coffee. Nearly 65% of Americans drink at least one cup of coffee a day.

So, how does coffee give you that pick-me-up? In a word, caffeine. How does caffeine do that? Well, it blocks a chemical in the brain called adenosine. The role of adenosine is to slow the speed of neurons firing in your brain. It helps you think calmly, allowing for careful deliberation. When caffeine blocks adenosine, neurons begin to fire more quickly, replicating the fight or flight response. This releases extra sugar into the blood stream along with dopamine, giving you a quick jolt of energy. That quick jolt can be good, but as in all things, moderation is key. Overdoing on caffeine can lead to "crash" periods and poor sleep

at night. So making sure you only consume the right amount is important. (nutritiontotheedge.com)

So how do we find out how much caffeine is in coffee? First we had to develop a method. We used an Agilent Technologies Series 1200 HPLC equipped with a Diode Array Detector. Linearity standards were purchased from Restek Inc. in Bellefonte, PA. An Eclipse Plus C18 column 150 x 4.6mm was chosen for the analysis.

Various mobile phase combinations using acetonitrile, methanol, 0.15 % acetic acid and water were evaluated. The mobile phase which provided the best peak shape was a 90% acetonitrile and 10 % water with an isocratic elution.

Next we established a working range with good linearity and precision. Our standard curve covered a range of 5 ppm to 500 ppm with a relative standard deviation of 1.1%. Precision of multiple replicates of the mid-level standard yielded a deviation of 0.3%.

With coffee shops being the fastest growing section of the restaurant industry, we decided we would test the caffeine content at some of the biggest coffee sellers. So how much caffeine is in your morning cup o' joe? Do you stop at Dunkin Donuts, Starbucks,

or McDonalds on your way to work to get your morning pick-me-up? Dynalene Lab Services used the method we developed to test the amount of caffeine in these three top coffee sellers (sorry, Tim Horton fans; we don't have the pleasure of having a Tim Horton's convenient to us).

Here's how much caffeine we found in 16 ounces of coffee (Table 1). That's a Grande at Starbucks, a medium at your local McCafe, and between a small and medium at Dunkin Donuts.

Not a fan of hot coffee? More a devotee of iced coffee, especially on those hot summer days? Don't worry, we tested the iced coffee at each of the establishments as well. Seems they do a pretty good job on not having their coffee watered down, with caffeine levels very similar to the hot version. Table 2 shows what we found.

What if your morning cup of coffee doesn't seem to give you that same boost? Could the coffee be different? We decided to measure the 'precision' of the coffee in relation to caffeine content. We purchased

Sample	mg of Caffeine	mg/fl. Oz.
DD Hot	248	15.5
Starbucks Hot	327	20.5
McD Hot	199	12.4

Table 1: Samples of hot black coffee were purchased from Dunkin Donuts, Starbucks and McDonald's. These samples were filtered and diluted 1:10 in water and analyzed on the HPLC for caffeine content.

a cup of hot black coffee from Dunkin Donuts over five different days. (There were lots of volunteers for this job!) What we found was that the caffeine content was very similar over the different days, with a relative standard deviation of less than 5%. So it's not the

coffee; there is some other cause. (Maybe you drink too much coffee in a day and you're not getting good sleep.)

On a budget? Not spending your hard earned money at a coffee shop? We can relate to that. So we tested how much caffeine is in a cup of coffee made using a Keurig machine. We used a middle-of-theroad medium roast. The findings show that the caffeine content comes in between Dunkin and Starbucks at 291 mg per 16 oz. cup of coffee. This will probably vary between coffee machines, as life experience has taught us that not all coffee makers will dispense the same amount of water through that K-cup.

Are you part of the roughly 35% of Americans who don't drink coffee? Don't like the taste? Prefer something colder and sweeter to get you going? We also tested

a Classic Coke to see how much caffeine it contains. Turns out Coke has much less caffeine than coffee: a 16 oz. coke has about 46mg of caffeine. So how does a Coke give you that jolt? Perhaps it's the sugar in it. But that's a discussion for a different article.

Sample	mg of Caffeine	mg/fl. Oz.
DD Cold	226	14.1
Starbucks Cold	304	19.0
McD Cold	183	11.4

Table 2: Samples of plain iced coffee were purchased from Dunkin Donuts, Starbucks and McDonald's. These samples were filtered and diluted 1:10 in water and analyzed on the HPLC for caffeine content.



Sample	mg of Caffeine	mg/fl. Oz.
DD Hot Prep 1	255	15.9
DD Hot Prep 2	232	14.5
DD Hot Prep 3	253	15.8
DD Hot Prep 4	245	15.3
DD Hot Prep 5	237	14.8

Table 3: Method reproducability test



#### **DID YOU KNOW?**

Antifreeze proteins help the arctic fish survive in extreme cold temperatures.

Antifreeze proteins (AFPs) or ice structuring proteins (ISPs) produced by certain vertebrates, plants, fungi and bacteria that permit their survival in subzero environments. AFPs bind to small ice crystals to inhibit growth and recrystallization of ice that would otherwise be fatal. There is also increasing evidence that AFPs interact with mammalian cell membranes to protect them from cold damage.

Unlike the widely used automotive antifreeze, ethylene glycol, AFPs do not lower freezing point in proportion to concentration. Rather, they work in a noncolligative manner. This phenomenon allows them to act as an antifreeze at concentrations 1/300th to 1/500th of those of other dissolved solutes. Their low concentration minimizes their effect on osmotic pressure. The unusual properties of AFPs are attributed to their selective affinity for specific crystalline ice forms and the resulting blockade of the ice-nucleation process.

www.en.wikipedia.org/wiki/Antifreeze\_protein



### GLYCOL REFRACTOMETER DO'S & DON'TS





- . Be sure the instrument is clean and dry before use
- Use enough fluid to cover the entire measuring prism
- Point the instrument at a light source when taking the reading
- Read from the appropriate side of the scale (ethylene or propylene glycol)
- Clean the instrument after each use

- Do not touch the measurement prism or bottom of the sample cover
- Do not get fluid on the eyepiece of the instrument
- Do not open the sample cover when taking a reading
- Do not test a sample that is too hot or too cold (between 70°F and 85°F is best)
- Do not drop the instrument or subject it to hard knocks

#### **DYNALENE NEWS**

#### Dynalene launches New desiccation system product line

Dynalene launches Fluidry® product line that consists of moisture removal (or desiccation)

systems which helps in maintaining low moisture content in hydrocarbons, silicone oils and transformer oil (insulation oil). Maintaining low moisture content in low- and hightemperature heat transfer fluids is crucial to attaining or maintaining designed system efficiency. Moisture, which can enter into a fluid system due to improper seals,



improper handling, system leaks, or malfunctioning driers, can cause system failures.

Fluidry® includes, SX, TX, HX and M series desiccation products which are available for a wide range of fluid flow rates. The systems are effectively designed for use in bench top or low flow rate applications to higher flow rate industrial applications. Some of our products can also be used in drying lubricants, paints, refrigerant drying, CO<sub>2</sub> drying in LPG, air, inert gas, natural gas and other atmospheric gases.

Dynalene is in the final phases of commercializing a moisture measurement system. This system can be used to measure the moisture content in the hydrocarbon-based fluid cooling loop. This measurement system can also be integrated with the Fluidry® desiccation system which can help customers in monitoring the moisture level in their fluid loop at all times.

### Dynalene contamination removal systems

Dynalene's new molybdate removal system was recently commissioned at a customer site in Tampa, Florida. With stringent regulation on the allowable contamination levels in waste water discharged to the drain, the customer was penalized by the water

authorities for failing to meet the requirement. The molybdate removal system has been successful in bringing the molybdate level in the feed water from about 10 ppm to an acceptable level of below 0.08 ppm. After the contamination removal, the treated water is suitable to be discharged to the drain without needing any additional treatments..

Dynalene's chloride removal system was recently commissioned at a customer site in Seabrook, New Hampshire. The system was used in a nuclear power plant to remove chloride from an ethylene glycol cooling loop in a diesel generator. The chloride removal system was directly connected to the diesel generator system for contamination removal, avoiding any need to remove the fluid from the cooling loop. This system helped the customer remove the contamination without shutting down the plant for maintenance.

Dynalene has expanded our contamination removal product portfolio during the past year. Along with chloride and molybdate removal, we offer products that can be used to remove selenite, arsenic, phosphate, sulfate, calcium, iron, manganese, and others.

#### Dynalene completes NSF Phase 1 project delivery

Dynalene Inc. collaborated with Lehigh University on the STTR proposal "Corrosion Inhibition of Stainless Steel Alloys in High-Temperature Chloride Salts for Concentrated Solar Power Applications", funded by the National Science Foundation (NSF). Phase I of the project spanned from July 2016



to August 2017 and a final report was submitted to NSF summarizing the project accomplishments. The goal of Phase I is to optimize an inhibitor package for molten chloride salt to prevent corrosion of stainless steel alloys at temperatures up to 750°C (for more details please see pg. 4). In a nutshell, the researchers successfully demonstrated growth of corrosion-resistant crystalline coating on stainless steel alloys when immersed in the inhibited molten chloride salt blends.

The exposed coupons were characterized at Dynalene and Lehigh University using their advanced electron microscopes and other equipment. A plan for validation of these inhibited molten chloride salt mixtures is under review, including long-term, large-scale and high-temperature corrosion testing on various steel alloys. In the near future, Dynalene plans to commercialize and provide the inhibited molten chloride salt blends for CSP and other high temperature applications.



### Temperature Range Reference Chart for Dynalene Heat Transfer Fluids



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.

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