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FINAL REPORT

DEVELOPMENT OF A FIRE-RESISTANT
WATER-BASE HYDRAULIC FLUID

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WASHINGTON 25, D.C.

E. H. Kober
H. F. Lederle
G. F. Ottmann
J. E. Pregler

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1965
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OLIN MATHIESON CHEMICAL CORPORATION
CHEMICALS DIVISION
NEW HAVEN, CONNECTICUT

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Water-Base Hydraulic Fluid

Final Report

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Contributing Staff:

E. H. Kober
H. F. Lederle
G. F. Ottmann
J. E. Pregler
S. A. Kolback
W. C. Ready

Prepared by: H. F. Lederle
H. F. Lederle
Sr. Research Chemist I

Approved by: G. F. Ottmann
G. F. Ottmann
Section Manager

Submitted by: E. H. Kober
E. H. Kober
Technical Director

Date: May 28, 1965

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I. ABSTRACT

A number of new base stocks for water-base hydraulic fluids was prepared and evaluated. The most promising materials - addition products of ethylene oxide and glycidol to bicyclic aromatic amines and phenols - were formulated with water soluble pour point depressants, viscosity index improvers, anti-wear agents, rubber swellers and antifoam agents. These fluids pass the proposed target specifications. Judged by Falex tests, they exhibit considerably better anti-wear properties than a currently used MIL-H-22072 (Wep) fluid.

II. INTRODUCTION AND OBJECTIVES

The requirement of the U. S. Navy for a fire-resistant water-base hydraulic fluid for shipboard use continues to be urgent despite the progress made during and since World War II.

Water-base hydraulic fluids, developed by the Navy to replace the flammable petroleum base fluids, consisted originally of a 50% aqueous solution of ethylene glycol and a polymeric thickener such as polyalkylene glycol, made from a blend of ethylene oxide and propylene oxide or sodium polymethacrylate. Since World War II, many other thickeners have been developed and ethylene glycol has been replaced by other low molecular weight glycols as well as glycerine. Formulated fluids of this type exhibit good fire resistance, provided excessive water loss does not take place; they have, in general, good corrosion resistance to the usual metals of construction, but galvanized iron and cadmium coated metals present problems; these fluids do not have any destructive action on sealing and packing materials presently used, but they are inferior to the petroleum base fluids with regard to lubrication properties. Summarizing, future developments in the field of fire-resistant water-base hydraulic fluids have to be concerned mainly with the improvement of (a) fire-resistant characteristics of the organic portion of the fluid, (b) lubricity, (c) retention of rust-

inhibiting properties in presence of sea water, and (d) decreased foaming tendency of the fluid.

To overcome the deficiencies of the fluids presently used, four classes of compounds were suggested as potential candidates for fire-resistant water-base hydraulic fluids.

- (1) Oxyalkylated hydroxymethylphosphine oxides,
- (2) Oxyalkylated haloarylamines and halophenols
- (3) Oxyalkylated toluene diamines, and
- (4) Oxyalkylated polyfluoroalcohols and polyfluoroglycols

These proposed classes of compounds contain a multiplicity of hydroxyl groups which were expected to provide for water solubility and for low flammability because of high hydroxyl to carbon ratios in the aliphatic moieties. Our four approaches have in common that a number of compounds - known to be hydrolytically stable and to impart fire-resistance of various degrees - have been chosen to serve as initiators for the synthesis of polyoxyalkylated compounds and as "built-in" antioxidants.

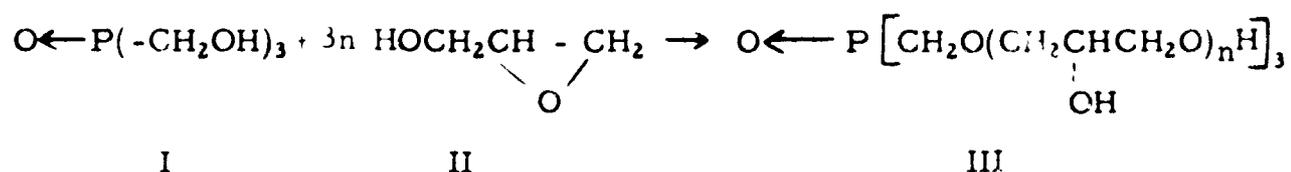
The project was carried out in three distinct phases. The first phase involved syntheses of various fluid base stocks and their preliminary evaluation with regard to water solubility, viscosity and pour point of the aqueous solution, flash- and flame points and autogenous ignition temperature. Products which come close to the viscosity specification and passed the other tests were selected for the second phase of the program and formulated with viscosity index improvers and pour point depressants to adjust viscosities and pour points to target specifications. In the third and last phase, the most promising candidates which had emerged from phase 2 were subjected to final formulation with additives such as corrosion inhibitors, rubber swelling agents, antifoam agents, lubricity improvers and antioxidants. The third phase was concluded with the preparation of four 2-quart samples of the most promising fluids and their final evaluations.

III. DISCUSSION

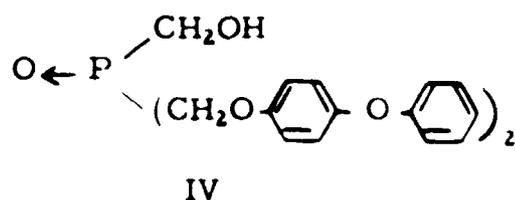
A. Preparation, Evaluation and Selection of Fluid Base Stocks

1. Oxyalkylated Hydroxymethylphosphine Oxides (Table 2)

While attempts to oxyalkylate trishydroxymethylphosphine oxide(I) with ethylene oxide failed, reaction with glycidol (II) proceeded readily. The reaction products (III) had relatively low autogenous ignition temperatures, and consequently were eliminated from further investigations.



Preparation of (IV) followed by reaction with glycidol also gave products



having low autogenous ignition temperatures and, consequently, products of this type were not further investigated.

2. Oxyalkylated Haloarylamines and Halophenols (Table 3)

Polyhalogenated monocyclic aromatic amines failed to react with ethylene oxide, propylene oxide or glycidol in the presence of either basic or acidic catalysts. Hence, no further work along these lines was carried out.

The polychlorinated phenols readily added ethylene or propylene oxides at 160°C, and glycidol at 105°C. Surprisingly, these products gave low autogenous ignition temperatures and, consequently, were not further investigated.

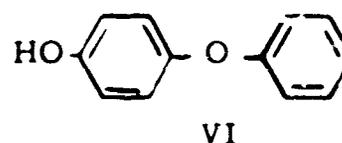
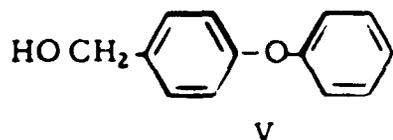
3. Oxyalkylated 2,4-Toluene Diamines (Table 4)

Water soluble products were readily obtained from 2,4-toluene diamine by addition of ethylene oxide, glycidol, or by propylene oxide, followed

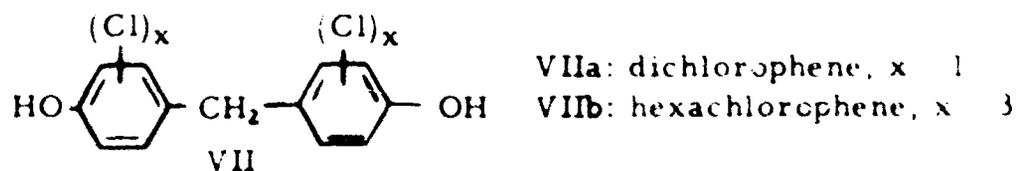
by glycidol. Large amounts of glycidol had to be added to attain aqueous solutions which had viscosities of less than 2000 cs at 25°F after being adjusted to 28 ± 3 cs at 150°F. This, in turn, lowered the autogenous ignition temperature below acceptable levels. Because reproducibility of viscosities could not be achieved, work on this system was terminated.

4. Oxyalkylated Aromatic Dicyclic Amines (Table 3, 6) and Dicyclic Phenols (Tables 4, 5)

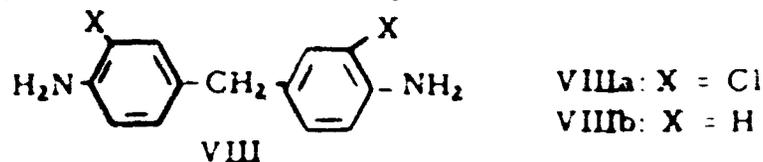
In general, oxyalkylated products derived from dicyclic aromatic initiators were found to have higher autogenous ignition temperatures than those derived from monocyclic aromatic compounds. Adducts of hydroxymethyldiphenyl ether (V) had lower autogenous ignition temperatures than those of hydroxydiphenyl ether (VI) and, thus, were not considered for



further investigations. Chlorinated dicyclic phenols, such as hexachlorophene (VIIa) and dichlorophene (VIIb) either failed to react with alkylene oxides or, if reaction occurred, the resulting fluids had low autogenous ignition temperatures.



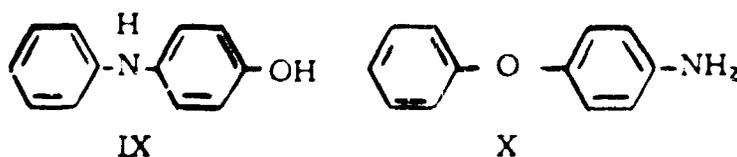
In contrast thereto, adducts of methylene bis(2-chloroaniline), (VIIIa), not only had satisfactory autogenous ignition temperatures, but also



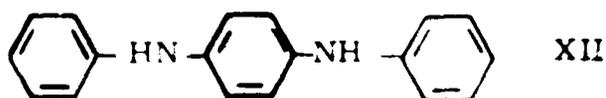
relatively low viscosities at 25°F in aqueous dilution. Products from methylene dianiline (VIIIb) had good autogenous ignition temperatures but poor viscosities and pour points. Attempts to correct this by ethylene oxide

addition improved the viscosities and pour points, but lowered the auto-ignition temperature to unacceptably low levels.

Products from p-anilinophenol (IX) proved superior to those from p-phenoxyaniline (X), and, consequently, products derived from (X) were not further investigated.

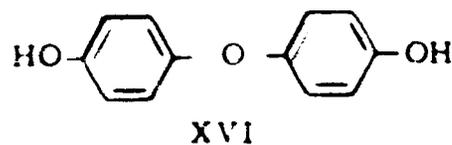
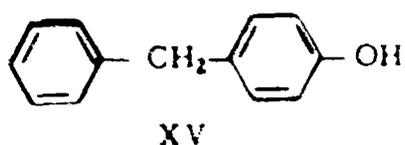
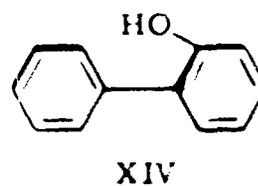
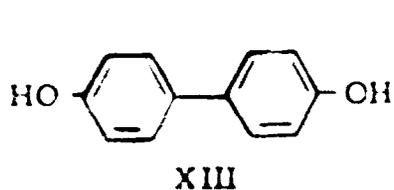


Adducts of oxydianiline (XI), and N,N'-diphenyl-p-phenylene diamine (XII)

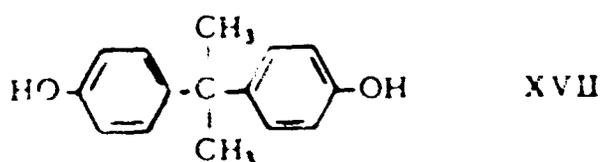


had sufficiently attractive properties to warrant further investigation.

Products derived from biphenol (XIII), o-phenylphenol (XIV), α-phenyl-p-cresol (XV), and dihydroxydiphenyl oxide (XVI) were not good enough to warrant further studies.



However, adducts of bisphenol A (XVII) had high specific gravities and high auto-ignition temperatures, and, consequently, they were further evaluated.

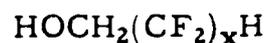


5. Oxyalkylated Polyfluoroalcohols and Polyfluoroglycols (Table 2)

Water soluble products were obtained by oxyalkylation of hexafluoropentane diol (XVIII), octafluoropentanol (XIX, x = 4), and dodecafluoroheptanol (XIX, x = 6), but none of these products had sufficiently high spontaneous ignition temperatures to warrant further investigations.



XVIII



XIX

B. Formulation with Viscosity Index Improvers and Pour Point Depressants

Based on the physical properties, discussed in Section A, several products were selected for formulation and further evaluation. These products were:

- (1) Oxyalkylated hydroxydiphenyl oxide (VI), (HDO 10-2)[⊕]
- (2) Oxyalkylated Bisphenol A (XVII), (BA 18-4)[⊕]
- (3) Oxyalkylated N,N'-diphenyl-p-phenylene diamine (XII), (DPD 18-4)[⊕]
- (4) Oxyalkylated 4,4'-methylene bis(2-chloroaniline) (VIIIa), (MOCA 12-8)[⊕]
- (5) Oxyalkylated anilinophenol (X), (AP 18-2)[⊕]
- (6) Oxyalkylated oxydianiline (XI), (ODA 12-4)[⊕]

1. Viscosity Index Improvers (Tables 7-13)

The nature of the water-base hydraulic fluids prohibited the use of commercially available hydrocarbon-soluble viscosity index improvers but required water soluble additives. In search for suitable candidates, a variety of alkylene oxide based products was evaluated.

[⊕] For the sake of brevity, an abbreviated nomenclature will be used from here on. The numbers following the letters indicate the moles of ethylene oxide and glycidol which have been added to the initiator.

The data compiled in Table 7 indicate that several of such products were effective for HDO 10-2. The most effective viscosity index improvers for this system were found to be Ucon 75-H-90,000, Ucon 75-H-150,000, product D-2220-60[⊕], Carbowax 6000^{⊕⊕} and Carbowax 20-M^{⊕⊕}. While Carbowax 20-M was effective in fairly low concentrations, it detrimentally affected the shear stability of some fluids. Ucon 75 H-150,000 is no longer available and product D-2220-60 is not available in commercial quantities. Consequently, Ucon 75 H-90,000 appears to be the best overall choice for improving the viscosity index of HDO 10-2.

Viscosity index improvers which were used with HDO 10-2 formulations were also effective with those of BA 18-4, (Table 8). The best overall performing additive appears to be Carbowax 6000.

In the case of DPD 18-4, Ucon 75-H-90,000 was found to be superior to Carbowax 6,000 (Table 9).

Ucon 75-H-90,000 was found to be the best viscosity index improver for MOCA 12-8 (Table 10). In this case, a viscosity at 25°F as low as 409.4 cs was obtained. This low viscosity prompted us to attempt some formulation without any viscosity index improvers.

The best overall viscosity index improver for AP 18-2 formulations is Ucon 75-H-90,000. While Carbowax 20-M gave a lower viscosity at 25°F., it is slightly shear-unstable (Table 11).

Ucon 75-H-90,000 is also the best viscosity index improver for ODA 12-4 formulations (Table 12). In Table 13, the results of evaluating several product mixtures are given. No advantage over the individual components was observed.

⊕ Nonylphenol, to which ethylene and propylene oxide in a 60/40 weight % ratio was added. Final viscosity 2220 cs/100°F.

⊕⊕ Carbowax 6000 and Carbowax 20-M are polyethylene glycols of molecular weight 6000 and 20,000, respectively.

2. Pour Point Depressants (Tables 14-18)

The data compiled in Tables 7 to 13 show that addition of viscosity index improvers to the selected base stock fluids resulted in pour points above 0°F. Since the proposed target specifications (Table 1) require a pour point maximum of 0°F., formulation with pour point depressants was indicated. It was anticipated that addition of pour point depressants would also lower the crystallization points below 0°F[⊕].

Substantial decreases of both the pour point and crystallization point of the fluids were obtained with ethylene glycol and tetraethylene glycol as pour point depressants. However, both compounds lowered the autogenous ignition value of the fluid base stock substantially. Marked pour point depressions were experienced with glycerin, which did not affect the autogenous ignition too unfavorably. 1,2,6-Hexanetriol, depending on its concentration, caused either undesirably high crystallization points or high viscosities at 25°F. Thiodiethylene glycol and the formal of triethylene glycol monomethyl ether also lowered the autogenous ignition point of the base stocks to below acceptable levels. Both tetrafluoropropanol and hexafluoropentane-1,2-diol failed to lower the pour points of the fluids at all. It was therefore concluded that glycerin represented the best overall compromise.

Examination of the results compiled in Tables 14 to 18 show that in each case satisfactory crystallization and pour points of the fluids were obtained with glycerin. In the case of the DPD 18-4 formulation product 801297-17 (Table 16) organic solids precipitated upon storing. This eliminated DPD 18-4 from further consideration. While ODA 12-4 formulations were generally satisfactory, the formation of solid polymers could

⊕ In this report the crystallization point is defined as the temperature at which the first crystals (presumably ice) started to form within the fluid.

not be avoided in larger batch preparations. For this reason, ODA 12-4 was not further evaluated.

An interesting effect was observed in the case of the MOCA 12-8 formulation. Without glycerin (Table 10), Ucon 75-H-90,000 was the best viscosity index improver but lost its effectiveness with glycerin present (product 801298 B, Table 17). In the latter case, Carbowax 6000 was the preferred additive (product 810305-10).

C. Final Formulation and Evaluation

Based on the results obtained in the second phase of our investigations, the following fluids were selected for further study:

Fluid Type 1 (product 810450, Table 15):

BA 18-4 base stock	38.4%
Carbowax 6000	9.6%
Glycerin	15.6%
Water	36.4%

Fluid Type 2 (product 810436, Table 14):

HDO 10-2 base stock	38.7%
Ucon 75-H-90,000	9.7%
Glycerin	10.3%
Water	41.3%

Fluid Type 3 (product 810457, Table 16):

AP 18-2 base stock	36.0%
Ucon 75-H-90,000	9.0%
Glycerin	10.3%
Water	41.3%

Fluid Type 4 (product 814353, Table 17):

MOCA 12-8 base stock	40.0%
Glycerin	18.0%
Water	42.0%

Fluid Type 5:

Moca 12-8 base stock	31.5%
Glycerine	19.1%
Carbowax 6000	8.6%
Water	40.7%

1. Static Corrosion Tests (Table 19)

Fluids 1 to 4 (see above) were each formulated with four different anticorrosion packages and tested against 7 different metals. Each fluid was also tested without an anticorrosion package added. The static corrosion test was performed according to Military Specification MIL-H-22072 (Wep) and amendments, December 31, 1963(1). The permissible weight change of each test coupon is 4 mg. per coupon. The following four anti-corrosion or inhibitor packages were selected for the tests:

Package 1: Borax · 5 H₂O, 1.5%; Nacap (50% aqueous solution of the sodium salt of mercaptobenzothiazole), 0.5%.

Package 2: Potassium phosphate K₂HPO₄, 2.0%; aminomethylpropanol, 0.25%; Nacap 0.6%.

Package 3: Liquibor 524 (ethylene glycol-borax condensate), 1.0%; Nacap, 0.5%.

Package 4: Borguard (propylene glycol-butynediol-borax condensate), 2.5%; Nacap, 0.5%.

The results of the tests are compiled in Table 19 and indicate that at least two formulations of each of the four products passed the static corrosion test.

2. Stirring Corrosion Tests (Table 20)

The fluid formulations which passed the static corrosion test were subjected to the stirring corrosion test according to MIL-H-22072(Wep)(1). This test permits a maximum weight change of 7 mg. per test coupon. In addition, successive pairs of panels (e.g. panels 1 and 2, 3 and 4) were in electrical contact with each other. Results are given in Table 20. While Fluid Type 1 (BA 18-4) with inhibitor package 2 passed on a weight change basis, a phase separation occurred shortly after the start of the test, thus eliminating this formulation from further consideration. Also eliminated was the formulation of Fluid Type 1 (BA 18-4) with package 3 as inhibitor

due to excessive weight change of several test coupons. On the basis of the static corrosion tests, we then chose the next best formulation (inhibitor package 4) which did not undergo phase separation under test conditions, (Table 19), for further evaluations.

Fluid Type 2 (HDO 10-2) formulated with inhibitor package 3 passed the test and was selected for further formulation.

Two versions of Fluid Type 3 - uninhibited and formulated with inhibitor package 4 - passed the stirring corrosion test. The fluid containing package 4 was chosen for additional evaluations on the basis of its less corrosive action on aluminum.

Fluid Type 4 containing inhibitor package 4 failed in the case of galvanized steel only. Difficulties in resynthesizing the MOCA 12-8 base stock having a viscosity which allows formulation without a viscosity improver kept us from evaluating other inhibitor systems.

3. Rubber Swelling Tests (Table 21)

The first three runs listed in Table 21 show that the fluid types 1 to 3 exceeded the permissible 5% rubber shrinkage (1). However, a series of tests indicated that isodecanol, tridecylalcohol or heavy oxo fraction[⊕] are effective rubber swellers in 1% concentration in fluids of type 1. Since both tridecyl alcohol and heavy oxo fraction were less soluble than isodecanol, the latter was chosen for the final formulations. However, it should be pointed out that isodecanol is not as effective in a type 2 fluid (last run, Table 21).

[⊕] octyl alcohols, 5%; nonyl alcohol, 10%; decyl and higher alcohols, 35%; esters, 45%; soaps, 5%.

4. Falex Wear Tests (Table 22)

Since lack of lubricity is one of the shortcomings of the presently used fluids, special attention was focused on better wear characteristics. The tests were performed in a Falex Wear Tester and were directly compared with the MIL-H-22072 (Wep) Fluid which was evaluated for reference purposes. A large take-up and a relatively high weight loss of the metal parts signified poor wear characteristics of the MIL-H-22072 fluid. Our fluids, not formulated with an antiwear agent, were similar or even inferior. Therefore, formulation with an antiwear agent was imperative.

The first substantial improvement for a fluid of type 2 was obtained with 0.4% stearic acid. Since this compound was not sufficiently soluble in all formulations, octanoic acid was employed as substitute and proved to be superior to stearic acid in 0.5% concentration. It was therefore included in all final formulations. The corrosion inhibitor packages employed apparently also contribute to the antiwear properties. Thus, a fluid of type 3, containing package 4 but no octanoic acid, was much superior to the MIL-H-22072 Fluid and was even more improved on addition of octanoic acid. It should be observed that all final formulations described in this report exhibited substantially better wear properties than the MIL-H-22072 (Wep) type fluid.

5. Foaming Characteristics (Table 23)

The results of this test are compiled in Table 23. They show that foaming of our products is easily inhibited or controlled by the addition of 5 to 10 drops of Dow-Corning Antifoam B per quart of fluid.

6. Sonic Shear, Compatibility with Sea Water, High Temperature Stability, Low Temperature Crystallization and Evaporation Residues (Tables 21, 24)

The results of the sonic shear tests (Table 24) show that all products tested did not exceed the permissible maximum viscosity change

of 10% at 150°F. The product formulated with Carbowax 20-M came rather close to failing, presumably due to the relatively high molecular weight of the Carbowax (20,000). For this reason, Carbowax 20-M was eliminated as potential viscosity index improver (see also Section B, 1).

Data obtained from the sea water compatibility tests, using 10% sea water per formulation (Table 24) indicate that all fluids would probably be operable under these conditions since only traces of fine crystals had formed at 0°F. Test results for high temperature stability, low temperature crystallization, and evaporation residue are given in Table 21. All products tested passed the specifications.

7. Effect of Anti-Oxidants on the Autogenous Ignition of HDO Base Stocks (Table 25)

In Table 25, the effect of various anti-oxidants on the autogenous ignition temperature of HDO 10-2 base stocks are compiled. Since slightly higher autogenous ignition values were observed in only a few cases, it is concluded that the anti-oxidants tested are not effective in types of fluids developed under this program.

8. Reproducibility of Base Stock Preparation (Table 26)

Table 26 lists the viscosities, hydroxyl numbers and autogenous ignition temperatures of different preparations of the fluid base stocks used in the final fluid formulations. Close reproducibility of these physical properties is required in order to obtain reproducible fluid formulations. Examination of the data indicate that the viscosities, autogenous ignition temperatures and hydroxyl numbers check reasonably well among individual preparations of the same base stock with the exception of one MOCA 12-8 preparation (run 8073b4).

9. Pump Tests (Table 27)

This test is designed to evaluate wear on the pump parts and shear stability of the fluids. Since the pump system described in

MIL-H-22072 was not available at this laboratory, we adopted a modified SAE procedure designed for the evaluation of central hydraulic systems fluids (2). This test uses a 1960 Ford power steering pump (eight cylinder, for use without air conditioning), Ford Motor Company part no. COAA3A691-R, (Eaton Mfg. Co. no. ER-10919-7). The pump was operated at 3000 ± 100 r.p.m., the discharge pressure being maintained at 800 psi for 60 seconds and at 75 p.s.i. for 30 seconds. The fluid inlet temperature was maintained at approximately 150°F. by means of a heat exchanger. Figure 1 gives a schematic illustration of the system. The pumps were run for 100 hours with a sample of the fluid being withdrawn for analysis after 50 hours. The weight changes of the pump parts were obtained; and the fluids (including the 50 hour sample) were examined for changes in physical properties.

The data listed in Table 27 show that some water loss occurred during all of the tests. This throws considerable doubt on any conclusions regarding shear stability since a water loss would counteract any viscosity decrease due to shearing action. Another consistent trend is a lowering of the pH in each case

Of the products which completed the test, the fluid of type 1 appears to be superior. Weight losses on the cam ring, roller vanes and rotor were relatively low. The fluid of type 2 gave considerably higher wear, especially on the roller vanes. The pump was just barely able to deliver 800 lbs. at the end of the test. The fluid of type 3 gave a relatively high weight loss on the cam ring. This is the only product where a slight shear instability can definitely be proven. In the 50 hour sample, the viscosity decreased by 2 cs. despite a water loss. At the end of the run, only 200 lbs. pressure could be obtained. Results obtained with fluid of type 4 are inconclusive, since the test fluid leaked from a defective seal in the solenoid sometime between 50 and 100 hours.

10. Final Formulations (Table 28)

Our final formulations are given in Table 28. With the exception of product 4, all materials meet the specifications shown in this table. Product 4 was reformulated to give product 5 when it became apparent that both poor reproducibility of the base stock composition and the use of inhibitor package 4 increased the viscosities of the formulated fluid at 25 F to unacceptably high levels.

Fluids of types 1, 2, 3 and 5 have been submitted to the U. S. Navy Marine Engineering Laboratory in two quart quantities for further evaluation. While the fluids of Table 28 are our best formulations to date, they should not necessarily be construed as having the optimum compositions. These products could undoubtedly be improved.

IV. CONCLUSIONS AND RECOMMENDATIONS

1. The objective of this project, the development of two fire-resistant water base hydraulic fluids, was accomplished.
2. The completely formulated fluids have passed all of the proposed target specifications but have not been subjected to a pump test according to MIL-H-22072 (Wep).
3. The lubricity of the developed fluids is much superior to that of the MIL-H-22072 (Wep) fluid as judged by the results obtained in the Falex Wear Test.
4. A sequence of superiority of the four submitted fluids cannot be established due to the missing results of a meaningful pump test.
5. It is anticipated that the present fluid compositions could be improved by further formulation studies.

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VI. EXPERIMENTAL

A. Synthesis of Base Stocks

In view of the large number of base stocks prepared during this project, only the preparation of the four optimum products which were used in the final formulations will be described here. For the synthesis of the other base stocks, the individual bimonthly reports should be consulted(3), (4),(5),(6).

1. Synthesis of BA 18-4 Base Stock

To a 2000 ml. three neck flask, equipped with stirrer, drying tube, nitrogen inlet tube, thermometer, jacketed addition funnel and an ice-water cooled condenser with a dry ice condenser on top, there was charged 34.2 g. (1.5 moles) of bisphenol A and 8.0 g. (4.28 per cent by weight based on bisphenol A) of powdered potassium hydroxide. The mixture was heated to 140°C with stirring. A total of 1188 g. (27 moles) of ethylene oxide was added dropwise starting at 140°C and at such a rate that the temperature of the reaction mixture was maintained between 140 and 150°C. The addition of ethylene oxide was terminated after a molar ratio of ethylene oxide to bisphenol A of 18:1 had been reached. The temperature of the reaction mixture was then lowered to 105°C. and 444.0 g. (6.0 moles) of glycidol was added in a dropwise manner at such a rate that the pot temperature remained under 120°C. When the addition of glycidol had been completed, the reaction mixture was maintained at 105°C. for an additional hour. The resulting product was then freed. Low boiling components were then removed from the resulting product by heating to 225°C in a vacuum of 0.1 mm. Hg. A total of 1906 g. of liquid product (hydroxyl number - 348), which corresponds to 96.6 per cent of the theoretical yield, was obtained.

2. Synthesis of HDO 10-2 Base Stock

A 2000 ml. three-necked flask equipped with stirrer, drying tube, nitrogen inlet tube, thermometer, jacketed addition funnel and ice-water cooled condenser with a dry-ice condenser at the top was charged with 233.0 g. (1.25 moles) of p-phenoxy phenol and 3.0 g. (1.29 per cent by weight based on p-phenoxy phenol) of powdered potassium hydroxide. The mixture was heated to 140°C; stirring was started when the p-phenoxy phenol had completely melted. A total of 550.0 g. (12.5 moles) of ethylene oxide was added dropwise starting at 140°C and at such a rate that the temperature of the reaction mixture was maintained between 135°C. and 160°C. The addition of ethylene oxide was terminated after a ratio of 10 moles of ethylene oxide per mole of p-phenoxy-phenol had been reached. The reaction flask was then cooled to a temperature

of 105°C and 185 g. (2.5 moles) of glycidol was added in a dropwise manner while the pot temperature was held below 120°C. After the glycidol addition had been completed, the reaction mixture was kept at 105°C for another hour. Low boiling components were then removed by heating the reaction mixture to 225°C under a vacuum of 0.1 mm Hg. A total of 953.3 g. of liquid product (hydroxyl number - 211) was obtained which corresponds to 98.7 per cent of the theoretical yield.

3. Synthesis of AP 18-2 Base Stock

A 300 ml. three-necked flask equipped with a stirrer, drying tube, thermometer, nitrogen inlet tube, jacketed addition funnel and ice-water cooled condenser with a dry-ice condenser at the top, was charged with 39.0 g. (0.2 moles) of p-anilinophenol and 0.1 g. (2.56 per cent by weight based on p-anilinophenol) of powdered potassium hydroxide. The mixture was then heated to 110°C with stirring. A total of 158.4 g. (3.6 moles) of ethylene oxide was added dropwise starting at 110°C and at such a rate that the temperature of the reaction mixture remained below 150°C. The addition of ethylene oxide was terminated after a molar ratio of ethylene oxide to p-anilinophenol of 18:1 had been reached. The reaction mixture was then cooled to a temperature of 103°C and 29.6 g. (0.4 moles) of glycidol was added in a dropwise manner and at such a rate that the temperature remained below 110°C. After the addition of the glycidol had been completed, the reaction mixture was held at 105°C for one hour. In the next step low boiling components were removed from the resulting product by heating to 225°C under a vacuum of 0.1 mm Hg. A total of 214.7 g. of fluid product (hydroxyl number - 202) was obtained, which corresponds to 94.5 per cent of the theoretical yield.

4. Synthesis of MOCA 12-8 Base Stock

A 500 ml. three-necked flask equipped with stirrer, drying tube, nitrogen purge, thermometer, jacketed addition funnel and ice-water cooled condenser with dry-ice condenser at the top, was charged with 80.2 g.

(0.3 moles) of 4,4'-methylene bis(2-chloroaniline) and 3.0 g. (3.75 per cent by weight based on 4,4'-methylene bis(2-chloroaniline) of powdered potassium hydroxide. The mixture was heated to 110°C; stirring was started when the 4,4'-methylene bis(2-chloroaniline) had completely melted. A total of 158.4 g. (3.6 moles) of ethylene oxide was added dropwise starting at 110°C and at such a rate that the temperature of the reaction mixture was maintained at below 120°C. The addition of ethylene oxide was terminated after a ratio of 12 moles of ethylene oxide per mole of 4,4'-methylene bis(2-chloroaniline) had been reached. The reaction flask was cooled to a temperature of 105°C and 178.0 g. (2.4 moles) of glycidol was added in a dropwise manner while the pot temperature was held under 110°C. After the addition of the glycidol had been completed, the reaction mixture was maintained at 105°C for another hour. Low boiling components were then removed from the reaction mixture by heating to 225°C under a vacuum of 0.1 mm. Hg. A total of 408.8 g. of liquid product (hydroxyl number - 451) was obtained, which corresponds to 98.4 per cent of theory.

B. Formulation Studies

Except for the adjustment of viscosities, the formulation experiments were straightforward and need not be further described. A typical calculation for adjustment of viscosities is given in Bimonthly Report No. 4 (6).

C. Evaluations

All evaluations were carried out by well known ASTM procedures, or as described in specification MIL-H-22072 (Wep) (1). Exceptions were as follows:

1. Pour Points

Only two cold temperature baths, one maintained at -10°F, the other at -100°F were used.

2. Crystallization Points

Performed like the pour points. The temperature at which crystallization started was defined as the crystallization point.

3. Autogenous Ignition Temperatures

A few drops of the compound were introduced into a porcelain crucible contained in an electric furnace (approx. 9.5 cm. high, 4.5 cm. diameter, equipped with a thermocouple). Measurements were started somewhat below the expected autogenous ignition temperature. The temperature was then raised at an approximate rate of 5°F per minute, and the temperature at which the first flash was observed was recorded. A clean crucible was used for each determination. The autogenous ignitions without water were performed in similar fashion. While this procedure was not as accurate as the ASTM procedure, it was much less time consuming and it gave reasonably reproducible results. ⊕

4. Corrosion Tests

The panel preparation and other test procedures of MIL-H-22072 (Wep)(1) were used. The metal specimens received from Robert K. Johnston, San Antonio, Texas, had to be cut to the right dimensions. This left some of the edges and the center hole of the galvanized steel specimens unplated. These panels were regalvanized before use. In place of the metals employed in MIL-H-22072 (Wep), the following were used: Aluminum (QQ-A-250/4b), phosphor bronze (QQ-B-750, Comp. A), bronze (MIL-B-1654IA(Wep)), galvanized steel (QQ-S-698 Gr. 1009, zinc electroplated per QQ-Z-325A, Type II, class 1), monel (Monel 400, QQ-N-281, class A), copper (QQ-C-576 b), silver brazing alloy (MIL-B-15395 A(Ships), Gr. IV) and steel (QQ-S-698, Grade 1009).

The same metals were used in the stirring corrosion test. However, in the stirring test, the following metals were connected by means of stainless steel washers: Brazing alloy and phosphor bronze, phosphor bronze and steel, brazing alloy and steel, bronze and galvanized steel, bronze and aluminum, copper and aluminum, steel and aluminum.

⊕ Under these conditions, ethylene glycol ignited at 650°F.

5. Rubber Swelling

Buna N rubber O rings (Precision Rubber Products Corp., Dayton, Ohio, AN 6227-14, marking BR) were used. Run at 158°F for one week.

6. Foaming Characteristics

The foam tests were performed according to ASTM procedure D-892 at 75°F.

7. Sonic Shear Test

Performed by the procedure of Nejak and Dzuna (7).

8. Falex Wear Test

M-10 type journals and V-blocks were used in this test. The pressure on the journal was raised by 100 lbs. every fifteen minutes, until a total of 800 lbs. had been reached. At the end of the test, the weight loss on journal and jaws was determined. For a more detailed procedure, the literature should be consulted (9)(8).

9. High Temperature Stability and Low Temperature Crystallization Tests

Performed as described in MIL-H-22072 (Wep) (1).

10. Evaporation Residue

A 5 ml. sample of the fluid was evaporated at 212°F in a Petri dish. After cooling to room temperature, the nature of the residue and its solubility in water were checked.

11. Compatibility with Sea Water

To a small amount of the fluid, an additional 10% of sea water was added. Pour points, crystallization points and viscosities at 150°F and 25°F were then determined. An aliquot of the sample was then heated in a test tube. If a phase separation below the boiling point occurred, the temperature was recorded.

12. Pump Test

Equipment

(a) Eaton Mfg. Company power steering pump (ER-10919-7)

(b) Pump drive 10 Hp electric motor.

- (c) Thermometer to record temperature.
- (d) Time activated solenoid control valve.
- (e) Pressure gauge 0-150.
- (f) Flex-o-pulse timer to switch solenoid control valve.
- (g) Pressure gauge 0-1000.
- (h) Water cooled heat exchanger to maintain fluid temp.
- (i) Necessary copper tubing, valves and connectors to assemble pumping circuit as shown in Fig. 1.

Operating Conditions

Pump speed constant at 3000 RPM \pm 100 RPM
Pump discharge pressure- cycle 800 \pm 20 psi for 60 sec.
" " " " 75 \pm 15 psi for 30 sec.
Fluid inlet temp. 150°F \pm 5.
Test duration, 100 hrs. with a sample withdrawn at 50 hrs.
Fluid volume, 2 quarts.

Procedure

The pump was disassembled, cleaned, and the following parts were weighed: the cam ring, the rotor and the six roller vanes taken as a unit. The pump was reassembled and inserted into the circuit as shown in Figure 1. The fluid to be tested (2 quarts) was introduced into the reservoir while slowly turning the pump drive wheel by hand until both the reservoir and the system were full. The Flex-o-pulse timer was set and the pump started. The pressures of 800 lbs. or 75 lbs. were obtained by partially closing valves A and E respectively (Fig. 1). Since the 1000 lb. gauge had a tendency to break, valve D was kept closed, except for occasional checking. The temperature of the solution was adjusted by circulating cool water thru the heat exchanger. The test was not started until the temperature of the fluid in the reservoir was 150°F. The duration of the test was 100 hrs. with samples taken at 50 hours. The samples were checked for changes in pH, specific gravity, appearance, and viscosity. At the conclusion of the test the pump was disassembled and the parts checked for wear and weight losses.

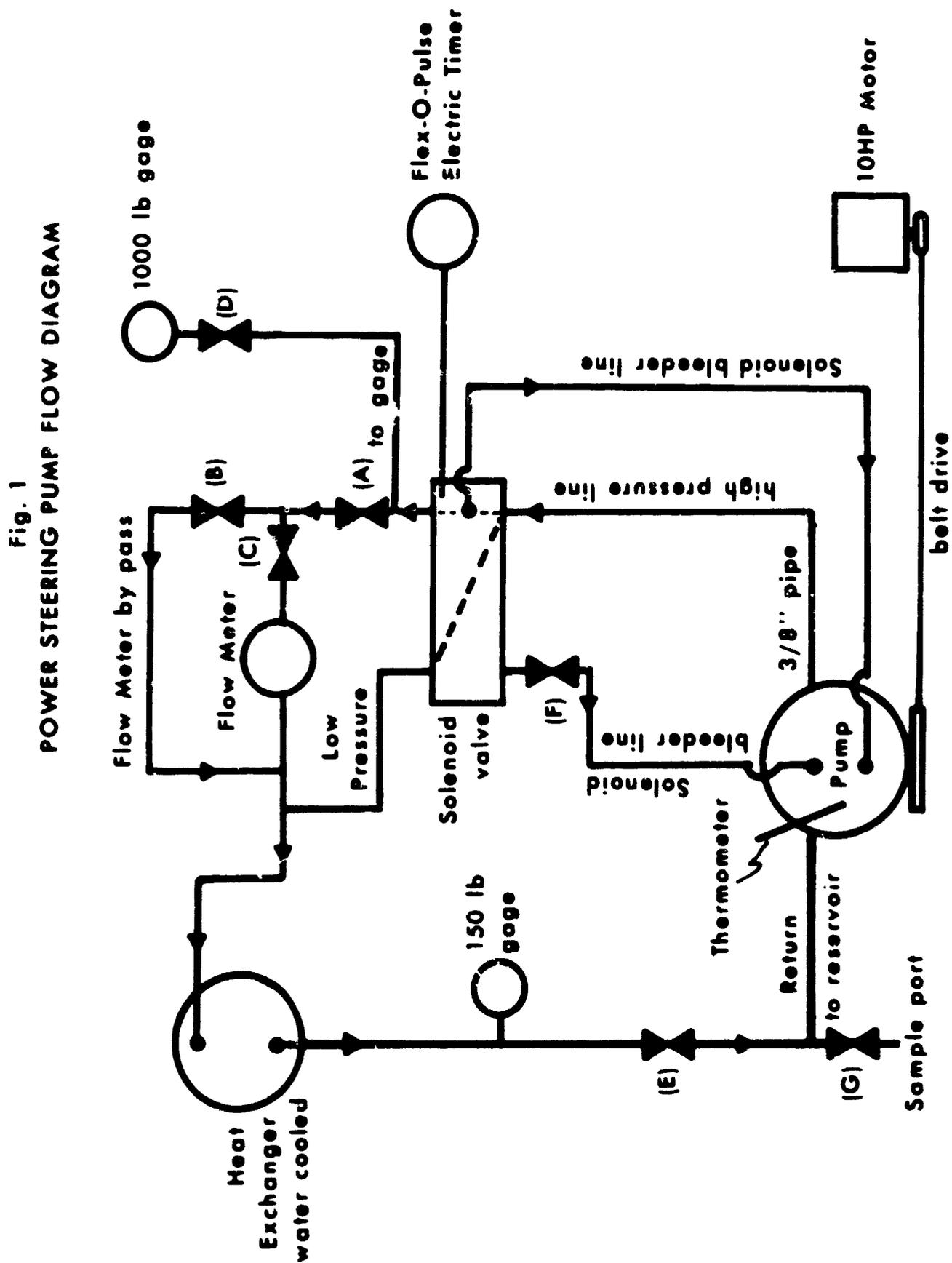


TABLE 1

Summary of Test Methods and Target Objectives for Water-Base
Hydraulic Fluids

	<u>Target Objective</u>
1. Fire Resistance	
a) Flash Point	450 F (min.)
b) Fire Point	550 F (min.)
c) Autogenous Ignition Temperature	900 F (min.)
d) Compression Ignition (5000 psi)	Nonflammable
2. a) Viscosity at 25 °F	850 cs (max.)
b) Viscosity at 150 °F	25 - 31 cs
3. Pour Point	0 °F (max.)
4. Shear Stability	<u>±</u> 10% at 150 °F
5. Specific Gravity at 60/60 °F	1.6 (max.)
6. Lubricity (5000 psi)	Operate in Screw and Variable Piston Pump
7. Compatibility with Metals	Steels, Copper, Copper Alloys, Bronzes, and Aluminum
8. Compatibility with Buna N Rubber	Compatible
9. Compatibility with Sea Water	Operable with 10% dilution
10. Stability	
a) Storage	-20 to 110 F
b) Use	25 to 180 F
11. Foam Test	No stable foams
12. Toxicity	Minimize Use Hazards
13. Fluid Residues	Removable by flushing with water

TABLE 4

Miscellaneous Hydroxyalkyl Substituted Compounds

Reference Number	Initiator Type	Moles Glycol Added	Hydroxyl No. Functional	Water Miscibility, Room Temp., 150°F	Sea Water Miscibility, 212°F	Flash Pt., °F (a)	Fire Pt., °F (a)	Autog. Ign., °F	Water Solution			
									% Base	K. Vis., cs. 25°F	Pour Pt. °F.	
688867-68	C ₁₂ F Glycol	4	663	sol.	→	> 530	> 530	825	62	3388	-20	
688867-74	C ₁₂ F Alcohol	2	489	sol.	→	450	460	800	82	2091	0	
688867-82	C ₁₂ F Alcohol	6	576	sol.	→	380	450	725	75	2251	-25	
740164	100% C ₁₂ F	4	620	sol.	→	> 520	> 520	750	72	2100	-20	
740176	75% C ₁₂ F	3	672	sol.	→	> 428	> 428	775	77	2934	-25	
612181	75% C ₁₂ F	26	630	sol.	→	not det.	→	825	62	25.4	1307	-15

(a) A bubble or temperature indicator indicates a boiling point, no flash

- (b) 1,1,1,3,3,3-hexafluoro-2-propanol
- (c) 1,1,1,3,3,3-hexafluoro-2-propanol
- (d) 1,1,1,3,3,3-hexafluoro-2-propanol
- (e) 1,1,1,3,3,3-hexafluoro-2-propanol
- (f) 1,1,1,3,3,3-hexafluoro-2-propanol

U. S. PATENT OFFICE
 1,1,1,3,3,3-Hexafluoro-2-propanol

TABLE 1
Hydroxyalkyl Substituted Aromatic Amines

Reference Number	Analysis Type	Moles Added		Hydroxyl No. Exampl.	Hydroxyl Inerity	Water Miscibility, Deionized		Flash Pt., °F (c)	Fire Pt., °F (c)	Autog. Ign., °F	Water Solution			
		EO ^a	Glycol (b)			Room Temp, 150°F	Miscibility 212°F				% Base	K.Vis., cs. 150°F	Pour Pt. °F	
60059-00	MDA (d)	0	4	893	910	sol.	→	> 540	> 540	925	63	25.4	3864	+20
612121	"	0	0	591	590	sol.	→	> 490	> 490	750	63	31.1	1917	-20
607329	ADA	16	0	202	198	sol.	→	520	520	925	70	25.5	2060	-30
740187	ODA	0	2	869	905	sol.	→	not det.	→	1050	62	25.2	3603	+20
740195-96	"	16	0	218	248	sol.	→	535	> 500	900	75	25.5	2293	-20
612147	"	12	0	371	438	sol.	→	> 470	> 470	875	64	28.1	1360	-20
612114-15	POA (f)	0	4	664	700	sol.	→	> 490	> 490	850	58	31.1	6093	+20
612116-17	"	18	0	122	265	sol.	→	540	> 550	775	64	28.6	1165	-20
612112	DPD (g)	24	0	162	210	sol.	→	> 520	> 520	900	65	30.8	2815	-20
612150	"	12	0	302	363	insol.	→	not det.	→	975	not det.	→	→	→
60011-11	POA (h)	0	4	1015	1070	sol.	→	> 550	> 550	875	70	25.9	1458	-20
612164 A	"	0	0	1036	1070	sol.	→	not det.	→	825	69	29.2	5338	0
612177	"	0	0	972	1070	sol.	→	not det.	→	→	70	27.0	4520	-5
612178	"	0	0	not det.	→	sol.	→	not det.	→	→	66	26.5	3209	0
612169	"	0	0	902	942	sol.	→	not det.	→	800	66	29.0	1925	-15
612171	"	0	12	878	889	sol.	→	not det.	→	825	65	26.8	1997	-20
612174	"	0	0	826	827	sol.	→	not det.	→	810	63	27.0	1396	-20
60014-16	"	2	2	681	690	sol.	→	not det.	→	→	→	→	→	→
60020-21	"	2	2	690	622	sol.	→	585	590	850	69	25.6	12,078	-20
60017-19	"	0	0	503	240	sol.	→	not det.	→	→	→	→	→	→
60022-23	"	4	0	825	673	sol.	→	550	555	875	78	30.6	3289	-25
60045-54	"	0	0	554	583	sol.	→	500	550	875	71	27.8	2499	-20
60139	"	0	3.43	657	598	insol.	→	not det.	→	→	→	→	→	→
60157	"	0	6.16	350	389	insol.	→	not det.	→	→	→	→	→	→
60158	"	0	7.67	306	325	insol.	→	not det.	→	→	→	→	→	→

a. Ethylene oxide
b. Propylene oxide
c. A mixture of temperature indicates a boiling point, no fire or flash point being observed.
d. 1,4-Dioxane
e. Aniline
f. Methylolamine
g. 2,4-Diaminophenol
h. 2,4-Diaminophenol

TABLE 4

Hydroxyalkyl Substituted Halogenated Phenols

Reference Number	Phenol Type	Moisture Added		Hydroxy No. Found	Hydroxy No. Theoretical	Water Miscibility, detemized		Flash Pt., °F (1)	Fire Pt., °F (1)	Autog. Ign., °F	% Base	K. Vis., cs.		Pour pt., °F
		HO (a)	PO (b)			Room temp. 150°F	210°F					150°F	25°F	
60000-01	2,4,6- TMC ⁽¹⁾	0	0	522	535	sol.	→	530	560	900	73	29.2	8553	0
60000-10	"	0	0	562	570	sol.	→	545	560	850	69	26.2	5303	0
60000-50	"	0	0	601	658	sol.	→	555	580	850	67.5	27.8	2607	-20
60000-07	"	0	1	481	471	sol.	→	450	540	875	63	29.2	7463	0
60000-56	"	10	0	151	88	sol.	→	495	515	875	not det.	→	→	→
60000-50	"	6	0	282	567	sol.	→	535	575	850	73	29.7	3332	-20
60000-05	2,4,6,2,4,6- TC ⁽¹⁾	0	0	505	528	sol.	→	495	550	825	not det.	→	→	→
60000-70	"	0	0	566	695	sol.	→	480	>480	800	nd	28.0	3989	-10
60000-52	2,4,6,2,4,6- TC ⁽¹⁾	0	0	476	495	sol.	→	>450	>450	875	59	27.7	14,792	20
60000-03	2,4,6,2,4,6- TC ⁽¹⁾	0	0	600	590	sol.	→	>400	>400	800	65	27.0	4585	+5
60000-1	2,4,6,2,4,6- TC ⁽¹⁾	0	0	471	400	sol.	→	>450	>450	750	72	31.3	4814	-20
70014	DC ⁽¹⁾	18	0	108	106	sol.	→	540	>580	750	80	30.4	3925	+5
70015	DC ⁽¹⁾	18	0	313	299	sol.	→	520	520	775	80	29.9	4035	+5

(a) 5% by volume in water

(b) 10% by volume in water

(c) 2,4,6-trichlorophenol

(d) 2,4,6-trichloro-3-hydroxyphenol

(e) 2,4,6-trichloro-2,4,6-trihydroxyphenol

(f) 2,4,6-trichloro-2,4,6-trihydroxyphenol

(g) 2,4,6-trichlorophenol

(h) 2,4,6-trichloro-3-hydroxyphenol

(i) A 2 indicates a temperature, indicates a boiling point, no fire or flash point being observed.

TABLE 5

Hydroxylated Substituted Diphenyl Ether, Diphenyl Ether, Diphenyls, Biphenols, Bisphenols and Methylene Phenols

Reference Number	Phenol Name	Molar Ratio	Hydroxyl Content, %	Water Miscibility, 150°F	Water Miscibility, 210°F	Sea water Miscibility, Room temp.	Flash Pt., °F	Fire Pt., °F	Autog. Ign., °F	% Base	K. Vis., 150°F	Pour Pt., °F	
													sol.
6000001	CHINOL	10	0	not det.	sol.	sol.	not det.	not det.	750	80	20.0	2380	-20
6000002	CHINOL	10	0	not det.	sol.	sol.	not det.	not det.	750	not det.			
6000003	CHINOL	10	4	70.4	640	sol.	550	> 550	875	69	26.5	2292	-20
6000004	CHINOL	10	0	25.2	168	sol.	550	> 550	900	82	27.0	2476	-25
6000005	CHINOL	10	7	3.1	275	sol.	510	> 535	875	70	28.0	2306	-25
6000006	CHINOL	10	4	61	582	sol.	480	590	975	64	25.4	7115	0
6000007	CHINOL	10	8	40	70	sol.	565	> 565	900	87	27.0	2577	0
6000008	CHINOL	10	2	2.5	178	sol.	500	> 510	900	72	25.9	2100	-20
6000009	CHINOL	10	2	211	217	sol.	> 530	> 530	900	75	25.7	2092	-20
6000010	CHINOL	10	4	25.9	256	sol.	not det.	not det.	850	69	28.4	2284	-20
6000011	CHINOL	10	6	29.9	315	sol.	not det.	not det.	850	66	28.0	1972	-20
6000012	CHINOL	10	6	31.5	41.9	sol.	510	500	875	65	29.7	2439	-20
6000013	CHINOL	10	12	not det.	not det.	sol.	not det.	not det.	1025	not det.			
6000014	CHINOL	10	14	not det.	not det.	sol.	530	> 530	950	62	27.2	3209	0
6000015	CHINOL	10	8	456	496	sol.	510	510	850	61	30.6	gel	-20
6000016	CHINOL	10	0	690	706	sol.	540	> 560	975	65	28.5	3567	0
6000017	CHINOL	10	0	130	126	sol.	535	> 560	800	80	27.5	2618	-20
6000018	CHINOL	10	0	not det.	not det.	soluble below 57°F	not det.	not det.	not det.				
6000019	CHINOL	10	0	not det.	not det.	soluble below 120°F	not det.	not det.	not det.				
6000020	CHINOL	10	0	not det.	not det.	soluble below 153°F	not det.	not det.	not det.				
6000021	CHINOL	10	0	not det.	not det.	soluble below 176°F	not det.	not det.	not det.				
6000022	CHINOL	10	0	not det.	not det.	soluble below 190°F	not det.	not det.	not det.				
6000023	CHINOL	10	0	12.4	120.4	sol.	> 520	> 520	875	79	30.4	2747	-20
6000024	CHINOL	10	4	346	261	sol.	> 500	> 500	900	70	28.6	2304	-20
6000025	CHINOL	10	0	not det.	not det.	insol.	not det.	not det.	not det.				
6000026	CHINOL	10	4	6.21	6.43	sol.	490	490	775	57	30.6	9170	+15
6000027	CHINOL	10	4	3.77	60.2	sol.	480	515	850	64	23.1	5209	+10
6000028	CHINOL	10	2	2.3	1.99	sol.	495	565	750	80	28.1	3714	-10
6000029	CHINOL	10	4	6.67	5.88	sol.	495	520	625	58	25.1	2791	+20
6000030	CHINOL	10	6	6.53	6.12	sol.	495	> 510	1000	58	30.8	5054	+15
6000031	CHINOL	10	2	1.99	6.88	sol.	490	> 490	750	78	30.9	3365	-15

1. The water miscibility data were obtained at a heating rate of 10°C/min. The fire point is the first point at which a flame is observed.

2. The fire point is the first point at which a flame is observed.

3. The fire point is the first point at which a flame is observed.

4. The fire point is the first point at which a flame is observed.

5. The fire point is the first point at which a flame is observed.

6. The fire point is the first point at which a flame is observed.

7. The fire point is the first point at which a flame is observed.

8. The fire point is the first point at which a flame is observed.

9. The fire point is the first point at which a flame is observed.

10. The fire point is the first point at which a flame is observed.

TABLE 6

Hydroxyethyl Substituted 4,4'-Methylene Bis(2-Chloroaniline) (MOCA)

Molecular Weight	Molar Weight	Molar Weight	Hydroxy %	Water Miscibility / Deionized Water Temp. 150°F	Flash Pt. °F	Fire Pt. (b) °F	Autog. Ign. °F	Water Solution			
								% Base	K. Vis., ca. 150°F	Pour Pt. °F	
60000	600	600	0.00	insol.	not determined	not determined	1150	not determined	not determined		
61200	612	612	0.00	sol.	500	500	825	50	25.0	1775	+20
62400	624	not determined	not determined	sol.	not determined	not determined	850	50	28.1	1165	+25
63600	636	636	0.00	sol.	not determined	not determined	850	48	28.7	Froze	→
64800	648	648	0.00	sol.	515	> 530	950	56	29.9	1687	+15
66000	660	660	0.00	sol.	> 490	> 490	875	47	26.5	1141	+20
67200	672	not determined	not determined	insol.	not determined	not determined	not determined	not determined	not determined	not determined	→
68400	684	not determined	not determined	sol., below 185°F	not determined	not determined	not determined	not determined	not determined	not determined	→
69600	696	not determined	not determined	sol.	not determined	not determined	not determined	not determined	not determined	not determined	→
70800	708	708	0.00	sol.	465	> 485	850	62	28.9	1439	0

(a) Not reported sample
 (b) A boiling point is indicated when a boiling point, or two or flash points being observed.

TABLE 7

Formulation of HDO 10-2⁽¹⁾ Base Stock (807314) with V.I. Improvers

Product Code	Base Stock, %	V.I. Improver % Type	Water %	Viscosity, cs.		AIT °F	Crystal. Pt. °F	Pour Pt. °F
				150°F	25°F			
810406	73.8	-- None --	24.2	25.1	2480.5	--	Not determined	--
810407	63.5	6.3 Carbowax 6000	31.4	27.5	1814.7	900	0	-20
810413	47.9	12.0 Carbowax 6000	40.1	25.5	1016.4	975	Not determined	
801252-1	45.6	11.4 Ucon 75- H-90,000	43.0	26.3	709.8	950	+20	+20
801252-13	50.0	7.5 Ucon 75- H-150,000	42.5	25.4	1027.0	930	Not determined	
801261-3	45.0	9.0 Ucon 75- H-150,000	46.0	27.2	823.5	950	+15	+10
801253-16	47.2	11.8 Ucon 50- HB-5100	41.0	26.6	1102.0	950	Not determined	
801255-11	51.1	12.9 Polyglycol 15-1000	36.0	26.2	1312.0	975	Not determined	
810409	48.0	12.0 D-2220 -60(2)	40.0	26.1	607.3	975	+5	-10
801257-10	49.6	12.4 E-1050(3)	38.0	26.1	1211.0	900	Not determined	
810415	51.2	12.8 T-1050 -70(4)	36.0	25.5	1383.1	950	Not determined	
810411	41.9	10.5 454419(5)	47.6	27.4	1318.6	1000	Not determined	
810476	51.8 ⁽⁷⁾	5.2 CW 20-M ⁽⁶⁾	43.0	25.6	1118.7	1000	Not determined	
810480	45.2 ⁽⁷⁾	6.8 CW 20-M ⁽⁶⁾	48.0	25.4	848.2	925	+25	+20

- (1) Hydroxydiphenyl oxide to which 10 moles of ethylene oxide followed by 2 moles of glycidol have been added.
- (2) Random addition product of ethylene and propylene oxide to nonylphenol.
- (3) Random addition product of ethylene and propylene oxide to methanol.
- (4) Random addition product of ethylene and propylene oxide to tripentaerythritol.
- (5) Mixed block-random polymer of ethylene and propylene oxide, thickened with diepoxides.
- (6) Carbowax polyethylene glycol molecular weight 20,000.
- (7) Base stock 807360.

TABLE 8

Formulation of BA 18-4 Base Stock (807315) with V. I. Improvers⁽¹⁾

Product Code	Base Stock %	V. I. Improver %	V. I. Improver Type	Water %	Viscosity, cs.		AIT °F	Crystal. Pt. °F	Pour Pt. °F	Comment
					150 °F	25 °F				
810417	66.0	--	None	34.0	28.0	2001.8	----	Not determined	-	-
810420	44.8	11.2	Carbowax 6000	44.0	25.4	784.1	925	+10	+5	-
801266-15	41.6	10.4	Ucon 75-H-90,000	48.0	26.9	624.5	950	+15	+15	-
801264-1	44.1	8.9	Ucon 75-H-150,000	47.0	27.1	718.8	925	+5	+5	-
801270-1	49.6	12.4	Ucon 50-HB-5100	38.0	26.9	1601.6	925	Not determined	Not determined	Separated at 150 °F
810423	48.0	12.0	Polyglycol 15-1000	40.0	26.4	1176.0	950	Not determined	-	-
810427	42.9	15.1	Polyglycol 15-1000	42.0	25.7	961.5	850	Not determined	-	-
810419	44.6	11.1	D-2220-60	44.3	25.7	867.5	975	+10	+5	-
801268-10	48.0	12.0	E-1050	40.0	27.4	1198.0	925	Not determined	Not determined	Separated at 150 °F
801271	47.2	11.8	T-1050-70	41.0	26.2	1124.0	875	Not determined	-	-
801272-1	50.4	12.6	454419	37.0	25.4	2361.2	975	Not determined	Not determined	Separated at 150 °F
810475	48.2 ⁽²⁾	4.8	CW 20-M	47.0	25.4	778.8	875	+20	+15	-

(1) BA 18-4: Bisphenol A to which 18 moles of ethylene oxide, followed by 4 moles of glycidol, have been added. For other abbreviations see Table 7.

(2) Base stock 807373.

TABLE 9

Formulation of DPD 18-4 Base Stock (807320) with V.I.Improvers (1)

Product Code	Base Stock %	V.I.Improver %	V.I.Improver Type	Water %	Viscosity, cs.		AIT ° F.	Crystal. Pt. °F	Pour Pt. °F	Comment
					150°F	25°F				
810426	51.0	--	None --	49.0	25.1	1548.4	--	Not determined	--	Solid separated
810431	40.2	10.0	Carbowax 6000	49.8	26.3	1110.6	1000	Not determined		Separated at 150°F.
801276-77	36.0	9.0	Ucon 75-H- 90,000	55.0	26.8	686.7	1000	+15	+25	-
801275-7	36.0	9.0	Ucon 75-H- 150,000	55.0	25.9	638.2	975	+20	+30	-
801279-2	46.4	11.6	Ucon 50- HB-5100	42.0	28.1	2432.0	975	Not determined		-
801269	40.0	10.0	D-2220-60	50.0	25.0	992.2	975	Not determined		Separated at 150°F
801278	43.2	10.8	E-1050	46.0	25.3	1564.7	975	Not determined		Separated at 150°F
801282-83	41.6	10.4	T-1050-70	48.0	26.1	1279.9	1025	Not determined		-
801281-1	42.4	10.6	454419	47.0	26.5	3193.3	1000	Not determined		-

(1) DPD 18-4: N,N'-Diphenyl-p-phenylene diamine to which 18 moles of ethylene oxide followed by 4 moles of glycidol have been added. For other abbreviations, see Table 7.

TABLE 10

Formulation of MOCA 12-8 Base Stock (807322) with V.I. Improvers (1)

<u>Product Code</u>	<u>Base Stock %</u>	<u>V.I. Improver</u>		<u>Water %</u>	<u>Viscosity, cs.</u>		<u>AIT °F.</u>	<u>Crystal. Pt. °F.</u>	<u>Pour Pt. °F.</u>	<u>Comment</u>
		<u>%</u>	<u>Type</u>		<u>150°F</u>	<u>25°F.</u>				
740192	62.0	--	None	38.0	28.9	1439.0	850	None	0	-
801290	39.2	9.8	Carbowax 6000	51.0	25.0	631.9	950	+20	+20	-
801283-84	34.4	8.6	Ucon 75-H-90,000	57.0	26.2	409.4	900	+30	+25	-
801286-17	36.9	7.6	Ucon 75-H-150,000	55.5	25.3	467.4	950	+25	+25	-
801284	44.8	11.2	Ucon 50-HB-5100	44.0	25.3	1570.2	925	Not determined	Not determined	Separated at 150°F.
801291	44.0	11.0	Polyglycol 15-1000	45.0	25.5	1235.3	925	Not determined	Not determined	Separated at 150°F.
801288-10	43.2	10.8	D-2220-60	46.0	27.5	1382.1	900	Not determined	Not determined	Separated at 150°F.
801289	44.0	11.0	454419	45.0	27.2	3096.7	925	Not determined	Not determined	Separated at 150°F.

(1) MOCA 12-8: Methylene bis (o-chloroaniline) to which 12 moles of ethylene oxide followed by 8 moles of glycidol have been added. For other abbreviations, see Table 7.

TABLE 11
Formulation of AP 18-2 Base Stock with V.I. Improvers⁽¹⁾

Product Code	Base Stock % Code	V.I. Improver		Water %	Viscosity, cs		AIT °F	Crystal. Pt. °F.	Pour Pt. °F
		%	Type		150 °F	25 °F			
807329	70.0 807329	--	None --	30.0	25.5	2060.0	Not det.	None	-30
814306	47.2 807335	11.8	Carbowax 6000	41.0	26.8	922.3	900	0	-15
814366	46.4 807374	11.6	Carbowax 6000	42.0	25.2	829.0	950	+10	-5
810471	36.8 807374	9.2	Carbowax 20-M	54.0	25.4	484.3	975	+20	+20
810472	51.4 807374	5.1	Carbowax 20-M	43.5	25.7	845.1	975	+15	+10
810442	41.6 807329	10.4	Ucon 75-H- 90,000	48.0	25.3	631.9	975	+15	+5
814307-15	50.4 807335	12.6	Ucon 50- HB-5100	37.0	25.0	1427.8	1000	Not determined	
810443	48.0 803329	12.0	I-2220-60	40.0	25.9	1106.7	1000	Not determined	

(1) AP 18-2: Anilinophenol to which 18 moles of ethylene oxide followed by 2 moles of glycidol have been added. For other abbreviations, see Table 7.

TABLE 12

Formulation of ODA 12-4 Base Stock (807319) with V.I.Improvers⁽¹⁾

<u>Product Code</u>	<u>Base Stock %</u>	<u>V. I.Improver %</u>	<u>V. I.Improver Type</u>	<u>Water %</u>	<u>Viscosity,cs.</u>		<u>AIT °F.</u>	<u>Crystal. Pt. °F</u>	<u>Pour Pt. °F</u>	<u>Comment</u>
					<u>150°F</u>	<u>25°F</u>				
632347	64.0	-- None	---	36.0	28.1	1336.0	875	None	-20	-
810444	44.0	11.0	Carbowax 6000	45.0	25.3	761.1	950	+15	+10	-
801273-22	40.0	10.0	Ucon 75-H -90,000	50.0	25.4	589.7	1000	+20	+20	-
801293-24	53.6	13.4	Ucon 50-HB-5,100	33.0	26.2	2255.5	950	Not determined	-	-
810445	51.5	12.9	Polyglycol 15-1000	35.5	25.5	2000.1	875	Not determined	Precipitated at 150°F	-
801291-92	47.2	11.8	D-2220-60	41.0	26.2	1262.9	975	Not determined	-	-
801294	50.4	12.6	454419	37.0	26.7	1988.5	1000	Not determined	-	-
801296	42.4	10.6	454470 ⁽²⁾	47.0	25.2	1887.2	975	Not determined	-	-

(1) ODA 12-4: Oxydianiline to which 12 moles of ethylene oxide followed by 4 moles of glycidol have been added.
 454470: Mixed block-random polymer of ethylene and propylene oxide thickened with resorcinol diglycidyl ether.
 For other abbreviations, see Table 7.

(2) See footnote 5, Table 7.

TABLE 13

Formulation of Base Stock Mixtures with V. I. Improves (1)

<u>Product Code</u>	<u>Base Stock %</u> <u>Type</u>	<u>V.I.Improver %</u> <u>Type</u>	<u>Water %</u>	<u>Viscosity, cs</u> <u>150°F</u> <u>25°F</u>	<u>AIT °F</u>	<u>Crystal. °F</u>	<u>Pour Pt. °F</u>
807351	(22.0 HDO 10-2 (807323) (22.0 MOCA 12-8 (807321)	11.0 Carbowax 6000	45.0	26.2 819.2	900	+15	+15
807351	(20.8 HDO 10-2 (807323) (20.8 MOCA 12-8 (807321)	8.4 Ucon 75- H-90,000	50.0	27.0 606.4	900	+20	+20
807347	(21.65 HDO 10-2 (807323) (21.65 MOCA 12-8 (807321)	10.7 D-2220-60	46.0	28.0 854.0	925	+20	+15

(1) For abbreviations, see Tables 7 and 10.

TABLE 14

Formulation of 11DO 10-2 Base Stock with V. I. Improvers
and Pour Point Depressants(1)

Product Code	Base Stock % Code	V. I. Improver % Type	Pour Point Depressant % Type	Water %	Viscosity, cs. 150°F 25 F.	AIT °F	AIT °F Without Water	Crystal. Point °F	Pour Point °F	
810428	807514	10.1 Green 75-11-90,000	10.0 Ethylene glycol	40.0	25.4	730.5	950	825	-15	-35
810422	"	9.4 "	21.2 "	31.8	25.7	754.9	925	800	None detected	-50
810436	"	9.7 "	10.3 Glycerine	41.3	27.5	840.9	950	900	0	-10
810432	"	8.4 "	22.8 "	34.2	27.8	1033.8	975	900	-35	-60
810437	"	9.6 "	10.8 1,2,6-Hexane-triol	41.4	25.9	900.3	825	825	+15	-35
810434	"	8.4 "	22.8 "	34.2	26.8	1216.9	950	975	-10	-45
810435	"	9.5 "	21.2 Thiodiethylene glycol	31.8	25.3	753.2	725	700	0	-5
810430	"	9.6 "	20.8 Tetrafluoropropanol	20.8	27.1	635.4	975	950	+20	+15
814310	807527	10.8 D-2220-60	9.2 Glycerine	36.8	25.2	1135.2	1000	Not det.	0	-25

(1) See Table 1 for abbreviations.

TABLE 15.
Formulation of BA 18-4 Base Stock (807315) with V. I. Improvers
and Pour Point Depressants (1)

Product Code	Base Stock %	V. I. Improver %	V. I. Improver Type	Pour Point Depressant %	Pour Point Depressant Type	Water %	Viscosity, cs.		AIT °F.	AIT Without Water °F.	Crystal Point °F.	Pour Point °F.
							150-F	25-F				
810450	38.4	5.6	Carbowax 6000	15.6	Glycerine	36.4	25.4	861.2	950	925	-15	-40
810441	36.8	9.2	Icon 75-11-10,000	10.8	"	43.2	26.0	678.6	975	900	0	-10
810440	32.6	6.2	"	17.7	"	41.3	25.3	653.8	900	875	0	-30
810451	37.6	9.4	"	10.6	Tetraethylene Glycol	42.4	26.1	701.6	900	825	0	-10
810447	36.0	9.0	"	22.9	"	33.0	27.0	1115.9	975	850	None observed	-40
810455	40.0	10.0	"	10.0	TM Formal (2)	40.0	26.5	947.0	950	850	+5	-25
810454	34.4	6.6	"	22.8	"	34.2	25.1	908.4	925	825	-25	-35
810449	40.9	8.2	"	23.6	Hexafluoro-pentandiol	35.5	27.0	918.1	925	750	+20	-20
810458	41.6	10.4	D2220-60	9.6	Glycerine	38.4	26.1	1017.3	1000	850	-15	-35
810459	42.4	10.6	"	9.4	1,2,6-Hexane-triol	37.6	27.2	1236.1	950	875	-15	-35

1. For abbreviations, see Table 8.

2. TM Formal: Formol of triethylene glycol monomethyl ether.

TABLE 16

Formulation of DPD 18-4 (807320), ODA 12-4 (807319) and AP 18-2 (807335)
 Base Stocks with V. I. Improvers and Pour Point Depressants(1)

Product Code	Base Stock %	Base Stock Type	V. I. Improver %	V. I. Improver Type	Pour Point Depressant %	Pour Point Depressant Type	Water %	Viscosity, cps 150°F	Viscosity, cps 25°F	AIT °F	AIT Without Water °F	Crystal Pt. °F	Pour Pt °F	Comment
807317	72.6	DPD	7.4	Icon 75-1499,000	16.9	Glycerine	44.1	25.0	627.5	1000	900	-5	-5	Solids precipitated
807322	34.6	ODA	6.6		17.1		30.9	25.5	781.3	950	950	-5	-25	
807327	36.0	AP	9.0		16.5		38.5	26.4	816.4	1000	925	-10	-30	
807339	42.8	AP	10.7	Carbowax 6000	9.3		37.2	25.7	912.5	950	850	-20	-45	
807337	33.2	AP	4.3	Carbowax 20-M	15.75		36.75	25.5	903.2	975	875	0	-30	

(1) Pour abbreviations, see Tables 9, 11 and 12.

TABLE 17

Formulation of MOCA 12-B(807322) Base Stock with V.I. Improvers and Pour Point Depressants(1)

Product Code	Base Stock %	V.I. Improver % Type	Pour Pt. Depressant % Type	Water %	Viscosity, cs 150°F 25°F	AIT °F without water	Crystal. Pt., °F	Pour Pt., °F	Comment
M14300-5	48.4	12.2 Carbonax 4000	15.0 Glycerin	23.4	25.7 836.3	900	-15	-45	-
M14300-10	33.0	4.4 "	17.4 "	40.6	27.0 806.9	925	0	-20	-
M10450	36.1	7.6 "	24.8 1,2,6-Hexane-triol	37.2	25.1 971.2	900	-5	-35	-
M01200B	35.0	5.8 5,000 75-11-90 (800)	16.8 Glycerine	39.2	26.3 1464.4	850	-5	-25	Cloudy at 150°F.
M10400	43.5	None	0.0 "	48.0	25.4 790.6	950	+15	+10	
M14300	40.0	None	16.0 "	42.0	25.7 849.1	925	0	-5	

(1) For abbreviation, see Table 10.

(2) Product 807322.

TABLE 18

Formulation of Base Stock Mixtures with V. I. Improvers and
Pour Point Depressants (1)

Product Code	Base Stock		V. I. Improver		Pour Pt. De- pressant		Water %	Viscosity, cs.		AIT without water °F.	AIT °F.	Cryst. Pt. °F.	Pour Pt. °F.
	%	Type	%	Type	%	Type		150°F.	25°F.				
807351	19.7	HDO 10-2 (807323)	9.9	Ucon 75 11-90,000	15.2	Glycerin	35.6	25.5	630.8	975	975	+10	-10
807352	19.7	MOCA 12-8 (807322)											
807353	18.0	HDO 10-2 (807323)	9.0	D-2220- 60	16.4	Glycerin	38.4	27.2	922.0	975	975	0	-25
807354	18.0	MOCA 12-8 (807322)											

(1) For abbreviations, see Table 13.

TABLE 19

Static Corrosion Tests

Fluid Type	Metal Panel	Weight Change, Gm. Per Panel			
		Package No. 1 (1)	Package No. 2 (2)	Package No. 3 (3)	Package No. 4 (4)
Type 1, BA 16-4 Formulation 807365: Base: 38.4% CW 6000: 9.6% Glycerin: 15.6% Water: 36.4%	1. Aluminum	Uninhibited -.0002 (5)(11)	-.0356 (5)(12)	-.0002	+.0001
	2. Phos. Bronze	-.0450(doubtful)	-.0002 (6)	+.0002	+.0002 (17)
	3. Bronze	-.0003 (6)	-.0004	-.0001	+.0008 (17)
	4. Steel, Galv.	-.0002	+.0028 (6)(13)	-.0002	None (17)
	5. Monel	None	+.0003 (6)	-.0003	-.0053
	6. Copper	-.0001	-.0004	None	+.0003
	7. Brazing Alloy	-.0001	N	-.0016 (16)	-.0005 (16)
	8. Steel	None	+C (6)	-.0007 (15)	-.0005
Type 2, H10 10-2 Formulation 810465: Base 38.7% (Ucon 75-H- (90000 9.7% Glycerin: 10.3% Water : 41.3%	1. Aluminum	-.0008 (6)(18)	-.0203 (5)(8)	+.0001 (6)	-.0001
	2. Phos. Bronze	-.0001	-.0003 (8)(9)	+.0002 (6)	+.0001
	3. Bronze	-.0001	-.0002 (6)	-.0018 (6)	+.0001 (6)
	4. Steel, Galv.	-.0020 (5)(19)	+.0032 (8)(9)(13)	-.0001	-.0004 (6) (12) (21)
	5. Monel	-.0001	+.0004 (6)(9)	None (6)	-.0003
	6. Copper	-.0001	-.0002 (8)(9)	+.0001 (11)	None
	7. Brazing Alloy	-.0001 (15)	-.0002 (8)(9)	-.0011 (11)	-.0005
	8. Steel	-.0002 (15)	-.0002 (8)(9)	-.0013 (14)	-.0004
Type 3, AP 18-2 Formulation 810467: Base 36.0% (Ucon 75-H- (90,000 9.0% Glycerin 16.5% Water 38.5%	1. Aluminum	-.0030 (6)(22)	-.0330 (8)(10)(12)	+.0001 (6)	+.0003 (7)
	2. Phos. Bronze	+.0002	+.0001 (8)(9)	+.0004 (6)	+.0003 (7)
	3. Bronze	None	None (6)(9)	+.0005 (6)	+.0006 (7)
	4. Steel, galv.	None	+.0043 (8)(9)(23)	+.0003	+.0001 (23)
	5. Monel	+.0002	+.0090 (8)(9)	-.0221 (6)	+.0001 (23)
	6. Copper	+.0002	+.0003 (8)(9)	+.0006 (6)	+.0006 (7)
	7. Brazing Alloy	+.0002	+.0003 (8)(9)	None	-.0001 (15)
	8. Steel	+.0003	+.0005 (8)(9)	+.0003 (13)	+.0002 (15)
Type 4, MOCA 12-8 Formulation 810470: Base 40.0% Glycerin 18.0% Water 42.0%	1. Aluminum	-.0084 (5) (24)	-.0066	-.0001 (11)	-.0001
	2. Phos. Bronze	-.0002	None	+.0004	-.0003
	3. Bronze	-.0003	+.0003	None	-.0001
	4. Steel, Galv.	-.0001	+.0017	-.0001	+.0001
	5. Monel	-.0001	+.0003	-.0001	+.0001
	6. Copper	-.0002	None	-.0001	+.0001
	7. Brazing Alloy	-.0001	+.0003	None	+.0004
	8. Steel	-.0002	-.0014 (6) (24)	+.0002 (6)	+.0004

(1) Package 1: Borax 5 H₂O, 1.5%, Nacap, 50% solution, 0.5%

(2) Package 2: Potassium phosphate, K₂HPO₄, 2.0%; aminomethyl propanol 0.25%; Nacap 0.6%

(3) Package 3: Liquibor 524, 1.0%; Nacap 0.5%

(4) Package 4: Borguard 2.5%; Nacap 0.5%

(5) White salt deposit

(6) Sludge

(7) Slight sludge

(8) Liquid separation

(9) Dispersed by inverting four times

(10) Product gelled

(11) Green film

(12) Black spots

(13) Moderate etching

(14) Very slightly pitted

(15) Few black spots

(16) Few black pitted areas

(17) Brown film

(18) Etched with multiple black spots

(19) About 1/8 of area attacked. Remainder like new. No pitting.

(20) Dark gray film on one side.

(21) Blue film

(22) Heavy black film

(23) Dark gray film

(24) Black film

TABLE 20
Stirring Corrosion⁽¹⁾

Position	Metal	Fluid 1: BA 18-4 Formulation 807365		Fluid 2: H ₂ O 10-2 Formulation 810465		Fluid 3: AP 18-2 Formulation 810467		Fluid 4: MOCA 12-8 Formulation 807389	
		Package No. 2 ⁽⁸⁾	Package No. 3 ⁽⁸⁾	Package No. 4	Uninhibited	Package No. 4	Package No. 4 ⁽⁸⁾	Package No. 4 ⁽⁸⁾	
1	Brazing Alloy	-0.0008	-0.0049	-0.105	-0.0003	-0.0029	-0.0063 ⁽¹⁶⁾		
2	Phos. Bronze	-0.0005	-0.131	-0.0001	-0.0008	-0.0030	-0.0006 ⁽⁶⁾		
3	Phos. Bronze	-0.0008	-0.011 ⁽⁴⁾	-0.014	-0.0012	-0.0045 ⁽⁵⁾	-0.0005 ⁽¹¹⁾		
4	Steel	+0.0001	-0.004 ⁽⁴⁾	-0.184 ⁽⁸⁾	-0.0007	-0.0003	-0.0019 ⁽¹¹⁾		
5	Brazing Alloy	-0.0020	-0.085 ⁽⁴⁾	-0.048	-0.0011	-0.0051	-0.0046		
6	Steel	+0.0002	-0.003 ⁽⁴⁾	-0.049 ⁽⁹⁾	-0.0004	-0.0002	-0.0001		
7	Bronze	-0.0030	-0.079	+0.009	-0.0012 ⁽¹²⁾	-0.0011	-0.0001 ⁽⁶⁾		
8	Gal. Steel	+0.0007	-0.019	-0.025 ⁽¹⁰⁾	-0.0005	-0.0012	-0.0173 ⁽¹⁷⁾		
9	Bronze	-0.027 ⁽³⁾	-0.103	+0.005	-0.0044	-0.0018	-0.0028		
10	Aluminum	+0.0003 ⁽⁴⁾	-0.006	+0.0004 ⁽⁴⁾	-0.0035 ⁽¹³⁾	-0.0003	-0.0010 ⁽¹¹⁾		
11	Copper	-0.015 ⁽³⁾	-0.022 ⁽⁶⁾	+0.002	-0.0001	-0.0008	+0.0001 ⁽⁵⁾		
12	Aluminum	-0.0001	-0.005	+0.0001 ⁽⁴⁾	-0.0005	-0.0001	-0.0012		
13	Steel	-0.0002 ⁽⁵⁾	+0.001 ⁽⁴⁾	-0.0001	+0.0006 ⁽¹⁴⁾	-0.0002 ⁽⁴⁾	-0.0003		
14	Aluminum	-0.0001 ⁽⁴⁾	-0.0014 ⁽⁴⁾	+0.0007 ⁽¹¹⁾	-0.0038 ⁽¹⁵⁾	None	-0.0016 ⁽⁴⁾		

(1) For the composition of the inhibitor packages and formulations, see Table 19; weight changes given in gm. per panel

(2) Contained 0.5% octanoic acid in addition to package No. 4.

(3) Black film

(4) Few minor corroded areas

(5) Dark film

(6) Dull

(7) Black spots, minor pitting

(8) Black corroded and pitted areas

(9) Black corroded areas

(10) Gray etched surface

(11) Few black corroded areas

(12) Few lightly corroded areas

(13) Black spots

(14) Copper colored film

(15) Few black areas, minor pitting

(16) Green film

(17) Gray corroded areas

(18) Phase separation soon after start

(19) Emulsified

TABLE 21

Rubber Swelling, Stability and Evaporation Tests

Fluid Type	Reference	Inhibitors	Anti-Wear Agent	Rubber Sweller	Rubber Swelling, %	High Temperature Stability	Low Temperature Crystallization		Evaporation Residue	
							Pass	Not det.	Character	Water Solubility
Type 1; 807365	BA	None	None	None	-11.1, -12.2, -11.7	Not det.	Pass	Not det.	Waxy solid	Soluble
Type 2; 810465	HDO	"	"	"	-10.8, -9.1, -9.8	"	"	"	Liquid	"
Type 3; 810467	AP	"	"	"	-10.7, -11.4, -9.7	"	"	"	Viscous liquid	"
Type 4; 810470	MOCA	"	"	"	-2.5, -2.7, -3.4	"	"	"	Viscous liquid	"
Type 1; 807387	BA	Pkg. 3	0.5% octanoic acid	"	-7.5, -8.5, -9.5	Pass	Not det.	Not det.		
"	"	"	"	1% isodeca-nol	-0.5, -2.2, -1.6	Not det.	"	"		
"	"	"	"	1% trideca-nol(oxo)	+10.0, +10.3, +10.5	"	"	"		
"	"	"	"	1% heavy oxo fraction ⁽¹⁾	+3.5, +2.6, +3.0	"	"	"		
Type 2; 810465	HDO	None	3% Pyronate sulfonate	"	-6.3, -5.8, -3.6	"	"	"		
"	"	"	None	1% trideca-nol(oxo)	-4.8, -5.3, -4.5	"	"	"		
Type 3; 810467	AP	"	0.5% stearic acid	None	-9.1, -9.1, -9.1	Thin film, probably stearic acid	"	"		
Type 4; 807389	MOCA	Pkg. 4	0.5% octanoic acid	"	-0.2, +1.5, +1.2	Pass	Pass	Pass	Not det.	
Type 2; 810490	HDO	Pkg. 3	"	"	Not det.	"	Not det.	Not det.		
Type 3; 810492	AP	Pkg. 4	"	"	Not det.	"	"	"		
Type 2; 810490	HDO	Pkg. 3 + Dow Corning anti-foam B	"	1% isodeca-nol	-6.9, -7.0, -6.6	Not det.	Pass	Not det.	Not det.	

(1) See footnote, p. 11.

TABLE 22

Falex Wear Tests

Type	MIL-H-22072 (Wep) Fluid	Inhibitor Pkg.	Anti-Wear Agent % Type	Max. Temp. °C.	Take-up Notches	Weight Loss, %	AIT °F.
		Unknown	Unknown	138	461	1.13	Not det.
Type 1 (807365)		Uninhibited		118	549	2.78	Not det.
" 2 (810465)		"		145	647	2.37	"
" 3 (810467)		"		123	609	2.67	"
" 4 (810470)		"		128	616	2.60	"
" 2 (810465)		None	3 Pyronate ⁽³⁾	120	461	2.38	950
"		"	3 Hexafluoropentanedioi	148	437	2.21	950
"		"	3 C ₇ Tri-H-fluoro alcohol	135	370	1.67	975
"		"	3 C ₉ Tert. Fluoro alcohol	148	593	2.07	975
"		"	3 Tetrachlorobutanol	140	432	2.12	900
"		"	0.5 [NP(OC ₆ H ₄ CH ₂ OH) ₂] ₃	135	614	2.50	950
"		"	0.5 Duponol C(4)	140	603	2.43	950
"		"	0.5 Stearic Acid	113	139	0.69	925
"		"	0.5 Octanoic Acid	97	131	0.61	950
" 1 (807365)		"	0.4 Stearic acid	103	405	1.80	950
" 1		"	0.5 Octanoic Acid	103	136	0.60	925
" 1 (807387)		3	0.5 "	110	31	0.092	900
" 1 (807397) ⁽¹⁾		4	0.5 "	113	53	0.11	950
" 2 (810495) ⁽¹⁾		3	0.5 "	135	73	0.16	1000
" 3 (810467)		None	0.5 Stearic Acid	127	264	0.90	975
"		None	0.5 Octanoic Acid	97	150	0.60	1000
"		4	None	140	182	0.60	975
" 3 (810492) ⁽¹⁾		4	0.5 Octanoic Acid	119	53	0.14	950
" 3 (810492)		4	0.5 "	115	50	0.14	1000
" 4 (807389) ⁽²⁾		4	0.5 "	131	46	0.12	950
" 4 (807395)		4	0.5 "	120	53	0.15	950

(1) Also contained 1% isodecanol and 5 drops/qt. of Dow-Corning Antifoam B.

(2) Also contained 5 drops/qt. of Dow-Corning Antifoam B.

(3) A water soluble hydrocarbon sulfonate.

(4) Sodium lauryl sulfate.

TABLE 23

Foaming Characteristics at 75°F

Fluid	Inhibitors	Defoamer Drops/qt. Type	Foam Vol. ml.		Time Required to Obtain 10 ml.
			After Blowing	10 min. after Blowing	
Type 1; 807387	Pkg. 3 + 0.5% octanoic acid	None	395	283	38'28"
"	"	2 Dow-Corning Antifoam B	375	232	25'25"
"	"	5 "	330	52	13'4"
"	"	10 "	225	0	5'47"
"	Pkg. 4 + 0.5% octanoic acid + 1% isodecanol	5 "	148	0	2'13"
Type 2; 810490	Pkg. 3 + 0.5% octanoic acid + 1% isodecanol	None	469	262	20'10"
"	"	5 oleyl alcohol	444	188	19'10"
"	"	5 each (oleyl alcohol (Pluronic L-101	325	187	26'9"
"	"	5 each (oleyl alcohol, (Pluronic L-101, (Antifoam B	260	0	6'39"
"	"	5 Dow-Corning Antifoam B	207	0	5'29"
Type 3; 810492	Pkg. 4 + 0.5% octanoic acid	None	223	70	16'25"
"	"	5 Dow-Corning Antifoam B	239	0	7'30"
Type 4; 807389	Pkg. 4 + 0.5% octanoic acid	None	358	227	33'27"
"	"	5 Dow-Corning Antifoam B	82	0	1'36"
"	"	10 "	40	0	0'50"
Type 5; 810499	(807389 reformu- lated with CW6000	5 "	390	109	15'2"
"	"	10 "	106	0	1'27"

TABLE 24

Sonic Shear and Compatibility with Sea Water⁽¹⁾

Fluid Type	Sonic Shear		Reference Number	Compatibility with 10% Sea Water				Reference Number	
	Viscosity, cs. Before	Viscosity Change % After		Miscibility	Cryst. Pt. °F	Pour Pt. °F	Viscosity, cs. 150°F		Viscosity, cs. 25°F
Type 1, BA	25.9	+ 4.2	807365	Miscible	+ 5	-25	19.9	491.1	810450
Type 2, HDC	26.6	+ 1.1	810465	Separates at boiling point	+ 5	-15	23.6	577.7	810436
Type 3, AP	27.2	+ 3.7	810467	"	+ 5	-15	20.2	475.8	810457
Type 4, MOCA	26.1	+ 1.1	810470	Miscible	+ 5	0	19.9	484.5	814355
Type 5, formula- lated 215, CW 20-M	25.5	- 9.4	814395	Not det.					

(1) Products contained V.I. improvers and pour point depressants, but no additives.
 (2) Polyethylene glycol of molecular weight 20,000.

TABLE 25

Effect of 0.5% Antioxidants on the Autogenous Ignition of HDO Base Stock

<u>Product Type</u>	<u>Additive</u>	<u>A.I.T.</u>
HDO 632360	None	900, 925
" "	Phenothiazine	925
" "	N,N-diphenyl-p-phenylene diamine	925
" "	Phenyl-β-naphthylamine	925
" "	Age Rite Resin D ⁽¹⁾	925
" "	2,6-Di-tert.-butyl phenol	925
<hr/>		
HDO 807314	None	900, 925
" "	Antioxidant 2246 ⁽⁴⁾	925
" "	(Butylated hydroxyanisole (BHA))	925
" "	(Butylated hydroxy toluene (BHT))	925
" "	Age Rite White ⁽³⁾	925
" "	5-Ethyl-10, 10-diphenylphosphazasiline	925
<hr/>		
HDO 807360	None	850
" "	Pyrogallol	850
" "	Cupric acetyl acetate	925
" "	Anti-oxidant 702 ⁽²⁾	925
" "	Cobaltic acetyl acetate	850
" "	Benzanilide	900
" "	Tetraphenyl lead	850
" "	Phthalimide	900
" "	Phenylbenzoate	900
" "	Tetraphenyl tin	875
" "	n-Propyl gallate	925
" "	Anthraquinone	900
" "	Triphenyl phosphite	925

(1) Polymerize trimethyldihydroquinoline (R. T. Vanderbilt Co.)

(2) 4,4'-Methylene bis 2,6-di-tert.-butylphenol (Ethyl Corp.)

(3) N,N'-Di(2-naphthyl)-p-phenylenediamine (R. T. Vanderbilt Co.)

(4) 2,2'-Methylene bis (4-methyl-6-tert. butyl phenol) (American Cyanamid Co.)

TABLE 26

Reproducibility of Base Stock Preparation

<u>Product</u>	<u>Reference No.</u>	<u>Viscosity, cs. 150°F.</u>	<u>AIT °F.</u>	<u>Hydroxyl No. Found</u>	<u>Theory</u>
BA 18-4	807315	434.1	875	255,253	226
"	807336	421.3	850	265,263	"
"	807370	448.4	850	251,251	"
"	807373	480.6	900	250,249.5	"
"	807394	469.2	850	253,256	"
HDO 10-2	807314	113.0	950,925	208	217
"	807323	105.5	850	225,224	"
"	807327	116.7	850	218,220	"
"	807360	119.4	850	214,216	"
AP 18-2	807329	206.2	925	202,204	198
"	807335	230.1	950	195,195	"
"	807355	231.6	850	186,187	"
"	807356	216.0	900	193,194	"
"	807358	216.6	850	188,189	"
"	807374	220.0	875	197,200	"
MOCA 12-8	807322	6180	875	451,451	486
"	807364	7943	825	439,438	"
"	807377	6220	875	439,440	"
"	807390 I	6449	Not det.	453, 452	"
"	807390 II	6298	"	449,453	"

TABLE 27

PUMP TESTS

Fluid Type	Inhibitor Prg.	Anti-Wear Agent	Time Hrs.	K. Vis., cs. 150 F.	pH	Sp. Gravity	% H ₂ O	Pump Weight Loss, %		Remarks
								Cam Ring	Roller Vanes Rotor	
Type 1, BA (807352)	3	0.5% octanoic acid	0	25.0	7.3	1.12	39.2	-	-	Slight wear on cam ring
			50	26.0	7.2	1.11	37.9	-	-	
			100	27.8	6.3	1.26	36.3	0.49	0.09	0.05
Type 2, BDO (807352)	3	0.5% octanoic acid	0	26.1	7.2	1.10	41.0	-	-	Pump barely delivered 800 lbs. at end of test
			50	26.3	6.5	1.11	41.1	-	-	
			100	29.2	6.3	1.10	40.0	0.88	2.56	0.22
Type 3, AP (807352)	None	0.5% stearic acid	0	27.5	11.0	1.11	39.4	-	-	Pump delivered only 200 lbs. at end of run
			50	25.5	7.4	1.10	36.2	-	-	
			100	27.8	6.2	1.10	35.8	1.4	0.5	0.1
Type 4, MCOA (807352)	4	0.5% octanoic acid	0	25.8	7.5	1.15	39.3	-	-	Fluid lost between 50 and 100 hours, due to leaking gasket
			50	40.7	7.0	1.14	32.5	-	-	
			100	42.7	6.7	1.15	Not det.	0.00	0.09	0.00

TABLE 28

Final Formulations

Composition	Type 1 (807397)	Type 2 (810495)	Type 3 (810500)	Type 4 (807395)	Type 5 (810499)
Base Stock	BA 16-4, 35.87%	HDO 10-2, 36.64%	AF 18-2, 34.05%	MOCA 12-8, 40.68%	MOCA 12-8 30.72%
Carboxax 6000	8.94%	-	-	-	8.39%
Ucon 75-H-90000	-	9.18%	8.51%	-	-
Glycerin	14.56%	9.75%	15.59%	18.27%	18.63%
Water	36.18%	41.62%	37.48%	37.61%	39.67%
Isodecanol	1.00%	0.91%	0.97%	-	-
Olefinic Acid	0.49%	0.48%	0.47%	0.49%	0.38%
Burguard	2.45%	-	2.37%	2.46%	1.85%
Isqubar 524	-	0.94%	-	-	-
Soap	0.49%	0.48%	0.52%	0.49%	0.37%
Dow-Corning Antifoam B	5 drops / quart	10 drops / quart			

Physical Properties

K Viscosity @ 150 F	25.2	27.0	25.4	25.8	25.7
K Viscosity @ 25 F	855.4	763.4	812.6	1,096.7(s)	788.0
pH	7.15	7.25	7.20	7.50	7.38
Specific Gravity	1.11	1.10	1.11	1.15	1.13
Co. p. with sea water	Soluble to boiling pt.	Soluble below 211 F.	Soluble below 203 F.	Soluble to boiling pt.	Soluble to boiling pt.
Airgenous Ign. T.	950	1000	950	950	975
Pour Point, F (1)	-40	-5	-35	-30	-30
Flash Point, F (6)	>500	>530	>520	>515	>515
Fire Point, F (6)	>500	>530	>520	>515	>515

(1) Refers to the base stock.

(2) Began to crystallize at -17 F.

(3) Began to crystallize at 27 F.

(4) Began to crystallize at 0 F.

(5) Without additive package 4, 304.2 cc.

(6) Products boiled at the indicated temperatures without flash or fire points.