Evaluation of High Ester Content PAO Based Single Hydraulic Fluid in High Humidity Conditions

November 1994

By Ellen M. Purdy
USA Tank Automotive Command
Mobility Technology Center Belvoir

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<table>
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Mobility Technology Center Belvoir
Fuels and Lubricants Division
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Section I  Introduction

The Army currently uses three military specification hydraulic fluids for its ground equipment; MIL-H-6083 (OHT); MIL-H-46170 (FRH); and MIL-H-5606 (OHA). In an attempt to decrease the logistic burden of using three distinct fluids, a single fluid was developed by the Belvoir Research, Development, and Engineering Center (BRDEC). The fluid is required to maintain the same level of fire resistance available from MIL-H-46170 (FRH), as well as provide the same level of low temperature operability and elastomer (seal) swell as MIL-H-5606 (OHA) and MIL-H-6083 (OHT), petroleum based hydraulic fluids which have relatively little fire resistance. Single hydraulic fluid (SHF), will use the same chemistry as FRH, but provide the same low temperature viscosities as OHA and OHT. Development of SHF is an attempt to provide the best of the three fluids while eliminating their deficiencies in one single fluid.
Section 2  Background

SHF is formulated primarily as a polyalphaolefin (PAO) and ester based fluid. The ester in the fluid formulation is intended to act as an elastomer swell agent. Traditionally, PAO based fire resistant hydraulic fluids are formulated with diesters with a limit of 30% by weight being the maximum allowed under MIL-H-46170. This limitation has been imposed due to the ester’s propensity to absorb water from the atmosphere. This absorption of water increases the likelihood of corrosion and affects the low temperature viscosity. The 30% limit represents a compromise between the tendency of the fluid to absorb water and the amount of elastomer swell required by hydraulic systems sealing materials. Usually the ester in typical PAO/ester based fluids provide a volume swell for the standard NBR-L rubber of 15% - 19% which is used for qualification acceptance testing. There is a debate among hydraulic system users that this is insufficient elastomer swell to prevent excess leakage, and that the minimum amount of acceptable swell is 19%. 4,5,6,7,8

SHF requirements do not impose an ester content limit, but do require the minimum 19% elastomer swell. If a limit is to be included in the specification requirements for SHF, it will most likely be increased to 40% ester. To determine that this increased limit is not detrimental to fluid performance, formulations of varying ester contents were subjected to humidification then tested against the required performance targets for SHF.
Four formulations for SHF were prepared with ester contents varying from 34% to 40%. The ester used in these formulations is an isodecyl ester rather than a diester. Isodecyl esters were chosen for SHF because they do provide a significant level of elastomer swell, but also maintain high flash points and low viscosities at low temperatures. Typical viscosity values for these esters are 750 cSt at -54°C with a 170°C flash point. Diesters typically exhibit viscosities above 7,000 cSt at -54°C but have excellent flash points of 200°C or higher. The 4 formulations summarized in Table 1 below are formulations developed by the Fuels and Lubricants Division which successfully passed all SHF proposed performance requirements.

<table>
<thead>
<tr>
<th>COMPONENT</th>
<th>SHF1</th>
<th>SHF2</th>
<th>SHF3</th>
<th>SHF4</th>
</tr>
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<tbody>
<tr>
<td>% 2 cSt PAO Basestock</td>
<td>15</td>
<td>14.5</td>
<td>11.2</td>
<td>10.8</td>
</tr>
<tr>
<td>% 4 cSt PAO Basestock</td>
<td>45</td>
<td>43.5</td>
<td>44.8</td>
<td>43.2</td>
</tr>
<tr>
<td>% Isodecyl Ester</td>
<td>34</td>
<td>36</td>
<td>38</td>
<td>40</td>
</tr>
<tr>
<td>% Corrosion Inhibitor</td>
<td>3</td>
<td>3</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>% Anti-Wear Additive</td>
<td>2</td>
<td>2</td>
<td>2</td>
<td>2</td>
</tr>
<tr>
<td>% Antioxidant</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
</tr>
</tbody>
</table>

The above formulations were tested against SHF performance requirements with results summarized in Table 2 below. The tests identified in Table 2 were deemed to be the fluid characteristics and/or performance most likely affected by excessive water absorption. This data establishes baseline performance against which the humidified fluid performance will be evaluated.

<table>
<thead>
<tr>
<th>Test</th>
<th>SHF1</th>
<th>SHF2</th>
<th>SHF3</th>
<th>SHF4</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flash Point</td>
<td>186°C</td>
<td>185°C</td>
<td>185°C</td>
<td>186°C</td>
</tr>
<tr>
<td>Flash Point</td>
<td>200°C</td>
<td>196°C</td>
<td>197°C</td>
<td>193°C</td>
</tr>
<tr>
<td>Pour Point</td>
<td>-65°C</td>
<td>-65°C</td>
<td>-65°C</td>
<td>-65°C</td>
</tr>
<tr>
<td>100°C Viscosity</td>
<td>2.56 cSt</td>
<td>2.57 cSt</td>
<td>2.59 cSt</td>
<td>2.56 cSt</td>
</tr>
<tr>
<td>40°C Viscosity</td>
<td>9.12 cSt</td>
<td>8.70 cSt</td>
<td>8.77 cSt</td>
<td>8.07 cSt</td>
</tr>
<tr>
<td>-40°C Viscosity</td>
<td>665 cSt</td>
<td>636 cSt</td>
<td>668 cSt</td>
<td>630 cSt</td>
</tr>
<tr>
<td>-54°C Viscosity</td>
<td>3427 cSt</td>
<td>3033 cSt</td>
<td>2887 cSt</td>
<td>2979 cSt</td>
</tr>
<tr>
<td>Low Temp Stability</td>
<td>pass</td>
<td>pass</td>
<td>pass</td>
<td>pass</td>
</tr>
<tr>
<td>Ox/Corr (FTM 5308)</td>
<td>pass</td>
<td>pass</td>
<td>pass</td>
<td>pass</td>
</tr>
<tr>
<td>Corrosion (hrs/hrs)*</td>
<td>272/336</td>
<td>248/240</td>
<td>214/377</td>
<td>272/336</td>
</tr>
<tr>
<td>Water Sensitivity</td>
<td>&gt; 90%</td>
<td>&gt; 90%</td>
<td>&gt; 90%</td>
<td>&gt; 90%</td>
</tr>
<tr>
<td>Evaporation Loss</td>
<td>33%</td>
<td>33%</td>
<td>30%</td>
<td>32%</td>
</tr>
</tbody>
</table>

*These values represent the number of hours before failure on a sandblasted surface (first number) and a polished surface (second number).
The 4 test fluids were subjected to humidification at 85% relative humidity with the water content determined daily (see Appendix A for humidification procedure). When an asymptotic rate of humidification was exhibited, the fluids were removed from the humidified environment for performance evaluation. Figure 1 shows the water absorption of the 4 SHF formulations. Over a total period of 288 hours, the maximum water content for the fluids never exceeded 0.25% water by weight. While the differences in water content among the four fluids are minimal, the formulation containing the highest amount of ester (40%) did exhibit the greatest water absorption.

![Graph showing water absorption of SHF formulations over time]

Figure 1. Water Absorption of SHF Formulations

4 Evaluation of High Ester Content PAO Based Single Hydraulic Fluid in High Humidity Conditions
Section 4  Results

The viscosities of the humidified fluids are summarized in Table 3. Included in this table are the SHF performance targets. As can be seen, all of the fluids except SHF3 met the viscosity requirements for SHF even when humidified. The viscosities at the lower temperatures, although within requirements (except SHF3 at -54°C) are significantly greater than the baseline results. It would be expected that if the viscosity increases were due only to the additional amount of water in the fluids, SHF4 would exhibit the highest viscosity at the low temperatures, but such is not the case.

Table 3. Humidified Fluid Viscosities

<table>
<thead>
<tr>
<th>FLUID</th>
<th>40°C</th>
<th>100°C</th>
<th>-40°C</th>
<th>-54°C</th>
</tr>
</thead>
<tbody>
<tr>
<td>SHF Requirement</td>
<td>19.5 max</td>
<td>2.5 min</td>
<td>800 max</td>
<td>3500 max</td>
</tr>
<tr>
<td>SHF1</td>
<td>8.62</td>
<td>2.58</td>
<td>710</td>
<td>3131</td>
</tr>
<tr>
<td>SHF2</td>
<td>8.88</td>
<td>2.57</td>
<td>702</td>
<td>3445</td>
</tr>
<tr>
<td>SHF3</td>
<td>8.80</td>
<td>2.54</td>
<td>702</td>
<td>3721</td>
</tr>
<tr>
<td>SHF4</td>
<td>8.74</td>
<td>2.57</td>
<td>695</td>
<td>3356</td>
</tr>
</tbody>
</table>

Flash point/fire point and pour point results are summarized in Table 4 along with the SHF target requirements. As can be seen, all 4 humidified formulations were well above the minimum flash/fire point requirements for SHF, but exhibited little difference from baseline data obtained from the non-humidified fluids.

Table 4. Flash/Fire Point and Pour Point

<table>
<thead>
<tr>
<th>FLUID</th>
<th>FLASH</th>
<th>FIRE</th>
<th>POUR</th>
</tr>
</thead>
<tbody>
<tr>
<td>SHF Requirement</td>
<td>180°C min</td>
<td>190°C min</td>
<td>-60°C min</td>
</tr>
<tr>
<td>SHF1</td>
<td>182°C</td>
<td>190°C</td>
<td>-66°C</td>
</tr>
<tr>
<td>SHF2</td>
<td>182°C</td>
<td>196°C</td>
<td>-66°C</td>
</tr>
<tr>
<td>SHF3</td>
<td>84°C</td>
<td>198°C</td>
<td>-66°C</td>
</tr>
<tr>
<td>SHF4</td>
<td>184°C</td>
<td>196°C</td>
<td>-66°C</td>
</tr>
</tbody>
</table>

Also no perceptible change in pour point was exhibited for the humidified fluids when compared to the non-humidified fluids.

One of the more critical tests performed on the humidified fluids involved low temperature stability. The humidified samples were subjected to -54°C for a period of 72 hours after which they were removed and examined for any signs of instability. Each of the fluids exhibited no signs of gelling, separation, or precipitation of additives. No
crystallization was present which was the most important criteria. Some synthetic fluids that absorb water will exhibit signs of crystallization indicating a separation of the water from the fluid at low temperatures. The humidified fluids provide the same stability at low temperatures as the unhumidified SHF.

Another significant test for synthetic hydraulic fluids involves light transmittance (see MIL-H-46170 for procedure). The percentage of light transmittance of a fluid is a measure of its sensitivity to water. This water sensitivity test was developed specifically to detect hydraulic fluid formulations which may contain water sensitive additive ingredients. The test involves doping 250 ml fluid samples with 0.5 ml of reagent grade water and allowing the fluids to sit for a period of 24 hours. An untreated sample is used to set the light transmittance standard at 100%, then the doped sample is tested. A fluid is considered sufficiently insensitive to water if the transmittance is 90% or greater. The humidified fluids exhibited transmittance rates between 96% and 100%, which further supports the stability of the high ester content formulations.

Evaporation Loss was tested for the humidified fluids. It was expected that if the fluids did pick up significant amounts of water, the evaporation loss would increase. As can be seen in Table 5, no significant change in evaporation occurred for the humidified fluids. This is not unusual since the greatest water content for the four test fluids was less than 0.25%.

Table 5. Evaporation Loss for Humidified Fluids

<table>
<thead>
<tr>
<th>FLUID</th>
<th>SHF</th>
<th>SHF1</th>
<th>SHF2</th>
<th>SHF3</th>
<th>SHF4</th>
</tr>
</thead>
<tbody>
<tr>
<td>% evaporation</td>
<td>35% max</td>
<td>31.6</td>
<td>32.0</td>
<td>31.1</td>
<td>30.6</td>
</tr>
</tbody>
</table>

Two final tests performed on the humidified fluids involve rust protection and oxidation/corrosion stability and are the two performance aspects which could be most significantly degraded by the presence of water. The humidity cabinet test is a severe test of a fluid's ability to inhibit corrosion. Steel panels which are polished on one side and sandblasted on another are subjected to a minimum 100 hours in the humidity cabinet. Failure occurs for each side when 3 rust spots greater than 1 mm in diameter appear. Each of the humidified fluids passed the 100 hour requirement but did exhibit a loss of protection due to the higher water content versus the un-humidified fluids. None of the fluid samples provided sufficient protection beyond 172 hours. This is a significant reduction in protection given that each of the un-humidified fluids provided at least 210 hours of protection.

The final test performed on the humidified fluids was Federal Test Method 791-5308 which was conducted for 168 hours (7 days) at 135°C (maximum operating temperature identified for SHF). The metal coupons of steel, magnesium, aluminum, cadmium, and copper showed no signs of oxidation or corrosion with minimal weight changes below the +0.2 mg/sqcm (+0.6 mg/sqcm for copper) requirement.
Table 6. Viscosity Change for Humidified Fluids

<table>
<thead>
<tr>
<th>FLUID</th>
<th>Initial Vis</th>
<th>Final Vis</th>
<th>% Change</th>
</tr>
</thead>
<tbody>
<tr>
<td>SHF1</td>
<td>8.62 cSt</td>
<td>9.69 cSt</td>
<td>12.41%</td>
</tr>
<tr>
<td>SHF2</td>
<td>8.88 cSt</td>
<td>9.66 cSt</td>
<td>8.78%</td>
</tr>
<tr>
<td>SHF3</td>
<td>8.80 cSt</td>
<td>9.44 cSt</td>
<td>7.27%</td>
</tr>
<tr>
<td>SHF4</td>
<td>8.74 cSt</td>
<td>9.35 cSt</td>
<td>6.98%</td>
</tr>
</tbody>
</table>

With the exception of SHF1, the humidified fluids exhibited acceptable viscosity changes of less than 10% (see Table 6), although the changes are somewhat higher than those normally exhibited by non-humidified fluids. The amount of water absorption does appear to have a slight affect on the viscosity change of the fluid, but the amount of ester does not seem to directly influence this change. SHF1 has the least amount of ester yet the greatest change in viscosity after being subjected to high humidity and the severe conditions of the FTM-791-5308 test.
Section 5  Conclusions

From the test results discussed above, it can be concluded that the use of isodecyl esters at treat rates between 34% and 40% do not adversely affect the fluid's performance. While water absorption does occur over time when the fluid is subjected to high humidity, very little water is actually picked up. Previous work in the development of these formulations reveals that ester contents of 34% to 40% provide elastomer swell for NBR-L rubber in the range of 19% to 22% which meets the requirements for SHF. Since the humidified fluids were able to perform satisfactory when evaluated against SHF requirements, there is no reason to limit the ester content to below 40%.
References


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ATTN WL/POSF
ATTN WL/POSL
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WRIGHT PATTERSON AFB
OH 45433-7750
AIR FORCE WRIGHT LAB
ATTN WL/MLSE
2179 12TH ST STE 1
WRIGHT PATTERSON AFB
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