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## Low Temperature Lubricant Fluidity Study

New internal research investigating low temperature fluidity additives

### Abstract

Low temperature lubricant fluidity is a critical consideration for various lubricant applications, from industrial refrigeration compressors to freezer chains. Improving low temperature fluidity in lubricants can improve system performance, decrease energy consumption, and reduce issues related to lubricant thickening. The goal of this project was to better understand the effect of additive concentration and to use the knowledge gained to improve production formulations. This study was performed internally by SLFi engineering staff, guided by the tutelage of Jim Sandler.

### Introduction

Improving low temperature fluidity in lubricants is a major consideration when striving for continuous formulation improvement. Lubricants with lower pour points can provide a wide range of benefits for various low temperature applications. To continue to improve lubricant formulations, detailed analysis of both the additives and base stocks must be performed.

Typically, the low temperature fluidity of lubricants is determined via pour point testing. The American Society for Testing and Materials has developed a set of procedures, D97, that specify standard test methods for the pour point of petroleum products. This test, while used industry wide, is not without flaws. In essence, the procedure dictates that a fluid is chilled within a tube which is then inverted. The lowest temperature at which the operator notices movement within the fluid is marked as the pour point of the fluid.

The downside of this test is that it is visually determinate. Repeatability has an error of +/- 3°C while the error for reproducibility increases to +/- 4°C. As well, the viscosity of the fluid at temperatures near the pour point is not tested. While the test allows production of data sheet specifications, it has been found to lack usefulness when performing additive testing. This is due to the fact that the rate at which the viscosity increases as the fluid approaches pour point is just as critical as the pour point temperature itself.

For this study, a different approach was used so the viscosity of the fluid as it approaches the pour point could be tracked. To perform this, a Brookfield Engineering DV2T-LV rotary viscometer was used. Not only will the viscometer allow data logging of the viscosity of the fluid as it approaches pour point, but it also operates at a higher precision with accuracy of +/-1% and repeatability error of +/- 0.2%.

The Brookfield DV2T-LV rotary viscometer is supplied with 4 different spindles to measure various viscosity ranges. The spindle used to measure very low viscosities has a large amount of surface area interacting with the fluid, while the opposite is true for higher viscosity fluids. The

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counter-rotating force applied to the spindle as it rotates through the fluid is measured by a displacement sensor within the machine. Thicker viscosity fluids will exhibit greater resistance to rotation compared to lighter viscosities.



*Figure 1: Brookfield DV2T-LV Viscometer used for the study.*



*Figure 2: Brookfield DV2T-LV Viscometer spindle selection. Lightest viscosity range on the bottom going to heaviest at the top.*

## Testing

Testing was performed on a variety of formulated lubricants as well as pure base stocks. To better understand how additive dosing affects fluidity, samples were made with various concentrations of a single additive in identical base stocks. Large batches of the samples were made to minimize any dosing error that could arise using different batches.

To chill the fluid, a dry ice and cryogenic heat transfer fluid bath was used. Dry ice is slowly added to the bath prior to introducing the sample to chill it to a predetermined temperature of  $-65^{\circ}\text{C}$ ,  $\pm 2^{\circ}\text{C}$ . During testing, the bath was maintained at the prescribed temperature by slowly adding additional dry ice pellets. Tests were performed on 300mL samples within 400mL Griffin form beakers. An inverted petri dish was used to elevate the sample within the bath to help equalize cooling around the sample.

Data logging of the viscosity was started once the room temperature sample was placed into the chilled bath and its temperature had lowered to  $0^{\circ}\text{C}$ ,  $\pm 5^{\circ}\text{C}$ . Testing concluded once the fluid reached a predetermined end point viscosity. This end point viscosity was determined by the maximum viscosity for the range of the selected spindle. The two end points utilized were

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1,200,000 cP, for the #63 spindle, and 6,000,000 cP, for the #64 spindle. These two spindles measure the upper portion of the DV2T-LV viscometer's scale.

The Brookfield DV2T-LV viscometer was pre-programmed with a stepping test procedure that would reduce the spindle's rotational speed as the viscosity increased. Two different programs were used, one for each spindle. The same programs were used throughout the testing. Dynamic viscosity data points were logged every second in centipoise. The data logging would automatically stop once the end point viscosity was reached.

A single operator performed all of the testing for this study, under supervision of the testing coordinator. This was done to help minimize any variables introduced by human error and technique. The operator was tasked with ensuring the sample was not contaminated by the liquid from the cooling bath, as well as maintaining the cooling bath's prescribed temperature of -65°C.

Testing was performed within SLFi's laboratory. This room is air conditioned to 25°C, +/- 1°C. The average humidity in the room varied unpredictably, but was typically greater than 60%. A shade was used to prevent direct sunlight from striking the chilled bath, minimizing any error due to radiative heating. As well, the base of the bath was lightly insulated to minimize heat transfer from the metal table.

Various test fluids were used to determine the effect of additive concentration on the low temperature fluidity. These samples had a predetermined amount of additive in a standard base fluid. Two common refrigeration compressor lubricants that are currently available on the market were tested for additional comparison.

## Results

The test results were saved to comma separated value files to be analyzed on a personal computer. These results tables were used to produce various graphical displays of the test data. The data was specifically organized to evaluate two different objectives.

The first objective was to quantify the effect of additive concentration on the low temperature fluidity of the fluids. Only the SLFi prepared samples were compared for this evaluation. Typically, with the additive that was analyzed, there is a specific region of concentration that the additive is most effective. Over- or under-dosing of the additive negates the positive effects of it. Understanding this effective region is critical for determining the acceptable margin of error for additive dosing when blending the final lubricant. The resulting data from this portion of the experiment is priority information.

The next step of the analysis was to compare the low temperature fluidity of SLFi products to that of leading competitors. Samples of a leading naphthenic and paraffinic mineral oil ammonia refrigeration lubricant were obtained by SLFi staff on the open market. These samples were run on the viscometer, along with a sample from a production batch of SLFi's R717AC-68 product. The table and graph shows the results of this test.

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Table 1: Low Temperature Fluidity Results - Three Ammonia Refrigeration Lubricants

Lubricant	Dynamic Viscosity at Specified Temperature (cP)									
	0°C	-5°C	-10°C	-15°C	-20°C	-25°C	-30°C	-35°C	-40°C	-45°C
Naphthenic	1,083	1,760	2,736	4,322	6,813	20,450	51,885	182,400	779,572	3,662,823
Paraffinic	635	937	1,353	1,975	3,063	5,388	13,815	46,331	247,586	>2,500,000
SLFi R717AC-68	309	511	808	1,157	1,754	2,585	4,131	8,266	28,884	267,977

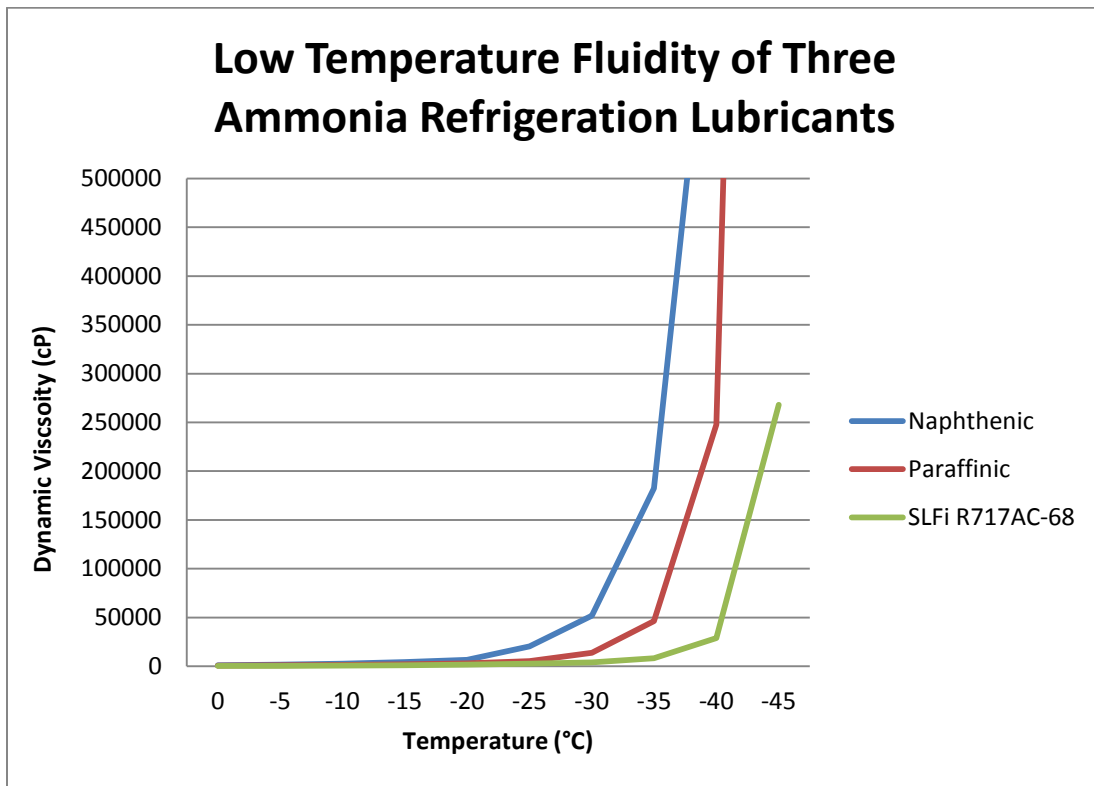


Figure 3: Low temperature fluidity results in graphical form.

The results clearly indicate the advantage of SLFi's advanced formulation in regards to low temperature fluidity. The naphthenic fluid began to approach pour point, exhibiting a dramatic rise in viscosity, at approximately -30°C. The standard paraffinic showed similar characteristics at approximately -35°C. At -45°C, SLFi-R717AC-68 is just under 300,000 cP while the competitor fluids are well beyond their pour point.

A more important consideration is the viscosity of the fluids as they approach their pour point. Each fluid undergoes a dramatic exponential rise in viscosity past a certain temperature. The



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location of this rise has been termed as the viscosity knuckle. Once the lubricant has reached this point, it has become significantly more resistant to flow. Once the viscosity surpasses approximately 1,500,000 cP, fluid mobility is severely restricted.

## **Conclusions**

While this study has not been concluded in full, initial results are promising. SLFi has successfully developed a method for studying the critical low temperature fluidity of a fluid using methods not commonly employed by the industry. This has enabled more detailed analysis of the behavior of lubricants as they approach their solidification point, allowing further understanding of the fluid and the effect of additives.

For initial analysis, three common ammonia refrigeration lubricants were chosen. Low temperature fluidity of these lubricants is critical to ensuring consistent oil return and prevents fouling of heat exchangers due to lubricant solidification. The data collected in this initial study has shown that current SLFi formulations can offer, on average, a 143% improvement in low temperature fluidity compared to naphthenic based formulations and 92% for paraffinic based. This will result in improved oil return and overall system efficiency resulting from the reduced energy losses due to viscous drag forces within the system.

SLFi will continue to research new and innovative techniques, such as the low temperature fluidity testing described here in, to continuously improve lubricant formulations. As testing continues, future reports will be drafted summarizing progress.