Use of CTFE as an Additive to Enhance Fire Resistance of Single Hydraulic Fluid

November 1994

By Ellen M. Purdy
USA Tank Automotive Command
Mobility Technology Center Belvoir
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Use of CTFE as an Additive to Enhance Fire Resistance of Single Hydraulic Fluid

A Polyalphaolefin/ester base fire resistant hydraulic fluid was reformulated to include chlorotrifluoroethylene (CTFE) as an additive to enhance fire resistance. CTFE was added to the formulation at 2%-10% by weight. Each of the formulations were evaluated for performance and flammability. Addition of the CTFE was found to promote excess foaming when tested for foaming characteristics. The formulations were adjusted to include an anti-foam additive to counteract this effect. The new formulations were found to provide satisfactory performance and improvement in fire resistance. Addition of CTFE increased the flash and fire points of the fluid. In addition, the autoignition temperature, hot manifold spray ignition temperature were both increased above what is available from MIL-H-461470, the current military specification fire resistant hydraulic fluid.

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Use of CTFE as a Additive to Enhance the Fire Resistance of Single Hydraulic Fluid
Section 2  Technical Approach

The approach for this endeavor involved determining the proper additive level of CTFE to the previously formulated SHF. The solubility of CTFE could not be exceeded such that additives precipitated out while under extreme conditions, the metallurgy of current hydraulic systems was not attacked, or the elastomer materials degraded. In order to insure that each of these problems was avoided, the formulation/evaluation effort proceeded in a systematic manner to allow each aspect of the fluid's performance to be evaluated individually.

The first step was to determine the solubility limits of the CTFE, and thus bound the formulation possibilities. Upon determining the maximum amount of CTFE that effectively remained in solution, the point of marginal performance return was identified. This optimization technique placed a further limit on the amount of CTFE that was incorporated into the formulation. Many times, when formulating a fluid, a point is reached where additional amounts of additive result in very little improvement of performance - the point of diminishing returns. After these criteria were established, the formulations were prepared and evaluated. The evaluation was conducted sequentially with increasing complexity of functional performance analysis. Such an approach allowed the formulation to be increasingly optimized as the evaluation progressed.
Section 3  Results

Initial screening of the solubility limits of the CTFE was conducted to determine the maximum amount that would go into solution in SHF. CTFE was found to be soluble up to 20% by weight before precipitation was evidenced. Formulations with incremental additions of CTFE were prepared and tested for flash and fire point to identify the point of diminishing returns on performance. Flash and fire points were chosen as evaluation criteria because the objective of the exercise was to improve fire resistance. An increase in these characteristics is usually (but not necessarily always) indicative of an increase in the fire resistance of the fluid. Test results indicated that very little gain in flash and fire point was available beyond 10% addition of CTFE. With this limitation identified, five formulations were developed for further investigation (see Table 1).

Table 1. Formulations to be Investigated

<table>
<thead>
<tr>
<th>COMPONENT</th>
<th>SHFC1</th>
<th>SHFC2</th>
<th>SHFC3</th>
<th>SHFC4</th>
<th>SHFC5</th>
</tr>
</thead>
<tbody>
<tr>
<td>2 cSt PAO</td>
<td>15%</td>
<td>15%</td>
<td>15%</td>
<td>15%</td>
<td>15%</td>
</tr>
<tr>
<td>4 cSt PAO</td>
<td>45%</td>
<td>45%</td>
<td>45%</td>
<td>45%</td>
<td>45%</td>
</tr>
<tr>
<td>Ester</td>
<td>32%</td>
<td>30%</td>
<td>28%</td>
<td>26%</td>
<td>24%</td>
</tr>
<tr>
<td>Corrosion Inhibit</td>
<td>3%</td>
<td>3%</td>
<td>3%</td>
<td>3%</td>
<td>3%</td>
</tr>
<tr>
<td>Anti-Wear</td>
<td>2%</td>
<td>2%</td>
<td>2%</td>
<td>2%</td>
<td>2%</td>
</tr>
<tr>
<td>Anti-Oxidant</td>
<td>1%</td>
<td>1%</td>
<td>1%</td>
<td>1%</td>
<td>1%</td>
</tr>
<tr>
<td>CTFE</td>
<td>2%</td>
<td>4%</td>
<td>6%</td>
<td>8%</td>
<td>10%</td>
</tr>
</tbody>
</table>

The PAO based fluid is composed of a blend of 2 cSt and 4 cSt PAO basestocks and an ester which enable the fluid to meet viscosity, fire resistance, and elastomer swell requirements. The ester used for this fluid is an iso-decyl ester which has a high flash point, low viscosity at low temperatures and provides the desired seal swell. When incorporating the CTFE into the formulation, the ester content was reduced by the same weight percentage that the CTFE was added. The reduction in ester content will create a slight decrease in the elastomer swell, but this effect should be offset by the elastomer swell characteristics of the CTFE, thus no loss of performance should occur.

The above formulations were evaluated for flash and fire point, viscosities, pour point, evaporation loss, wear characteristics, foaming, and low temperature stability. Results from these tests were compared against requirements for the desired Single Hydraulic Fluid (see Table 2) to determine if a level of performance improvement has been obtained. Table 2 compares the performance requirements for the current fire resistant hydraulic fluid (FRH, MIL-H-46170) and SHF. It should be noted that SHF provides superior low temperature performance, oxidation/corrosion stability, and seal swell over FRH. The results of the evaluation of the CTFE formulations against SHF performance are summarized in Tables 3-6 below.
Table 2. Requirements for Desirable Military Hydraulic Fluid

<table>
<thead>
<tr>
<th>PERFORMANCE TEST</th>
<th>MIL-L-46170</th>
<th>SHF</th>
</tr>
</thead>
<tbody>
<tr>
<td>Oxidation/Corrosion</td>
<td>168 hrs @ 121°C</td>
<td>168 hrs @ 135°C</td>
</tr>
<tr>
<td>ASTM D4636, #3</td>
<td>vis. &lt; 10%</td>
<td>vis. &lt; 10%</td>
</tr>
<tr>
<td>Corrosion Inhibition</td>
<td>100 hrs</td>
<td>100 hrs</td>
</tr>
<tr>
<td>ASTM D1748</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Galvanic Corrosion</td>
<td>10 days</td>
<td>10 days</td>
</tr>
<tr>
<td>FTM 5322</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Low Temp Stability</td>
<td>72 hrs @ -54°C</td>
<td>72 hrs @ -54°C</td>
</tr>
<tr>
<td>FTM 3458</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Pour Point</td>
<td>-60°C</td>
<td>-60°C</td>
</tr>
<tr>
<td>ASTM D97</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Viscosity @ 40°C</td>
<td>19.5 cSt max</td>
<td>19.5 cSt max</td>
</tr>
<tr>
<td>ASTM D445</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Viscosity @ 100°C</td>
<td>3.4 cSt min</td>
<td>2.5 cSt min</td>
</tr>
<tr>
<td>ASTM D445</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Viscosity @ -40°C</td>
<td>2600 cSt max</td>
<td>800 cSt max</td>
</tr>
<tr>
<td>ASTM D445</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Viscosity @ -54°C</td>
<td>report</td>
<td>3500 cSt max</td>
</tr>
<tr>
<td>ASTM D445</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Solid particle Count</td>
<td>10,000 max @</td>
<td>10,000 max @</td>
</tr>
<tr>
<td>MIL-H-46170</td>
<td>5-25 micrometers</td>
<td>5-15 micrometers</td>
</tr>
<tr>
<td>Solid particle count</td>
<td>250 max @</td>
<td>1,000 max @</td>
</tr>
<tr>
<td>MIL-H-46170</td>
<td>26-50 micrometers</td>
<td>16-25 micrometers</td>
</tr>
<tr>
<td>Solid particle count</td>
<td>50 max @</td>
<td>150 max @</td>
</tr>
<tr>
<td>MIL-H-46170</td>
<td>51-100 micrometers</td>
<td>26-50 micrometers</td>
</tr>
<tr>
<td>Solid particle count</td>
<td>10 max @ over</td>
<td>20 max @</td>
</tr>
<tr>
<td>MIL-H-46170</td>
<td>100 micrometers</td>
<td>51-100 micrometers</td>
</tr>
<tr>
<td>Solid particle count</td>
<td>5 max @ over</td>
<td>100 micrometers</td>
</tr>
<tr>
<td>MIL-H-46170</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Acid Number</td>
<td>0.2 gm KOH/gm max</td>
<td>0.3 gm KOH/gm max</td>
</tr>
<tr>
<td>ASTM D664</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Elastomer Swell</td>
<td>15% - 25%</td>
<td>19% - 30%</td>
</tr>
<tr>
<td>FTM 3603</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Use of CTFE as a Additive to Enhance the Fire Resistance of Single Hydraulic Fluid
Table 2. Requirements for Desirable Military Hydraulic Fluid - continued

<table>
<thead>
<tr>
<th>PERFORMANCE TEST</th>
<th>MIL-L-46170</th>
<th>SHF</th>
</tr>
</thead>
<tbody>
<tr>
<td>Evaporation Loss</td>
<td>5% max</td>
<td>35% max</td>
</tr>
<tr>
<td>ASTM D972</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Steel on Steel Wear</td>
<td>0.3 mm max @</td>
<td>0.3 mm max @</td>
</tr>
<tr>
<td>ASTM D4172</td>
<td>10 kg load</td>
<td>10 kg load</td>
</tr>
<tr>
<td>Steel on Steel Wear</td>
<td>0.65 mm max @</td>
<td>0.65 mm max @</td>
</tr>
<tr>
<td>ASTM D4172</td>
<td>40 kg load</td>
<td>40 kg load</td>
</tr>
<tr>
<td>Foam Characteristics</td>
<td>65 ml max</td>
<td>65 ml max</td>
</tr>
<tr>
<td>ASTM D892</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Water Content</td>
<td>500 ppm max</td>
<td>100 ppm max</td>
</tr>
<tr>
<td>ASTM D1744</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Flash Point</td>
<td>219°C min</td>
<td>180°C min</td>
</tr>
<tr>
<td>ASTM D92</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Fire Point</td>
<td>246°C min</td>
<td>190°C min</td>
</tr>
<tr>
<td>ASTM D92</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Autoignition Temp</td>
<td>343°C min</td>
<td>325°C min</td>
</tr>
<tr>
<td>ASTM E659</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Hi Temp/Hi Press Ignt</td>
<td>no continuation of burning when ignition source is removed</td>
<td>no continuation of burning when ignition source is removed</td>
</tr>
<tr>
<td>FTM 6052</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Flame Propagation</td>
<td>0.3 cm/sec max</td>
<td>0.3 cm/sec max</td>
</tr>
<tr>
<td>MIL-H-83282</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Storage Stability</td>
<td>12 months</td>
<td>12 months</td>
</tr>
<tr>
<td>FTM 3465</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**VISCOSITY CHARACTERISTICS**

When comparing the results of the viscosity determinations for the above formulations to the original SHF formulation without the CTFE, there is a consistent and significant decrease in viscosity at -54°C (see Table 3). Viscosities at the other temperatures are consistent with results for the original formulation. The one exception is the 100°C viscosity for SHFC5, which is below the minimum desirable requirement for the hydraulic fluid (see Table 2), and thus not acceptable. All other viscosities are satisfactory when compared to the criteria established in Table 2. Given that the viscosities remain the same except for the decrease at -54°C, the addition of the CTFE has caused an improvement in performance of the fluid. Viscosities below 3500 cSt are imminently desirable as long as the fluid maintains at least 2.5 cSt at 100°C. Formulations 1-4 meet this requirement.
Pour, Flash, and Fire Point Characteristics

No perceptible change in pour point was evidenced when comparing the CTFE formulations to the non-CTFE formulation. The flash and fire points, however, for the CTFE formulations all show an increase over that obtained for the original formulation. The non-CTFE formulation exhibits a flash point of 186°C and fire point of 197°C. The addition of CTFE in 2% to 8% by weight results in a minimum 6°C increase for flash point and 7°C increase for fire point. SHFC5 which contains 10% CTFE only exhibited a 2°C increase for flash point and 3°C increase for fire point. Given the unacceptable high temperature viscosity and minimum improvement in flash and fire point, SHFC5 is not recommended as a candidate for improved fire resistance SHF.

Table 4. Pour, Flash, and Fire Point (°C)

<table>
<thead>
<tr>
<th>FLUID</th>
<th>POUR</th>
<th>FLASH</th>
<th>FIRE</th>
</tr>
</thead>
<tbody>
<tr>
<td>SHFC1</td>
<td>below -65</td>
<td>192</td>
<td>204</td>
</tr>
<tr>
<td>SHFC2</td>
<td>below -65</td>
<td>194</td>
<td>204</td>
</tr>
<tr>
<td>SHFC3</td>
<td>below -65</td>
<td>192</td>
<td>204</td>
</tr>
<tr>
<td>SHFC4</td>
<td>below -65</td>
<td>192</td>
<td>208</td>
</tr>
<tr>
<td>SHFC5</td>
<td>below -65</td>
<td>188</td>
<td>200</td>
</tr>
</tbody>
</table>

Low Temperature Stability, Evaporation Loss, and Wear Characteristics

When all 5 formulations were tested for low temperature stability they met the criteria established in Table 2. When the test was repeated for verification, however, SHFC3 exhibited a permanent precipitation of additives. The precipitate would not return to solution upon heating the sample. This may be anomalous behavior in that the precipitate only appeared in this one sample of SHFC3. Other preparations of SHFC3 passed the low temperature stability test.
While only one of the formulations (SHFC1) met the requirements for evaporation loss, the results are still acceptable. Because hydraulic fluids are used in closed systems, the evaporation loss is not of critical importance to the fluid’s performance abilities. MIL-H-6083 allows up to 70% evaporation when tested at a lower temperature than the SHFC fluids were tested. An evaporation loss of 40% is still satisfactory for military hydraulic fluid, thus the candidates are still acceptable for consideration as an improved fluid. The results of the wear tests also indicate the fluids provide satisfactory performance. The 4-Ball Wear test exhibits good correlation with fluid performance in hydraulic pump endurance tests. That each of the fluids met the 65 mm maximum wear scar requirements suggests that they should perform well in future pump testing.

Table 5. Low Temp Stability, Evaporation Loss and Wear Characteristics

<table>
<thead>
<tr>
<th>FLUID</th>
<th>LOW TEMP ST</th>
<th>EVAP LOSS (%)</th>
<th>WEAR SCAR (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SHFC1</td>
<td>Pass</td>
<td>33.6%</td>
<td>0.533</td>
</tr>
<tr>
<td>SHFC2</td>
<td>Pass</td>
<td>35.9%</td>
<td>0.465</td>
</tr>
<tr>
<td>SHFC3</td>
<td>Pass</td>
<td>37.7%</td>
<td>0.473</td>
</tr>
<tr>
<td>SHFC4</td>
<td>Pass</td>
<td>40.2%</td>
<td>0.474</td>
</tr>
<tr>
<td>SHFC5</td>
<td>Pass</td>
<td>40.5%</td>
<td>0.467</td>
</tr>
</tbody>
</table>

FOAMING CHARACTERISTICS

When the fluids were tested for foaming characteristics, they all failed significantly. For each sequence of foaming, the first number represents the volume of foam generated during the aeration period, while the second number represents the volume of foam remaining after a 10 minute settling period. The maximum volume of foam allowed is 65 ml. None of the fluids could meet this requirement for all three sequences of testing (see Table 6). The CTFE acts as a pro-foamant when in the presence of the PAO/ester blend, thus an anti-foamant additive was necessary in the formulation.

Table 6. Foaming Characteristics (ml)

<table>
<thead>
<tr>
<th>FLUID</th>
<th>SEQUENCE I</th>
<th>SEQUENCE II</th>
<th>SEQUENCE III</th>
</tr>
</thead>
<tbody>
<tr>
<td>SHFC1</td>
<td>20 - 0</td>
<td>70 - 0</td>
<td>200 - 0</td>
</tr>
<tr>
<td>SHFC2</td>
<td>190 - 0</td>
<td>70 - 0</td>
<td>230 - 0</td>
</tr>
<tr>
<td>SHFC3</td>
<td>330 - 0</td>
<td>80 - 0</td>
<td>200 - 0</td>
</tr>
<tr>
<td>SHFC4</td>
<td>370 - 0</td>
<td>60 - 0</td>
<td>300 - 0</td>
</tr>
<tr>
<td>SHFC5</td>
<td>530 - 0</td>
<td>60 - 0</td>
<td>230 - 0</td>
</tr>
</tbody>
</table>

Use of CTFE as a Additive to Enhance the Fire Resistance of Single Hydraulic Fluid 7
Due to time constraints, no attempt at optimization of the formula was made when determining the additive amount of anti-foam agent. Most treat rates require anywhere from 0.01% to 0.5% in order to control foaming. It was decided to add 0.5% anti-foam agent to determine if the foaming could be brought under control. When the new formulations were prepared and tested, no foaming developed at all during the aeration period. This suggested that 0.5% is significantly more additive than is required. Future efforts will include optimizing the amount of additive required for desired fluid performance.

CORROSION INHIBITION PERFORMANCE

The fluids were tested for both corrosion resistance in high temperature and humidity, and under galvanic conditions. Table 7 summarizes the results for both types of tests. The high temperature/high humidity test requires separate evaluation for a polished side of the test panel as well as a sandblasted side. In each case, the sandblasted side exhibited rust spots at a much earlier time than the polished side, yet the performance is satisfactory for both sides in that the fluid is only required to provide protection for 100 hours for each side. Each of the fluids also provided satisfactory galvanic corrosion protection, in that no signs of pitting, etching, or discoloration were evident on the steel test pieces at the end of the 7 day test.

Table 7. Corrosive Protection

<table>
<thead>
<tr>
<th>FLUID</th>
<th>SANDBLAST</th>
<th>POLISHED</th>
<th>GALVANIC</th>
</tr>
</thead>
<tbody>
<tr>
<td>SHFC1</td>
<td>487 hrs</td>
<td>551 hrs</td>
<td>168 hrs</td>
</tr>
<tr>
<td>SHFC2</td>
<td>168 hrs</td>
<td>641 hrs</td>
<td>168 hrs</td>
</tr>
<tr>
<td>SHFC3</td>
<td>-</td>
<td>-</td>
<td>168 hrs</td>
</tr>
<tr>
<td>SHFC4</td>
<td>431 hrs</td>
<td>584 hrs</td>
<td>168 hrs</td>
</tr>
<tr>
<td>SHFC5</td>
<td>217 hrs</td>
<td>635 hrs</td>
<td>168 hrs</td>
</tr>
</tbody>
</table>

FIRE RESISTANCE PERFORMANCE

As has been discussed above, the fluid formulations (with the exception of SHFC5) exhibited satisfactory laboratory performance. The formulations are of no advantage, however, unless the addition of CTFE results in a significant increase in fire resistance. While the fire and flash points did show an increase, these two characteristics are not entirely indicative of a fluid’s resistance to burning. Table 8 summarizes the results of the flammability testing (SHFC3 was not tested due to appearance of precipitation noted in low temperature stability evaluation). In each case, the fluids exceeded the minimum requirements for fire resistance under MIL-H-46170, which is the Army standard for fire resistant hydraulic fluid. The Linear Flame Propagation test determines how fast the burning fluid moves once it has ignited. The maximum propagation allowed is 0.3 cm/sec, yet each of the fluids exhibited no more than 0.23 cm/sec. The Hot Manifold
Spray Ignition Test (HMS Ignition) determines the temperature of a hot manifold at which the fluid will ignite upon contact with the manifold. MIL-H-46170 fluids exhibit HMS Ignition temperatures of 504°C while the CTFE containing fluids exhibited temperatures of 549°C or greater. The Auto Ignition Temperature of the fluids ranged from 353°C to 359°C which is 10 or more degrees higher than that required by MIL-H-46170. It is readily evident that the addition of CTFE to the PAO based hydraulic fluids does provide a measure of improved fire resistance.

Table 8. Fire Resistance

<table>
<thead>
<tr>
<th>FLUID</th>
<th>PROPAGATION</th>
<th>HMS IGNITION</th>
<th>AUTO IGNITION</th>
</tr>
</thead>
<tbody>
<tr>
<td>SHFC1</td>
<td>0.23 sec</td>
<td>549°C</td>
<td>357°C</td>
</tr>
<tr>
<td>SHFC2</td>
<td>0.20 sec</td>
<td>616°C</td>
<td>359°C</td>
</tr>
<tr>
<td>SHFC3</td>
<td>–</td>
<td>–</td>
<td>–</td>
</tr>
<tr>
<td>SHFC4</td>
<td>0.20 sec</td>
<td>627°C</td>
<td>353°C</td>
</tr>
<tr>
<td>SHFC5</td>
<td>0.20 sec</td>
<td>616°C</td>
<td>354°C</td>
</tr>
</tbody>
</table>

Use of CTFE as a Additive to Enhance the Fire Resistance of Single Hydraulic Fluid
Section 4 Conclusions

Initial laboratory and flammability evaluations reveal a significant improvement in SHF containing CTFE in fire resistance as well as other areas noted above. CTFE is known to cause excessive seal swell and to some extent deterioration of elastomeric materials, and also corrosion problems with certain metallurgy commonly found in existing hydraulic systems. These aspects of the fluid should be explored thoroughly via compatibility studies to insure that no long term problems with the fluid show up at a later time. The fluid should also undergo functional testing such as pump endurance testing and dynamic seal testing. Such tests represent close approximations of the actual conditions under which the fluid is expected to perform, thus they also would provide information regarding any possible long term problems with the addition of CTFE to SHF formulations. Successful performance in the compatibility studies and functional testing would substantiate the ability of a PAO/ester based fluid to provide superior fire resistance as well as low temperature operability.
References


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