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THE DEVELOPMENT OF THE HYDROLUBE
NON-INFLAMMABLE HYDRAULIC FLUIDS

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ABSTRACT

This report concerns the development of non-flammable hydraulic fluids suitable for use in aircraft. A survey of the few types of fluids appearing essentially non-flammable from specially devised flammability tests, indicated that aqueous fluids held the most promise. Research on this kind of fluid led to the development of the Hydrolubes, which are defined as polymer-thickened corrosion-inhibited aqueous fluids having one or more of the glycols as the major constituents.

Inflammability, freezing point and viscosity measurements led to the selection of ethylene glycol as the freezing point depressant to be used with the water, in concentrations of approximately fifty percent. In order to have a fluid with the proper viscosity and viscosity-temperature characteristics, (V.I.) it was considered necessary to thicken the glycol-water mixture with a linear polymer. Many potential polymeric thickeners were investigated, the most suitable being polyalkylene glycol derivatives. The solutions thickened with this polymer are known as the Hydrolube U fluids.

Corrosion inhibitors (both liquid phase and vapor phase) had to be developed for the polymer-thickened, glycol-water mixtures. In military aircraft, eight different metals and twelve dissimilar metal contacts were known to exist, and these, if possible, had to be protected. While the search for the most suitable inhibitor or combination of inhibitors necessitated carrying out tests with hundreds of corrosion inhibiting materials, several very promising inhibitor combinations were obtained.

During early mechanical screening tests (which were carried out concurrently with laboratory tests on many fluid compositions), it became apparent that some wear-reducing additives would be necessary in the Hydrolubes. Oiliness or wear-reducing additives of the type that would form hydrophobic films on steel were studied for this purpose. These invariably belonged to the "Colloidal electrolyte" class. After laboratory methods were devised for examining these hydrophobic films, many such compounds were examined, and it was concluded that the substituted ammonium carboxylates derived from organic acids having eight or more carbon atoms would be the most suitable. Even these, however, did not produce fluids with greatly improved lubricating properties until special means were developed for solubilizing or dispersing the wear additives.

Hydrolube U appears on the basis of laboratory and Naval flight tests to hold much promise as a suitable hydraulic fluid for aircraft. The more recently developed Hydrolubes U-2 and U-3 show greatly improved lubricating properties (although somewhat less desirable corrosion inhibiting properties), as compared with Hydrolube U. These improved fluids will give greater freedom from wear in the present hydraulic systems.

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new laboratory method was developed for measuring the flammability hazard involved in the presence of oil sprays or mists in the atmosphere. A large number of hydraulic fluids and new fluids of promise were studied, and the conclusions relative to their flammability resistance were verified by numerous incendiary firing tests carried out at Dahlgren Proving Grounds through the cooperation of the Bureau of Ordnance. These flammability studies have been described elsewhere. (1,2)

4. Chemical research was undertaken at this laboratory in early 1943 to develop less flammable hydraulic fluids, and it was later decided to emphasize the production of an aqueous-base fluid. The help of K.E. Walker of the Ammonia Division of the DuPont Company was obtained in the search for available chemicals to serve as suitable polymer thickeners and corrosion inhibitors. At the request of this laboratory, the results of the investigations of that organization were later summarized in a company report by Walker, Harford and Wholey. (4) In the meantime this laboratory concluded that no available polymer would be entirely satisfactory, and a simple theoretical argument led to the selection of a new polyalkylene glycol copolymer. While the desired copolymer had never been made, it was a homologue of the new Ucon synthetic oils, and the cooperation of the Carbide & Carbon Chemicals Corporation was obtained in its synthesis. This resulted in the production of the polymer used in Hydrolube U. Later, the cooperation of the Rohm & Haas Company made it possible to obtain four newly prepared water-soluble polymers, one of which was used in the development of Hydrolube A. Both Hydrolubes U and A were found suitable for use in fighter planes in the summer of 1945, and preparations were underway to carry the problem into the production stage when the war ended. As its adoption was then no longer pressing, production was postponed for a while in order to attempt to further improve the fluids with respect to wear preventative properties. However, in the summer of 1946, the incidence of a number of serious fires in commercial and military transport planes aroused an increased interest in these fluids. Hence, a summary of progress in the development of non-flammable fluids for aircraft (3) was prepared for military and industrial consumption and was released and distributed widely by the Bureau of Aeronautics and the Civil Aeronautics Administration. In the latter part of that report, an outline was given of the development and properties of Hydrolubes U and A. It is the purpose of this report to present a more detailed technical account of the development of the various fluids investigated, including Hydrolubes U and A. It is proposed at a later date to present a report on research in progress the past year on the improvement of

the anti-wear properties of the Hydrolubes in which emphasis will be given to wear tests, to bench tests and flight tests, and to the modification and redesign of mechanical equipment found advantageous in order to obtain the maximum effectiveness of the Hydrolube fluids.

5. It is desirable here to define what is meant by a "Hydrolube" fluid. The name "Hydrolube" was selected by this laboratory early in the war as a generic name for any hydraulic fluid consisting of a polymer-thickened, corrosion-inhibited, aqueous solution having one or more of the glycols as major organic components. It was found convenient to identify the different Hydrolubes by a letter following the generic name; thus the names Hydrolube A and Hydrolube U were used to distinguish the fluids thickened with "Acrysol" (sodium polymethacrylate) polymers from those thickened with "Ucon" (polyalkylene glycol) copolymers.

C. Known Facts Bearing on the Problem

6. Prior to this investigation no aqueous fluids had been developed which were suitable at low temperatures for use as aircraft or ordnance hydraulic fluids or as lubricants in modern equipment. The ordinary aqueous-base cutting fluids and textile lubricants, which are oil-in-water emulsions, were considered unsatisfactory for the proposed uses. For not only do such fluids break or freeze at temperatures likely to be encountered during use, but their viscosities also vary markedly, depending upon the cleanliness of the system and the degree of dispersion of the oil in the aqueous mixture. Ethylene glycol solutions containing small amounts of water, a corrosion inhibitor, and a copper de-activator were used during World War II as aircraft engine coolants (specification AN-E-2b) and as hydraulic fluids in aircraft arresting systems. But such compositions are unsatisfactory for the uses contemplated, due to their high freezing points, their large temperature coefficients of viscosity (low V.I.) and their limited flammability resistance. Corrosion inhibited solutions of glycerine in water have been used for years in naval gun recoil systems (specifications O.D. 1914 and O.S. 1914) and in numerous industrial hydraulic systems not exposed to the extremes of temperature. At the end of World War II, it was found that the German cruiser, the Prinz Eugen, was fitted with some hydraulic equipment operated with a corrosion inhibited glycerine-water solution. However, this and similar solutions are only suitable for use in slow-speed, piston-operated hydraulic equipment unexposed to the extremes of temperature and made mainly from ferrous metals.

7. Antifreeze glycol-water and alcohol-water solutions are widely used in automobile radiators when inhibited against corrosion by compounds such as borates, phosphates, amines and amine salts, sulfonates, and 2-mercaptobenzothiazole. Unfortunately, too little is known of their mode of operation as inhibitors. The reliable published data relative to the physical and chemical properties involved in their action as inhibitors are so limited that the problem of inhibiting the corrosion of any new water-base, low freezing point fluid is solvable at the present only by the use of a time-consuming and essentially empirical experimental approach. The principal outside sources of information used were the confidential report of Walker, Harford and Wholey (4) of the DuPont Company, and the patent literature.

THE CHOICE OF THE AQUEOUS BASE FLUID

A. Flammability Considerations

8. The use of water as a fire resistant component in the hydraulic fluid compositions is based on the results of the earlier flammability investigation (1,2) of this laboratory. For convenience of reference, Table I has been prepared from these reports. Of the pure freezing point depressants studied, it will be noted that only the glycols have reasonably high resistance to the fire hazards considered. The glycols alone do not appear satisfactory, however, for even if the required low freezing point is obtained by the use of a mixture of homologous glycols, the problem of obtaining a sufficiently low viscosity at -40°F to decrease the low temperature torque cannot be properly solved. Moreover, greater flammability resistance to a wide variety of fire hazards is desirable, as is greater freedom from the deleterious effects of the pure glycols on rubber packings. It will be noted that the spray flammability limit of ethylene glycol-water solutions becomes very high when 45% or more (by volume) of water is present. The same is true for aqueous solutions of diethylene glycol, "methyl carbitol" (the monomethyl ether of diethylene glycol), "carbitol" (the monoethyl ether of diethylene glycol), "ethyl cellosolve" (the monoethyl ether of ethylene glycol) and "butyl cellosolve" (the monobutyl ether of ethylene glycol). However, 45% water is not sufficient to adequately increase the spray flammability limit of methyl cellosolve.

B. Freezing Points and Other Considerations

9. With 45% or more (by volume) water present in the hydraulic fluid, it is evident that a freezing point depressant is needed. Considerations relative to obtaining a low enough freezing point must influence the decision as

to the most suitable proportions of the water and the freezing point depressant. Furthermore, a linear polymer must be added to the aqueous base fluid (to the mixture of water and freezing point depressant) in order to increase the viscosity at 130°F as well as to increase the viscosity index. At a conference with the representatives of the Bureau of Aeronautics, it was agreed that the viscosity of the finished aircraft hydraulic fluid should not be less than 10 centistokes (csks.) at 130°F, and not over 2,000 csks. at -40°F, and that the freezing point should be not higher than -50°F, and as low as -65°F if possible. For use on ships, the Bureau of Ordnance requested that the viscosity should not be less than 10 csks. at 210°F and not over 215 csks. at 0°F, while the pour point should be below -40°F. Aside from these specifications, it was realized that the following factors should be considered in the selection of base fluids:

(a) The base fluid should not contain materials whose boiling points are below that of water, and it is preferable that they should be much higher;

(b) It should be as stable as possible to thermal oxidation in the atmosphere;

(c) It should have as low a viscosity at 130°F and at -40°F as possible in order to obtain the highest possible V.I. when thickened with a polymer;

(d) It should be capable of being inhibited against the corrosion of common metals used in hydraulic systems;

(e) It should not attack rubber or plastic parts exposed in present equipment to contact with the hydraulic fluid;

(f) It should be capable of large volume production in the near future or in time of war.

10. The selection of the freezing point depressant was made on the basis of a number of simple considerations. These data are summarized in Tables II and III. The lower molecular weight alcohols such as methyl, ethyl and isopropyl alcohols were eliminated because of their low boiling points. The higher molecular weight alcohols having high enough boiling points were too insoluble in water. Similarly, the higher boiling ethers were eliminated because of their low solubility in water. The "carbitols" of higher boiling points than methyl carbitol were eliminated because their aqueous solutions containing enough water to render them sufficiently non-flammable and low in freezing point were too viscous at -40°F to permit adequate V.I. improvement (See Table II). Even methyl carbitol-water mixtures containing 30% to 40% water had viscosities of from 400 to 300 csks. in contrast to the much lower viscosities of glycol-water solutions; moreover, the methyl-carbitol-water mixtures were more deleterious to rubber packings than the

glycol-water solutions. "Cellosolves" with molecular weights above ethyl cellosolve were too viscous and were poor freezing point depressants. Ethyl cellosolve, with a boiling point of 135°C, was much less promising than ethylene glycol, with a boiling point of 197°C. Dimethoxytetraethylene glycol-water solutions containing 30% or more water had viscosities at -40°F of more than 950 csks. and hence were far too viscous for use. Aqueous mixtures of beta-(methoxy methoxy) ethanol containing slightly more than 30% water had freezing points which were too high, and mixtures having such low proportions of water were also too flammable. Of the homologous series - ethylene glycol, diethylene glycol, triethylene glycol, etc., the first was preferred because its viscosity was least; its attack on packings was least; its boiling point was adequately high (197°C); and its availability was the best. Of the homologous series - ethylene glycol, propylene glycol, butylene glycol, etc., the first was preferred for the same reasons. Thus it was concluded that the most suitable base fluid would be some mixture of ethylene glycol and water.

11. A graph of the freezing point vs. the percentage composition for ethylene glycol-water solutions will be found in Plate 1. If the data from this graph are expressed in the nearest whole numbers, the eutectic mixture freezes at -65°F and contains 67% by volume glycol and 33% by volume water. It was not considered desirable to use the eutectic mixture, however, since any evaporation of water from the hydraulic fluid would cause a rapid increase in the freezing point. A more reliable base fluid should contain somewhat more water than the eutectic mixture, for any small loss of water through evaporation would then merely cause an additional decrease in the freezing point. The spray flammability and incendiary fire tests (1,2) demonstrated that at least 40% by volume water was necessary to assure complete freedom from the propagation of flames in the liquid spray in the atmosphere. In the former test, 40% oxygen was needed for an 80/20 glycol-water mixture, and over 80% for a 50/50 mixture. These considerations led to the decision to use a mixture of 55% by volume of ethylene glycol and 45% by volume of water as a base stock in developing a Hydrolube for use as a hydraulic fluid. Such a fluid has a freezing point of -55°F. Lower freezing points could no doubt be obtained by the introduction of a third component such as one of the higher glycols or ethyl carbitol, methyl carbitol, ethyl cellosolve, etc., but such mixtures have not been given full consideration to date, in order to avoid complicating the development program by problems such as availability or increased rates of attack on hydraulic packings and plastic parts.

C. Some Properties of the Base Fluid Selected

12. The viscosity-temperature characteristics of the 55/45 (volume ratio) glycol-water solution selected for the base fluid and of those having the volume ratios 70/30, 60/40, 50/50 and 40/60 have been graphed on ASTM viscosity chart paper in Plate 2. From the graph for the 55/45 ratio, the following viscosities have been read off for future reference:

Viscosity-Temperature Characteristics of 55/45 Base Stock

<u>Temperature in °F</u>	<u>Viscosity in csks.</u>
210	0.79
130	1.74
100	2.60
77	3.75
32	10.0
0	25.0
-20	52.0
-40	134.0

The following constants of the 55/45 base fluid are also collected here for convenience of reference:

Spontaneous ignition temperature:	897°F
Spray flammability limit:	between 67% and 80% oxygen
Specific gravity:	25°C/4°C 1.085
Boiling Point:	228°F (760 mm.)
Freezing point:	-55°F

POLYMERS FOR THICKENING AND V.I. IMPROVEMENT

A. General Information

13. Linear polymers have been much used by the petroleum industry to improve the viscosity index of lubricating and hydraulic oils. They have also been used as pour point depressants for engine lubricants. One of the early research problems encountered here was to find or to develop water soluble (or dispersable) linear polymers suitable for improving the viscosity and V.I. of the glycol-water base fluid. It was hoped that the polymers would also serve to depress the freezing point. In the search for suitable polymers a few guiding principles were used. Increasing the molecular weight of the polymer lowers the weight concentration of polymer necessary to obtain a given viscosity rise and viscosity index. As the molecular weight increases beyond approximately 15,000, the rate of shear breakdown or

mechanical degradation of the polymer becomes increasingly important, the effect becoming most disturbing at average molecular weights of 50,000 or more.⁽⁵⁾ This means that the viscosity increase and V.I. improvement obtained by the addition of high polymers will decrease with time due to the mechanical working in the hydraulic systems. Hence, the value of a V.I. improver is not determined until a mechanical degradation test of some kind has shown that the rate of shear breakdown is sufficiently small. It was also desirable to avoid using a polymer thickened fluid which would leave a hard or a very tacky residue after the evaporation of the water and glycol. Such a residue is undesirable since it can cause cementing of threaded fittings, seizing of sliding parts and accelerated failures of rubber packings. Thus a polymer which is a liquid or a soft waxy solid over the temperature range of interest is more likely to be satisfactory than one which is tacky, glassy or crystalline.

B. Polymers Studied

14. The first survey of water soluble, linear polymers was made at the recommendation of this laboratory by Walker, et al.⁽⁴⁾ Their report listed some twenty polymers, among which were carbowax, polyvinyl alcohol, glycol cellulose, methyl cellulose, abopon, gum arabic, gum ghatti, agar agar, methyl starch and the sodium, potassium and ammonium salts of polyacrylic and polymethacrylic acids. Solutions in the base fluid of each of these polymers were tested in the hydraulic bench tests with Pesco gear pumps. While none of the fluids containing polymers below 15,000 average molecular weight showed evidence of shear breakdown, the most promising polymers were found to be either Carbowax 6000, a solid polyethylene glycol polymer made by the Carbide & Carbon Chemicals Corporation, or any one of the alkali salts of polymethacrylic acid. A fault with the Carbowax polymer was its limited solubility in the base fluid at temperatures below -25°F.

15. After this laboratory had obtained sufficient hydraulic equipment to conduct its own experiments and tests on wear and mechanical stability, the first work was on the Hydrolubes containing the polymers recommended by Walker⁽⁴⁾ and his co-workers. Subsequently experiments were made using the Carbide and Carbon Chemical Corporation's new polyalkylene glycol derivative (Ucon LB) and one of its copolymers (Ucon HB)⁽⁶⁾. As the former was found too insoluble in the base fluid and the latter was found not sufficiently soluble over the entire temperature range desired, attention was shifted to obtaining a copolymer which could be expected to have sufficient solubility in water. Thus the manufacturers were urged to prepare a similar copolymer having an ethylene

oxide content half way between that of the Carbowax and that of the Ucon HB copolymer. The resulting copolymer designated by the producer as "Ucon" 75 H* (see footnote) was found suitable for the preparation of fluids superior to those made from Carbowax. Not only did the new polymers remain in solution from -50°F up to the highest temperatures used, but it also caused a greater V.I. improvement than Carbowax 6000, and was not broken down by the shearing action of pump systems. When it was later learned that the Rohm and Haas Company had developed new water soluble polymers from polyacrylic and polymethacrylic acids, their cooperation was obtained and four polymers were prepared and examined. Of these polymers only the "Acrysol" G-3667 and G-3650** (sodium polymethacrylate) were found sufficiently promising for use in the Hydrolubes. It was also found that these types of polymers when used in concentrations up to 25% in the 55/45 glycol-water base mixture did not decrease the flammability resistance of the fluid.

16. The final choice of a suitable polymer included the consideration of the residue remaining after the evaporation of volatile constituents from the Hydrolube. When the water and glycol had evaporated from an exposed metal surface covered with a thin layer of each of the Hydrolube fluids, the fluid containing the Ucon polymer left a viscous, oily residue, while the fluid containing the sodium polymethacrylate polymer became increasingly tacky and finally left a hard, adherent resin. The V.I. improvement by the former polymer was less than by the latter, the difference being due to the higher average molecular weights of the latter. However, solutions of the former showed no evidence of shear breakdown, while those of the latter evidenced considerable shear breakdown. The study of both polymers was continued in order to determine whether there were any important differences in the wear preventative properties of the resulting hydraulic fluids. The two types of water-glycol base fluids were given the names "Hydrolube U" and "Hydrolube A", the "U" and "A" denoting that the polymers added were the Ucon copolymer and the Acrysol polymer respectively. In each case the viscosity at 130°F was adjusted to 10 csks., and the base stock was a solution of 55% by volume of commercial ethylene glycol and 45% by volume of distilled water.

(Footnotes)

* In order to indicate the relative average molecular weight of the Ucon polymer, the producer measured its viscosity at 100°F and included the value obtained as a part of the serial designation for the polymer. Thus the first laboratory preparation of the polymer supplied this

laboratory was Ucon 75-H-26,000 - the 26,000 being the viscosity of the polymer in S.U.S. at 100°F. Later laboratory preparations of these polymers possessed higher average molecular weights and were designated Ucon 75-H-35,000, Ucon 75-H-54,000, etc. The first batch from pilot plant production was Ucon 75-H-69,400, and this is the Ucon polymer used for almost all of the work on Hydrolube U reported here.

** The numbers following "Acrysol G" were assigned by the manufacturer to designate the particular batch of Acrysol polymer shipped. No indication was given as to the molecular weights of the various batches.

C. Properties of Thickened Base Stock

17. In Table IV will be found the viscosities of various polymer-thickened, uninhibited Hydrolube fluids. All of the fluids were prepared so as to have viscosities as near 10 csks. at 130°F as possible in order to match the viscosity of the petroleum hydraulic fluid of AN-VV-O-366b specification at that temperature. Of the seven promising polymers listed, only polymers #4 and #5 gave fluids with viscosities much below 1000 csks. at -40°F. However, because of the extremely small quantities of the polymers needed to obtain the desired viscosities at 130°F, they were considered likely to exhibit excessive shear-breakdown during use. A suitable decrease in the degree of polymerization could possibly make these polymers more suitable. Polymers #1 and #3 were considered unsuited for further study at the time, because cloudiness and precipitation would occur in the Hydrolubes made with them. Although polymer #2 formed a clear solution in the glycol-water mixture, it precipitated when the various promising inhibitors were added; moreover, the polymer concentrate was shipped in a mixture of beta-(methoxy methoxy) ethanol and water, because of its inadequate solubility in water, and even the mixtures as shipped were found to separate into two phases after long standing. Hence, this polymer was considered unsatisfactory for use in glycol-water mixtures of interest here and especially in the presence of the amine phosphate-MBT-DIPAN inhibitor combinations. This left polymers #6 and #7, both of which produced Hydrolube fluids warranting further study and evaluation. Therefore, Hydrolubes containing these polymers were carried through the various laboratory and mechanical studies and tests to flight testing in naval aircraft.

18. Evaporation of water from the Hydrolubes during operation would bring about an increase in viscosity; notwithstanding, no significant viscosity increase of any Hydrolube tested has been noted in the bench tests, mock-up or flight tests conducted to date. Shear-breakdown of the polymer

during operation would cause a decrease in viscosity; even so, no decrease in viscosity has been noted with the Ucon-thickened Hydrolubes. While considerable shear-breakdown was noted in the Hydrolube A fluids, it was not considered too great to be tolerated in aircraft systems. The rate of shear-breakdown of this polymer was observed in a bench test using a Pesco gear pump, and the results are given in Plate 3. It will be observed after 3,000 cycles of the fluid through the pump that the decrease in viscosity becomes a linear function of the number of cycles. In the system used, 8,500 cycles corresponded to 100 hours of pump operation.

19. As all of the Hydrolube fluids investigated were found to supercool readily, it was difficult to obtain satisfactory freezing point determinations in the conventional manner. Therefore, the method used consisted in freezing the mixture, if possible, in a mixture of dry ice and acetone, and in allowing the frozen mixture to stand a day or two at the desired test temperature (-50°F or -60°F usually) to ascertain whether or not it would thaw. The freezing point of uninhibited Hydrolube U was thus found to be between -50°F and -60°F . As stated earlier, polymethacrylic acid neutralized with an alkali metal hydroxide was found to be a fairly satisfactory polymer for the Hydrolubes. In general, fluids with freezing points approximately 10°F lower can be obtained with sodium polymethacrylate as a thickening agent than with the Ucon 75-H type. The freezing point of the uninhibited Hydrolube A is below -60°F .

THE PROBLEM OF CORROSION INHIBITION

A. Introduction

20. Inhibiting corrosion in an aqueous fluid for use in present aircraft hydraulic systems is much more difficult than inhibiting an aircraft engine anti-freeze coolant. Difficulties arise because of: (a) the wider variety of metals and metallic couples present in the hydraulic systems; (b) the much lower temperatures to which the fluid is exposed, thus making it necessary to eliminate many inhibitors which are not sufficiently soluble at low temperatures; and (c) the desirability of obtaining vapor phase corrosion inhibition.

21. Discussions with the representatives of the Bureau of Aeronautics and with producers of aircraft equipment revealed that present-day military planes (and presumably commercial and private planes) are likely to contain the majority of the following metals: steel, copper, aluminum, 52 S σ aluminum, 24ST aluminum, lead-bronze, tin-bronze,

brass, zinc-plated steel, and cadmium-plated steel. In addition, the handling of the fluids in cans during storage and shipment is likely to expose the fluid to contact with tin and 50/50 lead-tin solder. The pairs of metals usually in direct metallic contact while immersed in the hydraulic fluid are: steel against brass, steel against bronze, steel against copper, steel against zinc-plated steel and cadmium-plated steel, steel against aluminum, solder against copper, solder against steel, solder against bronze and brass, cadmium-plated steel against aluminum, and zinc-plated steel against aluminum.

22. The vapor phase corrosion of steel was considered a potential hazard in some of the aircraft hydraulic systems, since they were not designed for use with aqueous fluids, and since it was possible that some steel parts would not always be completely immersed in the fluid. It was also conceivable that such an exposure of the steel parts could happen during storage, shipment, installation, adjustment or repair of hydraulic components. Corrosion inhibition during storage and shipment needed especial consideration, for in such instances equipment coated with petroleum oils or greasy or waxy rust preventatives would create a serious problem in the cleaning of parts prior to their installation in the aqueous hydraulic systems. Hence the utilization of the Hydrolubes was held up for over a year while this laboratory sought a vapor phase corrosion inhibitor or some means of circumventing such difficulties.

23. In order to have a satisfactory aqueous hydraulic fluid for use in aircraft, the inhibitor or combination of inhibitors used should be thermally stable to oxidation at temperatures as high as 180°F. They should be effective in small concentrations, e.g. from 0.1% to 5%. They should be soluble in the base fluid at temperatures ranging from at least -50°F to 180°F. They should not interfere either with the thickening action or solubility of the polymer thickener, or give rise on oxidation to detrimental products. Finally, they should not cause the fluid or its vapor to be too toxic for safe use when the fluid leaks out or is spilled in poorly ventilated compartments.

24. At the recommendation of this laboratory, K.E. Walker of the Ammonia Department of the DuPont Company undertook a search for suitable corrosion inhibitors for the Hydrolubes, especially the Carbowax thickened fluids. His work was concerned mainly with the commercially available compounds which had already appeared satisfactory or promising as corrosion inhibitors for radiator anti-freeze compositions.

(4) In the summer of 1944 this laboratory learned of the discovery by the Shell Development Company of California of

the ability of certain crystalline preparations to inhibit the rusting of a steel object when wrapped in paper impregnated with these materials. This kind of rust inhibition occurred through a vapor phase transfer process. Upon request the Shell Company made available a sample of material designated as V.P.I.-220, the main ingredient of which was found to be diisopropylammonium nitrite. A study was made of the possibility of using this compound or one of its homologues as an additive in the Hydrolubes, in the hope of obtaining adequate vapor phase inhibition to the corrosion of steel. This proved so promising when as much as 1% to 2% of the nitrite was used that the diisopropylammonium nitrite (hereafter abbreviated to DIPAN) was adopted as an ingredient in many of the compositions described here. An intensive investigation of a large number of corrosion inhibitors and polymeric additives was then made with DIPAN present, in order to obtain a fluid having adequate inhibition in the aqueous as well as vapor phase. Due to the various interferences found between the additives, this was a laborious and slow process.

25. The use of certain amine salts as liquid phase inhibitors and of DIPAN as a vapor phase inhibitor made it necessary to use a copper deactivator. This led to a study of a variety of known copper deactivators and to the search for new ones having more suitable properties. As the more satisfactory inhibited solutions were found, they were examined in hydraulic pump tests for wear, corrosion, foaming and other properties. The need for better wear preventative properties was apparent at an early date, and many specially prepared additives and rust inhibitors capable of adsorption were used with the thought that thereby the wear would decrease. Also the need for having the Hydrolubes free from tacky properties eventually caused a shift of emphasis away from solutions containing the salts of polyacrylic or polymethacrylic acids to fluids containing the Ucon 75-H polymers as the thickening agent. The very great need for speed of research during the war in an effort to obtain a usable non-flammable hydraulic fluid did not always make for a fundamental or systematic study of corrosion inhibition. For these reasons the numbers of combinations of additives tried with the glycol-water base fluid were very large. This in turn accounts for the large number of tables.

B. Corrosion Test Methods

26. It is not probable that a single laboratory corrosion test can be designed to exactly duplicate what happens to a fluid in hydraulic systems, especially where high speed hydraulic pumps are involved. However, the laboratory

corrosion tests have been very helpful in eliminating a largenumber of potential corrosion inhibitors from consideration, and the results have given a fair idea of the relative amounts of corrosion likely to take place in systems aside from places where lubrication is a factor. Long term, static, storage tests with the actual component parts of the hydraulic system in contact with the Hydrolube are desirable. Such tests are now in progress at the Naval Air Materiel Center at Philadelphia, Pennsylvania.

27. (i). For broad screening purposes a static type of corrosion test was used first. This involved the complete immersion of the polished metal specimens (1" x 3/8" x 1/16") in a given volume of fluid (usually 20 ml.) in such a manner that they remained idle in the fluid but in mettalic contact in the same order as they were likely to be in actual hydraulic systems.

(ii). The second method used was an accelerated type of corrosion test similar to that described by Walker et al. (4). It involved stirring the polished metal specimens at about 1800 r.p.m. in approximately 400 ml. of solution with the metals again in metallic contact with one another. The rectangular metal strips (1" x 3/8" x 1/16" with a 1/4" hole in the center were placed on a 6 mm. O.D. Pyrex glass rod which was flattened at one end. The metal strips were crossed at right angles to one another to serve as blades of a stirrer and were bound securely with a fine nichrome wire (see diagram - Plate 4). This was often used as a quantittive test with the metals being weighed at the beginning and end of each test.

(iii). The third method was a vapor phase corrosion test involving the suspension of a polished rectangular specimen (1 1/2" x 1/2" x 3/32") in a closed vessel directly over a given volume of fluid (usually 10 to 20 ml.). The metal specimen was suspended with its long axis vertical, so that the bottom of the strip was about 1/4" above the liquid level. The vessel containing the specimen was kept in an oven at the desired temperature.

(iiii) The fourth method was a rocking bomb or autoclave test which involved suspension of the metal specimens in contact with one another in a given volume of solution and subjecting the fluid to pressures ranging from 500 to 2,000 p.s.i. and elevated temperatures. The fluid was agitated during the test by rocking the entire apparatus (see Plate 5).

28. The metal specimens used in the static⁽ⁱ⁾ and accelerated corrosion tests⁽ⁱⁱ⁾ were: 52 S0 aluminum, cadmium-plated steel, mild steel, lead bronze, 50-50 lead-tin solder, yellow brass, copper and zinc-plated steel. By placing specimens of the above metals on a glass rod next to one another in the proper order, as described below, metal to metal contacts were established as follows: mild steel against cadmium-plated steel, lead bronze, yellow brass, copper, 50-50 lead-tin solder, 52 S0 aluminum, and zinc-plated steel; and 50-50 lead-tin solder against yellow brass, lead bronze and copper. The metal order used was: aluminum, cadmium-plated steel, bronze, solder, brass, steel, copper, solder, steel, aluminum, zinc-plated steel and steel. These metals and metal-to-metal combinations were chosen primarily because all were reported in military aircraft hydraulic equipment, and secondarily because it was evident that future hydraulic systems were likely to be made of metals and alloys taken from this list. For the vapor phase test⁽ⁱⁱⁱ⁾, mild steel strips were used. For test ⁽ⁱⁱⁱⁱ⁾, the rocking bomb test, the same metals were used as those listed above for tests (i) and (ii).

29. All metals were polished with #100 alumina paper (Behr-Manning Co., Philadelphia, Pa.), were boiled in C.P. benzene, and were dried before using them in the corrosion tests. The pH of each of the fluids tested was determined before and after the corrosion tests. For these measurements a Beckman pH meter (Laboratory Model G) with standard glass and calomel electrodes was used. Where metallic corrosion was measured quantitatively, the specimens were weighed after polishing as described above. At the end of the tests and before final weighings were made, each specimen was carefully cleaned of any loose corrosion products by rubbing it with a soft rubber eraser, was boiled again in C.P. benzene and was dried. From the weight loss and other physical data, the corrosion was calculated and expressed in inches of penetration per year. In calculating the corrosion results, too much emphasis was not placed on the calculated inches of penetration per year, since the assumption of a linear relationship between the time and rate of corrosion was not justified in all cases. Moreover, no attempt was made to quantitatively measure pitting corrosion which nearly always occurred where aluminum was attacked, as well as in some other cases. Other variations in corrosion rates may result from slightly different cleaning techniques of the metal specimens after the corrosion tests, from inevitable variations in the speed of stirring and the degree of aeration of the fluid. Aeration was not excessive in the accelerated corrosion tests since there was only a small clearance between the stirring shaft and the rubber stopper, and it was necessary to seal and lubricate the "bearing surface" occasionally with a drop or two of ethylene glycol.

C. Results of Early Corrosion Tests in the Absence of DIPAN

30. The results of the quantitative accelerated corrosion tests are listed in Tables 5, 6 and 7. In examining these tabulated corrosion data, it must be borne in mind that quantitative accelerated corrosion tests were always preceded by certain preliminary corrosion tests. The static corrosion test was first used in making such preliminary studies. The unsatisfactory additives or additive combinations found in this test were regarded as unworthy of further study as inhibitors for aircraft and ship ordnance hydraulic fluids, but any promising additives found were rerun in the quantitative accelerated tests. As research progressed it was found expedient to discard the static corrosion tests entirely and to use the qualitative accelerated corrosion tests in which some of the metals were weighed, for in some instances corrosion that did not show up in a week or more in the static corrosion tests was found to show up in a day or two when the qualitative accelerated corrosion tests were used. On the basis of corrosion results with this latter test, fluid compositions containing the less promising inhibitors and inhibitor combinations were considered unsatisfactory for use as hydraulic fluids and were therefore not rerun in the quantitative accelerated corrosion tests.

31. For brevity only certain high points in the results obtained with the various corrosion tests are described in this and the next section. Among the general classes of inhibitors studied were borates, alkali metal phosphates, sodium mercaptobenzothiazole, substituted ammonium phosphates, substituted ammonium carboxylates, alkylammonium salts of dicarboxylic acids, alkali metal salts of hydrocarbon sulfonic acids, quaternary ammonium phosphates, potassium fluoride and various combinations of these compounds both with and without the vapor phase inhibitor, DIPAN. In several instances compounds of other types were synthesized and subsequently tested as corrosion inhibitors. Compounds of the various classes which were synthesized for use in the Hydrolubes are listed in Table 8. The corrosion inhibitors were tried in the base stock consisting of ethylene glycol (55% by volume) and water (45% by volume), and in a reference fluid containing either the Ucon or the Sodium polymethacrylate polymers. In the work of Walker et al⁽⁴⁾, compounds such as the borates, the alkali metals phosphates, sodium nitrite, sodiumsilicate, potassium dichromate and several other promising inhibitors were investigated but none were tried in the solutions of later interest at NRL (especially the fluids containing the Ucon polymer or DIPAN). The corrosion tests described in the tables at the end of this report are not a repetition of the work of Walker's laboratory.

Borax (Sodium Perborate)

32. Quantitative accelerated corrosion tests have shown borax to be a fairly satisfactory corrosion inhibitor in glycol-water base mixtures. With 1.0% borax in the stated base fluid, copper, brass, aluminum and steel were protected; cadmium was heavily attacked; and both Pesco bronze and solder were slightly attacked. When the polymer thickener, Carbowax, was used in the base stock containing 1.0% borax, corrosion of zinc was observed in addition to corrosion of cadmium, bronze and solder. It might be stated here that the Carbowax and Ucon polymers, which are polyalkylene glycol derivatives should have no effect upon most inhibitors, for these compounds are not as surface active as the other additive compounds. In the instance just described, and in other instances as well, the corrosion results obtained with the same inhibitor in either Carbowax or Ucon thickened Hydrolubes differed from those obtained when the inhibitor was used in glycol-water base stock. This difference is attributed to the impurities, e.g. aldehydes or other foreign organic constituents in the polymers. Walker⁽⁴⁾ has suggested that higher percentages of borax would probably improve the corrosion inhibition of glycol-water mixtures; however, the fluids inhibited with borax alone would not have vapor phase inhibition. Furthermore, all pump tests made at this laboratory using Carbowax or Ucon-thickened Hydrolubes containing borax have revealed appreciable corrosion and poor lubrication of the pumps. Hence, borax is not considered to be a satisfactory corrosion inhibitor for most types of Hydrolubes.

Borax and Sodium Mercaptobenzothiazole (MBT)

33. It has been known for some time that borax-inhibited Hydrolubes containing 0.1% to 0.2% MBT inhibited corrosion better than did the borax-inhibited Hydrolubes containing no MBT. This inhibitor combination in glycol is standardized for use in the Navy Department Specification 51C39 for an antifreeze composition. It is also the inhibitor combination for glycol in Army Specification 4-1116. Although no bench tests or other mechanical evaluation tests have been made on fluids inhibited with this combination, as indicated earlier, it is very unlikely that this type of fluid would be satisfactory as an aircraft hydraulic fluid. It would not inhibit vapor phase corrosion nor would it perform any better as a pump lubricant than a number of the Hydrolubes inhibited with borax alone.

Alkali Metal Salts of Phosphoric Acid (NaH_2PO_4 , Na_2HPO_4 , K_2HPO_4 , etc.)

34. When slightly less than 0.4% of sodium dihydrogen phosphate was used as a corrosion inhibitor in a Carbowax-thickened Hydrolube, Pesco bronze, solder and copper showed a slight chemical attack. One of the aluminum specimens and the zinc-plated steel specimen, although showing no changes in weight, did show signs of electrolytic corrosion at the points of contact with cadmium-plated steel and steel respectively. Cadmium, as is often the case when salts of phosphoric acid are used, acquired a black, adherent and protective coating. In this quantitative accelerated corrosion test, steel was also protected. Walker et al (4) tried 2.5% dipotassium hydrogen phosphate in 70% glycol-30% water solutions and observed corrosion of brass and copper and protection of steel and aluminum. From these results it appeared doubtful that alkali metal phosphates alone would be satisfactory corrosion inhibitors for the Hydrolubes. Here again there would be no vapor phase corrosion inhibition, and in the liquid phase there would be corrosion of copper, certain brasses and bronzes, and solder, as well as electrolytic corrosion between aluminum and zinc-plated steel.

Borax-Alkali Metal Phosphate-MBT Combinations

35. In quantitative accelerated tests several fluids containing the inhibitor combination (0.3% borax; 0.3% sodium dihydrogen phosphate; 0.1% MBT) adequately inhibited liquid phase corrosion. Aluminum, cadmium-plated steel, mild steel, lead bronze, solder, brass and copper all were protected in glycol-water or Carbowax-thickened glycol-water mixtures. Zinc-plated steel corroded slightly but did not lose weight. When this combination of inhibitors was tried in a Ucon-thickened Hydrolube, the corrosion results were nearly the same, except that lead-bronze and solder were slightly attacked. Lack of vapor phase inhibition, instability of the fluid to light (causing the precipitation of mercaptobenzo-thiazyl disulfide), and poor lubricating qualities again left considerable to be desired in the fluid.

Sodium Nitrite

36. Corrosion tests on Hydrolube compositions inhibited with sodium nitrite alone were not tried at this laboratory. However, in tests by Walker et al, (4) a Hydrolube thickened with polyvinyl alcohol and inhibited with 1.0% sodium nitrite was reported to give heavy corrosion of copper and brass. The only other metals tested were aluminum and steel, and they were not attacked.

Alkali Metal Salts of Aliphatic and Aromatic Hydrocarbon Sulfonic Acids

37. Several sulfonic acid derivatives in concentrations ranging from 0.2% to 2.0% showed some slight promise as inhibitors in glycol-water and Ucon-thickened glycol-water mixtures. When either sodium-2-ethylhexane sulfonate or sodium dodecane sulfonate was used in such fluid compositions, cadmium was corroded, in most instances aluminum was discolored, copper was corroded at its contact with solder and steel was protected. In similar tests with sodium decylbenzene sulfonate (Santomerse D-Monsanto), cadmium was corroded at its contact with aluminum, while copper, solder and steel were protected. Igepon AP Extra, a sulfonated ester, having the formula, $\text{CH}_3-(\text{CH}_2)_7-\text{CH}=\text{CH}-(\text{CH}_2)_7\text{COO}-\text{CH}_2-\text{SO}_3\text{Na}$, appeared to be a fairly good inhibitor in the stated base mixture, but copper was corroded at its points of contact with solder. Of the sulfonic acid derivatives which were tried as corrosion inhibitors, only the sodium 2-ethylhexane sulfonate gave clear solutions. The turbid solutions which resulted when any of the other derivatives were used indicated insufficient solubility of the hydrocarbon sulfonates of higher molecular weight. Furthermore, since these tests were only static corrosion tests of 5 to 7 days duration, it appeared doubtful that any of the sulfonates would be entirely satisfactory as corrosion inhibitors. Apart from use as corrosion inhibitors, the hydrocarbon sulfonates were also of interest as possible copper deactivators and will be discussed again in Section F. As these sulfonates have long aliphatic chains, they were considered as possible anti-wear additives and are discussed as such in the next section. The greater resistance to hydrolysis of the alkali metal salts of these compounds than other soaps would be advantageous in wear preventatives for aqueous fluids.

Substituted Ammonium Phosphates (Amine Phosphates) with and without MBT

38. The amine phosphates have been much used in recent years as aqueous inhibitors. Triethanolammonium phosphate was adopted early in the war as the inhibitor for specification AN-E-2 aircraft antifreeze and engine coolant glycol. When it was soon found that this amine salt was very corrosive to copper and copper containing alloys, the aircraft coolant specification was modified to incorporate the use of a copper deactivator (sodium mercaptobenzothiazole) with the triethanolammonium phosphate. (Specifications AN-E-2b). Walker et al⁽⁴⁾ tried 2.5% triethanol ammonium phosphate and 0.2% MBT in a 30% glycol-70% water solution and found no corrosion of aluminum, mild steel, copper, brass and 50-50 solder. In other tests made by Walker and his

associates, diethylethanolammonium phosphate and triethanolammonium phosphate were both found to corrode copper and copper-containing alloys in the absence of MBT, although such corrosion with the former salt was less than with the latter. No results were made available by these investigators on the use of diethylethanolammonium phosphate with MBT. This combination was examined at this laboratory with a vapor phase inhibitor present, and the results are discussed in paragraph 55. The use of 0.5% of o-hydroxybenzyl-ethanolammonium phosphate and 0.1% MBT in a Ucon-thickened glycol-water base mixture was found to result in a fairly promising fluid. In a quantitative accelerated test, zinc was only slightly corroded, aluminum corroded slightly at its points of contact with cadmium, while solder evidenced a very slight loss in weight but appeared in good condition. Steel, lead-bronze, brass and copper were all well inhibited, and a black, adherent coating formed on cadmium. It is likely that a higher percentage of this amine phosphate might better inhibit corrosion. The many other tests made with the substituted ammonium phosphates involved the addition of DIPAN and are discussed in paragraphs 54 and 55.

Quaternary Ammonium Phosphates

39. A concentration of 0.5% triethylbenzylammonium phosphate in a Ucon-thickened Hydrolube inhibited the corrosion of copper, solder and steel. Both aluminum and cadmium were corroded at their points of contact, and in the case of aluminum there was a slight loss in weight. Lead-bronze was also slightly corroded. When a concentration of 2.5% of this inhibitor was used, the results were not as satisfactory, for copper and lead-bronze were heavily corroded, while solder, steel, cadmium and aluminum were slightly corroded. Turbid fluids resulted in each of these 7-day accelerated tests.

Substituted Ammonium Carboxylates

40. The use of 0.5% diamylammonium caprylate with 0.1% MBT in a Hydrolube thickened with sodium polymethacrylate polymer protected copper, steel and aluminum, although the latter was stained. Cadmium was corroded and solder showed contact corrosion with copper. These results were taken from a 7-day accelerated test. The use of 0.5% diamylammonium caprylate with 0.1% beta-(2-mercaptobenzothiazyl) propionic acid appeared even less promising from the results of a comparable test. Solder was heavily corroded, cadmium was also corroded, and aluminum was pitted. A very slight weight loss was observed with copper, and steel was discolored. The combination of 0.5% diamylammonium caprylate, 0.1% MBT and 2.5% diethylethanolammonium phosphate in an

Acrysol-thickened Hydrolube in a 7-day accelerated test inhibited the corrosion of copper, steel (darkened) and aluminum. Solder was slightly corroded at its contact with copper, and cadmium was corroded. The use of 0.8% diamylammonium laurate, 0.1% MBT and salicylal ethanolamine in Ucon-thickened Hydrolubes better inhibited corrosion than the combinations just mentioned. When a concentration of 0.1% salicylal ethanolamine was used, steel and aluminum were well inhibited, copper and solder were only slightly attacked, and alluminum was corroded. When 0.4% salicylal ethanolamine was tried in a similar test, the corrosion of steel, aluminum and copper was stopped, and the attack on solder was slight; but cadmium was still corroded. The corrosion results obtained with 0.8% diamylammonium laurate, 0.1% MBT and 0.4% o-hydroxybenzyl-ethanolamine (reduced salicylal ethanolamine) were the same as those of the preceding run, except that solder was more heavily corroded. Tests on 0.8% diamylammonium laurate, 0.1% diethylethanol ammonium phosphate and 0.1% MBT showed good protection of copper, solder and steel. Aluminum, though having no loss in weight, was discolored, and cadmium was corroded. All of the corrosion results with diamylammonium laurate combinations in Ucon-thickened Hydrolubes were taken from 7-day accelerated tests.

Potassium Fluoride

41. This compound was used by the Germans as a corrosion inhibitor during World War II in glycerine-water recoil fluids. It was said to be especially effective in protecting the lighter metals (presumably magnesium, aluminum and their alloys). A concentration of 2% potassium fluoride was tried at this laboratory in a Ucon-thickened, glycol-water mixture. In an accelerated corrosion test lasting 10 days, copper, solder, lead-bronze, steel, cadmium and aluminum were protected. Only solder was heavily corroded. Of course, this fluid gave no vapor phase inhibition to steel.

D. Results of Corrosion Tests in the Presence of DIPAN

42. The preceding discussion dealt with inhibitors considered essentially from the standpoint of their effectiveness in liquid-phase corrosion inhibition. Most of them did not afford protection to steel surfaces not immersed in the fluid but exposed to fluid vapors (water vapor particularly). Early in the research on the Hydrolubes when the need for a vapor phase inhibitor became evident, the use of some free amine as a vapor phase inhibitor was considered. Morpholine and a few of the other less volatile amines were tried but were found unsatisfactory for that purpose. Even if such compounds inhibited corrosion in the vapor phase, the amine

concentrations required in the liquid phase so raised the pH that the liquid-phase corrosion of aluminum and some of the other metals became serious.

43. When the first sample of the Shell preparation of DIPAN was received, it was immediately tried in a Carbowax-thickened, borax-inhibited Hydrolube and was found to protect steel specimens suspended immediately above the fluid.⁽⁷⁾ These tests were carried out in ovens at various temperatures. At temperatures of 120°F or below, the vapor-phase protection of steel was complete, but at oven temperatures of 140°F and higher, vapor-phase corrosion began to appear (for details of the test method, see paragraph 27). The DIPAN was identified as diisopropylammonium nitrite. A number of other amine nitrites were also synthesized and studied at NRL⁽⁸⁾, and several others were found to be effective as vapor phase inhibitors. Of the amine nitrites investigated, DIPAN was preferred because of its satisfactory vapor phase inhibition at temperatures up to 120°F, its greater solubility in the Hydrolubes, and its greater vapor pressure assuring vapor phase inhibition for a considerable distance above the liquid level. For the Hydrolube application it was found more suitable to use the pure amine nitrite without the sodium bicarbonate buffer used in impregnating Kraft paper.⁽⁷⁾ A number of liquid-phase corrosion tests carried out in the presence of DIPAN are described below.

DIPAN

44. The use of diisopropylammonium nitrite alone was not satisfactory in inhibiting corrosion in the Hydrolubes. For example, ethylene-glycol base stock containing 2.0% DIPAN caused heavy corrosion of solder, lead bronze and cadmium in a quantitative accelerated corrosion test. Zinc-plated steel was somewhat attacked, brass and copper were tarnished, and aluminum and steel were inhibited. When 1.7% DIPAN (impure) was tried in a Ucon-thickened Hydrolube of the composition indicated in Table VI, bronze, copper, solder, zinc, brass, aluminum and steel were all adequately protected, but cadmium was heavily attacked. The good corrosion results obtained in this accelerated corrosion test were attributed to the presence of impurities present in the early experimental samples of the DIPAN and Ucon polymer. This sample of DIPAN supplied by the Shell Development Company was buffered with approximately 1.0% sodium bicarbonate, and analyses by this laboratory revealed that it contained traces of other inorganic impurities. On the other hand, the Ucon polymer 75-H-54,000 furnished by the Carbide and Carbide Chemicals Corporation contained 0.1% of the sodium salts of phosphoric acid. Subsequent batches of the DIPAN

and Ucon polymer received from the manufacturers were free from the stated inorganic impurities. When a comparable test was made using a Hydrolube containing 1.7% DIPAN (pure) and the Ucon polymer 75-H-69,400 (unneutralized), the corrosion results differed radically from those obtained with the aforementioned fluid. For as shown in Table VII, this latter Ucon-thickened Hydrolube corroded cadmium, bronze, solder, copper and zinc. Brass was tarnished, and again aluminum and steel were protected. Hence a search was made for inhibitors which afforded protection to these metals in the liquid phase, but which at the same time did not interfere with the vapor phase inhibiting action of DIPAN. This involved trying numerous compounds, although almost all of them fall into the two broad classes already discussed.

DIPAN with Borax; DIPAN with Alkali Metal Phosphates;
DIPAN with Sodium Polymethacrylate

45. All of these combinations were tried in quantitative accelerated corrosion tests (Tables V and VI) and were found entirely unsatisfactory as inhibiting combinations. All were less promising than these inhibitors in the absence of DIPAN. The combination of 1.7% DIPAN and 1.0% Borax in a Ucon-thickened Hydrolube protected steel, copper and brass. Aluminum gained slightly in weight and would be amply inhibited. Solder and bronze were slightly corroded, while cadmium and zinc-plated steel were more heavily corroded. When 1.7% DIPAN was tried in a Hydrolube thickened with 5.1% sodium polymethacrylate, only steel and aluminum were inhibited. Copper solder and cadmium were heavily attacked, and zinc was slightly attacked. A Ucon-thickened Hydrolube containing 1.7% DIPAN and 0.4% dihydrogen phosphate only inhibited the corrosion of brass and solder. Steel, lead-bronze, zinc and cadmium were all attacked.

DIPAN with Alkali Metal Salts of Aliphatic Hydrocarbon
Sulfonates

46. These additive combinations were tried only in static corrosion tests of 7 days duration. When 0.5% to 1.0% of Santomerse D (essentially sodium decylbenzene sulfonate) and 2.0% DIPAN were used in Ucon-thickened Hydrolubes, copper, steel, cadmium and aluminum were protected. But solder was attacked, and a heavy precipitate was observed in the test mixtures. Although this precipitation could result from the corrosion products, it was more probably due to the insufficient solubility of the sulfonate. Sodium-2-ethyl-hexane sulfonate in concentrations ranging from 0.1% to 2.0% and 2.0% DIPAN in Ucon-thickened fluids appeared to inhibit the corrosion of copper, solder and steel, but cadmium and aluminum were corroded. The addition of 0.5% to 2.0% Igepon

AP Extra and 2.0% DIPAN to Ucon-thickened, glycol-water mixtures resulted in fluids which corroded copper, solder, steel, cadmium and aluminum.

DIPAN with Carboxylic Acids and Substituted Ammonium Carboxylates

47. The substituted ammonium salts of caprylic, capric and lauric acids were examined for their possible use in the Hydrolubes to inhibit corrosion and at the same time to improve the wear properties of the fluids. A few hydroxy carboxylic acids were examined in Ucon-thickened Hydrolubes containing 2.0% diethylethanolammonium phosphate and 0.1% MBT. Separate tests were made with 0.5% beta-hydroxy pelar-gonic acid, 0.4% myristic alpha-hydroxy acid, 0.4% 9,10 epoxystearic acid and 0.4% 9,10,12 trihydroxy stearic acid in the stated Hydrolube composition. In all of these 7 day static corrosion tests steel, solder and aluminum appeared to be protected. Copper likewise appeared to be protected, except in the test with 9,10 epoxystearic acid. In all cases cadmium was much corroded.

48. The following substituted ammonium laurate salts in the concentrations indicated were each tried in Ucon-thickened base mixtures containing 2.0% DIPAN:

- 1.0% diethylethanolammonium laurate
- 1.0% triethanolammonium laurate
- 1.0% monoisopropanolammonium laurate
- 1.0% diisopropanolammonium laurate
- 1.0% triisopropanolammonium laurate
- 1.0% tripropylammonium laurate
- 1.0% di-n-butylammonium laurate
- 0.5% monoamylammonium laurate
- 1.0% diamylammonium laurate
- 0.25% N-ethylmorpholine laurate
- 0.25% N-phenylmorpholine laurate

In each of these 7 day static corrosion tests, cadmium was attacked and solder was corroded at its contact with copper. In all but a few cases, copper and aluminum were discolored (and probably corroded). However, steel was always protected. Of the substituted ammonium laurates thus examined, 0.5% diamylammonium laurate and 0.25% N-ethylmorpholine laurate appeared to afford the best protection to copper and aluminum in addition to steel. Of the two, diamylammonium laurate was preferred for further research because of its greater solubility and lower freezing point. However, even diamylammonium laurate had too limited a solubility in the Hydrolubes unless it was solubilized as discussed later in paragraphs 67 and 68.

49. If 0.5% of diamylammonium laurate and 2.0% of DIPAN were to be used in a Ucon-thickened Hydrolube, it seemed advisable to use a copper deactivator. It was believed that sodium mercaptobenzothiazole might be satisfactory not only to protect copper but also to diminish the corrosion of alloys containing lead. However, a quantitative corrosion test with 0.1% MBT showed that only copper and steel were protected. Cadmium, lead-bronze, solder, aluminum and zinc were all considerably corroded. Other promising copper deactivators (described later in Section F) were tried but with no better results.

50. It was then decided to try an antioxidant in a Ucon-thickened Hydrolube containing a mixture of diamylammonium laurate, DIPAN and MBT. The antioxidant was intended to improve the stability of the MBT and possibly to help the corrosion inhibition. Here, the results were encouraging. A concentration of 0.1% of an antioxidant N-4-98 (obtained from Mr. Harvey Fife of the Mellon Institute Fellowship of the Carbide and Carbide Chemicals Corporation and claimed to be a stabilizer or antioxidant for MBT) was tried and found to protect copper, steel and aluminum. The attack on solder was very slight, but there was still considerable corrosion of cadmium, lead-bronze, brass and zinc. While the corrosion results from this quantitative accelerated test were an improvement over those of earlier runs, this antioxidant was not considered suitable for further use because of its very limited solubility in the Hydrolube.

51. Other antioxidants were tried including hydroquinone, various amino phenols and salicylaldehyde. None of these appeared particularly promising when used in Ucon-thickened Hydrolubes containing 2.0% DIPAN and 0.1% MBT. When 0.2% hydroquinone was tried in the stated base fluid, only aluminum and copper were protected. Steel was corroded at its contact with cadmium, and both solder and cadmium were heavily attacked. In a comparable 7 day accelerated test with 0.2% p-aminophenol in the base fluid, only aluminum was inhibited. Copper, solder, steel and cadmium were attacked. The same type of test made with 0.2% salicylaldehyde in the base fluid showed corrosion of copper, solder and cadmium. Only steel and aluminum were inhibited.

52. In the search for suitable antioxidants, 0.4% salicylal ethanolamine, which originally had been prepared as a possible copper deactivator, was tried in a quantitative accelerated test. The results with this concentration of salicylal ethanolamine were comparable with those obtained with the Mellon Institute antioxidant, N-4-98. Copper, steel and aluminum were protected; the corrosion of solder was slight; and cadmium, lead-bronze, brass and zinc were corroded.

considerably. Salicylal ethanolamine was much more soluble in the Hydrolube than was antioxidant N-4-98. Furthermore, it had the desirable property of acting as a solubilizer for the diamylammonium laurate. With as much as 0.4% of this compound present in the Hydrolube, nearly 1.0% of the laurate could be added and would not flocculate out, whereas in the absence of the salicylal ethanolamine even 0.5% diamylammonium laurate produced a turbid solution. This is discussed further in paragraph 67.

DIPAN with Substituted Ammonium Dicarboxylates

53. When a concentration of 0.5% of dibutylammonium succinate was tried in a Ucon-thickened Hydrolube inhibited with 2.0% DIPAN and 0.1% MBT, only copper showed protection. In this 7 day accelerated test, solder, steel, cadmium and aluminum were corroded.

DIPAN with Substituted Ammonium Phosphates (Amine Phosphates)

54. Monoisopropanolammonium phosphate, diisopropanolammonium phosphate, triisopropylammonium phosphate and butyldiethanolammonium phosphate were each tried in a concentration of 1.7% in a Ucon-thickened Hydrolube inhibited with 2.0% DIPAN. In these 7 day accelerated corrosion tests, the only test metals were copper and solder, and both of these metals were corroded considerably. In comparable tests, the use of 2.0% diethylethanolammonium phosphate or 0.5% N-ethylmorpholine phosphate gave better protection to the copper and solder. Hence, both of the latter compositions were rerun in quantitative tests (see Table XIV).

DIPAN with Substituted Ammonium Phosphates (Amine Phosphates) and MBT

55. As discussed in paragraph 38, certain of the substituted ammonium phosphates when used with a small percentage (0.1% to 0.2%) of MBT were very good corrosion inhibitors for all the metals under consideration. It was found by this laboratory that the unusual protective power of the amine phosphates was not affected by the presence of as much as 2.0% DIPAN. No higher percentages of DIPAN were tried. These amine phosphates were the only class of inhibitors found that were able to afford protection to all of the metals in the presence or absence of DIPAN. For example, when 1.7% DIPAN, 2.0% diethylethanolammonium phosphate and 0.1% MBT were used in a Ucon-thickened fluid, it was found in a quantitative accelerated test that there was good corrosion protection of aluminum, cadmium, steel, lead-bronze, brass, copper and zinc at temperatures as high as 160°F.

There was vapor phase protection also of steel at temperatures up to 120°F. That DIPAN seems to have no effect on any of the metals tested is probably due to the strong adsorption of the phosphate ion or the amine phosphate molecule on the surface of the various metals. Other satisfactory results with this inhibiting combination were obtained by replacing the MBT with different copper deactivators, and these results are discussed in paragraph 59. A solution of 2.5% triethanolammonium phosphate, 2.0% DIPAN and 0.1% MBT in the Acrysol-thickened fluid protected aluminum, steel, solder, brass, copper and zinc. The corrosion of lead-bronze was slight, but that of cadmium was serious.

E. Vapor Phase Corrosion Inhibition at Higher Temperatures

56. Early experiments demonstrated that all fluids containing DIPAN gave complete vapor phase inhibition at 120°F. But when the temperature rose to 140°F, a slight amount of corrosion appeared on the metal strips, and at 160°F the rate of corrosion became significant. Some combinations of additives with DIPAN in the Hydrolube appeared worse than others. The results of the tests conducted at 160°F are given in Table IX. The type of test used was described earlier in paragraph 27. It was discovered that at least a few compounds in combination with DIPAN gave complete protection at 160°F to metal specimens during the test period. These are listed in tests 6, 8 and 9. Furthermore, this protection with DIPAN and salicylal ethanolamine will continue if a soap, e.g. diamylammonium laurate, is added. (See test No. 9). But the addition of diethylethanolammonium phosphate again produces corrosion at this elevated temperature. It is significant that all the compounds which tend to stabilize the DIPAN and make it useful at higher temperatures have structures like familiar antioxidants. More research is evidently desirable.

F. Copper Deactivators

57. Sodium mercaptobenzothiazole was the first compound suggested and used by Walker et al (4) in the Hydrolubes as a copper deactivator. Although it proved adequate in the laboratory corrosion tests, several difficulties soon arose. It was found that the fluids containing MBT would become turbid, and precipitation eventually occurred after the fluid was exposed to sunlight. This is a photo-chemical reaction in which the MBT is converted into the mercaptobenzothiazyl disulfide, and it is accelerated by ultraviolet light. It is believed that this oxidation reaction takes place in fluids containing MBT after long operation in a hydraulic pump system. It was also believed that an improvement in the Hydrolube composition would result if an effective and more oxidation-stable copper deactivator could be found.

58. The many compounds tried as copper deactivators are listed in Tables X through XIII. These compounds were tried both in the Acrysol-thickened and the Ucon-thickened Hydrolubes with or without other corrosion inhibiting combinations. It will be observed from comparable runs in Tables X and XI that an Acrysol-thickened fluid containing 2.0% DIPAN was very corrosive to copper, while a Ucon-thickened fluid containing 2.0% DIPAN attacked copper much less. Hence the use of a deactivator in either of these fluids was not always sufficient to determine its effectiveness as a copper deactivator. On the other hand, when either the Acrysol-thickened fluid or the Ucon-thickened fluid contained the inhibiting combination of diethylethanolammonium phosphate and DIPAN, in the absence of a copper deactivator, the rates of attack on copper were of the same magnitude. The few compounds protecting copper in both of these polymer-thickened fluids containing the substituted ammonium phosphate-DIPAN combination were considered the most effective copper deactivators. The best fluids from the standpoint of corrosion are the Ucon-thickened Hydrolubes containing diethylethanolammonium phosphate, DIPAN and a copper deactivator.

59. In addition to MBT, the most suitable copper deactivators for this fluid appear to be:

- (a) β -(2-Mercaptobenzothiazyl) propionic acid
- (b) Potassium salt of 2-mercaptothiazoline
- (c) Potassium salt of disalicylal-o-phenylene-diamine
- (d) $\propto$ Benzoin oxime

Of the four compounds listed (a) is considered the best, as it will protect copper as well as MBT. It was suggested to this laboratory by Walker (4) who made available a small sample. It is likely to be more stable toward oxidation than MBT. The compounds (b), (c) and (d) have a limited solubility in the Hydrolube and are not quite as effective as either compound (a) or MBT. However, they are considerably more effective than other compounds tested, and it would be much better to have one of these compounds present than to have no copper deactivator at all.

THE SEARCH FOR WEAR PREVENTATIVES OF THE HYDROPHOBIC TYPE

60. Early hydraulic pump tests at 1,000 and 1,500 p.s.i. revealed that the rates of wear of the gears of the Pesco aviation gear pumps were considerably higher with the Hydrolubes than with the petroleum aircraft hydraulic fluid (specification AN-VV-O-366b). The rates of wear of the bronze bushings were made low when suitable corrosion inhibitors for bronze were used. The better aqueous fluids

such as Hydrolube U and Hydrolube A actually caused less wear to the bronze bushings than the petroleum fluid.

61. Since none of the usual aqueous corrosion inhibiting combinations tried were able to decrease greatly the rates of wear (or spalling) of the gears, research was directed toward improving the boundary lubricating properties of Hydrolube compositions by the addition of one or more compounds having wear reducing properties. The compounds considered for anti-wear additives had to be examined for low temperature solubility in the Hydrolubes and for compatibility with corrosion inhibitors and polymer thickeners. After a number of wear additives were discarded because of their marked corrosive action on the metals known to exist in hydraulic systems, it was realized that some compromise would have to be made between the maximum corrosive inhibition and the maximum wear prevention.

62. The limited, available information on aqueous lubricants necessitated starting this search with a consideration of fundamentals. The types of compounds considered here as the most promising oiliness or wear-reducing agents were the aliphatic compounds containing a carbon chain of at least eight carbon atoms (preferably unbranched) and a hydrophilic group capable of adsorbing on the metal surface. When a monolayer of such a compound is completely adsorbed on a metal surface, the molecules are oriented in such a manner that a compact or closely packed film is formed in which the hydrophilic end of each molecule is in contact with the metal surface and the carbon chain (the hydrophobic portion) extends away from the metal surface. Such long carbon chains extending from the metal surface are flexible and at the same time are able to decrease the probability of metal-to-metal contact. Compounds containing eight (8) or more carbon atoms would not be expected to be particularly soluble in aqueous fluids. Since many of the most suitable compounds with such properties belong to the class known as "colloidal electrolytes", it was recognized that a solution of such substances would have to be examined carefully for any tendency it might have to precipitate or be filtered out while in use in aircraft hydraulic equipment.

63. For the purpose of screening out the more promising wear-additives from the large number of compounds considered, a very simple laboratory test was devised. This test was designed to screen out the less adsorptive compounds. In this test a steel specimen of approximately $3\frac{1}{4}$ " x $\frac{1}{2}$ " x $\frac{1}{16}$ " was polished with #100 alumina paper, boiled in C.P. benzene and dried. A fine stream of freshly distilled water was then run slowly over the strip to ascertain if it was "wetted" or if the water tended to recede and stand in drops on the metal

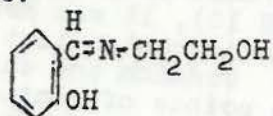
surface. If the latter were the case, it indicated that the specimen was not properly cleaned and the above cleaning operation was repeated until the water wetted the strip. Absolute cleanliness of all apparatus and glassware used had to be observed at all times. The metal specimens were handled with platinum-tipped forceps, previously cleaned by heating in a flame until white hot. Once cleaned, they were not allowed to remain long in the air. After cleaning the metal strip and ascertaining that its surface was free from foreign material, it was immersed in about 20 ml. of the test fluid which was then maintained at either 100° or 140°F. The strip was carefully withdrawn periodically and freshly distilled water was run over it. The hydrophobic nature of the film formed was judged qualitatively by the degree the water "wetted" the strip. The time necessary to immerse the strip in the test solution in order for it to acquire a hydrophobic film varied from a few seconds up to several hours, depending upon the concentration and degree of dispersion of the hydrophobic material and whether or not other polar additives were present in the test solution.

64. The results of the hydrophobic film tests just described are given in Tables XIV, XV and XVI. The higher molecular weight carboxylic acids and especially their metal and amine salts, esters of carboxylic and phosphoric acids, sulfonated esters, long chain alkyl sulfates, alkyl and alkaryl sulfonates, alkylamines, alkyl amides and amine salts of phosphoric acid were among the types of compounds tried as promising anti-wear additives. The amines, carboxylic acids and their esters were found to be practically insoluble in the Hydrolubes if the alkyl chain contained eight or more carbon atoms. Therefore, it was decided to drop them from further consideration, at least until other more readily soluble or dispersible additives had been thoroughly investigated. These included some of the metal and amine salts of the carboxylic acids, the alkyl sulfates, and the alkyl and alkaryl sulfonates as compounds to be considered further.

65. It was decided to examine first the amine salts of the carboxylic acids. Of the carboxylic acids used to make such salts (these included caprylic, capric, lauric, myristic, oleic, ricinoleic and certain hydroxy stearic acids), it was decided that lauric acid was the most suitable. Fluids containing lauric acid (neutralized with an amine) showed considerably less tendency to foam than those containing salts of higher molecular weight acids. Many amines were reacted with lauric acid, and as explained in paragraph 48 diamylammonium laurate was found to be one of the least corrosive of the amine laurates tested in the Ucon-thickened Hydrolubes.

66. In glycol-water mixtures where rapidly adsorbed films were obtained, it was necessary to use such a high concentration of the film-forming compound that turbidity resulted. Nevertheless, several hydraulic gear and piston pump tests were run with turbid, Ucon-thickened Hydrolubes containing substituted ammonium laurate salts in order to determine whether the presence of a hydrophobic film forming additive was beneficial to the lubrication of the pumps. Sufficient improvement resulted in the wear preventative characteristics to encourage further work on the hydrophobic type of anti-wear additive in Hydrolube U. Since the solutions were turbid at the beginning of these tests and showed considerable flocculent material after being run in pumps for 100 hours, it was believed that further improvement might be made if either a more soluble hydrophobic material could be found, or if some small change in the solvent could be made which would increase the solubility or dispersability of the hydrophobic material. After much search it was concluded that no hydrophobic film-forming compound would be found which was sufficiently soluble and stable over the temperature range desired in the ethylene glycol-water base mixture or in the Ucon-thickened glycol-water mixture.

67. Efforts were therefore centered on finding a way to solubilize the hydrophobic additives. Several types of compounds were found effective in solubilizing substituted ammonium carboxylate salts. Salicylaethanolamine (hereafter briefly referred to as SMEA) was found to have some value in this respect. It is a condensation type of compound formed from salicylaldehyde and ethanolamine and has the following structure:



A concentration of 0.4% SMEA was found to solubilize about 0.8% diamylammonium laurate in Ucon-thickened glycol-water mixture at room temperature and still allowed the laurate to adsorb out as a hydrophobic film. Without SMEA in the solution, even 0.2% diamylammonium laurate produced a turbid solution. Hydraulic bench tests were then run on fluids containing the laurate and the solubilizer and a considerable decrease was observed in the pump wear rates. However, the solubilization effect diminished after the fluid had been heated some time at 140°F or higher. As the search was continued for other solubilizers, it was found that certain materials such as butyl carbitol, β -(methoxymethoxy)-ethanol, butyl cellosolve and hexylene glycol were solubilizers for the ammonium laurate. Since these materials had to be added in considerable quantities to solubilize appreciable quanti-

ties of the laurate, it was decided to replace 10% to 15% of the ethylene glycol of the base mixture with one or more of these solubilizers while the water content was maintained the same, to keep the fluid non-flammable.

68. When 12% of the glycol was replaced by a mixture consisting of 10% butyl cellosolve and 2% hexylene glycol (2-methyl,2-4 pentanediol--a new commercial product of the Shell Development Company), it was found that between 1.0% and 2.0% of diamylammonium laurate could be dissolved and would remain so after prolonged heating at 160°F. The hexylene glycol appeared necessary to keep the amine laurate in solution at the lower temperature (-50°F). Since this type of fluid formed good hydrophobic films, several mechanical evaluation tests were made with it. These investigations are not complete as yet, but with certain modifications in the bench tests using the Vickers piston pumps, the wear properties of this fluid have been shown to be superior to those of Hydrolube U.

69. Other classes of hydrophobic materials have been examined in the laboratory, but mechanical tests have not been completed on them. Of the alkyl sulfates, only a few have been studied to date. These included the commercial wetting agents such as "Duponols" and "Tergitols", principally high molecular weight alcohol sulfates neutralized with alkali metal hydroxides. Dupanol C, (sodium lauryl sulfate), was found to give a good hydrophobic film in a Ucon-thickened Hydrolube containing 2.0% DIPAN, if a concentration of 0.5% was used. However, due to the fact that this fluid gelled at -40°F and was fairly alkaline (pH 10), it was not considered satisfactory. Only a static corrosion test lasting three days was run with this fluid. Cadmium and copper were corroded, steel was corroded at the points of contact with cadmium, and a heavy sediment remained in the solution. More work needs to be done on this fluid. None of the other alkali metal-organic sulfates examined gave hydrophobic films.

70. The alkali metal salts of alkane sulfonic acids like the salts of decane, dodecane and tetradecane sulfonic acids did not form hydrophobic films. Sodium decylbenzenesulfonate was found to give a hydrophobic film from its solution in ethylene glycol-water, but no such film was observed from Ucon-thickened base stock. The same was true when the Ucon polymer had been percolated through alumina gel and silica to remove any possible polar impurities. References have been made from time to time on the interference of certain compounds with the formation of hydrophobic films.

71. It will be observed, for example, in Tables XIV through XVI that none of the hydrophobic film-forming additives gave

films with the sodium polymethacrylate polymer present or when a salt of phosphoric acid was present. In fact, as little as 0.01% of diethylethanolammonium phosphate prevented the amine carboxylate salts from forming hydrophobic films. It is suggested that these interferences may be due to the preferential adsorption of the interfering substance on the metallic surface. On the other hand, some additives such as DIPAN may be able to co-adsorb with the wear additive. More research will be necessary in order to fully understand the effects of different additives when used with the hydrophobic film forming materials.

RESULTS OF HYDRAULIC TESTS WITH HYDROLUBES

72. A number of Hydrolube fluid compositions having the most promising properties with respect to inhibition against corrosion, viscosity-temperature characteristics, freedom from foaming, and low freezing and pour points were prepared in batches of five gallons and were subjected to various mechanical tests in the hydraulic systems. These tests included:

- (a) Hydraulic bench tests using Pesco gear pumps.
- (b) Hydraulic bench tests using Vickers piston pumps.
- (c) Other hydraulic tests.

The results of some of the many bench tests are summarized in Tables XVII and XVIII, with the operating conditions for each test listed at the top of each table. Only the high points of these tests and their chemical interpretation will be discussed here. A more complete account and discussion of experimental work on the hydraulic and lubricating properties of the Hydrolubes will be given in a subsequent report.

A. Gear Pump Tests

73. In the first bench tests, Pesco (1P-349N) aviation gear pumps operating at 1800 and 3600 r.p.m. were utilized to circulate fluid continuously through a hydraulic bench test system. The system was constructed where possible with standard AN aircraft parts. A brass reservoir (approx. 1 gal. capacity) and a line-type filter (AN6234-3) having a filter element (AN6235-3), were used in these tests. The hydraulic tubing was usually either 24ST aluminum or 52SO aluminum, although copper tubing was used in a few runs. The fittings were of the standard AN type (24ST aluminum). After the fluid passed through the pump and was compressed to 1000 or 1500 p.s.i., it was passed through a Vickers Model C-167-E relief valve and dropped to 25 p.s.i. It then circulated through a copper coil immersed in a thermostated water bath

(serving as a heat exchanger), through a rotometer (Fisher and Porter type No. 7), and finally flowed back into the reservoir where the fluid pressure was again atmospheric. The entire system usually contained between three and four gallons of fluid. The thermostat controlling the water bath for the heat exchanger was immersed in the fluid in the reservoir and could be set for any temperature desired. This was usually 100°F, 140°F or 180°F. The length of the tests varied from 100 to 500 hours, depending on the program planned. The Pesco gear pump used had two gears and four bushings which were weighed at the beginning of each run after they had been cleaned and dried with a suitable solvent. Similar weighings were made periodically during the run - usually at the end of 100 hour intervals.

74. It was found in all of the early bench tests that the gear teeth had a greater tendency to "spall" or flake off in spots and to wear unevenly than is usually characteristic of gears run with petroleum fluids. This may be caused by intergranular corrosion accelerated by stresses, which occurred underneath the coating of the nitrided steel gear teeth surface. It is more likely due to the inadequate load carrying capacity of the adsorbed film. Related observations are that gear teeth polished with abrasive cloth before use exhibited greatly decreased wear rates, (Walker et al),⁽⁴⁾ and that gears broken in by first running them in a hydraulic pump on petroleum fluid for 50 to 100 hours evidenced much smaller wear rates, (this laboratory). Something analogous to this "break-in" was found to take place when the Hydrolube fluids were run on newly manufactured gears. At any given temperature of operation of the pump tests, the highest rates of wear occurred during the first 50 hours, whereas after the first 100 hours the wear rates became much lower.

75. However, no pump failures ever occurred in any of the bench tests using gear pumps operated at 1000 or 1500 p.s.i. At the end of all the tests the pumps were operating satisfactorily with little loss in the original volumetric efficiency. The wear rates on the bushings were low when the fluids used showed the best corrosion resistance in the stirring tests of paragraph 27. On the other hand, the wear rates of the gears were fairly high even when the fluid temperature was only 100°F. At temperatures of 140°F, the wear rates were further increased, and at 180°F, they became very high. Increasing the pump speeds from 1800 to 3600 r.p.m. increased the wear rates somewhat. When the fluid pressure was increased from 1000 to 1500 p.s.i., the wear rates on the gears also increased. The only wear additive tried that gave appreciable lubrication improvement to the Hydrolubes in the Pesco gear pump runs was

diamylammonium laurate, which was especially effective when used in the presence of salicylaethanolamine, (see Table XVII, Runs 4, 5, 6 and 14.)

B. Piston Pump Tests

76. Numerous hydraulic bench tests have been run on the Hydrolubes utilizing Vickers (Model PF-2713-10) aviation pumps in the place of the Pesco gear pumps. In most of these tests, the remainder of the equipment was the same as that used for gear pump tests as described in paragraph 73. However, in the last runs the brass reservoir and line-type filter were replaced by a combination aluminum reservoir and micron filter, the unit used being a Grumman No. 56208 reservoir from a F8F Navy airplane. Most of the tests were carried out at pressures of 1000 p.s.i., while the pumps were run at 3600 r.p.m. The majority of the tests were made at 100°F, although a few have been run at 140° and 180°F. In nearly all instances the piston pump tests were continued until the pumps failed. In a number of early runs which lasted approximately 100 hours, the breakdown was generally due to a ball bearing failure. Such failures sometimes occurred in the bearing on the drive shaft end of the pump and sometimes in the small bearings supporting the cylinder block.

77. In immersion corrosion tests at 160°F, with ball bearings of a similar type to those used in the Vickers piston pump, evidence of a slight amount of corrosion was found to develop with most of the fluids after 168 hours. Yet the same corrosion tests lasting several months showed very little increase over the corrosion observed in the first week. It is possible that either the ball bearing failures encountered are due to stress corrosion, or that overloading and inadequate load carrying capacity of the adsorbed film are responsible. In order to obtain a truer picture of what goes on, several ball bearing testing machines have been built and put into operation. The details of this and other machines together with the results obtained from them will be described in a forthcoming report. It was found that the piston pump life was increased by changing to the Grumman reservoir mentioned above. With the modifications just described, it was possible to increase the pump life from 100 to 500 hours with Hydrolube U when its temperature was kept at 100°F. If the fluid temperature was raised to 140°F, the pump life fell to about 100 hours again (with the improvements). No comparable tests at 140°F with the old style reservoir and filter were made. Further improvements in the piston pump life were obtained when additional fluid was made to cycle past the ball bearings within the pump at the rate of about 0.3 gallons per minute. Even at 140°F with Hydrolube U, the pump life was extended to 235 hours (the Grumman F8F reservoir

being also used in this test). The best piston pump runs made to date have been with the Ucon-thickened Hydrolubes containing diamylammonium laurate as a wear preventative. In bench tests having the improvements described above (for example in Run 12 of Table XVII, the piston pump was in satisfactory operating condition at the end of 1000 hours, at which time the test was discontinued. This was a five-fold increase in the pump operating time over that obtained using Hydrolube U under similar conditions. Runs 14, 15, 16 and 17 of Table XVII show the effect of higher pressures on piston pump life when the various Hydrolubes are used.

C. Other Hydraulic Tests

78. After the hydraulic bench tests had been completed, long tests were made on the two fluids (Hydrolube U and Hydrolube A) in mock-ups of aircraft hydraulic systems. In these tests a number of standard aircraft hydraulic parts were operated intermittently. The test periods varied from several weeks up to nine months, and the fluid temperatures ranged from -20°F to 140°F . These tests, which were carried out both at this laboratory and at Naval Air Materiel Center in Philadelphia, demonstrated that the Hydrolubes were quite promising for service use in aircraft.

79. Flight tests were then started with Hydrolube A and Hydrolube U. Hydrolube A was tested in the hydraulic system of a Grumman F6F fighter plane and after a four month period and a total of 100 flight hours, all of the hydraulically operated parts were still in satisfactory operating condition. Hydrolube U was then put in the hydraulic systems of 10 new Chance-Vought 4-F-U fighter planes, and AN-VV-O-366b hydraulic fluid was installed in another group of 10 planes. The flight tests to date with these planes have indicated Hydrolube U is also suitable for use in fighter planes. These tests have been going on for about 12 months and are still in progress. A detailed report of all flight tests made with the Hydrolubes, including the problems connected with converting a hydraulic system from petroleum or castor-oil base fluids to Hydrolubes, will be discussed in the forthcoming report already mentioned.

SOME PROPERTIES OF SEVERAL PROMISING HYDROLUBES

A. General Properties and Composition of Hydrolubes

80. From the results already described, it was concluded that two Hydrolube compositions deserved careful testing in aircraft hydraulic systems. The two fluids selected were Hydrolube A and Hydrolube U. For the purpose of comparison, the compositions, pH, viscometric properties, surface tensions, and freezing (or pour) points are given in Table XIX,

along with the corresponding data on the present petroleum aircraft hydraulic fluid used by the Army and Navy. It must be borne in mind that Hydrolube A and Hydrolube U represent only the best compositions to date that have been carried through extensive laboratory and mechanical evaluation tests to flight tests in aircraft. The more recently developed fluids Hydrolube U-2 and Hydrolube U-3 have been tried only in hydraulic gear and piston pump tests and have shown greatly improved lubricating properties but somewhat less desirable corrosion inhibiting properties. The compositions of these fluids are also given in Table 19. In addition to these Hydrolubes described, many other Hydrolubes can be made by varying the additives. For example, other amine phosphates have been shown to be nearly as effective as the triethanolammonium phosphate or the diethylethanolammonium phosphate. Likewise, other copper deactivators such as β (2-mercaptobenzothiazyl) propionic acid and the potassium salt of 2-mercaptothiazoline have been shown to be nearly as good as the sodium mercaptobenzothiazole.

B. Foaming Characteristics of Hydrolubes

81. The foaming characteristics of Hydrolube A and Hydrolube U have been measured and compared with those of the petroleum fluid. The method for making such measurements consisted of passing clean, dry air at a constant rate through a sintered glass disc and into the test fluid. The height to which the foam rose in a definite interval of time and the rate of fall of the foam after turning off the air were noted. From the results of these tests the Hydrolube A fluid showed less tendency to foam than the petroleum fluid, while in contrast the Hydrolube U fluid showed a slightly greater tendency to foam than did the petroleum fluid. However, no objectionable effects have been traced to excessive foaming of either of the Hydrolubes during mock-up or flight tests.

C. Effect of Hydrolubes on Packings

82. The effects of the Hydrolubes on rubber packings have been studied by the High Polymer Section of this laboratory. Three different rubber compositions meeting Army-Navy Specification AN-HH-P-114-B for O-rings and manufactured by the Goodyear Rubber Company, the Arrowhead Rubber Company, and the Linear Packing Company were tested with Hydrolube A, Hydrolube U and the petroleum fluid for changes in tensile strength, elongation, durometer hardness and volume. The results of these tests are tabulated in Table 20. In nearly all of these tests the Hydrolubes proved to be comparable to or better than the petroleum fluid. The Hydrolubes were definitely better with regard to volume change, as both

Hydrolubes caused far less swelling of the various types of rubber than did the petroleum fluid. Hydrolube A was slightly better than Hydrolube U in this respect.

D. Oxidation Stability of the Hydrolubes

83. Only a few oxidation tests were run with the Hydrolubes. These were carried out in a rocking autoclave (American Instrument Company Micro Bomb) at 160°F for 100 hours, under 1000 p.s.i. air pressure. Metal test strips as indicated in Plate 5 were arranged on a glass rod which was sealed into the cap of a glass liner, which in turn fitted inside the bomb. In each of these tests 80 ml. of fluid was used. As the result of the test Hydrolube U showed very little change in pH nor did it become appreciably corrosive to the metals tested. Aluminum, cadmium-plated steel, mild steel, Pesco lead-bronze, copper and yellow brass were amply inhibited, while solder and zinc-plated steel showed almost negligible attack. In a comparative test, Hydrolube U-2 dropped slightly in pH and heavily corroded many of the test metals. Mild steel was the only metal adequately protected; yellow brass and copper showed a limited attack, and the other metals (cadmium-plated steel, zinc-plated steel and solder) were corroded considerably.

E. Effect of Altitude on Hydrolubes

84. In an attempt to determine the effect of altitude on the Hydrolube U fluid, a hydraulic bench test was made in which the reservoir was connected to a vacuum system and operated at a reduced pressure of 20 inches of mercury (corresponding to 10,000 feet altitude). This was the maximum pressure differential (external to internal) at which the Pesco pump would operate. The temperature of the fluid in the reservoir varied from 100° to 160°F. No significant change was observed in the flow rate. The only noticeable effect was a slight hazing of the fluid. This test was confirmed by flight tests which operated satisfactorily to altitudes of 36,000 feet. The details of these tests will be given in the report on wear.

F. Corrosion Inhibition

85. According to the standardized laboratory corrosion test mentioned in Paragraph 26, both Hydrolube A and Hydrolube U are inhibited at temperatures as high as 160°F against the corrosion of the following metals: steel, copper, 52S0 aluminum, lead-bronze (Pesco), tin-bronze (Vickers), yellow brass, 50-50 lead-tin solder, and zinc-plated steel. Whereas Hydrolube U is inhibited against the corrosion of cadmium-plated steel, Hydrolube A will show a very slow rate

of attack. Also the following series of metals in metallic contact will not be corroded when immersed in either of the two fluids at temperatures as high as 160°F: steel against brass, bronze, copper, solder, zinc-plated steel and cadmium-plated steel; solder against bronze, brass and copper; aluminum against cadmium-plated steel. The metallic contacts of aluminum against steel and aluminum against zinc-plated steel will be very slightly corroded when immersed in Hydrolube A, while the same contacts immersed in Hydrolube U will not be corroded.

G. Hydrolube Residues

86. Although the evaporation of water from the Hydrolube fluids within the hydraulic system itself is not considered a problem, there are places such as on the threaded pipe fittings and extended cylinders, where a thin film of fluid may exist and where some evaporation may take place. The nature of the residue resulting from such evaporation is important. In the case of Hydrolube A, a hard, resinous (tacky) residue will eventually result. To date the full effects of this property have not been fully investigated, but it was considered that indicative difficulties were encountered in the Naval Air Materiel Center tests where some threaded fittings previously wetted by the Hydrolube eventually seized. This property would be more objectionable in some types of hydraulic equipment than in others. In contrast, the evaporation of Hydrolube U leaves a non-hardening but viscous liquid which is much less tacky. No objectionable reports about seized or tight fittings have ever been received from mock-up or flight tests with this fluid.

CONCLUSIONS AND RECOMMENDATIONS

87. The most suitable composition for aqueous, non-flammable fluid found to date is a base fluid (55% by volume ethylene glycol and 45% by volume water) to which a special "Ucon" polyalkylene glycol copolymer (Carbide & Carbon Chemical Corporation Ucon-75H) has been added. The Ucon polymer thickens the fluid to the proper viscosity and viscosity-temperature characteristics. The most promising corrosion inhibiting combination was found to consist of diethylethanolammonium phosphate, diisopropylammonium nitrite (for vapor phase inhibition) and sodium mercaptobenzothiazole (for copper protection). Hydrolube U embodies this composition. Another promising fluid, Hydrolube A, was obtained using sodium polymethacrylate as thickener in the place of the Ucon polymer.

11/1/94	100	Forward NTN announced to the public
11/1/94	0.1b	

88. It is concluded that these aqueous hydraulic fluids have shown enough promise in laboratory, mock-up, and flight tests to deserve serious consideration for use in certain types of military aircraft. Hydrolube U has been more extensively tested and appears the more promising.

89. It should be noted that both fluids will give a more limited life in existing Hydraulic pumps than is encountered with the petroleum fluids. However, for planes having hydraulic systems operating at 1500 p.s.i. or lower, the Hydrolubes should be quite satisfactory provided the storage tank temperature does not greatly exceed 140°F. The greater safety from fire hazards and the otherwise good performance characteristics make the Hydrolubes deserving of much more than casual service tests.

90. In order to improve pump life, further research and testing is in progress on wear reducing additives. Research already completed has indicated that wear additives having a hydrophobic type of structure can improve the life of the present hydraulic pumps. Certain modifications in the hydraulic set-up are also recommended for improvement in the life of the Vickers piston pumps where Hydrolubes are used. These would include the utilization of an F-8-F reservoir with an internal micron filter and also the utilization of an extra line to force a steady flow of hydraulic fluid continuously by the ball bearings within the pump.

91. It is recommended that consideration be given the redesign of aircraft hydraulic pumps and to possible metallurgical changes in the existing hydraulic pumps, in order that optimum performance can be obtained with the Hydrolubes. In this connection, it would be advantageous to eliminate overloaded ball bearings and as many as possible of the wide variety of metals now used in the construction of aircraft hydraulic systems and equipment. Special efforts should be made to eliminate cadmium and zinc-plated steel, magnesium, and alloys having an appreciable lead content such as the bronze used in Pesco hydraulic pumps. This would make the use of promising wear reducing agents such as the amine carboxylates much more feasible.

92. Further tests such as long term corrosion tests (especially with component aircraft hydraulic parts), mock-up and flight tests will be recommended as the more promising Hydrolube fluids are developed.

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Appendix A

Synthesis of Compounds for Use in Hydrolubes

A large number of compounds were considered and tried as possible corrosion inhibitors, copper deactivators, solubilizers, antioxidants, etc. for the Hydrolubes. Many of these compounds were not available commercially and were therefore synthesized at this laboratory. The compounds of these various classes synthesized for use in the Hydrolubes are listed in Table VIII.

Among the first compounds prepared at NRL for use in the Hydrolubes were the derivatives of salicylaldehyde. The original interest in such derivatives was due to their prior use as copper deactivators. A compound of that type, disalicylal-o-phenylenediamine, was supplied to this laboratory by Walker et al⁽⁴⁾ and was claimed to give a fairly satisfactory copper deactivation in aqueous solutions. While the results of corrosion tests at this laboratory confirmed their claims concerning the protection of copper, the compound was considered unworthy of further study because of its extreme insolubility in Hydrolube compositions. As attention was focused on the synthesis of salicylaldehyde derivatives which might be more soluble in the Hydrolube, a small sample of disalicylal-ethylenediamine was prepared and tried. But it was also found difficultly soluble in the Hydrolube and did not adequately protect copper. In the search for effective copper deactivators having a higher degree of solubility in the Hydrolubes, other derivatives of salicylaldehyde were made.

The effectiveness of the salicylaldehyde derivatives as metal deactivators has been attributed by many investigators to the ability of these compounds to form a chelate structure when a metal replaces the hydrogen of the phenolic group:



where M is some metal, e.g. copper.

This structure is known to be very stable in the case of certain metallic derivatives. The fact that a large number of compounds are capable of chelation with various metals is the basis for their use as organic reagents in the analyses for these metals.⁽⁹⁾ Other compounds might give derivatives capable of forming a similar type of chelate structure are levulinic acid and acetoacetic acid (ester).

Aside from the derivatives just discussed, alpha benzoin oxime and beta benzoin oxime were synthesized for their possible use as copper deactivators. The other compounds of similar structure tried for their usefulness as copper deactivators were obtained from Eastman Kodak Company.

Appendix B

Preparation of Hydrolube Compositions

The finished Hydrolube fluids were prepared by mixing the weighed quantities of the various constituents, using rapid stirring (1800 r.p.m.). In most instances the polymer thickener was first blended with the ethylene glycol-water base stock. The other ingredients were then added and the resulting solution heated, if necessary, to facilitate the dissolving of certain additives. Such heating was required only when solid materials were in such a physical state that they were not readily wetted by the fluid. When an amine phosphate was used as an inhibitor, the appropriate amine and phosphoric acid were usually added separately, in the proportion of $2\frac{1}{2}$ moles of amine to 1 mole of phosphoric acid. The few primary and secondary amine phosphates listed in Table VIII were synthesized according to the method by Ralston⁽²⁵⁾ and added directly to the Hydrolube fluid. When an amine laurate compound was used as an inhibitor, it was prepared beforehand by the method given in Table VIII and added as such. Whenever DIPAN and/or MBT, or any of its derivatives were used, they were added last to the fluid. For small quantities (less than $\frac{1}{2}$ gal.) filtering was accomplished by using either a medium or coarse fritted glass filter. The filtering of larger quantities of fluid was carried out mechanically by pumping the fluid with a Viking gear pump through an aircraft filter (a Purelator or Skinner type).

Table I

Flammability of Glycols and Aqueous Mixtures

Fluid Composition (% by volume)		Viscosity csks. at 100°F	Flash Points (C.O.C.) °F	S.I.T.	Spray Flamm. (% Oxygen)
% Ethylene glycol	% Water				
100	0	8.7	240	856.	40.
90	10	6.9		862.	
85	15	6.0			
80	20	5.1		871.	48.
75	25	4.4			
70	30	3.8		880.	
65	35	3.2		885.	67.
60	40	2.8		892.	
50	50	2.2		903.	> 80.
40	60	1.6		918.	
30	70	1.3		934.	
20	80	1.1		956.	
% Diethylene glycol					
100	0	16.0	280 (300)	900.	42.
80	20				50.
65	35				> 80.
% Triethylene glycol					
100	0	18.5	330.*	730.	25.
% Tetraethylene glycol					
100	0	22.5		687.	
% Propylene glycol					
100	0	19.6	230 (235)	835.	55.
% Dimethoxy tetraethylene glycol					
100	0	2.5	285.*		12.
55	45				> 80.
% Diethylene glycol monethyl ether					
100	0	2.8	210*	790.	12.
55	45				60.

Table I (Continued)

Flammability of Glycols and Aqueous Mixtures

Fluid Composition (% by volume)			Viscosity csks. at 100°F	Flash Points (C.O.C.) °F	S.I.T. °F	Spray Flamm. (% Oxygen)
% Ethylene glycol	% Glycol	% Water				
monomethyl ether						
100	0	0	1.3	115.*		12.
55	0	45				29.
30	25	45				> 80.
% Ethylene glycol						
monoethyl ether						
100	0	0	2.2 at 68°	135.*		12.
55	0	45				51.
30	25	45				> 80.
% Ethylene glycol						
monobutyl ether						
100	0	0	2.4	165.*		12.
55	0	45				56.
30	25	45				> 80.

* Manufacturers' data
When available fire point is given in parentheses

Table II
Viscosities of Aqueous Base Mixtures

Composition of Base Mixture (% By Volume)		Viscosity csks. @ 100°F	Viscosity csks. @ -40°F
70%	Ethylene glycol, 30% water	3.91	
65%	Ethylene glycol, 35% water	3.6	
60%	Ethylene glycol, 40% water	2.91	162.4
55%	Ethylene glycol, 45% water	2.05	133.7
50%	Ethylene glycol, 50% water	2.25	Frozen
70%	Methyl cellosolve, 30% water	1.92	88.1
60%	Methyl cellosolve, 40% water	2.00	103.0
70%	Cellosolve, 30% water	2.51	-
60%	Cellosolve, 40% water	2.00	253.0
60%	Butyl Cellosolve, 40% water	2.86	Solid
70%	Methyl carbitol, 30% water	3.5	399
60%	Methyl carbitol, 40% water	3.0	310
55%	Methyl carbitol, 45% water	-	Frozen
50%	Carbitol, 50% water	-	-
70%	Carbitol, 30% water	4.0	730
60%	Carbitol, 40% water	3.5	Frozen
70%	B-(Methoxymethoxy) Ethanol, 30% water	2.47	123
60%	B-(Methoxymethoxy) Ethanol, 40% water	2.35	Frozen
70%	Dimethoxy tetraethylene glycol, 30% water	4.08	965
60%	Dimethoxy tetraethylene glycol, 40% water	3.72	953

Table III

Some Physical Constants of Common Freezing Point Depressants *

Compounds	Boiling Point @°C @ 760 mm	Solubility in H ₂ O % by Weight @ 20°C	Viscosity Cskts. @ 20°C	Flash Point °F	Freezing Point @ °C
Methanol	64.5	Complete	0.75	60	-97.8
Ethanol	78.3	"	1.51	70	-114.4
Isopropanol	82.3	"	2.98	70	-85.8
Butanol	117.7	7.7	3.67	115	-89.8
Methyl amyl alcohol (4-methyl pentanol-2)	131.8	1.7	0.81	115	-
2-Ethyl butanol	148.9	0.43	-	135	-
Hexanol-2	157.2	0.58	6.64	165	-51.6
Ethyl ether	34.5	6.9	0.35	-40	-116.2
Isopropyl ether	68.5	0.90	0.47	15	-60.
n-Butyl ether	142.2	0.03	0.90	100	-95.2
n-Hexyl ether	226.2	< 0.01	-	170	-
Ethylene glycol	197.2	Complete	21.0	240	-12.3
Diethylene glycol	245	"	35.7	290	- 6.5
Triethylene glycol	287.4	"	42.7	330	- 9.4
Propylene glycol	188.2	"	56.5	225	-
Dipropylene glycol	231.8	"	104.2	280	-
Hexylene glycol (2-methyl- 2,4-pentanediol)**	199	"	-	210	-
Methyl cellosolve	124.5	"	1.78	115	< -70
Cellosolve	135.1	"	2.20	135	< -70
Diethyl cellosolve	121.4	21.0	0.79	95	-
Butyl cellosolve	171.2	Complete	7.12	240	< -100
Methyl Carbitol	194.2	"	3.79	200	-
Carbitol	201.9	"	4.55	210	< -76
Diethyl carbitol	188.9	"	-	160	-
Butyl carbitol	230.4	"	6.01	240	-
Dimethoxy tetraglycol	275.8	"	4.01	285	-21.4

* From Carbide and Carbon Chemical Corporation's catalogue, twelfth edition (p.8,16,30,36)

** Shell Development Company

Table IV

Viscosities of Polymer-thickened, Glycol-water Mixtures

Weight Percent Polymer in glycol-water* Base Stock	Viscosity in Centistokes				Comments
	130°F	100°F	0°F	-40°F	
1.25% Acrylate polymer ^a G-3360	10.58	15.19	-	937.5	Solution cloudy; polymer partially precipitates on long standing
2.75% Acrylate polymer ^a G-3380	10.01	15.69	183.6	1083	Small amount of β (methoxymethoxy ethanol used to solubilize polymer
3.70% Acrylate polymer ^a G-3381	9.914	15.63	197.4	1230	Solution cloudy; polymer partially precipitates on long standing
0.12% Acrysol G.A. ^b	10.07	15.73	146.7	791	Solution clear
0.29% Acrysol G.S. ^c	9.84	16.19	145.7	793	Solution clear
5.45% Methacrylate polymer ^d G-3667	9.97	15.49	177.9	1125	Solution clear
10.68% Ucon 75-H-69400 ^e	9.94	16.33	219.6	1476	Solution clear

* Volume ratio - Ethylene glycol: water = 55:45

a Polyacrylic acid derivatives prepared specially for NRL by Rohn & Haas Co.

b Trade name for Ammonium polyacrylate polymer manufactured by Rohn & Haas Co.

c Trade name for Sodium polyacrylate polymer manufactured by Rohm & Haas Co.

d Polymethacrylic acid derivative specially prepared for NRL by Rohm & Haas Co.

e Polyalkylene glycol derivative specially prepared for NRL by Carbide & Carbon Chemicals Corp.

Table V
Accelerated Corrosion Tests
160°F ± 2° for 136 Hours
Composition of Base Stock in which Inhibitors and Polymers
were tried:

Ethylene Glycol	Percent by Volume	Percent by Weight
Distilled Water	55.0	57.7
	45.0	42.3

Inhibitors (Percent by Weight in Base Stock Sol.)	Polymer Thickeners (% by wt. in Base Stock Sol.)	pH	Corrosion in Inches Penetration per Year x 10 ³														Final Appearance of Solution	Other Comments
			5280 Aluminum	Cadmium- Plated Steel	Mild Steel	Lead Bronze	50-50 Solder	Yellow Brass	Mild Steel	50-50 Copper	Mild Solder	5280 Aluminum	Zinc- Plated Steel	Mild Steel				
Blank	None	7.4 6.2	0	28	7.4	1.4	0.65	0	12	10.21	0.27	7.6	0	21	7.6			
2.0 BIPAN (a)	None	8.1	0	31	0	3.1	10	0	0	0	9.6	0	41.2	0.60	0		Solder corroded at copper and brass contacts	
0.9 Borax	None	7.5 7.2	0	17	0	4.1	4.1	0	0	0	3.9	0	0	0.12	0			
0.3 Borax 2.0 DIPAN	None	7.3 7.6	47	0	2.5	24	1.3	0	0	1.0	24	0	0	6.8	0			
1.0 Borax 0.35 NaH ₂ PO ₄ 3.8 DIPAN	None	7.5 7.7	0	0	0	3.1	10	0	0	0	14	0	0	0.56	0		Copper dis- colored	
0.3 Borax 0.3 NaH ₂ PO ₄ 2.0 DIPAN	None	7.0 7.3	210	3.3	1.0	0.58	2.8	0	0.78	0.50	3.3	1.0	4.3	1.8	1.7	Flocculant precipitate present	Steel discolored	
0.32 Borax 0.3 NaH ₂ PO ₄ 0.08 MBT	None	7.4 7.5	0	0	0	0	0	0	0	0	0	0	0	0	0	Turbid	Zinc discolored	
Blank	13.3 Ucon-75-H- 54000	7.6 7.4	0	11.6	0	0.90	0	+0.84	2.4	+0.69	0	0	0	8.3	0	Slightly turbid		
Blank	10.2 Ucon-75-H 69400	6.6 6.4	1.6	9.5	2.9	1.9	0.61	0	3.6	0	0.44	3.8	4.5	5.4	3.8	Orange, turbid		
1.0 Borax	13.1 Ucon-75-H- 54000	7.6 7.7	2.3	11	0	3.6	2.0	0	0	0	1.6	0	1.5	0.29	0	Slightly turbid		
1.7 Dicyclohexyl- amine nitrite	12.1 Ucon-75-H- 69400	6.8 8.7	41.5	24	+0.21	1.1	66	0	0	0	73	0	+0.13	1.5	0	White precip- itate present		
0.76 Borax 0.69 NaH ₂ PO ₄ 0.2 MBT	13.1 Ucon-75-H- 54000	7.7 7.7	0	0	4.9	0	0	0	0	0	0	0	0	0.37	0		Aluminum corroded at zinc contact	

Table V (Continued)

Inhibitors (Percent by Weight in Base Stock Sol.)	Polymer Thickeners (% by wt. in Base Stock Sol.)	pH In. Fin.	Corrosion in Inches Penetration per Year x 10 ³										Zinc- Plated		Final Appearance of Solution	Other Comments	
			5280 Aluminum	Cadmium- Plated Steel	Mild Lead Steel	50-50 Bronze	Yellow Brass	Mild Steel	Mild Copper	50-50 Solder	Mild Steel	5280 Aluminum	Mild Steel	Mild Steel			
1.61 DIPAN 0.2 MDT 1.44 Diethylethanol- amine 0.5 Phosphoric acid	10.5 Ucon-75-H 69400	9.1 7.5	0	0	0	0.20	0	0	0	0	0	0	0	+0.47	0	Dark brown, slightly turbid	Steel discolored
Blank	3.9 Methyl-benzyl- ammonium polyacrylate	8.0 8.0	4.8	5.4	0	0	0	+0.19	0	0.26	0	0.30	17	0	0.19		
Blank	5.26 Acrysol G-3667	8.5 8.7	11	3.9	0	0.39	12	0	0	0	9.4	0	3.3	0.47	0	Turbid	
2.0 DIPAN	5.15 Acrysol G-3667	8.3 7.6	0	9.9	0	17	4.8	0.82	0	24	3.7	0	0	0.28	0	Green, pre- cipitate present	
1.7 DIPAN 0.5 Phosphoric acid 1.57 Diethylethanol- amine	13.7 Acrysol G-3596A	8.8 8.2	35	6.3	0	1.9	0	0.50	0	49	0.23	0	12	0	0	Green-brown	
1.7 DIPAN 0.1 MDT 0.5 Diamylammonium caprylate	5.15 Acrysol G-3667	8.3 7.9	0	8.3	0	0.82	3.3	0	0	0	3.4	0	0	0.26	0	Green, turbid	
2.0 DIPAN 0.5 Diamylammonium caprylate 0.1 3 (2-Mercapto- benzothiazyl) phosphoric acid	5.15 Acrysol G-3667	9.1 7.5	0	12	0	0.14	8.0	0.32	0	0.15	7.6	0	0	0.33	0	Yellow, slightly turbid	
1.9 DIPAN 0.1 2-Mercaptothiaso- line 0.32 Sodium bicarbonate 1.0 Borax	0.1 Acrysol G-3359	7.5 7.8	2.1	43	0	2.9	0.72	0	0	0.37	0.56	0	0	3.1	0	Slight precipitate present	
1.8 DIPAN 0.02 MDT 1.9 Triethanolamine 0.45 Phosphoric acid	3.83 Acrysol G-3667	8.0 8.0	0	7.6	0	0.37	0	0	0	0	0	0	0	0	0	Clear	
1.8 DIPAN 0.02 MDT 3.7 Triethanolamine 1.0 Phosphoric acid	5.44 Acrysol G-3667	8.0 7.9	0	7.7	0	0	0	0	0	0	0	0	0	0	0	Deep red	

Table V (Continued)

Inhibitors (Percent by Weight in Base Stock Sol.)	Polymer Thickeners (% by wt. in Base Stock Sol.)	pH In. Fin.	Corrosion in Inches Penetration per Year x 10 ³												Final Appearance of Solution	Other Comments	
			5280 Aluminum	Cadmium- Plated Steel	Mild Steel	Lead Bronze	50-50 Solder	Yellow Brass	Mild Steel	50-50 Copper Solder	Mild Steel	5280 Aluminum	Zinc- Plated Steel	Mild Steel			
1.8 DIPAN	5.5 Acrysol	7.5 7.4	65	8.0	+0.26	0.89	0.37	0	+0.24	0.	0.26	+0.25	25	0	+0.34	Slight precipitate	
0.9 Borax	G-3667																
0.3 Sodium bicarbonate																	
0.09 2-Mercaptothia- zoline																	
Blank	21.0 Carbowax	8.0 8.1	0	2.3	0	0	0	0	0.79	0.24	0	0	0	7.5	0	Clear	Copper discolored
0.9 Borax	18.7 Carbowax	7.9 7.8	0.21	8.0	0	12	1.3	0	0	0	3.1	0	3.7	0.37	0		Steel discolored
2.0 DIPAN	18.0 Carbowax	7.5 7.7	0	14	0	0	0	0	0	0	0	0	0	0	0		Zinc discolored
0.4 NaH ₂ PO ₄	18.6 Carbowax	7.5 7.5	0	0	0	0.38	0	0	0	0.24	0.53	0	0	0	0	Asber, clear	Aluminum corroded with cadmium contact
1.0 Sodium nitrite	18.0 Carbowax	7.5 7.5	0	13	+0.26	2.6	30	0	0	0	21	0	0	1.3	0	Slightly turbid	Steel, copper and brass discolored
1.0 DIPAN																	
0.3 Borax	18.7 Carbowax	8.0 7.7	0	0	0	+0.40	0	0	0	0	0	0	0	0	0		Bronze discolored
0.3 NaH ₂ PO ₄																	
0.07 MBT																	

a Diisopropylammonium nitrite (pure) supplied by Shell Development Co.

b Sodium dihydrogen phosphate

c Sodium mercaptobenzothiazole

In. = Initial

Fin. = Final

Table VI

Accelerated Corrosion Tests
 160°F ± 2° for 336 Hours
 Composition of Reference Fluid in which Inhibitors were tried:
 Base Stock (55% by volume Ethylene Glycol; 45% by volume Water) ----- 87.5
 Polymer Thickener (Ucon 75-H-54,000)^a ----- 10.8
 Vapor Phase Inhibitor -DIPAN- (Diisopropylammonium Nitrite)^b ----- 1.7

Inhibitors (Percent by Weight) in Reference Fluid)	pH	Corrosion in Inches Penetration per Year x 10 ³										Zinc Plated Steel	Final Appearance of Solution	Other Comments
		5280 Aluminum	Cadmium Plated Steel	Mild Steel	Lead Bronze	50-50 Solder	Yellow Brass	Mild Steel	Copper	50-50 Solder	Mild Steel	5280 Aluminum		
Blank	7.6 8.4	0	31	0	0	0	0	0	0	0	0	0	0	
1.0 Borax	7.3	+0.9	36	0	1.7	13	0	0	0	12	0	0	33	0
0.1 MBT ^c	8.1	3.7	0	0	0	0	+0.29	0	0	0	0	0	0	0 Clear
1.0 Borax 0.1 MBT	7.4	0	8.4	0	0.92	0.24	0	0	0	0	0	0	0	0 Turbid
0.4 Sodium Dihydrogen phosphate	8.6	182	3.6	1.3	0.56	0.51	0	0.90	1.4	0	1.5	69	1.00	1.6 Precipitate present
1.0 Triethanolamine 0.3 Phosphoric acid 0.1 MBT	7.9	32	8.1	0.70	0.39	0.75	0.24	0.22	0	3.3	0.57	10	6.3	0.34 Clear

^a Ucon 75-H-54,000 as supplied by Carbide and Carbon Chemical Corp. contained approximately 0.1% Sodium Phosphates

^b DIPAN as supplied by the Shell Development Co. contained 1.0% Sodium Bicarbonate and traces of other inorganic materials

^c MBT (Sodium Mercaptobenzothiazole)

Table VII

Accelerated Corrosion Tests
 160°F ± 2° for 336 Hours
 Composition of Reference Fluid in which Inhibitors were tried:
 Base Stock (55% Ethylene Glycol by volume; 45% water by volume) ----
 Polymer Thickener (Ucon 75-H-69,400)^a -----
 Vapor Phase Inhibitor -DIPAN-(Diisopropylammonium nitrite)^b-----

Percent by Weight
 87.5
 10.8
 1.7

Inhibitors (Percent by Weight in Reference Fluid)	pH	Corrosion in Inches Penetration per Year x 10 ³														Final Appearance of Solution	Other Comments
		In. Fin.	5280 Aluminum	Cadmium Plated Steel	Mild Steel	Lead Bronze	50-50 Solder	Yellow Brass	Mild Steel	Copper Solder	50-50 Solder	Mild Steel	5280 Aluminum	Zinc Plated Steel	Mild Steel		
Blank	7.0 7.9		0	26	0	1.3	86	0	0	0.50	68	0	0	5.1	0	-	Brass discolored
0.22 MBT ^c																	
1.44 Diethylethanolamine	9.0		0	0	0	0.25	0	0	0	0	0	0	0	0	0	Clear	
0.48 Phosphoric acid																	
1.44 Diethylethanolamine																	
0.49 Phosphoric acid	8.9 7.5		0	0	0	1.2	2.1	0.27	0	21	2.0	0	0	0	0	Dark brown, clear	
0.12 MBT																	
0.97 Diisobutylammonium laurate	8.1 7.3		0.11	15	0	2.5	2.9	0.29	0	0.38	2.8	0	2.1	3.2	0	Flocculant Precipitate present	
0.12 MBT																	
0.5 Diamylammonium laurate	8.0 7.0		3.6	12	0	2.8	2.5	0.28	0	0	1.9	0	3.5	4.3	0	Flocculant Precipitate	
1.44 Diethylethanolamine																	
0.48 Phosphoric acid	8.8 7.8		0	0.34	0	0.29	0.40	0	0	19	1.8	0	2.7	3.1	0	Brown	Brass and steel discolored
0.1 Benzothiazole																	
1.39 Diethylethanolamine																	
0.50 Phosphoric acid	10.5 7.2		0	0	0	0	0	0	0	16	0	0	0	0	0		
0.52 Potassium hydroxide																	
0.06 Sodium carbonate	7.7 8.0		+1.2	11	+0.30	1.8	151	+0.22	+0.26	+0.26	133	+0.16	+1.4	0.84	+0.24	Precipitate	
0.06 Sodium carbonate																	
0.02 Trisodium phosphate	7.9 8.9		+0.53	11	0	2.1	8.0	0.16	0	0.18	10	0	0	+0.28	0	Murky orange	
0.025 Trisodium phosphate	8.2 8.2		0	11	0	3.2	20	0	+1.5	0	5.9	0	0	0	0	Precipitate	
0.21 K-GD 1320 ^d																	
0.48 Phosphoric acid	8.4		0	0	0	0.	0.58	0	0	0.23	0.39	0	0	+0.18	0	Deep red, clear	Vickers bronze was used
1.44 Diethylethanolamine																	
0.10 GD 1320 ^e																	
0.02 Potassium hydroxide	8.6 7.2		0	6.0	0	2.5	1.8	1.1	0	0.25	1.3	0	9.9	4.1	0	Flocculant Precipitate	
0.50 Diamylammonium laurate																	

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Table VII (Continued)

Inhibitors (Percent by Weight) in Reference Fluid)	pH In. Fin.	Corrosion in Inches Penetration per Year x 10 ³												Final Appearance of Solution	Other Comments	
		5280 Aluminum	Cadmium Plated Steel	Mild Steel	Lead Bronze	50-50 Solder	Yellow Brass	Mild Steel	Copper	50-50 Solder	Mild Steel	5280 Aluminum	Zinc Plated Steel			Mild Steel
0.10 GD 1499 ^f																
0.50 Diamylammonium laurate	8.0 7.2	0	4.2	0	1.8	2.8	0.18	0	0	2.7	0	3.1	3.5	0	White pre- cipitate present	All metal specimens discolored
0.03 Potassium hydroxide																
0.10 GD 1499																
0.14 Potassium hydroxide																
0.17 Phosphoric acid	9.3 7.1	0	1.4	0	0.54	3.8	0.13	0	0	4.6	0	0	0.14	0	Slightly turbid	Aluminum cor- roded against cadmium-plated steel
0.3 Monoethylamine																
0.10 GD 1320																
0.25 Potassium hydroxide																
0.5 Diamylammonium laurate	8.8 7.7	0	0.54	0	0.53	1.4	0	0	20	3.6	0	0	0.72	0		
0.10 Anti-oxidant N-4-98 ^g																
0.10 Anti-oxidant N-4-98																
0.5 Diamylammonium laurate	7.5 7.4	6.8	10	0	2.3	3.2	1.5	0	0.39	1.9	0	11	3.9	0	Brown Flocculant precipitate	Steel discolored
0.1 Anti-oxidant N-4-98																
0.5 Diamylammonium laurate	8.3 7.8	0	8.2	0	1.9	0.31	1.4	0	0	0.24	0	0	1.6	0		
0.1 MBT																
0.08 Anti-oxidant N-4-98																
1.6 Diethylethanalamine																
0.44 Phosphoric acid	9.1 8.5	0	0	0	0	0	0	0	0	0	0	0	+0.29	0	Orange	
0.1 MBT																
0.1 n-Tetradecylamine																
0.08 n-Tetradecylamine																
1.58 Diethylethanalamine	8.5 8.3	0	0	0	0	0.20	0	0	0	0	0	0	1.2	0	Deep orange	Fluid foams badly
0.44 Phosphoric acid																
0.05 MBT																
1.5 Diethylethanalamine																
0.5 Phosphoric acid																
0.06 Potassium salt of 2-Mercaptothia- zoline	8.5	0	0	0.27	0.38	0.62	0.16	0	0.32	0.39	0	0	1.1	0	Dark brown clear	
0.3 Sodium 2-Ethylhex- ane sulfonate	7.6 7.9	22	6.2	0	1.6	1.3	1.2	0	0	0.78	0	6.1	2.1	0		Copper discolored
0.5 Diamylammonium laurate																

Table VII (Continued)

Inhibitors (Percent by Weight in Reference Fluid)	pH In. Fin.	Corrosion in Inches Penetration per Year x 10 ³										Zinc			Final Appearance of Solution	Other Comments
		5280 Aluminum	Cadmium Plated Steel	Mild Steel	Lead Bronze	50-50 Solder	Yellow Brass	Mild Steel	50-50 Copper	Mild Solder	Steel	5280 Aluminum	Plated Steel	Mild Steel		
0.1 Sodium 2-Ethylhexane sulfonate																
0.5 Phosphoric acid	8.9 7.9	0	0	0	0	0.97	0	0	9.6	1.8	0	0	1.8	0	Very dark	Brass discolored
1.4 Diethylethanolamine																
0.2 Sodium lauryl sulfonate																
1.4 Diethylethanolamine	8.8 7.1	0	0	0	0	1.4	0	0	3.7	1.1	0	0	0	0		Brass discolored
0.5 Phosphoric acid																
0.1 Tergitol 4 h																
1.4 Diethylethanolamine	8.8 7.7	5.7	0	0	0	0.17	0.14	0	13	0.70	0	0	2.4	0		Aluminum pitted
0.5 Phosphoric acid																
0.3 Saccharin	7.1 7.7	1.5	6.7	0	1.4	52	0	0	0.31	55	0	0	6.0	0	Turbid	
0.18 2-Mercaptobenzo- thiazyl- β -prop- ionic acid	9.3 8.6	0	0	0	0.13	0	0	0	0	0	0	0	0	0	Clear	Brass discolored
1.44 Diethylethanolamine																
0.5 Phosphoric acid																
0.36 2-Mercaptobenzo- thiazyl- β -prop- ionic acid	9.2 7.8	0	0	0	0.19	0.64	0	0	0	0.35	0	0	0	0	Clear	
1.44 Diethylethanolamine																
0.5 Phosphoric acid																
0.45 N-Ethylmorpholine																
0.05 Phosphoric acid	7.8 7.7	0	0.51	0	0.29	0.83	0	0	2.6	0.26	0.19	0	1.22	0	Slight white ppt.	
0.2 Salicylal ethanol- amine																
0.8 Diamylammonium laurate	8.0 8.0	0	6.11	0	3.6	5.72	4.4	0	15	3.9	0	0	7.3	0	Very dark	
0.2 Salicylal ethanol- amine																
1.4 Diethylethanolamine	8.8 7.9	0	0	0	0.22	1.0	0.29	0	31	1.3	0	0	0	0	Very dark	
0.5 Phosphoric acid																
0.5 Salicylal ethanolamine																
0.8 Diamylammonium laurate	8.8 7.8	1.1	7.7	0	3.9	2.6	0	0	14	5.1	0	0.46	10	0	Dark, turbid	All metals discolored

Table VII (Continued)

Inhibitors (Percent by Weight in Reference Fluid)	pH In. Fin.	Corrosion in Inches Penetration per Year x 10 ³												Final Appearance of Solution	Other Comments	
		5280 Aluminum	Cadmium Plated Steel	Mild Lead Steel	50-50 Bronze	Yellow Solder	Mild Brass	Mild Steel	50-50 Copper	Mild Solder	5280 Steel	Zinc Plated Aluminum	Mild Steel			
1.0 Salicylal ethanola- mine																
0.8 Diamylammonium laurate	9.0 8.0	9.8	9.1	0	7.0	22	19	0	21	23	0	0	3.6	0	Dark, turbid	
0.4 Salicylal ethanola- mine																
0.8 Diamylammonium laurate	8.6 8.0	0	9.2	0	2.7	0.77	1.0	0	0	0.65	0	0	3.7	0	Flocculant Precipitate	
0.2 MBT																
0.4 Salicylal ethanola- mine	8.3 8.5	0.46	0	0	0	0.19	0	0	0.13	0.18	0	0	0.82	0	Turbid	
0.06 Phosphoric acid																
0.1 MBT																
0.2 Salicylaloxime																
1.4 Diethylethanola- mine	8.9 8.0	0	0	0	1.7	0.38	1.5	0	2.0	0.38	0	0	0	0	Brown, clear	
0.5 Phosphoric acid																
0.14 Salicylal-sec- butylamine																
1.4 Diethylethanolamine	9.1 7.9	0	0	0	0.82	0.66	0	0	23	2.3	0	0	0	0	Dark amber	
0.5 Phosphoric acid																
0.2 Benzoin oxime																
1.4 Diethylethanolamine	8.2 7.7	0	0	0	0.28	0	0	0	0.63	0.52	0	0	0	0	Brown, clear	
0.5 Phosphoric acid																

a Ucon 75-H-69,400 (unneutralized)

b Diisopropylammonium nitrite (pure)

c Sodium Mercaptobenzothiazole

d Potassium salt of disalicylal-o-phenylene Diamine

e Disalicylal-o-phenylene diamine

f Disalicylal-o-phenylene diamine (E.I. DuPont de Nemours)

g An Antioxidant supplied by Carbide and Carbon Chemical

Corporation

h Made by Carbide and Carbon Chemical Corporation

Table VIII
Compounds Synthesized for use in the Hydrolubes

Compound	Method of Preparation ^a	Physical Constants of Compound	Starting Materials and Source
Salicylaethanolamine	10,11	b.p.152-154°C (2mm.)	Salicylaldehyde ^b , m.p.1-2°C Monoethanolamine ^b , m.p.9-10°C
Salicylalisopropanolamine	11	b.p.150-153°C (8 mm.)	Salicylaldehyde ^b , m.p.1-2°C Isopropanolamine ^c , b.p.45°C/5mm.
Salicylaethylamine	11,12	b.p. 92°C(4mm.)	Salicylaldehyde ^b , m.p.1-2°C Ethylamine (Eastman 506-x)
Salicylaldoxime	13	No definite melting point	Salicylaldehyde ^b , m.p.1-2°C Hydroxylamine Hydrochloride ^b
Disalicylaltriureide	14	No definite melting point	Salicylaldehyde ^b , m.p.1-2°C Urea ^d , m.p. 132-134°C
o-Oxybenzalazin	15	m.p. 220-221°C	Salicylaldehyde ^b , m.p.1-2°C Hydrazine sulfate(Eastman 575)
Salicylaethylene-thioamino acid	16	No definite melting point	Salicylaldehyde ^b , m.p.1-2°C Ethylenediamine (Eastman 1915); 95-100% purity
Levulinic acid oxime	17	No definite melting point	Levulinic acid ^b , m.p.28-30.5°C Hydroxylamine hydrochloride m.p. 153-155°C
Levulinic acid semi-carbazon	18	m.p. 94-96°C	Levulinic ^b acid, m.p.28-30.5°C Semicarbazide hydrochloride (Eastman 226)
Levulinic acid phenylhydrazon	19	m.p. 105-107°C	Levulinic acid ^b , m.p.28-30.5°C Phenylhydrazine ^b , m.p.18-19°C
Levulinic acid 2,4 dinitrophenylhydrazon	20	m.p. 206-207°C	Levulinic acid ^b , m.p.28-30.5°C 2,4 Dinitrophenylhydrazine ^b m.p. 199-200°C
2-[2-(1-Carboethoxypropylidene)amino] ethanol	21	m.p. 30°C	Acetoacetic ester ^b , b.p.78-79°C(20mm.) ^b Ethanolamine ^b , m.p. 1-2°C
Bis [4-(2-Oxypentylidene) amino] ethylene	22	m.p. 110-111°C	Acetylacetone ^b , b.p.133-135°C Ethylenediamine (Eastman 1915) 95-100% purity
2-[4-(2-Oxypentylidene) amino] ethanol	23	m.p. 72-73°C	Acetylacetone ^b , b.p.133-135°C Ethanolamine ^b , m.p. 9-10°C
α Benzoin oxime	24	m.p. 150-151°C	Benzoin ^b , m.p. 135-136°C Hydroxylamine hydrochloride ^b
β Benzoin oxime	25,26	m.p.98.5-100.5°C	Benzoin ^b , m.p. 135-136°C Hydroxylamine hydrochloride ^b
1°Dodecylammonium phosphate	27		n-Dodecylamine ^e
2°Dodecylammonium phosphate	27		n-Dodecylamine ^e
1°Tetradecylammonium phosphate	27		n-Tetradecylamine ^e
2°Tetradecylammonium phosphate	27		n-Tetradecylamine ^e

Table VIII (Continued)

Inhibitor *	Reactants and Source
Ethanolammonium laurate	Ethanolamine ^b , m.p. 9-10°C Lauric acid ^d
Diethylethanolammonium laurate	Diethylethanolamine ^c , b.p. 153-165°C Lauric acid ^d
Triethanolammonium laurate	Triethanolamine ^c , 80% purity Lauric acid ^d
Di-n-propylammonium laurate	Di-n-propylamine ^f , b.p. 105-109°C Lauric acid ^d
Tri-n-propylammonium laurate	Tri-n-propylamine ^f Lauric acid ^d
Monoisopropanolammonium laurate	Monoisopropanolamine ^c , b.p. 45°C (5mm.) Lauric acid ^d
Diisopropanolammonium laurate	Diisopropanolamine ^c , b.p. 116°C (5mm.) Lauric acid ^d
Triisopropanolammonium laurate	Triisopropanolamine ^c , Lauric acid ^d
Di-n-butylammonium laurate	Di-n-butylamine ^f Lauric acid ^d
Tri-n-butylammonium laurate	Tri-n-butylamine ^f , b.p. 203-219°C, 99% purity Lauric acid ^d
Diisobutylammonium laurate	Diisobutylamine ^f , b.p. 136-140°C Lauric acid ^d , m.p. 35-39°C
Monoamylammonium laurate	Monoamylamine ^f , b.p. 102-104°C Lauric acid ^d , m.p. 35-39°C
Diamylammonium laurate	Diamylamine ^f , b.p. 175-218°C, 99% purity Lauric acid ^d
Triamylammonium laurate	Triamylamine ^f , b.p. 234-260°C, 99% purity Lauric acid ^d
Morpholine laurate	Morpholine ^c , b.p. 120-132°C, 98% purity Lauric acid ^d
N-Ethylmorpholine laurate	N-Ethylmorpholine ^c , b.p. 138°C Lauric acid ^d
N-Phenylmorpholine laurate	N-Phenylmorpholine ^c , m.p. 57°C Lauric acid ^d
Pyridine laurate	Pyridine ^d , b.p. 112-115°C Lauric acid ^d
Diethylethanolammonium oleate	Diethylethanolamine ^c , b.p. 158-165°C Oleic acid ^g
Diisopropylammonium oleate	Diisopropylamine ^f , b.p. 81-85°C Oleic acid ^g
Diamylammonium oleate	Diamylamine ^f , b.p. 175-218°C Oleic acid ^g

Table VIII (Continued)

Inhibitor *	Reactants and Source
Diethylethanolammonium stearate	Diethylethanolamine ^c , b.p. 158-165° Stearic acid ^d , m.p. 63-68°C
Diisopropylammonium stearate	Diisopropylamine ^f , b.p. 81°-85°C Stearic acid ^d
Diamylammonium stearate	Diamylamine ^f , b.p. 175-218°C Stearic acid ^d
Diethylethanolammonium ricinoleate	Diethylethanolamine ^c Ricinoleic acid ^h
Diisopropylammonium ricinoleate	Diisopropylamine ^f , Ricinoleic acid ^h
Diamylammonium ricinoleate	Diamylamine ^f Ricinoleic acid ^h

* In the preparation of all substituted ammonium carboxylates, stoichiometric quantities of the appropriate amine and carboxylic acid were reacted at 70°C. Since most of the resulting soaps were liquids at room temperature, purification by recrystallization was not attempted.

a Numbers indicate Bibliography references.

b Eastman Kodak Company

c Carbide and Carbon Chemical Corporation

d Eastman practical

e Armour and Company

f Sharples Chemicals

g Eastern Regional Research Lab. O-61-32

h City Chemical Corporation

Table IX

High Temperature Vapor Phase Corrosion Tests on Steel
 (160 F° for 336 hours)
 Composition of reference fluid in which additives were tried:
 10.2% Ucon polymer 75-H-69,400
 89.8% Ethylene glycol-water (55:45 volume ratio)

Test	Additives (% by weight)	Steel Corrosion (in. pen./yr. x 10 ³)	Comments
1	Blank	3.4	Metal corroded and pitted
2	1.7 DIPAN	0.42	Metal slightly corroded on end nearest fluid
3	1.7 DIPAN 2.0 Diethylethanolammonium phosphate	0.64	Metal corroded, especially on part nearest fluid
4	0.1 MBT 1.7 DIPAN 0.8 Diamylammonium laurate	0.90	Metal corroded only on part farthest from fluid
5	0.1 MBT 0.4 Salicylal ethanolamine	1.1	Metal badly corroded
6	1.7 DIPAN 0.1 MBT 0.4 Salicylal ethanolamine	0	Metal bright; no corrosion
7	1.7 DIPAN 0.1 MBT 0.1 o-Hydroxy-benzylethanolamine	0.10	Metal bright, except for two small corroded spots
8	1.7 DIPAN 0.1 MBT 0.1 Antioxidant N-4-98 (Carbide and Carbon)	0	Metal bright; no corrosion
9	1.7 DIPAN 0.1 MBT 0.4 Salicylal ethanolamine 0.8 Diamylammonium laurate	0	Metal bright; no corrosion
10	1.7 DIPAN 0.1 MBT 0.4 Salicylal ethanolamine 2.0 Diethylethanolammonium phosphate	4.4	Metal badly corroded

Table X

Compounds Tried as Copper Deactivators
In
7-Day Accelerated Corrosion Tests at 160°F

Fluid composition in which compounds were tried

37.3% Water
50.7% Ethylene glycol
10.3% Ucon polymer 75-H-69,400
1.7% DIPAN (Diisopropylammonium nitrite)

Compound (% by weight)	Source	pH		Copper Corrosion (in. pen./yr. x 10 ³)	Final Appearance of Solution	Comments
		In.	Fin.			
Blank (a)		7.0	7.9	0.50		
0.1 Benzoin	1*			0	Clear	
0.2 α -Benzoin oxime	1*			0		
0.2 β -Benzoin oxime	1*			0	Turbid	S.C.
0.2 2,4-Diketo quinazalinic acid	1*			0	Clear	
0.2 Disalicylal ethylenediamine	2*			3.5	Precipitate present	
0.2 Disalicylal triureide	1*		7.4	17.0		
0.2 Dithio oxamide	1*			34.0	Deep red	
0.2 Levulinic acid	1*			4.4		S.C.
0.2 1-Naphthylamine-8-sulfonic acid	1*			0	Deep purple	S.C.
0.2 2-Naphthylamine-8-sulfonic acid	1*			0	Light orange	S.C.
0.2 1-Naphthylamine-3,6,8-sulfonic acid	1*			0	Orange	S.C.
0.2 o-Phenylenediamine	1*	7.2	7.7	0	Deep red	S.C.
0.2 m-Phenylenediamine	1*	7.5	8.9	0.17	Deep red	S.C.
0.2 p-Phenylenediamine	1*	7.7	8.0	0.21	Deep red	S.C.
0.2 Pyrogalllic acid	1*	6.7	9.0	0.28	Black	*S.C.
0.2 Quinaldinic acid	1*			0.76	Turbid	S.C.

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Table X (Continued)

Compound (% by weight)	Source	pH		Copper Corrosion (in. pen./yr. x 10 ³)	Final Appearance	Comments
		In.	Fin.			
0.2 Reacetophenone (2,4,Dihydroxy acetophenone)	1*			1.5	Deep red	
0.2 Rhodanine	1			12.0	Clear	
0.2 Salicylal ethylene thiamino acid	L*		7.2	1.8		
0.5 Thiodiethylene glycol (Kromfax solvent)	3*	7.0	7.8	0.17	Precipitate present	
0.2 Benzil monoxime	1*			0		
0.3 Saccharin	1*			stained		*S.C.
0.3 Saccharin	1*	8.45	7.7	bright		*S.C.
0.2 Sodium lauryl sulfonate	1*			O.K.		
0.1 Octyl amine	4*	10.2	9.5	stained		*S.C.
0.2 Octyl amine	4*	11.4	9.6	bright (OK)		*S.C.
0.2 Decylamine	5*	10.1		stained		*S.C.
0.1 Isopropylamine	3*					

- (a) Test lasted 14 days
 L* Laboratory preparation
 1* Eastman Kodak Company
 2* E.I. DuPont de Nemours
 3* Carbide and Carbon Chemical Corporation
 4* Sharples Chemicals
 5* Armour and Company
 *S.C. Solder was corroded in this test

Table XI

Compounds Tried as Copper Deactivators
In
7-Day Accelerated Corrosion Tests at 160°F

Fluid composition in which compounds were tried

39.6% Water

53.9% Ethylene glycol

4.9% Acrysol polymer 3667 (Sodium polymethacrylate)

1.6% DIPAN (Diisopropylammonium nitrite)

Compound (% by weight)	Source	pH		Copper Corrosion (in. pen./yr. $\times 10^3$)	Final Appearance of Solution	Comments
		In.	Fin.			
Blank ^a		8.3	7.6	24.	Dark green	S.C.*
0.2 o-Amino cresol	1 ^b	8.3	7.9	7.7	Dark brown	S.C.
0.2 1-Amino-8-naphthol-3,6 sulfonic acid	1 ^b	8.2	7.6	4.7	Dark purple	S.C.
0.2 Anthranilic acid	1 ^b	8.0	8.0	6.3	Amber	S.C.
0.1 Benzoin	1 ^b			0.97	Clear	
0.2 α -Benzoin oxime	1 ^b			0.15	Turbid	
0.2 β -Benzoin oxime	1 ^b			0.36		
0.2 Cupferon	1 ^b	8.4	7.7	0.67	Brown, turbid	
0.2 Dimethyl glyoxime	1 ^b	8.3	7.9	7.6	Dark brown	
0.2 4,4-Diphenyl semicarbazide	1 ^b	8.1	6.9	1.1	Brown, turbid	
0.2 Dipicryl amine	1 ^b	8.0	7.8	1.1	Dark red	
0.2 Dithio oxime	1 ^b	8.2	7.9	54.	Bright green	
0.5 Thiodiethylene glycol (Kromfax Solvent)	2 ^b	8.2	7.6	28.	Bright green	
0.2 Melamine	1 ^b			2.0	Clear	
0.1 2-Mercapto-4-phenylthiazole	1 ^b			0.26	Turbid	
0.2 β -Naphthyl amine	1 ^b	8.2	8.0	0	Precipitate	All metals
0.2 1-Naphthylamine-8-sulfonic acid	1 ^b			2.7	present	corroded
0.2 2-Naphthylamine-2-sulfonic acid	1 ^b			0.13	Orange	
0.2 Phenolphthalein	1 ^b			1.2	Orange	

Table XI (Continued)

Compound (% by weight)	Source	pH		Copper Corrosion ³ (in. pen./yr.X10 ³)	Final Appearance of Solution	Comments
		In.	Fin.			
0.2 4-Phenyl thiosemicarbazide	1	8.0	7.4	1.5	Brown, turbid	S.C.
0.2 Sodium diethyl-dithio- carbamate	1	8.6	8.0	19.0	Green	S.C.
0.2 Sulfosalicylic acid	1	7.9	7.8	6.7	Green	S.C.
0.2 3-Hydroxy-2-naphthoic acid	1	8.0	7.6	19.	Black	

a Test lasted 14 days

1* Eastman Kodak Company

2* Carbide and Carbon Chemical Company

L* Laboratory preparation

SC Solder corroded

Table XII

Compounds Tried as Copper Deactivators
In7-Day Accelerated Corrosion Tests at 160°F
Fluid compositions in which compounds were tried:36.4% Water
49.7% Ethylene glycol
10.2% Ucon polymer 75-H-69,400
0.54% Phosphoric acid
1.50% Diethylethanolamine
1.66% DIPAN (Diisopropylammonium nitrite)

Compound (% by weight)	Source	pH		Copper Corrosion (in. pen./yr. x 10 ³)	Final Appearance of Solution	Comments
		In.	Fin.			
Blank ^a		8.9	7.5	21.	Clear	
0.2 Sodium mercaptobenzothiazole	2*	9.0		0.0	Black	
0.1 Benzothiazole	1*		7.8	19.	Dark brown	
0.5 Potassium hydroxide	3*	10.5	7.2	16.		
0.2 Potassium salt of disalicylal ethylenediamine	4*		8.4	0.23	Deep red, Clear	
0.1 Potassium salt of disalicylal-o-phenylene diamine	4*	-	8.4	0.23	Deep red, Clear	
0.06 Potassium salt of 2-mercaptobenzothiazoline	4*		8.5	0.32	Deep red, Clear	
0.1 Sodium 2-ethylhexane sulfonate	B*	8.9	7.9	9.6	Dark brown	
0.2 Sodium dodecane sulfonate	1*	8.8	7.1	3.7		
0.1 Tergitol 4	5*	8.8	7.1	13.		
0.2 (2-Mercaptobenzothiazyl) propionic acid	4*	9.3	8.6	0.0	Orange, clear	
0.2 Salicylal ethanolamine	L*	8.8	7.9	31.	Dark amber	
0.2 Salicylal aldexime	L	8.9	8.0	2.0	Dark amber	
0.15 Salicylal mono-sec-butylamine	L*	9.1	7.9	23.	Dark amber	

Table XII (Continued)

Compound (% by weight)	Source	Copper Corrosion		Final Appearance of Solution	Comments
		In.	Fin.	(in. pen/yr. $\times 10^3$)	
0.2 Mixed α and β benzoin oximes	L*	8.2	7.7	0.61	Deep red, clear
0.2 α Benzoin oxime	L*			0.33	
0.1 α Benzoin oxime	L*				
0.1 β Benzoin oxime	L*			0.45	
0.2 β Benzoin oxime	L*			1.8	Dark brown, clear
0.1 — Benzoin	L*			15.	
0.2 Phthalic acid	L*	8.3	7.9	Corroded	Dark brown S.C.
0.2 Taurine	L*	9.0	8.5	33.0	Green S.C.
0.2 Ethylenediamine acetyl acetone	L*	8.8	8.2	34.0	Green
0.5 Ammonium polysulfide	5*	9.3		Corroded	Black
0.5 Ammonium sulfide	5*	9.2		Corroded	Black
0.1 Sodium sulfide	5*	8.1		Corroded	Black
0.1 Duponol C ^b	4*	9.4		Discolored	Precipitate present
0.3 Duponol C ^b	4*	9.5		Corroded	Ppt. present
0.5 Duponol C ^b	4*	9.6		Corroded	Ppt. present
0.1 Santomerse D	6*	9.3		Discolored	Clear S.C.
0.3 Santomerse D	6	9.4		Discolored	Turbid
0.5 Santomerse D	6*	9.5		Discolored	Turbid
0.2 Quinoline ^b	1*	8.8	7.8	Discolored	Dark green
0.2 Salicylal isopropyl-ethanolamine	L*	9.1	7.4	Discolored	Dark amber S.C.
0.2 Potassium salt of salicylal-sec-butylamine	L*		8.1	Discolored	Turbid
0.1 Saccharin ^c	1*	7.8	7.6	Discolored	Dark green
0.3 Saccharin ^b	1*	8.6	8.3	Corroded	Green, turbid
0.5 Saccharin ^d	1*	8.7	8.5	Corroded	Clear
0.1 Thiourea ^d	1*	8.9	8.6	Discolored	Black
0.3 Thiourea ^d	1*	8.8	8.5	Discolored	Black
0.3 Sodium salicylate ^c	1*	8.8		Discolored	Turbid

Table XII (Continued)

L. Laboratory preparation
 B. Supplied by L.W. Beck of Lubrication
 Section of NRL
 1. Eastman Kodak Company
 2. R.T. Vanderbilt Company
 3. J.T. Baker Chemicals
 4. E.I. DuPont de Nemours
 5. Carbide and Carbon Chemical Corporation
 6. Monsanto Chemical Company
 a. Test lasted 14 days
 b. Test lasted 4 days
 c. Test lasted 3 days
 d. Test lasted 1 day
 S.C. Solder corroded

Table XIII

Compounds Tried as Copper Deactivators
In
7-Day Accelerated Corrosion Tests at 160°F
Fluid composition in which compounds were tried:

36.9% Water
50.3% Ethylene glycol
10.7% Ucon polymer 75-H-69,400
1.6% DIPAN (Diispropylammonium nitrite)
0.5% Diamylammonium laurate

Compound (% by weight)	Source	pH		Copper Corrosion (in. pen./yr. x 10 ³)	Final Appearance of Solution	Comments
		In.	Fin.			
0.2 Salicylal aldoxime	L	8.9	7.8	Discolored	Deep amber	S.C.
0.2 Salicylal ethanolamine	L	8.5	7.8	Discolored	Amber, turbid	
0.2 Salicylal isopropanolamine	L	8.2	7.8	Discolored	Precipitate present	
0.3 Sodium 2-ethylhexane sulfonate	B			Corroded	Turbid	S.C.
0.5 Benzoin oxime	L	7.2	7.6	Corroded	Precipitate present	
0.2 Sodium salt of levulinic acid phenylhydrazine	L	7.1	7.9	1.4	Brown, turbid	All other metals corroded

L Laboratory preparation
1 Eastman Kodak
B L.W. Beck of NRL Lubrication Section

Table XIV
Hydrophobic Films

Fluid Composition in which Additives were tried:
42.3% Water
57.7% Ethylene Glycol

Additives (% by Weight)*	Source	Film @ 140°F	Appearance of Solution @ 68°F
0.001 Lauric acid (10 ppm)	1	Good	Clear
0.5 Diamylamine	2	Slight	Clear
0.5 N-Ethylmorpholine	3	Slight	Clear
0.5 Zinc valerate	1	Slight	Turbid
0.5 Diisopropylammonium laurate	L	Good	Turbid
0.5 Mono-1-amy1 triethanolammonium phosphate	L	Slight	Clear
0.1 Decylammonium phosphate	L	Good	Turbid
0.1 Tetradecylammonium phosphate	L	Good	Turbid
0.5 Duponol 80	4	None	Clear
0.5 Duponol ES	4	None	Clear
0.5 Duponol WS	4	None	Clear
0.5 Tergitol 4	3	Slight	Clear
0.5 Tergitol 7	3	Slight	Clear
1.0 Sodium 2-ethylhexane sulfonate	B	None	Clear
0.5 Santomerse D	5	Fair	Clear
0.5 MP 189	4	None	Clear
0.5 Nacconol NR	7	None	Clear
0.5 Nacconol FSNO	7	None	Clear
0.5 Nekal A	6	None	Clear
0.5 Duponol C	4	Good	Clear
0.5 Daxad 23	8	None	Clear
0.5 Santomerse S	5	Good	Clear
0.5 Santomerse 3	5	Good	Clear
0.5 Arctic Syntex Beads	9	Slight	Turbid
0.5 Alkanol B	4	None	Turbid
0.5 Igepon AP Extra	6	Good	Clear
0.5 Sodium lauryl sulfonate	1	None	Clear
0.5 Emulsphor ELA	6	None	Clear
0.5 Opal Wax 10	6	None	Clear
5.5 [Diamylammonium laurate 0.2] [Acrysol 3667 5.3]	L 10	None	Clear
6.3 [Diamylammonium laurate 1.0] [Acrysol 3667 5.3]	L 10	None	Clear
4.8 [Sodium laurate 0.2] [Triethanolammonium phosphate 2.5] [Diisopropylammonium nitrite 2.0] [Sodium mercaptobenzothiazole 0.1]	1 L 11 12	None	Clear

* Multiple additives are indicated by brackets

1	Eastman Kodak Company	8	Dewey and Almy Chemical Company
2	Sharples Chemicals	9	Colgate-Palmolive-Peet Co.
3	Carbide and Carbon Chemical Corp.	10	Rohm and Haas Company
4	E.I. DuPont de Nemours	11	Shell Development Co.
5	Monsanto Chemical Company	12	R.T. Vanderbilt Corp.
6	General Dyestuff Corporation	B	L.W. Beck of NRL Lubrocaton Section
7	National Analine Corporation	L	Laboratory preparation

Table XV

Hydrophobic Films

Fluid Composition in which Additives were tried:

38.7% Water
 51.5% Ethylene Glycol
 10.7% Ucon Polymer 75-H-69,400

Additives (% by Weight)*	Source	Film @ 140°F	Appearance of Solution @ 68°F
0.5 Diisopropylammonium laurate	L	Good	Turbid
1.0 Diisopropylammonium laurate	L	Good	Turbid
1.2 [Diisopropylammonium laurate Diisopropylamine (excess)]	[1.0] [0.2] L	Slight	Clear
0.5 n-Heptylammonium phosphate	L	Slight	Clear
0.3 Dioctylammonium phosphate	L	Good	Turbid
0.4 Dimethyl-laurylammonium phosphate	L	Good	Turbid
0.5 Sodium lauryl sulfonate	1	None	Clear
0.5 Duponol C	2	Good	Clear
1.0 Santomerse D	5	Slight	Turbid
1.0 Igepon AP Extra	6	Slight	Clear
1.2 [Diisopropylammonium laurate Sodium mercaptobenzothiazole]	[1.0] [0.2] 12	Good	Turbid
2.5 [Diisopropylammonium laurate Borax]	[0.5] [2.0] L	Slight	Turbid
3.5 [Diamylammonium laurate Triethanolammonium phosphate]	[1.0] [2.5] L	None	Turbid
3.0 [Diamylammonium laurate Diethylethanolammonium phosphate]	[0.5] [2.5] L	Slight	Turbid

Fluid Composition in which Additives were tried:

37.0% Water
 50.5 Ethylene Glycol
 10.5 Ucon Polymer 75-H-69,400
 2.0 DIPAN

0.1 Octylamide	1	Slight	Clear
0.05 Dodecylamide	1	Slight	Clear
0.03 Tetradecylamide	1	Slight	Clear
0.5 Diethylethanolammonium laurate	L	Slight	Clear
1.0 Diethylethanolammonium laurate	L	Fair	Turbid
2.0 Diethylethanolammonium laurate	L	Good	Turbid
0.5 Triethanolammonium laurate	L	Fair	Turbid
1.0 Dipropylammonium laurate	L	Good	Turbid
1.0 Monoisopropanolammonium laurate	L	Good	Turbid
1.0 Diisopropanolammonium laurate	L	Good	Turbid
1.0 Triisopropanolammonium laurate	L	Good	Turbid
1.0 Tri-n-butylammonium laurate	L	Good	Turbid
1.0 Diisobutylammonium laurate	L	Good	Turbid
0.5 Diamylammonium laurate	L	Fair	Turbid
1.0 Diamylammonium laurate	L	Good	Turbid
0.5 N-Ethylmorpholine laurate	L	Good	Turbid
0.2 N-Phenylmorpholine laurate	L	Good	Turbid
1.0 n-Octylammonium phosphate	L	Slight	Turbid
1.0 Santomerse D	3	Slight	Turbid
1.0 Sodium 2-ethylhexane sulfonate	B	None	Clear
1.0 Barium di-2-ethylhexane sulfonate	B	None	Clear
1.0 Magnesium di-2-ethylhexane sulfonate	B	None	Clear
1.0 Potassium decane sulfonate	B	Slight	Clear
1.0 Magnesium di-n-decane sulfonate	B	Slight	Clear
0.5 Sodium lauryl sulfonate	B	None	Clear
0.5 Duponol C	4	Good	Clear
0.5 Tergitol 4	3	Slight	Turbid
0.5 Igepon AP Extra	6	Slight	Clear
0.4 Batyl alcohol	13	Slight	Clear
0.1 9,10 Dihydroxy decanol	14	Slight	Clear

Table XII (Continued)

- L. Laboratory preparation
- B. Supplied by L.W. Beck of Lubrication
Section of NRL
- 1. Eastman Kodak Company
- 2. R.T. Vanderbilt Company
- 3. J.T. Baker Chemicals
- 4. E.I. DuPont de Nemours
- 5. Carbide and Carbon Chemical Corporation
- 6. Monsanto Chemical Company
- a. Test lasted 14 days
- b. Test lasted 4 days
- c. Test lasted 3 days
- d. Test lasted 1 day
- S.C. Solder corroded

Table XIII

Compounds Tried as Copper Deactivators
In
7-Day Accelerated Corrosion Tests at 160°F
Fluid composition in which compounds were tried:

36.9% Water
50.3% Ethylene glycol
10.7% Ucon polymer 75-H-69,400
1.6% DIPAN (Diisopropylammonium nitrite)
0.5% Diamylammonium laurate

Compound (% by weight)	Source	pH		Copper Corrosion (in. pen./yr. x 10 ³)	Final Appearance of Solution	Comments
		In.	Fin.			
0.2 Salicylal aldoxime	L	8.9	7.8	Discolored	Deep amber	S.C.
0.2 Salicylal ethanolamine	L	8.5	7.8	Discolored	Amber, turbid	
0.2 Salicylal isopropanolamine	L	8.2	7.8	Discolored	Precipitate present	
0.3 Sodium 2-ethylhexane sulfonate	B			Corroded	Turbid	S.C.
0.5 Benzoin oxime	L	7.2	7.6	Corroded	Precipitate present	
0.2 Sodium salt of levulinic acid phenylhydrazine	L	7.1	7.9	1.4	Brown, turbid	All other metals corroded

L Laboratory preparation
l Eastman Kodak
B L.W. Beck of NRL Lubrication Section

Table XV (Continued)

Additives (% by Weight)*		Source	Film @ 140°F	Appearance of Solution @ 68°F	
0.6	Diamylammonium laurate	0.5	L	Good	Turbid
	Potassium salt of disalicylal-o-phenylene diamine	0.1	4		
0.5	Diamylammonium laurate	L	Good	Turbid	
0.5	Sodium-2-ethylhexane sulfonate	B	None	Clear	
0.6	Diamylammonium laurate	0.5	L	Slight	Clear
	Salicylal ethanolamine	0.1			
0.9	Diamylammonium laurate	0.8	L	Good	Clear
	Salicylal ethanolamine	0.1	L		
1.2	Diamylammonium laurate	0.8	L	Good	Clear
	Salicylal ethanolamine	0.4	L		
3.7	Diamylammonium laurate	0.8	L	None	Clear
	Salicylal ethanolamine	0.4	L		
	Diethylethanolammonium phosphate	2.5	L		
3.0	Diamylammonium laurate	0.5	L	None	Turbid
	Diethylethanolammonium phosphate	2.5	L		
1.5	Diamylammonium laurate	0.5	L	Slight	Turbid
	Sodium dihydrogen phosphate	1.0			
0.5	Tetradecylammonium phosphate	L	Good	Turbid	
2.7	Octylamide	0.2	1	Slight	Clear
	Diethylethanolammonium phosphate	2.5	L		
2.6	Dodecylamide	0.1	1	Slight	Clear
	Diethylethanolammonium phosphate	2.5	L		

* Multiple additives are indicated by brackets

13 H.N. Holmes of Oberlin College
 14 Eastern Regional Research Laboratories
 Other sources as indicated in Table XIV

Table XVI
Hydrophobic Films

Fluid Composition in which Additives were tried:

37.0% Water
50.5% Ethylene Glycol
10.4% Ucon Polymer 75-H-69,400
2.0% DIPAN (Diisopropylammonium nitrite)
0.1% MBT (Sodium Mercaptobenzothiazole)

Additives (% by Weight)	Source	Film @ 140°F	Appearance of Solution @ 68°F
0.5 Diethylethanolammonium laurate	L	Fair	Turbid
1.0 Diethylethanolammonium laurate	L	Good	Turbid
1.0 Diisopropylammonium laurate	L	Good	Turbid
0.8 Diamylammonium laurate	L	Good	Turbid
0.3 Diethylethanolammonium oleate	L	Good	Turbid
0.2 Diisopropylammonium ricinoleate	L	Good	Turbid
0.05 n-Dodecylamine	15	Good	Turbid
0.2 1° n-Decylammonium phosphate	L	Slight	Turbid
0.2 1° and 2° n-Decylammonium phosphate salts	L	Slight	Turbid
0.2 1° and 2° n-Tetradecylammonium phosphate salts	L	Good	Turbid
0.1 1° n-Tetradecylammonium phosphate	L	Good	Turbid

Fluid Composition in which Additives were tried:

36.4% Water
49.6% Ethylene Glycol
10.2% Ucon Polymer 75-H-69,400
1.7% DIPAN (Diisopropylammonium nitrite)
2.0% Diethylethanolammonium phosphate
0.1% MBT (Sodium Mercaptobenzothiazole)

0.3 Phosphatidic acid	5	Slight	Clear
0.3 Pyrophosphatidic acid	5	Slight	Clear
0.5 β Hydroxy pelargonic acid	14	Slight	Clear
0.4 α Hydroxy myristic acid	14	Slight	Turbid
0.5 α Hydroxy lauric acid	14	Slight	Turbid
0.05 α Hydroxy palmitic acid	14	Slight	Turbid
0.2 9,10 Dihydroxy stearic acid	14	Slight	Turbid
0.5 9,10,12 Trihydroxy stearic acid	14	Slight	Turbid
0.5 Diisobutylamine	3	Slight	Clear
0.1 Dodecylaminoethanol	16	Slight	Turbid
0.05 β Methyl- β monododecylamine propanol	16	Slight	Turbid
0.3 Laurylamide	1	Slight	Clear
0.3 Calcium butyrate	17	Slight	Turbid
0.3 Barium valerate	17	Slight	Turbid
0.3 Magnesium valerate	17	Slight	Turbid
0.5 Sodium caprylate	1	Slight	Turbid
0.3 Sodium palmitate	1	Slight	Turbid
0.5 Triethanolammonium laurate	L	None	Turbid
0.5 Diisopropylammonium laurate	L	None	Turbid
0.5 Dicyclohexylammonium laurate	L	None	Turbid
0.3 Diethanolammonium oleate	L	Slight	Turbid
0.3 Dicyclohexylammonium stearate	L	None	Turbid
0.1 n-Decylammonium phosphate	L	Good	Turbid
0.2 n-Tetradecylammonium phosphate	L	Good	Turbid
0.5 Tergitol 4	3	Slight	Clear
0.5 Tergitol 7	3	Slight	Clear
0.3 Ethyl methyl-ortho-phosphate	5	Slight	Turbid
0.3 Butyl methyl-ortho-phosphate	5	Slight	Turbid
0.3 Ethyl octyl-ortho-phosphate	5	Slight	Turbid
0.2 Mono-n-propyl calcium ortho-phosphate	5	Slight	Turbid
0.5 Tetrabutyl pyrophosphate	5	Slight	Turbid
0.6 Mono-1-amy1 triethanol ortho-phosphate	5	Slight	Turbid
0.1 9,10 Dihydroxy octadecanol	14	Slight	Turbid
0.03 Batyl alcohol	18	Slight	Clear
0.05 N-n-dodecyl sulfanilamide	18	Slight	Clear
1.6 1° Dodecylammonium phosphate 0.1	L	Good	Turbid
Triethyl phosphate 1.5	5		

15 Armour and Company

16 J.S. Pierce; University of Richmond

17 Eimer and Amend Incorporated

18 Calco Chemical Company

Other sources indicated in Table XIV

Table XVII
Bench Tests Using Vickers PF9-2713-10ZE Piston Pump

Run No.	Fluid Composition	Year Additive	Pressure in psi	Speed of Test in rpm	Temperature in °F	Time of Test in hrs.	Wear	
1	Acrysol G-3596A	13.7%	1000	3600	100	100	Shut down; no pump failure; ball and socket joints in Vickers pump very stiff; considerable insoluble material deposited on pump parts and on filter.	
	Glycol-water ^a	82.4						
	TEAP ^b	2.1						
	DIPAN ^c	1.7						
	MBT ^d	0.1						
pH = 8.8. Viscosity @ 130°F = 10.6 csks.								
2	Ucon 75-H-69400	10.22%	1000	3600	100	96.5	Ball bearings failed; green deposits over inside of pump.	
	Glycol-water	86.05						
	DEEAP ^e	2.04						
	DIPAN	1.65						
	MBT	0.04						
pH = 8.8. Viscosity @ 130°F = 10 csks.								
3	Ucon 75-H-69400	10.22%	1000	3600	100	104.5	Ball bearings failed; green deposits on inside of pump; fluid composition is same as run 2, except for slightly higher content of MBT; drain line is from pump housing to reservoir, instead of through internal relief valve in pump.	
	Glycol-water	86.09						
	DEEAP	2.04						
	DIPAN	1.65						
	MBT	0.10						
pH = 8.8. Viscosity @ 130°F = 10 csks.								
4	Ucon 75-H-69400	10.22%	1000	3600	100	163	Shut down; no pump failure; this represents improvement over run 2.	
	Glycol-water	85.90						
	DEEAP	2.04						
	DIPAN	1.66						
	MBT	0.10						
Antioxidant N-4-98 ^f 0.08								
pH = 8.8 Viscosity @ 130°F = 10 csks.								
5	Ucon 75-H-69400	10.22%	1000	3600	100	182	Ball bearings failed; black deposits on inside of pump after 50 hrs.	
	Glycol-water	86.09						
	DEEAP	2.04						
	DIPAN	1.65						
	K salt of G-1499 ^g	0.10						
pH = 8.8 Viscosity @ 130°F = 10 csks.								
6	Ucon 75-H-69400	10.7%	1000	3600	100	100	Ball bearings failed; purerator filter element attacked somewhat by fluid; fluid turbid at end of run.	
	Glycol-water	86.4						
	DIPAN	1.6						0.8% Diamylammonium laurate
	MBT	0.1						
	Salicylal ethanolamine	0.4						
	Diamylammonium laurate	0.8						
	pH = 8.6 Viscosity @ 130°F = 10.5 csks.							
					180	100		
					76			
					276 Total			

Table XVII (Continued)

Run No.	Fluid Composition	Wear Additive	Pressure in psi	Speed of Test in rpm	Temperature in °F	Time of Test in hrs.	Wear	
7	Acrysol G-3667							
	Glycol-water							
	DIPAN	0.33% Diamylammonium caprylate	1000	3600	100	100	Ball bearings failed. Considerable insoluble material deposited on pump parts and filter. Fluid showed considerable tendency to foam.	
	MBT	late			140	152		
	Diamylammonium caprylate	0.17% Diamylammonium laurate				252		
	Diamylammonium laurate					Total		
pH = 8.0 Viscosity @ 130°F = 9.97 csks.								
8	Ucon 75-H-69400							
	Glycol-water							
	DEEAP	None	1000	3600	100	541	Ball bearings failed. Considerable insoluble material (green) on pump parts and filter. F8F reservoir (with internal filter) used; as well as drain line from pump housing back to reservoir. (A repeat run of 448 hrs. @ 100°F was made. Disassembly showed thrust bearing to be failing.)	
	DIPAN							
	MBT							
	pH = 8.8 Viscosity @ 130°F = 10 csks.							
9	Ucon 75-H-69400							
	Glycol-water							
	DIPAN	0.8% Diamylammonium laurate	1000	3600	100	648	Ball bearings failed. F8F reservoir (with internal filter) used, as well as drain line from pump housing back to reservoir. Otherwise, all conditions were same as in run 6. Fluid turbid at end of run.	
	MBT							
	Salicylal ethanolamine							
	Diamylammonium laurate							
	pH = 8.6 Viscosity @ 130°F = 10.5 csks.							
10	Same as Run 6	Same as Run 6	1000	3600	140	147	Shut down. Pump had not failed. F8F reservoir and drain line used (Same as in runs 8 and 9). Thrust bearings were beginning to fail. Fluid had waxy, insoluble material floating on surface at end of run.	
11	Same as Run 8	None	1000	3600	140	96	Shut down. Pump had not failed. F8F reservoir and drain line used (same as in Runs 8, 9, 10). Thrust bearings were beginning to fail. Considerable insoluble material (green) on pump parts and filter.	
	12	Ucon 75-H-69400						
		Water						
Glycol								
Butylcellosolve								
2-Methyl-2,4 pentanediol								
DIPAN								
MBT								
	Diamylammonium laurate							
Viscosity @ 130°F = 10.7 csks.								

Table XVII (Continued)

Run No.	Fluid Composition	Wear Additive	Pressure in psi	Speed of Test in rpm	Temperature in °F	Time of Test in hrs.	Wear
13.	Same as Run 3 (specially filtered) through fine filter)	None	1000	3600	140	235	Pump shut down; ball bearings beginning to fail; P8F reservoir employed; fluid circulated through bearing case at rate of .33 gal/min.
14.	Same as Run 3 (Filtered through coarse filter)	None	1500	3600	140	137 3/4	Pump shut down; ball bearings beginning to fail; P8F reservoir employed; fluid circulated through bearing case at rate of .32 gal/min.
15.	Same as Run 12	Diamylammonium laurate	1500	3600	140	382	Pump shut down; ball bearings beginning to fail; P8F reservoir employed; fluid circulated through bearing case at rate of .33 gal/min.
16.	Ucon 75-H-69400 10.0% Water 36.9 Glycol 35.2 Butyl cellosolve 7.5 2-Ethyl 2,4 pentanediol 7.5 DIPAN 1.7 MBT 0.2 Diamylammonium laurate 1.0 pH = 8.1 Viscosity @ 130° - 10 csks.	Diamylammonium laurate	1500	3600	140	64.6	Pump shut down; ball bearings beginning to fail; no modifications on pump.
17.	Same as Run 13	None	3000	3600	140	15.1	Pump shut down; ball bearings beginning to fail; P8F reservoir employed; fluid circulated through bearing case at rate of .23 gal/min.

a Glycol-water in volume ratio of 55:45

b Triethylethanolammonium phosphate

c Diisopropylammonium nitrite

d Sodium mercaptobenzothiazole

e Diethylethanolammonium phosphate

f Antioxidant supplied by Carbide and Carbon Chemical Corp.

g Potassium salt of disalicylal-o-phenylenediamine

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Table XVIII
Bench Tests Using 1P349N Pasco Gear Pumps

Run No.	Fluid Composition	Wear Additive	Pressure in psi	Speed of Test in rpm	Temperature in °F	Time of Test in hrs.	Wear
1.	Carbowax 6000 Glycol-water ^a Borax	None	1000	3600	100	50	Gear weight losses averaged 100 mgs/50 hrs.; bushing weight losses averaged 100 mgs/50 hrs.; general appearance of gears and bushings very bad.
	pH = 7.5 Viscosity @ 100°F = 27 csks.						
2.	Ucon 75-H-69400 Glycol-water ^b DEKAP ^c MBT ^c DIPAN ^d	None	1000	1800	100 140 Total	300 200 500	Gear weight losses averaged 30 mgs/100 hrs.; (40 mgs 1st 100 hrs.); bushing weight losses averaged 1.5 mgs/100 hrs. (3 mgs. 1st 100 hrs.); no appreciable change in wear rates when temperature rose from 100° to 140°.
	pH = 8.8 Viscosity @ 130°F = 10 csks.						
3.	Same as Run 2	0.1% Tetradecylamine	1000	1800	100 140 Total	300 200 500	Conditions of test same as Run 2; wear rates showed no improvement over Run 2.
4.	Ucon 75-H-69400 Glycol-water ^b MBT DIPAN Diamylammonium laurate	0.5% Diamylammonium laurate	1000	1800	100 140 180 Total	200 100 100 400	Gear weight losses averaged 18 mgs/100 hrs. (50 mg 1st 100 hrs.); bushing weight losses averaged 4 mgs/100 hrs. (4 mg 1st 100 hrs.) Weight losses extremely low after 1st 100 hrs., even at 180°.
	pH = 8.0 Viscosity @ 130°F = 10.6 csks.						
5.	Same as Run 4	Same as Run 4	1000	3600	100	100	Gear weight losses averaged 60 mgs/100 hrs. (1st 100 hrs.); bushing weight losses averaged 10 mgs/100 hrs. (1st 100 hrs.)
6.	Ucon 75-H-69400 Glycol-water ^b MBT DIPAN Salicylal-ethanolamine Diamylammonium laurate	0.8% Diamylammonium laurate	1000	3600	100	100	Gear weight losses averaged 15 mgs/100 hrs. (1st 100 hrs.); bushing weight losses averaged 10 mgs/100 hrs. (1st 100 hrs.) These weight losses were lowest of any fluid tried for 1st 100 hr. period, and both gears and bushings appeared to have the least spalling of any run.
	pH = 8.6 Viscosity @ 130°F = 10.5 csks.						
7.	Acrysol O-3493-B Glycol-water ^b TEAP ^e MBT DIPAN	None	1000	1800	100	400	Gear weight losses averaged 50 mgs/100 hrs. (70 mgs. 1st 100 hrs.); bushing weight losses averaged 2 mgs/100 hrs. (5 mgs. 1st 100 hrs.); viscosity dropped considerably.
	pH = 8.0 Viscosity @ 130°F = 15 csks.						

Table XVIII (Continued)

Run No.	Fluid Composition(% by weight)	Wear Additive	Pressure in psi	Speed of Test in rpm	Temperature in °F	Time of Test in hrs.	Wear
8.	Acrysol G-3493-B Glycol-water TEAP MBT DIPAN	5.16 88.08 4.69 0.19 1.88	None	1000	1800	100 ----- 300 140 ----- 100 180 ----- 50 Total 450	Since broken-in gears were used, weight losses were low, except at 180°F; gear weight losses averaged 165 mgs./50 hrs. at 180°; viscosity dropped 2.5% during 1st 100 hrs.
pH = 8.1 Viscosity @ 130°F = 9.61 csks.							
9.	Same as Run 8	0.03% Dibutyl-amine 0.03% Diamyl-amine 0.03% Dioctyl-amine	1000 1500	3600	100 ----- 140 ----- Total 500	200 100 200 500	Gear weight losses averaged 50/mg/ 1st 100 hrs. When pressure was raised to 1500 psi, gear weight losses averaged 100 mg./ 100 hrs.@100°; at 1500 psi and 140°, gear weight losses averaged 280 mg./100 hrs.; bushing weight losses averaged 10 mg./100 hrs. Viscosity dropped.
10.	Acrysol G-3667 Glycol-water MBT DIPAN	5.2 92.95 0.10 1.75	0.5% Diamyl-ammonium caprylate	1000	3600	100 100	Gear losses in weight averaged 20 mgs/1st 100 hrs.; bushing weight losses averaged 6 mgs/1st 100 hrs.
pH = 8.0 Viscosity @ 130°F = 10 csks.							
11.	Same as Run 2 (Filtered through fine filter)	None	1000	3600	140	100	Gear weight losses averaged 12 mgs/100 hrs.; Bushing weight losses averaged 4 mgs/100 hrs.
12.	Same as Run 2 (Filtered through fine filter)	None	1000	3600	100	100	Gear weight losses averaged 7 mgs/100 hrs.; Bushing weight losses averaged 2.6 mgs/100 hrs.
13.	Same as Run 2 (Filtered through fine filter)	None	1000	3600	100	100	Gear weight losses averaged 7 mgs/100 hrs.; bushing weight losses averaged 2.5 mgs./100 hrs.; F8F reservoir employed.
14.	Ucon 75-H-69400 Water Glycol Butyl cellosolve 2-Methyl-2,4-pentanediol DIPAN MBT Diamylammonium laurate	10.0 36.9 38.2 10.0 2.0 1.7 0.2 1.0	Diamylammonium laurate	1000	3600	100 100	Gear weight losses averaged 6.3 mgs/100 hrs.; bushing weight losses averaged 16 mgs/100 hrs.; F8F reservoir employed.
pH = 8.1 Viscosity @ 130°F = 10 csks.							
15.	Same as Run 14	Diamylammonium laurate	1000	3600	100	100	Gear weight losses averaged 10 mgs/100 hrs. bushing weight losses averaged 21 mgs/100 hrs.; F8F reservoir employed.
a Glycol-water in volume ratio of 55:45 b Diethylethanolammonium phosphate c Sodium mercaptobenzothiazole d Diisopropylammonium nitrite e Triethylethanolammonium phosphate							

a Glycol-water in volume ratio of 55:45
b Diethylethanolammonium phosphate

c Sodium mercaptobenzothiazole
d Diisopropylammonium nitrite

e Triethylethanolammonium phosphate

Table XIX
Composition and Properties of 5 Aircraft Hydraulic Fluids

Hydrolube AComposition:

4.8% Sodium polymethacrylate
polymer G-3493-B
52.6 Ethylene glycol
38.8 Water
1.6 Triethanolamine
0.4 Phosphoric acid
1.6 DIPAN
0.2 MBT

<u>Temperature</u> <u>in °F</u>	<u>Viscosity</u> <u>in csk.</u>
130	9.6
100	15
0	180
-40	1260

Pour point: below -60°F
Surface tension: 48.2 dynes/cm.

Hydrolube U-2Composition

10.7% Ucon polymer 75-H-69,400
49.9 Ethylene glycol
36.5 Water
0.4 Salicylal ethanolamine
0.8 Diamylammonium laurate
1.6 DIPAN
0.1 MBT

<u>Temperature</u> <u>in °F</u>	<u>Viscosity</u> <u>in csk.</u>
130	10
-40	1563

Pour point: below -40°F
Surface tension: 30.6 dynes/cm.

Hydrolube UComposition:

10.2% Ucon polymer 75-H-69,400
49.7 Ethylene glycol
36.4 Water
1.5 Diethylethanolamine
0.5 Phosphoric acid
1.6 DIPAN
0.1 MBT

<u>Temperature</u> <u>in °F</u>	<u>Viscosity</u> <u>in csk.</u>
130	10
100	16
0	240
-40	1760

Pour point: below -50°F
Surface tension: 48.1 dynes/cm.

Hydrolube U-3Composition

10.0% Ucon polymer 75-H-69,400
38.2 Ethylene glycol
36.9 Water
10.0 Butyl cellosolve
2.0 2-Methyl -2,4-pentanediol
1.0 Diamylammonium laurate
1.7 DIPAN
0.2 MBT

<u>Temperature</u> <u>in °F</u>	<u>Viscosity</u> <u>in csk.</u>
130	10.1
-40	2310

Pour point: below -50°F

AN-VV-O-366bComposition

See Army-Navy Aeronautical
Specification Oil, Petroleum
Base (AN-VV-O-366b)

<u>Viscosity</u> <u>in csk.</u>	<u>Temperature</u> <u>in °F</u>
10	130
--	100
--	0
500 (max.)	-40

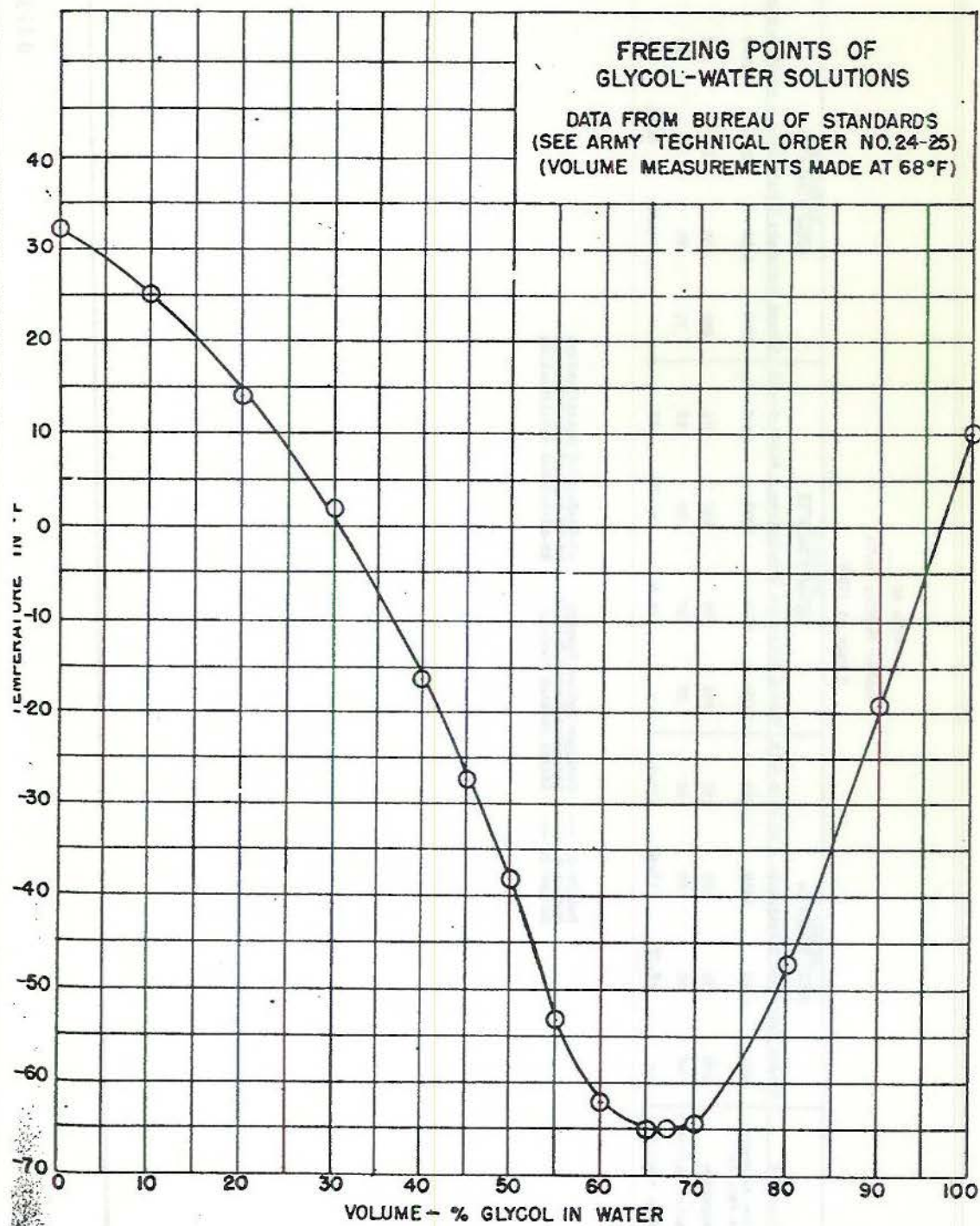
Pour point: below -75°F
Surface tension: 22.2 dynes/cm

Table XX
Rubber Packing Tests
7 Days at 158°F

Property	Rubber 8 After 7 Days in				Rubber 9 After 7 Days in				Rubber 10 After 7 Days in			
	Unaged Hydrolube	U Hydrolube A	AN-VV-Q-166b		Unaged Hydrolube	U Hydrolube A	AN-VV-Q-166b		Unaged Hydrolube	U Hydrolube A	AN-VV-Q-166b	
Tensile strength (lbs./sq. in.)	1257	1395	1435	1163	1688	1519	1908	1295	1860	1534	1762	1980
% Elongation	200	150	175	150	242	200	200	175	242	150	167	233
Durometer hardness	68	68	68	66	68	68	62	68	71	70	70	64
% Volume change	--	+7.27	+5.48	+10.6	-	+4.64	+2.18	+17	-	+6.36	+3.76	+11

Rubber 8 --- Goodyear Rubber Company AN-HH-P-114-B Specification
 Rubber 9 --- Arrowhead Rubber Company AN-HH-P-114-B Specification
 Rubber 10 --- Linear Packing Company AN-HH-P-114-B Specification

UNCLASSIFIED

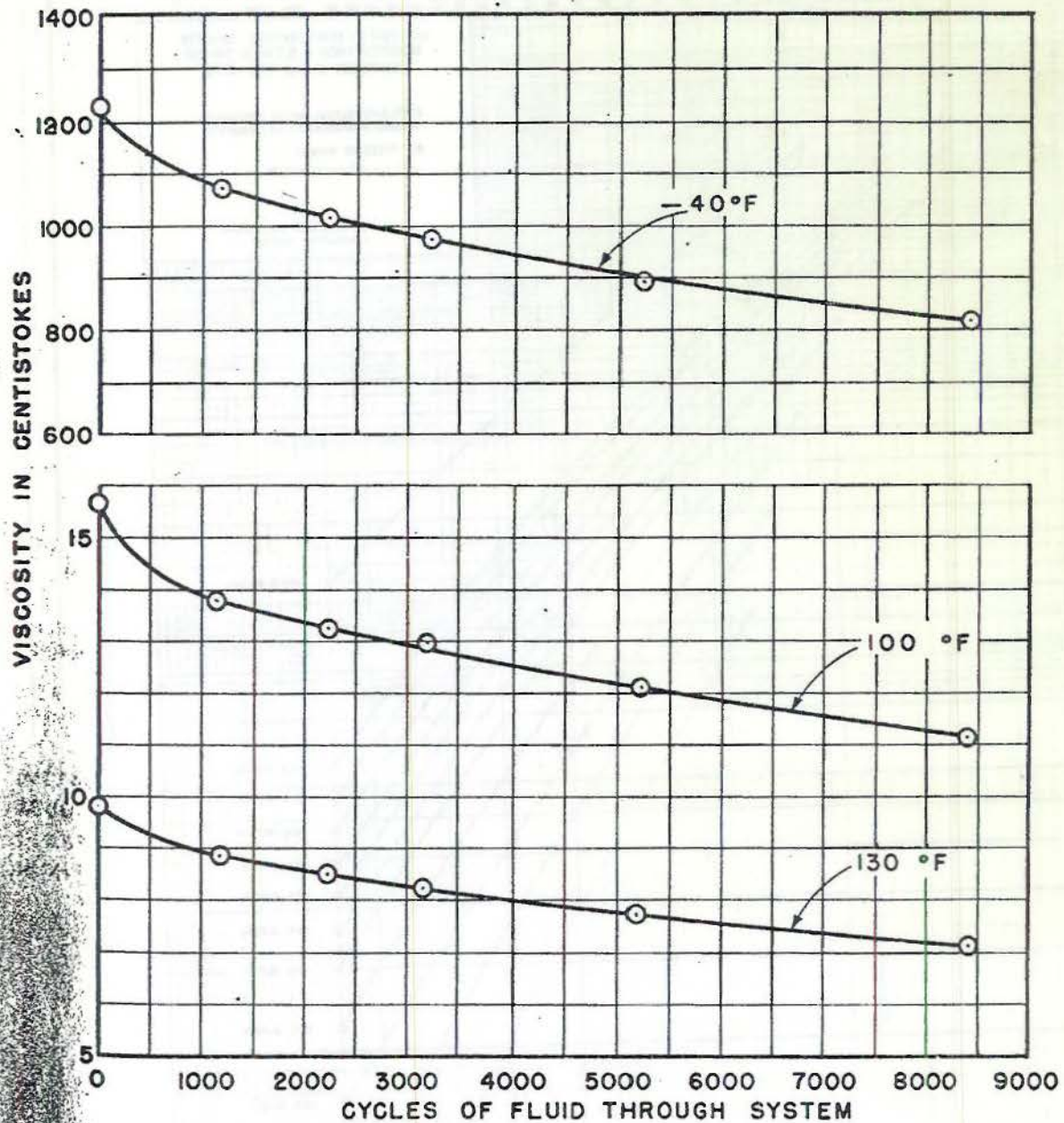


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PLATE I



SHEAR BREAK OF ACRY SOL G 3650 IN HYDROLUBE A
(HYDRAULIC BENCH TEST - USING PESCO IP349 PUMP)

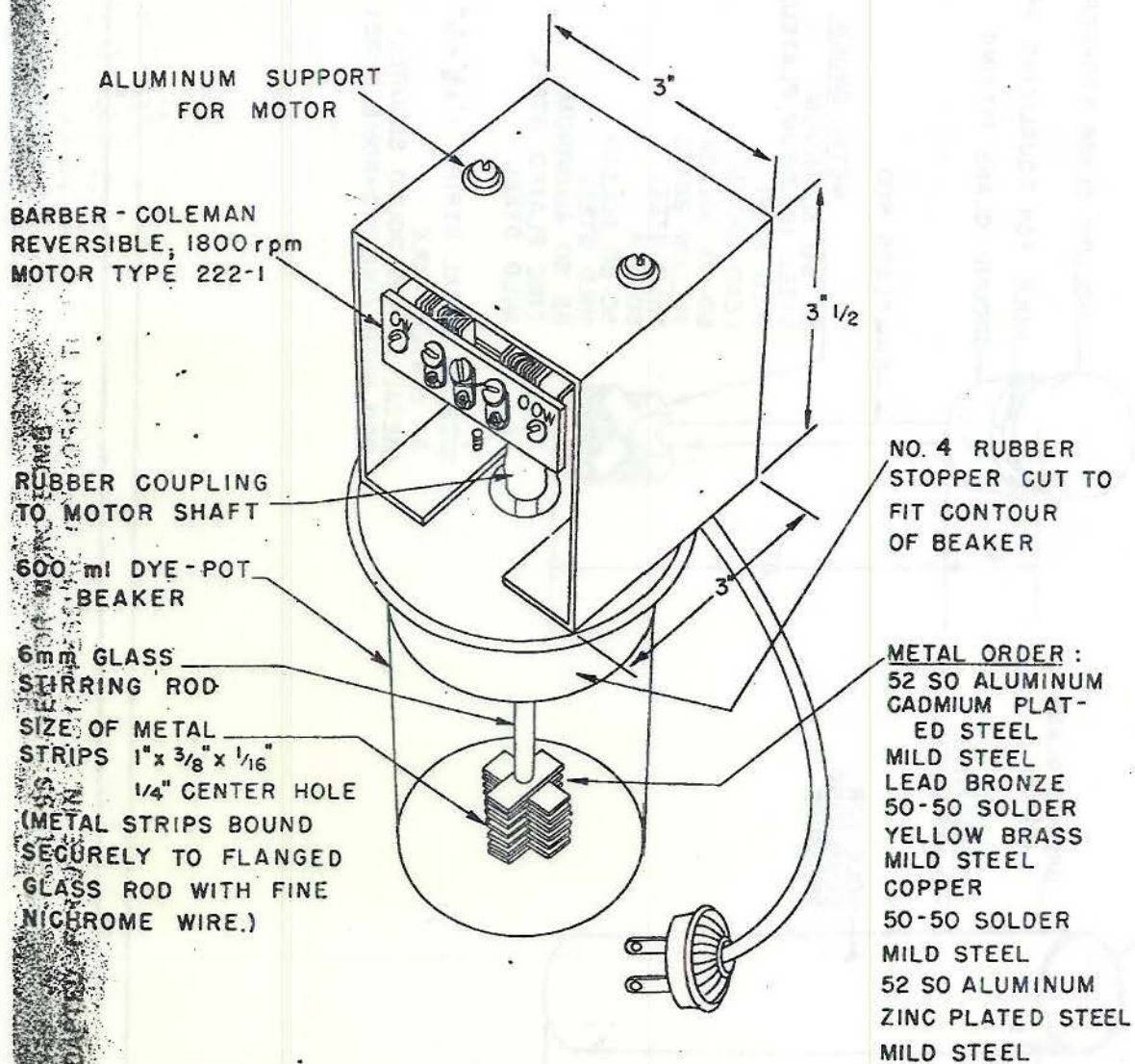


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PLATE 3

LIBRARY
NAVAL RESEARCH LABORATORY

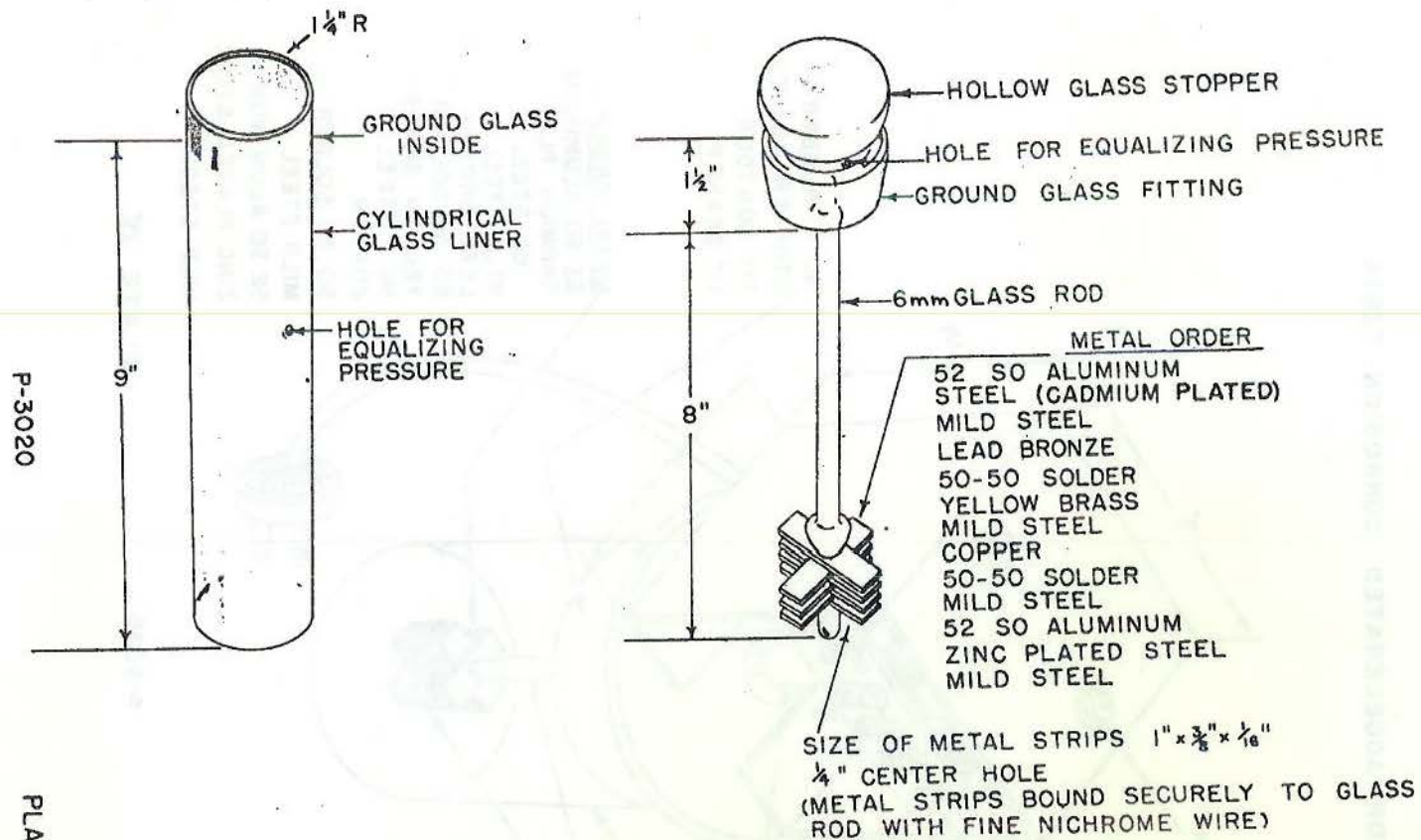
ASSEMBLY FOR ACCELERATED CORROSION TESTS



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PLATE IV

GLASS LINER FOR MICRO BOMB
(ADAPTED FOR USE IN HIGH PRESSURE CORROSION TESTS)



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PLATE V

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