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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

U.S. Army Tank-Automotive Command
Research, Development and Engineering Center
Warren, Michigan 48397-5000

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A Polyalphaelefn/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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By **Ellen M. Purdy**
USA Tank Automotive Command
Mobility Technology Center Belvoir
Fuels and Lubricants Division

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Section 1 Introduction and Background

The Army has long recognized the need for fire resistant hydraulic fluids for its armored systems. The fluids currently used by combat vehicles and artillery are MIL-H-6083 (OHT), Hydraulic Fluid, Petroleum Base, for Preservation and Operation, and MIL-H-46170 (FRH), Hydraulic Fluid, Rust Inhibited, Fire Resistant, Synthetic Hydrocarbon Base.[1,2] Each of these fluids has desirable characteristics, but exhibit qualities undesirable in a military hydraulic fluid. The OHT fluid has excellent low temperature properties but is characterized by a flash point of 80°C. FRH has a high flash point of 218°C, but poor low temperature performance. In order to improve both the performance of the fluid and its safety, improved formulations are required.

The objective of this endeavor was to provide the desired improved performance and increased safety by adding chlorotrifluoroethylene (CTFE) to a PAO based hydraulic fluid with good low temperature operability (Single Hydraulic Fluid, SHF).[3] Because CTFE is a completely non-flammable fluid, its addition as an additive to SHF was expected to result in a greater degree of fire resistance of the fluid. The difficulty in this approach, however, lay in the limited solubility of CTFE in PAO fluid, possible incompatibilities with other additives in the fluid, and incompatibilities with the metallurgy and elastomers of existing hydraulic systems.[4]

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

COMPONENT	SHFC1	SHFC2	SHFC3	SHFC4	SHFC5
2 cSt PAO	15%	15%	15%	15%	15%
4 cSt PAO	45%	45%	45%	45%	45%
Ester	32%	30%	28%	26%	24%
Corrosion Inhibit	3%	3%	3%	3%	3%
Anti-Wear	2%	2%	2%	2%	2%
Anti-Oxidant	1%	1%	1%	1%	1%
CTFE	2%	4%	6%	8%	10%

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

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

Table 2. Requirements for Desirable Military Hydraulic Fluid - continued

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

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.

Table 3. Viscosity Data (cSt)

FLUID	100°C	40°C	-40°C	-54°C
SHFC1	2.68	9.39	706	3234
SHFC2	2.68	9.50	63	2470
SHFC3	2.51	9.97	626	2927
SHFC4	2.58	9.06	649	2748
SHFC5	2.46	9.18	604	3083

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)

FLUID	POUR	FLASH	FIRE
SHFC1	below -65	192	204
SHFC2	below -65	194	204
SHFC3	below -65	192	204
SHFC4	below -65	192	208
SHFC5	below -65	188	200

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

FLUID	LOW TEMP ST	EVAP LOSS (%)	WEAR SCAR (mm)
SHFC1	Pass	33.6%	0.533
SHFC2	Pass	35.9%	0.465
SHFC3	Pass	37.7%	0.473
SHFC4	Pass	40.2%	0.474
SHFC5	Pass	40.5%	0.467

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)

FLUID	SEQUENCE I	SEQUENCE II	SEQUENCE III
SHFC1	20 - 0	70 - 0	200 - 0
SHFC2	190 - 0	70 - 0	230 - 0
SHFC3	330 - 0	80 - 0	200 - 0
SHFC4	370 - 0	60 - 0	300 - 0
SHFC5	530 - 0	60 - 0	230 - 0

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

FLUID	SANDBLAST	POLISHED	GALVANIC
SHFC1	487 hrs	551 hrs	168 hrs
SHFC2	168 hrs	641 hrs	168 hrs
SHFC3	–	–	168 hrs
SHFC4	431 hrs	584 hrs	168 hrs
SHFC5	217 hrs	635 hrs	168 hrs

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

FLUID	PROPAGATION	HMS IGNITION	AUTO IGNITION
SHFC1	0.23 sec	549°C	357°C
SHFC2	0.20 sec	616°C	359°C
SHFC3	–	–	–
SHFC4	0.20 sec	627°C	353°C
SHFC5	0.20 sec	616°C	354°C

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

1. Military Specification: MIL-H-6083, *Hydraulic Fluid, Petroleum Base for Preservation and Operation.*

2. Military Specification: MIL-H-46170, *Hydraulic Fluid, Rust Inhibited, Fire Resistant, Synthetic Hydrocarbon Base.*

3. "Development of a Single Hydraulic Fluid for Use in Army Ground Equipment," Ellen M. Purdy, Technical Report #2540, Belvoir Research, Development, and Engineering Center, October 1993.

4. "Information Compendium on Nonflammable Hydraulic Fluid and Design Requirements for its Adoption", Constance Van Brocklin, Technical Report #2486, Belvoir Research, Development, and Engineering Center, February 1990.

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CDR
MARINE CORPS SYS CMD
1 ATTN SSE
2033 BARNETT AVE STE 315
QUANTICO VA 22134-5010

CDR
BLOUNT ISLAND CMD
1 ATTN CODE 922/1
814 RADFORD BLVD
JACKSONVILLE
FLA 32226-3404

CDR
MARINE CORPS LOGISTICS BA
1 ATTN CODE 837
814 RADFORD BLVD
ALBANY GA 31704-1128

1 CDR
2ND MARINE DIV
PSC BOX 20090
CAMP LEJEUNNE
NC 28542-0090

1 CDR
1ST MARINE DIV
CAMP PENDLETON
CA 92055-5702

1 CDR
FMFPAC G4
BOX 64118
CAMP H M SMITH
HI 96861-4118

DEPARTMENT OF DEFENSE

ODUSD
1 ATTN (L) MRM
PETROLEUM STAFF ANALYST
PENTAGON
WASHINGTON DC 20301-8000

ODUSD
1 ATTN (ES) CI
400 ARMY NAVY DR
STE 206
ARLINGTON VA 22202

HQ USEUCOM
1 ATTN ECJU L1J
UNIT 30400 BOX 1000
APO AE 09128-4209

US CINCPAC
1 ATTN J422 BOX 64020
CAMP H M SMITH
HI 96861-4020

1 JOAP TSC
BLDG 780
NAVAL AIR STA
PENSACOLA FL 32408-5300

DIR DLA
1 ATTN DLA MMDI
ATTN DLA MMSB
CAMERON STA
ALEXANDRIA VA 22304-6100

CDR
DEFENSE FUEL SUPPLY CTR
1 ATTN DFSC Q BLDG 8
1 ATTN DFSC S BLDG 8
CAMERON STA
ALEXANDRIA VA 22304-6160

CDR
DEFENSE GEN SUPPLY CTR
1 ATTN DGSC SSA
1 ATTN DGSC STA
8000 JEFFERSON DAVIS HWY
RICHMOND VA 23297-5678

DIR ADV RSCH PROJ AGENCY
1 ATTN ARPA/ASTO
3701 N FAIRFAX DR
ARLINGTON VA 22203-1714

DIR ADV RSCH PROJ AGENCY
1 ATTN ARPA/ASTO
3701 N FAIRFAX DR
ARLINGTON VA 22203-1714

12 DEFENSE TECH INFO CTR
CAMERON STATION
ALEXANDRIA VA 22314

DEPARTMENT OF AIR FORCE

HQ USAF/LGSSF
1 ATTN FUELS POLICY
1030 AIR FORCE PENTAGON
WASHINGTON DC 20330-1030

HQ USAF/LGTV
1 ATTN VEH EQUIP/FACILITY
1030 AIR FORCE PENTAGON
WASHINGTON DC 20330-1030

AIR FORCE WRIGHT LAB
1 ATTN WL/POS
1 ATTN WL/POSF
1 ATTN WL/POSL
1790 LOOP RD N
WRIGHT PATTERSON AFB
OH 45433-7103

AIR FORCE WRIGHT LAB
1 ATTN WL/MLBT
2941 P ST STE 1
WRIGHT PATTERSON AFB
OH 45433-7750

AIR FORCE WRIGHT LAB
1 ATTN WL/MLSE
2179 12TH ST STE 1
WRIGHT PATTERSON AFB
OH 45433-7718

1 AIR FORCE MEEP MGMT OFC
615 SMSQ/LGTV MEEP
201 BISCAYNE DR STE 2
ENGLIN AFB FL 32542-5303

1 SA ALC/SFT
1014 ANDREWS RD STE 1
KELLY AFB TX 78241-5603

1 WR ALC/LVRS
225 OCMULGEE CT
ROBINS AFB
GA 31098-1647