

CONTRACT NO. NOba 90269
FINAL REPORT

MARCH 27, 1965

E. F. HOUGHTON & CO.

PHILADELPHIA, PA.



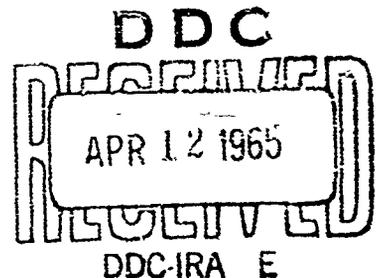
DEVELOPMENT OF FIRE RESISTANT
WATER BASED HYDRAULIC FLUIDS

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PHILIP RAKOFF
G. JOHN COLUCCI
ROBERT K. SMITH

DEPARTMENT OF THE NAVY
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WASHINGTON, D. C.



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G. JOHN COLUCCI
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ABSTRACT

Hydraulic fluids consisting of polyglycols, water and corrosion additives have been formulated whose properties meet or approach those proposed in the contract targets.

The fire resistance characteristics of the finished lubricant were studied with inhibitors based on boron, phosphorus, or chlorine containing materials. Nitrogen and highly conjugated ring structures were incorporated into the major fluid components.

Simulated hydraulic system tests have been used to establish the actual performance of the proposed hydraulic fluid base formulas.

INTRODUCTION

Water-glycol lubricants have been satisfactorily used by industry and the governmental agencies during the past ten years in shipboard hydraulic systems. However, the material remaining after evaporation of the water, in fluids meeting MIL-H-22072, is susceptible to ignition. The current objective is to impart fire resistance in both the lubricant, and the non-aqueous residue. The targets include a flash point of greater than 450°F, a fire point of greater than 450°F and an autogenous ignition temperature approaching 900°F.

Other required properties include:

Viscosity	850 cs max @ 25°F; 25-31 cs @ 150°F		
Pour Point	0°F maximum		
Shear Stability	After shearing, no more than $\pm 10\%$ change @ 150°F		
Specific Gravity	< 1.6 @ 60°/60°F		
Lubricating Ability	Must be capable of lubricating shipboard hydraulic pumps to pressures of 5000 psi (screw types and variable stroke piston)		
Compatibility	Fluids shall be compatible with the following alloys:		
	<u>Steel</u>	<u>Phosphor Bronze</u>	<u>Copper</u>
	QQS 633 MIL-B-857	QQF 330	QQN 281 MIL-T-873
	<u>Aluminum</u>	<u>Silver Brazing</u>	<u>Bronze</u>
	QQA 268	MIL-S-15395	MIL-B-16541
Rubber Compatibility	Must be compatible with Buna N Spec. MIL-P-5516		
Sea Water Contamination	Must be functional with 10% sea water		

Stability	Must be stable at storage temperatures of -20°F to +110°F and at use temperatures of +25°F to 180°F and filterable through a 5 micron filter without loss of properties
Foaming	Must not produce a stable foam
Toxicity	Must minimize hazards from use on shipboard
Cleaning	Fluid residues shall be removable by flushing with H ₂ O.

- The research effort has studied: (a) ignition inhibitors, and (b) polymer modification to increase fire resistance.

Water soluble compounds have been studied which contain phosphorous, nitrogen, chlorine or boron in the molecule. Many organic materials containing these elements are known to have fire resistance; in some cases, they can impart fire or ignition resistance to other organic systems. These materials have been evaluated in prototype water-glycol fluids for flash point, fire point, autogenous ignition temperature, solubility and viscosity increasing tendency. Autogenous ignition temperature increases as high as 100°F have been attained by the use of such inhibitors.

Utilizing commercially available materials, two blends have been developed upon which to base the target fluid; these are designated Hydraulic Fluid Base A and Hydraulic Fluid Base B. Base A is a blend consisting of an ethylene-propylene oxide condensate (molecular weight 10,000 to 20,000), an ethylene-propylene oxide condensate (molecular weight 800 to 1200), water and appropriate inhibitors; Base B contains an ethylene-propylene oxide condensate (molecular weight 15,000 to 25,000), an ethylene-propylene oxide condensate (molecular weight 800 to 1200), water and inhibitors.

To evaluate the lubricity of the candidate lubricants, a commercial vane pump and hydraulic system was chosen due to the difficulty of obtaining a suitable screw-pump during the time of the contract. The vane pump is more critical with respect to the lubricity characteristics of the test fluids than either a screw-pump or an axial piston pump.

SYNTHESIS OF IGNITION INHIBITORS

A major portion of the contract effort has been expended in the synthesis of compounds containing various elements which can inhibit autoignition or quench flame-induced combustion. Concurrently, the structure of the glycol portion used in the hydraulic fluid bases has been modified to increase its fire resistance. Finally, aromatic derivatives of polyoxyalkylene glycols were most successful in decreasing flammability characteristics of the finished hydraulic fluids.

The various compounds are discussed in the following sections in conjunction with their effects on the properties of water-glycol lubricants.

A. Boron Compounds

In the first phase of the contract, the inclusion of boron into organic materials yielded products which were water soluble and which inhibited the autoignition of a water-glycol fluid when used as additives.

Over forty complex structures were synthesized from boric acid or boric anhydride. The condensates formed were essentially either an alkoxy-borate complex or an alkanolamine borate ester. These compounds were polymeric in nature but did not appreciably alter the viscosity of the finished hydraulic fluid. Several boron-nitrogen compounds, when used as additives, substantially increased fire resistance. The following table summarizes appropriate data:

AUTOIGNITION INHIBITION OF A COMMERCIAL
WATER GLYCOL HYDRAULIC FLUID

	<u>AIT, °F</u>
Commercial Water Glycol Hydraulic Fluid (1)	765-775
(1) + 5% Diethanolamine borate	775-780
(1) + 10% Diethanolamine borate	860-870
10% Polyalkylene Glycol a 35% Polyalkylene Glycol b 10% DEAB 45% Water	875-895
10% Polyalkylene Glycol a 35% Propylene Glycol 10% DEAB 45% Water	930-950

B. Phosphate Compounds

Various phosphate organics were prepared and are classed as follows:

- (1) Metal alkyl phosphates
- (2) Aminopolyol - phosphate esters
- (3) Methoxy polyethylene phosphates

The most promising water-soluble phosphate candidate was the potassium salt of a dialkyl phosphate. The material is slightly alkaline when dissolved in water and has a relatively high auto-ignition temperature (1100°F - 1115°F). Further evaluation of the product in a water-glycol hydraulic lubricant showed that the auto-ignition of the hydraulic fluid was not substantially increased when this phosphate was used as an additive.

The aminopolyol-phosphate esters have flash and fire points in the 400°F to 500°F range and their autogenous ignition temperatures are only 700°F to 750°F. This is probably due to the fact that only a small percentage of phosphorous is present in the molecule and the resultant autoignition temperature is characteristic of the aminopolyol.

The methoxy polyethylene phosphates have relatively high flash and fire points (440°F - 570°F) and again low autogenous ignition temperatures (740°F - 760°F). When dissolved in water, the pHs of

their solutions are 1.5 to 2.5 . This property mitigates against formulation of a high quality hydraulic fluid.

C. Chlorinated Organics

Several methoxy polyethylene chlorinated aromatics were prepared which had flash and fire points in the range of 425°F to 500°F. Their autoignition temperatures were 800°F to 850°F. The water solutions are extremely acid, therefore, they are not desirable as additives.

The dialkyl chlorinated aromatics which we synthesized are insoluble in water and were eliminated from further testing.

D. Phenyl Ethers

Organic materials which are essentially hydroxy alkylene phenyl ethers have been prepared. These have autoignition temperatures greater than 950°F. The flash and fire points fall into the range of 220°F to 250°F which makes these ethers undesirable as flame retardants. Our experience to date has led us to believe that an inverse relationship exists between autoignition temperatures and flash points. The data summarized below tabulate the AIT results obtained when two aromatic ether derivatives designated HPPE (hydroxypropyl phenyl ether) and HPMPE (hydroxypropyl methyl phenyl ether) are added in 10 percent concentration to a water based hydraulic fluid test formula.

Test Formula

Polyglycol c	20.0%
Additive	10.0%
Polyglycol b	34.0%
Water	36.0%

<u>Additive</u>	<u>Autoignition Temperature</u>
None	820°F
HPPE	850°F
HPMPE	860°F

E. Miscellaneous Organics

The aminopolyol-methoxy polyethylene glycols and dibasic acid esters have flash and fire points of over 500°F; however, their autogenous ignition temperature only range between 700°F and 750°F.

A phenol-formaldehyde resin type, when added in 10 percent concentration to a water-glycol fluid raised the autoignition temperature from 820°F to 870°F. Further work of this type was suspended because the non-aqueous residues containing resins were very tacky and would cause seizure of hydraulic system components.

An attempt was made to alter the glycol portion of the proposed prototype hydraulic fluid by a condensation reaction using epichlorohydrin. The autoignition temperature increased only when the treated material was dissolved in water. This effect is shown below.

<u>Material</u>	<u>Autoignition Temperature</u>
Polyglycol b	780°F
Treated Polyglycol b	775°F
40% Polyglycol b 60% Water	835°F
40% Treated Polyglycol b 60% Water	870°F

When the Treated Polyglycol b was used as a replacement for the Polyglycol b in a fully compounded hydraulic fluid formula, the autoignition temperature was not appreciably raised.

F. Hydroxy Polyalkylene Derivatives of Bisphenol A

After synthesizing different types of compounds to inhibit autoignition tendencies, the conclusion was made that a polymer was needed which contained one or more phenyl groups in the molecule, and which was water soluble. Bisphenol A was selected as the starting material due to its availability, cost and chemical structure. Efforts were directed towards preparing derivatives which would be compatible with the water-glycol systems already being evaluated. Di hydroxy polyalkylene ethers of Bisphenol A were prepared by first forming the sodium salt of Bisphenol A and then reacting this product with polyoxyalkylene chlorides of different chain lengths.

The autoignition temperature of the prototype hydraulic fluid is increased from approximately 820°F to 870°F by substitution of such a Bisphenol A adduct for one of the major glycol components.

TABLE I

BASIC GLYCOL WATER BLENDS
(45% WATER)

Blend	Composition, vol. %				Viscosity, SUS AIT, °F	Cold Storage Test	Flash	Flare	AIT	Ignition Properties of Given Ratios without Water, °F		
A	Polyglycol a	16			100%	150%	Pass	24 hr.	Pass	480	570	775
B	Polyglycol b	38			329	152	Pass	Pass	Pass	480	570	760
C	Polyglycol c	45			342	152	Pass	Pass	Pass	495	570	760
D	Polyglycol d		10		335	152	Pass	Pass	Pass	350	490	815
E	Polyglycol e		15		348	160	Pass	Pass	Pass	440	490	690
F	Polyglycol f		14		Incompatible	Incompatible	Fail	Fail	Fail			
G	Polyglycol g		39		Incompatible	Incompatible	Pass	Pass	Pass			
H	Polyglycol h		41		363	810-825	Pass	Pass	Pass			
I	Polyglycol i		32		350	840	Pass	Pass	Pass			
			62.5*		308	850-880	Pass	Pass	Pass			

*Water content - 37.5%

HYDRAULIC FLUID FORMULATIONS

Our primary screening parameters for candidate fluids were fire resistance, viscosity and low temperature stability. Using these as a guide, many polyglycol-water combinations were prepared. Table I tabulates the physical test results of typical blends.

The data presented in Table II show the relative performances of the three bases for the target fluid. The pump was operated at 115°F - 125°F while delivering 5 GPM at 900 psi. Comparisons of wear rates during overnight test runs are summarized.

TABLE II

	<u>Ring Wear Rate</u>	<u>Vanes' Wear Rate</u>
Blend A	1.2 mg/hr	0.2 mg/hr
Blend B	1.9 mg/hr	0.4 mg/hr
Blend C	57.3 mg/hr	11.9 mg/hr

Either Blend A or Blend B shows an acceptable wear rate, while the rate associated with Blend C would lead to premature pump failure.

From this work, Bases A and B were chosen as the more promising combinations to study. Satisfactory corrosion inhibitors were compounded for these bases.

A preliminary check of the corrosivity of several fluid blends was conducted by examining the pump parts after test runs at 120° and 140°F. Blends containing the synthesized Glycol Borate additive, caused no visible corrosion to the pump parts. Blend C, which contains a water soluble, phosphate additive, caused heavy corrosion to the steel pump parts. Blends A and B, both containing various percentages of several polyalkylene glycols in a water formula, cause moderate to heavy corrosion to the steel pump parts.

TABLE III

WATER GLYCOL BASE STOCKS RATED FOR CORROSION
DURING VICKERS VANE PUMP RUNS

<u>Blend</u>	<u>Rating*</u>
A	4
B	6
C	10
J	1
K	1
L	1

*Ratings - 1 = Very good condition - No visible corrosion of pump parts

10 = Very poor condition - Rust and corrosion of pump parts is evident

An additive package containing 0.1% Benzotriazole and 1.0% Sodium Benzoate gave adequate protection and had the least effect on the lubricity of the base materials.

Table IV, which follows, shows this property.

TABLE IV

EFFECT OF ADDITIVE MATERIALS ON THE PUMP
PERFORMANCE OF THE BASE FLUIDS

<u>Fluid</u>	<u>Hours</u>	<u>Wear, mg</u>	
		<u>Ring</u>	<u>Vane Set</u>
Blend A + 1.0% Task C. J. (GAF)	16	237	4
	20	12	1
Blend A + 1.0% Sodium Benzoate	17	11	3
Blend A + 0.05% Benzotriazole	16	171	6
Blend A + 0.3% Sodium Nitrite	24	1267	817
Blend A + (1.0% Sodium Benzoate & 0.1% Benzotriazole)	17	260	28
	20	3	14
	37		
Blend A + (0.05% Benzotriazole & 1.0% Carbowax Oleate 6000)	65	18	37
Blend A + (0.5% Sodium Benzoate & 0.05% Benzotriazole)	24	153	55

Consequently, the additive package containing 1.0% Sodium Benzoate and 0.1% Benzotriazole was incorporated into the formula of Base A and B.

A program was initiated to modify Base A or B to obtain improved:

- (a) fire resistance
- (b) pump performance
- (c) corrosion inhibition of metals
- (d) low temperature stability
- (e) viscosity index
- (f) antifoam properties.

Each new class of fire retardants synthesized was checked in concentrations up to 10 percent of the bases. Among the earlier preparations, the glycol borates and the alkanolamine borates had the most effect on raising the autoignition temperature of the bases. Consequently, these materials were evaluated for their effect on the lubricity of the bases in the Vickers vane pump - all were found to be lacking in one respect or another. Generally, the glycol borates in concentration up to 10 percent had no adverse effects on lubricity but were not stable in the water system. These materials dropped out of solution during pump operation resulting in two phase fluids. In addition, the residues from the test fluids were crystalline and thus presented another obstacle to satisfactory hydraulic system operation.

The alkanolamine borates were not completely soluble at the effective concentration levels required for improved fire resistance of the base fluid. The only coupler which we found to provide solubility for these additives was inferior in its own fire resistance properties; thus negating the original effect of the alkanol amine borate.

At this point, it was decided to avoid these types of fire retardants as additives because of these compatibility and crystallinity problems.

Four test procedures have been used in our studies of corrosion inhibitors which are presented in Table V. We found the Dynamic Corrosion test to be the most critical one since it simulates actual system operations.

Since large quantities of fluid are needed for this dynamic test, our screening work has been divided between procedures 2 and 3.

TABLE V
CORROSION TEST PROCEDURES

- (1) Static liquid phase @ 130°F - MIL-H-19457 B, Procedure 4.4.4.1
- (2) Static liquid phase @ 158°F - MIL-H-19457 B, Procedure 4.4.4.1 but modified in the soak temperature which was raised to 158°F
- (3) Stirring corrosion @ 140°F - modified turbine oil rust test

Metal specimens are prepared in the same manner as in the static liquid phase corrosion tests above. Five specimens are placed in a glass rack which fits into 400 ml Berzelius beaker of the turbine rust test. The sixth panel is placed beside the rack in the beaker. Three hundred milliliters of fluid are added to the beaker to cover all metal specimens and supply a volume of fluid above the rack for stirring. Agitation is supplied by a shortened stirrer paddle which is attached to the drive spindle of the test apparatus. The stem of the paddle is shortened to provide clearance for the rack of specimens immersed on the bottom of the Berzelius beaker. The plastic beaker cover from the rust test apparatus is used also. The stirring speed of 1000 rpm + 50 is maintained during the test and the bath temperature is maintained at 140°F also.

- (4) Dynamic Corrosion Test - Reservoir of Vickers vane pump hydraulic circuit

The panels are prepared by the same procedure employed in the other tests. After weighing, the specimens in a glass rack are placed in the reservoir of a Vickers 5 GPM hydraulic test circuit which is being used in a lubricity study of the test fluid also. The rack is located in the return side of the reservoir where the test fluid circulates over the panels in turbulent flow.

At the present time, we are conducting endurance tests on Bases A and B and concentrating efforts on the evaluation of corrosion inhibitor systems since Hydraulic Fluid Bases A and B do not provide complete protection to all of the specification metals. Table VI summarizes the data obtained on Base Fluid A.

TABLE VI

Material

Polyglycol a	13.5%
Polyglycol b	40.4%
Water	45.0%
Sodium Benzoate	1.0%
Benzotriazole	0.1%

Viscosity @ 100°F = 69.8 cs
 @ 150°F = 29.5 cs
 @ 25°F = 789.0 cs

<u>Foam</u>	<u>Tendency</u>	<u>Stability</u>
@ 75°F	20 cc	45 sec collapse
@ 140°F	None	

Compatibility and Stability

@ room temperature	clear fluid
@ 0°F	remains clear and mobile after 24 hours
@ 0°F after 1 week	remains mobile and flows easily but some crystals develop and are suspended in the fluid
@ 160°F	remains clear - no separation of component materials

Compatibility with Synthetic Sea Water

Mixed with 15% synthetic sea water and allowed to stand

@ room temperature	remains clear - no floc is evident
--------------------	------------------------------------

TABLE VI continued

Pour Point	55°F
Specific Gravity @ 60/60°F	1.0935
AIT of the Fluid	* (a) 925 (b) 870 (c) 850
AIT of fluid residue evaporated to dryness in a 160°F forced circulation oven	850

**	
Flash and Fire points on the fluid residues	COC
	Flash Fire
	490 625

* a, b and c refer to 3 determinations on the same fluid
 ** Results obtained while continuously stirring the heterogeneous residues.

Static Liquid Phase Corrosion
 MIL-H-19457 Procedure
 One Week - 130°F

Metal	Spec.	Weight Change mg/cm ²	Appearance
Aluminum	QQA 250/4	+0.057	Light staining
Bronze	QQP 330	+0.015	Clean and bright
Steel	QQS 698	+0.014	Faces of specimen are clean and bright; fine rust specs on edges
Copper	QQC 576	+0.023	Clean and bright
Zinc	QQZ 285	-0.142	Dulled surface-crystalline salt accumulation on bottom of panel, when washed shows attack and pitting in this area
Brass	QQB 613	-0.023	Light staining

TABLE VI, continued

Stirring Liquid Phase Corrosion
Modified Turbine Oil Rust Test
(See Procedure 3 Table V)
90 Hours - 140°F

<u>Metal</u>	<u>Spec.</u>	<u>Weight Change</u> <u>mg/cm²</u>	<u>Appearance</u>
Aluminum	QQA 250/4	+0.020	Gray brown stain of panel surfaces
Bronze	QQP 330	+0.014	Clean and bright
Steel	QQS 698	-0.020	Clean and bright
Copper	QQC 576	-0.006	Clean, stained dull
Zinc	QQZ 285	-0.500	Clean and bright except at contact surfaces with glass rack where panel is pitted and chalked
Brass	QQB 613	-0.026	Clean, stained dull

Immersion Test at 160°F
One Week Duration

Test Material - Buna N Synthetic Rubber

<u>Specimen</u>	<u>% Volume Change</u>
a	-1.1%
b	-1.4%
c	-1.4%

Pesco Pump Test
MIL-H-19457 Procedure
5000 Cycles-1000 psi-100 ± 5°F

	<u>Viscosity @ 150°F</u>	<u>Water Content</u>
Start	29.7 cs	43.8%
End	32.4 cs	43.2%

Bench tests have indicated a need for corrosion inhibitors which will not crystallize from solution or cause excessive wear to pump parts during hydraulic operation. Several materials which are essentially aromatic carboxylates having alkylene groups of different chain lengths have been prepared to overcome this problem. These fluids are coded HPB, HDEB, and HTEB. Preliminary corrosion studies have shown that HTEB does inhibit zinc corrosion when used as an additive to the glycol hydraulic fluid. Pertinent data are shown in Table VII.

TABLE VII

Stirring Corrosion Test @ 140°F
(Modified Turbine Oil Rust Test - Procedure 3 Table V)

<u>Hydraulic Fluid</u>	<u>Test Specimen</u> (1"x2"x $\frac{1}{4}$ ")	<u>Weight Change</u>	<u>Appearance</u>
Base A	Zinc	-41.0 mg	Dull gray film Several black stains
Base A + 1.0% HTEB	Zinc	-11.0 mg	Clean

Dynamic Corrosion Test
(Conducted in the reservoir of the vane pump of the hydraulic circuit
Procedure 4 Table V)

<u>Hydraulic Fluid</u>	<u>Metal Specimens - Wt. Change and Appearance</u>					
	<u>Zinc</u>	<u>Steel</u>	<u>Aluminum</u>	<u>Brass</u>	<u>Bronze</u>	<u>Copper</u>
Base A	-30.1 mg	-0.4 mg	-0.1 mg	-0.9 mg	-0.3 mg	-0.6 mg
	White chalking and pitting	Clean and bright	Clean and bright	Clean and bright	Light stain	Clean and bright
Base A + 1.0% HTEB	- 4.9 mg	-6.2 mg	+0.7 mg	-0.4 mg	+0.3 mg	+0.4 mg
	White film	Dark stain	Bright	Slight stain	Slight stain	Bright

Presently, we are using modifications on procedure 2, Table V to accelerate these evaluations. Review of our test data shows the test panel observations to be as good a check of the inhibitor system as weight change measurements. Consequently, we have eliminated the weighing of the metals. Panels are observed after specified soak periods. The condition of test panels are noted for stains, deposits, etch, pitting and rust. Those inhibitor systems which prove best in this screening procedure are then checked in the dynamic corrosion tests (procedure 4 Table V) in a pump stand.

Endurance tests of bases A and B were set up in a pump stand to determine stability, lubricity, and corrosion inhibitor life of these fluids on extended operation at high temperature and pressure. At the conclusion of these runs, the performance of these fluids when contaminated with 10 percent sea water will be examined.

In these tests, in addition to the standard test circuit, a 5 micron filtration unit on the relief side of the throttle valve was added.

The test conditions at the pump were:

Throttle pressure	900 psi
Fluid Temperature	140°F
Output Theoretical	5 GPM
System Capacity	3½ gallons
Average Fluid Cycle Time	1½ passes per minute

In the run with Base A, the break-in wear for 18 hours was 16 mg/hr. The operating wear rate dropped to 3 mg/hr after 72 hours of operation and was down to 0.05 mg/hr after 260 hours of test.

The next inspection of the pump cartridge was made after an additional 500 hours of test. The wear rate had increased to 4 mg/hr during this period. During this operation period, the fluid began weeping from the rear shaft seal and a salting out of the sodium benzoate inhibitor occurred as these residues lost water. A build up of the cake around the shaft seal interfered with the seal in the pump when a restart of the system was attempted.

Dynamic corrosion tests of the target test metals were made in the reservoir during this endurance run. At 260 hours, of the target metals, only the galvanized steel panel was attacked. In the final examination at 760 hours, the steel panel was also found to be lightly corroded. We will try to determine if this rust resulted from contamination of the fluid, depletion of additive or oxidation of fluid components.

CONCLUSION

Extensive study of ignition inhibitors has led to the conclusion that ignition inhibition by the use of additives is not compatible with the requirements for a high flash point. Those materials which proved effective as ignition inhibitors simultaneously had high vapor pressures and low flash points. The fundamental reason for this phenomenon was not determined but may well be related to the requirement for a minimum inhibitory concentration in the vapor phase at ignition temperatures.

Alternate studies have shown that a fluid meeting the target requirements of flashpoint, viscosity, corrosion characteristics and related properties, as well as approaching an autogeneous ignition point of 900°F can be compounded. This fluid has performed sufficiently well in vane pumps that the recommendation is made that further pump testing in screw and axial-piston pumps be performed.

Preliminary work has also shown that polymer modification by the introduction of aryl groups in the chain may result in a fluid which surpasses the desired AIT of 900°F.