

Perm

ORGANIC CHEMICALS DIVISION RESEARCH

[Signature]

St. Louis Research Report No 2-72

FINAL REPORT
ON
FUNCTIONAL FLUIDS APPLICATION RESEARCH

JOB NO. 2-32-760-01-3505 (II)

February 16, 1960

Reported by: L. R. Stark



ST. LOUIS, MO

DSW 622033

MONSANTO CHEMICAL COMPANY
Organic Chemicals Division
St. Louis Research Department

St. Louis Research Report No. 2472

FINAL REPORT
ON
FUNCTIONAL FLUIDS APPLICATION RESEARCH

JOB NO. 2-02-760.01-3505 (II)

February 16, 1960

Reported by: L. R. Stark

Work done by: E. P. Cunningham
R. E. Hatton
H. W. Luebke
L. R. Stark

DSW 622034

STLCOPCB4096065

DISTRIBUTION FOR REPORT NO. 2472

1. File
2. T. M. Patrick - R. E. Hatton
3. M. C. Throdahl - file
4. Duplicate file
5. Central Technical Files - Creve Coeur
6. L. R. Stark
7. U. E. Klein
8. T. P. Sands - H. S. Litzsinger
9. Extra
10. Extra

This report contains confidential information which is the property of the Monsanto Chemical Company which shall be disclosed only to duly authorized persons. The recipient is held accountable for the filing and safe custody of this report which must be returned on demand.

DSW 622035

STLCOPCB4096066

TABLE OF CONTENTS

	<u>PAGE NO.</u>
INTRODUCTION	1
SUMMARY.....	1
I. Industrial Fire Resistant Type Fluids	1
II. High Temperature and Fire Resistant Aviation Type Fluids	2
III. Miscellaneous	2
CONCLUSIONS	3
DISCUSSION	3
I. Industrial Fire Resistant Type Fluids	3
A. Fire Resistant Fluid Base Stock Screening	3
B. Aroclor Fluid Formulation	3
C. OS-95 Formulation	5
D. Fire Resistant, Low Viscosity Hydraulic Fluid, OS-104	9
E. Phosphate Ester Formulation.....	15
F. Preservative Type Hydraulic Fluids.....	15
G. Rust Inhibited Pydraul F-9 and Pydraul 625.....	16
H. Steam Turbine Lubricant, OS-81.....	19
1. Rust Inhibited OS-81.....	19
2. OS-81 Resistant Coatings	19
3. Gas Turbine Lubricant Application.....	19
II. High Temperature and Fire Resistant Aircraft Type Fluids	21
A. High Temperature Fluid Screening and Formulation	21
1. General Screening	21
2. High Temperature Jet Lubricant Formulation	22
3. High Temperature Hydraulic Fluid Formulation	23
4. Polyphenyl and Polyphenyl Ether Mixtures.....	23
5. Miscellaneous	25
B. OS-45 Fluid Evaluation	26
1. Objective and Conclusion	26
2. Discussion of Experimental Work	27

DSW 622036

STLCOPCB4096067

TABLE OF CONTENTS (continued)

	<u>PAGE NO.</u>
C. Skydrol 500	32
1. Reformulation to Eliminate Dermatitis.....	32
2. Viscosity Index Improver.....	33
D. Silicate Weapons Lubricant.....	34
E. Skydrol 7000 - Low Temperature Viscosity.....	35
III. Miscellaneous.....	35
A. ASTM Cooperative Test Work	35
B. Synthetic Lubricants for Refrigeration Units...	36
C. Low Temperature Viscosity Study.....	36
D. Lubrication Study - Paraffinic Petroleum Oil...	36
E. Compressor Lubricant - Four-Ball Wear.....	37

APPENDIX

Table I	- OS-95 Physical and Functional Properties
Table II	- OS-104 Physical and Functional Properties
Table III	- OS-45 Fluid Evaluation
Table IV	- Rust Inhibited Pydraul F-9 and Pydraul 625
	ASTM Cooperative Tests
	Fire Resistance
	Lubricating Properties
	Stability

DSW 622037

STLCOPCB4096068

INTRODUCTION

The objective of this research was to provide an application program to maintain and expand our current functional fluid product lines. The scope of this work included bench scale evaluation of current fluids, evaluation of potential base stocks, and formulation of fluids with greater utility or competitive position from potential base stocks. This report presents the application studies completed during the period May, 1957 through December, 1958.

SUMMARY

I. Industrial Fire Resistant Type Fluids

Potential fire resistant industrial type fluid base stocks screened were of the classes phosphate ester, alkylated Aroclor, chlorinated biphenyl ether, and alkylated benzene. Of these a tertiary butyl triaryl phosphate exhibited qualities which would warrant further formulation efforts. This work is continued under the succeeding Job No. 3766.

Formulation work was done on an Aroclor base fluid in an effort to develop a satisfactory fluid of lower price than the current Pydrauls. Viscosity index improved versions were abandoned due to unfavorable cost or performance characteristics. This project resulted in a non-viscosity index improved, fire resistant, Aroclor base industrial type hydraulic fluid designated OS-95 and subsequently named Pydraul A-200 as a sales item.

A fire resistant, low viscosity hydraulic fluid, designated OS-104, was formulated to augment Pydraul 150 in systems requiring a greater degree of thermal and shear stability. During the period of this report OS-104 was not accepted by Development pending a review of market and fluid requirements.

A viscosity index improved phosphate ester fluid was formulated as a chlorine-free fire resistant fluid for the Navy and as a fire resistant, mineral oil insoluble fluid for a specific application. The project was discontinued when the fluid failed the Navy specification tests and the oil insoluble fluid application ceased to exist.

A preservative type fire resistant hydraulic fluid designated OS-100 was formulated for use by the U.S. Navy.

Rust inhibited versions of Pydraul F-9 and Pydraul 625, designated OS-101 and OS-102, respectively, were formulated for customers who experience rusting problems. Some unsuccessful work was done to find a non-foaming, non-copper attacking rust inhibitor for OS-81.

DSW 622038

Two coating materials were found which are resistant to OS-81. Various other information was supplied pertaining to the application of OS-81 as a fire resistant industrial gas turbine lubricant.

II. High Temperature and Fire Resistant Aircraft Type Fluids

Twenty experimental materials were screened for application as high temperature hydraulic fluids or lubricants. None of these base stocks appeared to be suitable for formulation to the applicable military specifications. Preliminary work was done to find low melting polyphenyl ether or polyphenyl mixtures for these applications. Again, these fluids did not possess the basic physical properties for application against existing specifications.

Two formulations, one based on bis(1-methylcyclohexylmethyl) sebacate and the other on 1,1-didodecyl-3,3-di(2-ethylhexyl)urea were formulated to meet specifications for a 450°F. to 550°F. high temperature fluid as outlined by a pump manufacturer's representatives. Full scale functional testing of these fluids was not completed during the period of this report.

A project was completed on the evaluation of the OS-45 type fluids to provide information on their performance and service life limits. The competitive fluids, Oronite 8515 and Oronite 8200, were included as a basis for comparison.

Reformulation of Skydrol 500 to eliminate a dermatitis causing additive resulted in an acceptable formulation subsequently adopted as Skydrol 500A.

Several tests were conducted on a new process Acryloid for Skydrol 500 with negative results. Further modified Acryloid samples from Rohm and Haas were evaluated under the succeeding Job No. 3767.

Formulation work on a silicate weapons lubricant to meet military specification MIL-L-14107 failed to provide a qualified product.

III. Miscellaneous

ASTM Cooperative tests were conducted on reference fluids supplied by an ASTM committee. Lubricating properties, fire resistance, and stability information was obtained on the fluids.

A minor amount of work was done to establish the level of lubricating properties desirable for refrigeration unit lubricants.

DSW 622039

Low temperature viscosity values were obtained on Skydrol 500 to check our procedure and results against values obtained at Douglas Aircraft Company.

Minor lubrication studies were conducted on iron treated petroleum oil and on compressor lubricants as a service to various individuals.

CONCLUSIONS

Appropriate application studies were conducted during the period May, 1957 through December, 1958. Future work of this nature will continue under a new job number.

DISCUSSION

I. Industrial Fire Resistant Type Fluids

A. Fire Resistant Fluid Base Stock Screening

Chlorinated diphenyl ether, dodecylbenzene, and three alkylated Aroclors were evaluated on the basis of low temperature viscosity, flash and fire points, molten metal ignition, and ASTM slope. Three Aroclor base stocks (1242, 1248, and 1254) were subjected to the same tests as reference fluids. The chlorinated dodecylbenzene exhibited the most desirable characteristics of the fluids listed; however, its fire resistance was inferior to that of Aroclor 1242. This fluid might find application as a diluent for the more fire resistant fluids to improve their low temperature characteristics. Test results appear in Table I.

Isopropyl triaryl phosphate (prepared by J.D. Sullivan) was considered to be less oxidatively or thermally stable than tricresyl phosphate (TCP) and was not considered further as a fire resistant fluid base stock. Tertiary butyl triaryl phosphate (St. L. 15407, JDS) exhibited oxidation corrosion characteristics equivalent or better than TCP. Further work on the t-butyl triaryl phosphates was done under a succeeding job number (3766). Oxidation corrosion and 4-Ball Wear data appear in Tables II and III.

B. Aroclor Fluid Formulation

A project was initiated to formulate an improved fire resistant industrial fluid based on Aroclors to broaden Monsanto's participation in this market, to meet competition, and to provide fluids of more desirable characteristics. The low cost of Aroclors together with their excellent fire resistance and good stability under hydrolytic, thermal, and oxidative conditions make them excellent candidates for fire resistant fluid base stocks. This project resulted in a non-viscosity index improved, fire resistant Aroclor base industrial type hydraulic fluid designated OS-95 and subsequently named Pydraul A-200 as a sales item.

DSW 622040

Initial formulation was directed at improving the viscosity index and lubricating properties of the Aroclor base stocks. A fluid equivalent or better than Pydraul F-9 in these respects was desired. Several formulations were prepared using Acryloid type viscosity index improvers (Acryloid 710, Acryloid HF-855, Acryloid HF-825, Acryloid HF-860). This approach was later abandoned due to the relatively large quantities of viscosity index improvers required. In addition to being shear unstable, these materials added considerably to the cost. These various formulations are presented in Table IV for record purposes. Included in this table are initial viscosities and four-ball wear and loss of viscosity on shearing for a few of the blends.

Another class of viscosity index improvers, the high molecular weight Ucon lubricants (Carbide), were found to be very shear stable. The first of these blends were made to establish the desired additive concentration and an Aroclor base stock combination. At that time a fluid was sought with a positive viscosity index and a 100°F. viscosity equivalent to 200 Saybolt Universal Seconds on an absolute basis. In addition the lubricating properties were to be equivalent to Pydraul F-9. Blends which approximated the viscosity requirements were as follows:

a.	Aroclor 1242	97.5% (w)
	Ucon 75-H-90,000	2.5%
b.	Aroclor 1242	67.0%
	Aroclor 1232	29.0%
	Ucon 75-H-90,000	4.0%

Other Ucon containing formulations appear in Table V. It was determined that the load carrying properties of the Ucon containing Aroclors as measured by the Almen machine were inferior to the base fluid.

Considerable formulation work was done to improve the load carrying properties without depreciating the good anti-wear characteristics. Several additive combinations were found which yielded Aroclor-Ucon blends with antiwear and load carrying properties equivalent to Pydraul F-9 in the four-ball and Almen tests. These blends appear in Table VI. It has since been questioned whether the Almen test is a good evaluation of E.P. properties and further whether or not such superior E.P. properties are necessary in a hydraulic fluid. This should be studied in the future.

DSW 622041

A revision in the fluid requirements to include a rust inhibitor further complicates the formulation problem. At that time, too, it was found that the Ucon viscosity index improvers created an emulsion problem and were dissolved from the Aroclor base into water. For these reasons and due to the additional cost of 2.5% to 4% of additive necessary, the Aroclor-Ucon blends were abandoned. It is recommended that the Ucon type of viscosity index improvers be further evaluated if future clarification of requirements for lubricating, emulsion, additive leaching by water, and anti-rust allow the use of such materials. In addition, their superior low-shear performance would warrant looking for materials of a similar nature but without the Ucon's shortcomings.

Due to the unpromising outlook for the viscosity index improved versions, a non-viscosity index improved Aroclor fluid was formulated. These blends appear in Table VII. The required fluid was to consist of a mixture of Aroclors as the base stock with appropriate additives as necessary to obtain the specified viscosity, lubricating properties, and anti-rust characteristics. The desired viscosity values were a 100°F. viscosity of 200 to 300 SUS absolute, 45°F. viscosity of 4500 SUS absolute maximum, and a 160°F. viscosity of 60 SUS absolute minimum. The pour point was to be less than 10°F. if possible. The lubricating properties were to be equivalent or superior to those of Pydraul F-9 and the anti-rust characteristics were to be of an acceptable level, as judged by the ASTM D-665 Steam Turbine Oil Rust Test. All other performance values were to be equivalent to those of Pydraul F-9.

The final blend representing these properties and designated OS-95 consists of 66% Aroclor 1248, 33% Aroclor 1242, 0.5% Santolube 393, 0.5% Santolube 70 and 50 ppm. D.F.C. 200 by weight. A discussion of the various OS-95 performance and physical properties follows in Section C.

C. OS-95 Formulation

1. General Requirements and Final Blend

An Aroclor base fire resistant, industrial type hydraulic fluid, OS-95, was formulated to meet viscosity, lubricating requirements, and antirust characteristics specified by the Sales Department. These requirements and the steps taken in formulating OS-95 appear in Section B, Aroclor Fluid Formulation. The final blend representing these properties consists of 66% (w) Aroclor 1248, 33% Aroclor 1242, 0.5% Santolube 393, 0.5% Santolube 70 and 50 ppm DCF-200. This fluid was transferred to the Development Department for full scale evaluation and testing in the field. Bench scale performance and physical properties for OS-95 appear in Appendix Table I and specific comments on the OS-95 characteristics appear below.

DSW 622042

2. Viscosity and Pour Point

The lower limit of the required 200 to 300 SUS absolute viscosity range was chosen as a basis for Aroclor base stock mixing. This would represent the most severe viscosity condition. A mixture of one part of Aroclor 1242 to two parts of Aroclor 1248 by weight exhibits the desired 100°F. viscosity of 200 SUS absolute. These proportions may have to be varied slightly to account for the differences in viscosities of individual Aroclor batches. This Aroclor mixture has a 45°F. viscosity of about 5000 SUS absolute which is slightly greater than the specified value and a 160°F. viscosity above the 60 SUS absolute minimum specified. The use of greater portions of higher Aroclors would bring the 45°F. viscosity above the 45°F. maximum and would result in higher pour point values.

3. Lubricating Properties

Four-ball wear tests on the Aroclor base stocks indicated wear values of 0.35 mm., 0.38 mm., and 0.82 mm. at loads of 4, 10, and 42 kilograms, respectively. These values indicated the necessity of an anti-wear additive to obtain an Aroclor fluid equivalent to Pydraul F-9. The work on the viscosity index improved version of the Aroclor fluid (Section above) indicated Santolube 393 to impart good anti-wear properties to Aroclor based fluids.

A Santolube 393 concentration of 0.5% was chosen on the basis of previous work. The anti-rust agent, Santolube 70, was also incorporated before further bench scale lubricating tests were performed since such additives may adversely affect lubricating properties. Additive concentrations of 0.5% (w) Santolube 393 and 0.5% (w) Santolube 70 gave acceptable Four-Ball Wear results superior to Pydraul F-9 and Almen E.P. results equivalent to Pydraul F-9.

Subsequent anti-rust tests indicated the Santolube 70 concentration could be cut from 0.5% to 0.05% to give adequate protection in the ASTM D-665 rust test. A 50 gallon blend was formulated on the 0.05% Santolube 70 basis and was found to have depreciated in Four-Ball Wear and Almen Load characteristics. Four-Ball Wear and Almen Load tests run on variations of the Aroclor formulation indicated that a Santolube 70 concentration of 0.5% returned the blend to the expected performance level. These results are outlined in Table I of the Appendix. The lubrication properties appeared to be better with the two additives, Santolube 393 and Santolube 70, together than with either alone. This apparent synergistic effect was not studied further.

Since it was requested that the fluids have lubricating performance equivalent to or better than Pydraul F-9, it was decided to submit the 0.5% Santolube 393, 0.5% Santolube 70 formulation to General Motors for their full scale pump tests. Any future work on this type fluid should include a study of the optimum Santolube 393 and Santolube 70 concentrations and the use of oiliness agents and anti-rust agents other than Santolube 70.

DSW 622043

4. Rust Inhibition

Previous work with rust inhibitors for Pydrauls has indicated that Santolube 70 is relatively effective as an inhibitor, as measured by the ASTM D-665 rust test. The Sales Department has found indications that additions of Santolube 70 to equipment with rusting problems is effective. For these reasons, Santolube 70 was chosen as the rust inhibiting additive for the Aroclor formulation.

The initial concentration of 0.5% Santolube 70 was reduced to 0.05% without deterioration in anti-rust properties, as judged by the ASTM D-665 rust test. Lubrication tests on the blend containing reduced amounts of Santolube 70 indicated a performance deterioration and the Santolube 70 concentration was increased to the original value of 0.5% by weight. Refer to Discussion, Section C above, concerning the fluid lubricating properties. The high concentration of Santolube 70 is undesirable from the standpoint of foaming tendencies and copper attack in systems where water is present.

5. Miscellaneous Properties

a. Appearance

OS-95 is a very light yellow liquid with a slightly hazy appearance. The haze is due to very small quantities of antifoamant, DCF-200.

b. Foam

The use of Santolube 70 in the OS-95 blend causes undesirable foaming. A concentration of 50 parts per million of Dow Corning Silicone DCF-200 - 350 cs. at 25°C. was used to inhibit foaming. This concentration is based on similar anti-foamant concentrations used in some of the current Pydrauls. No work was done on the determination of minimum amounts of defoamant necessary.

c. Neutralization Number

Unused OS-95 has a total acid number of about 0.8. This apparent high acidity is due to nature of the Santolube 393 and Santolube 70 present. The initial pH of the blend is greater than 7.

6. Toxicity

Toxicity tests have been run on OS-95 and are reported in a memo from J.T. Garrett to R.E. Hatton of February 26, 1958. This report indicates the Oral LD₅₀ to be 10,500 milligrams per kilogram and classifies the material as "practically non-toxic" from an oral ingestion standpoint. The skin Minimum Lethal Dose was found to be 625 to 1,250 milligrams per kilogram in rabbits which

DSW 622044

STLCOPCB4096075

8.

classifies the material as "slightly toxic" by skin absorption. The compound is only a mild eye irritant and a saturated atmosphere of OS-95 vapors is only slightly irritating to rats exposed for six hours. In summary, "The above information indicates that OS-95 is not a very seriously toxic product to handle and, therefore, no special handling precautions are necessary."

7. Compatibility

OS-95 appears to be completely miscible at all concentrations with Pydraul F-9, Cellulube 220, and Houghto-Safe 1120 at temperatures of 40°F., 75-80°F., and 120°F. No unusual depreciation of lubricating properties are expected from these mixtures. Shell four-ball wear and Almen E.P. values for the mixtures were generally between the values of the component fluids. The Almen and four-ball wear values for these fluids appear in Table VIII.

8. Full Scale Performance

At the time OS-95 was being developed in the laboratory, it was learned that the General Motors Corporation Process Engineering Staff had received an appropriation to do further test work on fluids and would include any new Monsanto fluid in their program free of charge. Subsequently a 550-pound lot of OS-95 was submitted for full scale pump tests by General Motors.

The General Motors test of OS-95 was conducted using a Vickers V104-D pump at 1000 psi. A preliminary report of this test was recorded in a memo to file by R. Davis, May 13, 1958.

"On May 2, 1958, Stu Litzsinger and I talked to John Panek of the GMC Tech Center on the telephone. John gave us the confidential results of his pump tests with OS-95. In summary, it looks as good as the best petroleum oil he has ever tested.

"After running 200 to 400 hours, a water cooler broke, letting water enter the fluid. It was separated, the pump examined and found to be in good shape, and the test continued. At 200 hours, there were 5 milligrams of wear on the ring; at 1000 hours, there were 31 milligrams of wear on the ring. The test will be continued to 2000 hours.

"The best of the petroleum hydraulic oils have 40 to 80 milligrams of wear on the ring at 2000 hours. We would be in about that range, but the test with OS-95 is even more severe than the ones run with oil because it is run with a Vickers V-104-D pump of the old style using an 11 gallon per minute ring. This is probably the most severe test that could be run with this type of pump."

DSW 622045

STLCOPCB4096076

On the basis of these good test results, OS-95 was placed in a Kux die casting machine at Chevrolet - Bay City Division with satisfactory results. A sample of OS-95 removed from this system was evaluated for antiwear, rusting, acidity, and viscosity. This fluid reportedly used for 260 hours, of which 14 hours were at 190°F., exhibits no significant change from new OS-95 except in color. These data appear in Table IX.

OS-95 was installed in a Johnson Motors Lester-Phoenix die casting machine. Development Department Call Report No. FF-414 of November 14, 1958 summarized results of this test as follows:

"A five-month service test (1800 hours) of OS-95 at Johnson Motors has shown conclusively that this fluid will offer satisfactory performance in a high-pressure hydraulic system as used in a Lester-Phoenix die casting machine. During this period of time, there has been no evidence of anything other than satisfactory operation, and nothing abnormal has been observed. Because of the success of this test, plans are to introduce OS-95 into the plant on a limited basis by using it as make-up for two machines presently operated on Pydraul F-9. If these machines operate satisfactorily for a period of two or three months, Johnson Motors would like to discontinue the purchase of Pydraul F-9 in favor of OS-95."

In conjunction with this test at Johnson Motors, a request was made by them to investigate the possibility of increasing the viscosity of OS-95 by approximately 50 SUS at 100°F. Subsequent calculations indicated this could be accomplished with a mixture of higher Aroclors but that the depreciation of low temperature properties was excessive. No further work was done with such a formulation.

9. Profitability

Profitability of OS-95 in comparison to Pydraul F-9 was determined by M. Dmytryszyn and reported in a memo of March 15, 1958 (MD to JHL, March 15, 1958).

D. Fire Resistant, Low Viscosity Hydraulic Fluid, OS-104

1. Fluid Requirements and Final Blend

Pydraul 150, a low viscosity fire resistant fluid designed for use in low temperature regulator systems, has inherent thermal and shear instability. Introduction of a competitive fluid, Houghto-Safe 1010, having greater shear and thermal stability, has resulted in the loss of Pydraul 150 sales. This necessitated the formulation of an improved Pydraul 150 type fluid. The Houghton fluid was being sold on the basis of thermal stability, such that a system failure and consequent hydraulic line overheating would cause Pydraul 150 to solidify while their fluid remains liquid, facilitating system clean-up. A shear and thermally stable, fire resistant

industrial type hydraulic fluid, designated OS-104, was formulated to meet viscosity, thermal stability, and lubricating requirements specified by the Sales and Development Departments. During the period covered by this report, OS-104 was not accepted by the Development Department pending a review of OS-104 and alternate formulations by Douglas Aircraft Company.

Fluid requirements were determined at a meeting of Sales, Development and Research on April 2, 1958. These requirements were specified in the memos, R. Davis to H.S. Litzsinger, April 7, 1958 and H.S. Litzsinger to E.P. Cunningham, April 11, 1958.

It was agreed that the Pydraul 150 supplement would be a blend of cresyl diphenyl phosphate (Santicizer 140) and tricresyl phosphate (TCP) and would not be V.I. improved. The general characteristics of this fluid were to be essentially those of the competitive product, Houghto-Safe 1010, and are as follows:

Viscosity	50 SUS at 150°F.
Pour Point	Less than -25°F.
Oxidation and Corrosion Resistance	To be equal or better than Houghto-Safe 1010 at 250°F.
Thermal Stability	Equal to or better than Houghto-Safe 1010 when exposed for 15 min. at 520°F.
Lubricating Properties	Equal to or better than Houghto-Safe 1010 as evaluated by the Shell 4-Ball, Almen, and Timken tests.
Foaming	To be checked and reported.
Rusting	To be checked and reported.
Flammability	To be checked by the three tests of SAE specification AMS 3150.

The only advantage such a blend might have over Houghto-Safe 1010 would be its better low temperature properties imparted by the Santicizer 140. It was suggested that a Ucon thickened and V.I. improved Santicizer 140 blend be considered to further increase the low temperature advantage.

The Santicizer 140-Ucon blend was subsequently abandoned due to emulsification problems and difficulties in obtaining adequate load carrying properties. However, anti-wear properties appear to be improved by the Ucon additive, 75-H-90,000.

DSW 622047

STLCOPCB4096078

It was found that the Santicizer 140-TCP mixture was deficient in lubricating properties. An extensive formulation study indicated the following formulation to be acceptable as judged by the specified qualities outlined above.

69.49% (wt.)	Santicizer 140
29.25%	Tricresyl Phosphate
0.25%	Dioleoyl malate (DOM)
0.1%	Dilinoleic acid (RT-67-A)
50 ppm.	DCF-200 Fluid

Some 270 formulations or formulation variations were prepared and tested for Almen E.P., four-ball wear, or 15 minute heat stability or each of these. Of the various additives tested, the following gave the best lubricating performance in the Santicizer 140-TCP base stock:

Santolube 393 (dithiophosphate ester)
 Oleic acid
 Vanlube AZ (Vanderbilt Co. zinc diamyl dithiocarbamate)
 Santolube 70 (Monsanto tetrapropenyl succinic anhydride-amine-fatty acid reaction product)
 Sarkosyl O (Geigy Chemical Corp. N-oleyl sarcosine)
 Aerolube 93-C (dithiophosphate ester)
 Aroclor 1232 (chlorinated biphenyl)
 Stan-Add 121 (barium salt of P_2S_5 -isobutylene R.P.)
 N-30 (bis, beta, chloroethyl vinyl phosphonate dibutyl hydrogen phosphite)
 Alkaterge T (Commercial Solvents Corp. - weakly cationic amine)
 M-434 (Monsanto-experimental gear oil additive blend)
 RT-52-A (Dihydroabietyl malate)
 Dioctyl malate
 RT-67-A (dilinoleic acid)

Most of these suffered considerable degradation in the 15 minute heat test. Several antioxidants were tried without any improvement. A better definition of fluid requirements may indicate that much of this work was unwarranted. It is recommended that such projects be better defined in the future and that they be assessed periodically during development to see if the proposed requirements are still realistic.

2. Viscosity and Pour Point

It was desired that the Pydraul 150 supplement have a low temperature fluidity advantage over Houghto-Safe 1010. It was Askania's desire (primary use of Pydraul 150 is in Askania regulator equipment) that the fluid be shear stable, operate down to -10°F. and have viscosities of 3000-4000 SUS at 0°F. and 50 SUS at 150°F. The 50 SUS at 150°F. value was easily attained by a Santicizer 140-TCP blend and a Santicizer 140-Ucon VI improved blend. However, subsequent viscosity checks indicated that the 3000-4000 SUS at 0°F. value was not obtained for the Houghton fluid, Santicizer 140-TCP, or Santicizer 140 alone.

DSW 622048

OS-104 has low temperature fluidity properties superior to Houghto-Safe 1010. The pour points and low temperature viscosities are tabulated in the Appendix Table II.

3. Lubricating Properties

Santicizer 140, the principal base stock of OS-104, was found to be deficient in its lubricating properties compared to Houghto-Safe 1010. These deficiencies were improved only slightly by addition of TCB. It was found that the aryl phosphate esters have relatively poor lubricating properties compared with the alkyl phosphates. Lubricating data, tabulated in Table X for various phosphate esters illustrate this difference in lubricating properties.

It is necessary to retain the aryl phosphates for this application due to their better thermal stability. Various additives may be compounded with the thermally stable aryl esters to impart improved lubricating characteristics. However, most of the additives examined were thermally unstable giving a very dark colored fluid on heating or giving solid decomposition products. Also, most of the more common lubricating additives used in petroleum type fluids were ineffective in the Santicizer 140-TCP system. The objective of the formulation study was to improve the antiwear properties of the base fluid while retaining or improving the load carrying (E.P.) properties consistent with acceptable thermal stability. The OS-104 formulation satisfies these objectives. Lubricating data are tabulated in Appendix Table II for OS-104 and in Table X for the non-additive treated phosphate esters and for the additives separately.

As was indicated in Section 1 above, future laboratory and service correlation should yield more realistic information on the lubricating requirements of hydraulic fluids. If requirements are less severe than specified, several of the milder lubricating additives might allow better performance in the thermal stability tests.

4. Thermal Stability and Oxidation-Corrosion Characteristics

The thermal stability requirement of the Pydraul 150 supplement was prompted by a Houghton advertising "gimmick" in which the two fluids are heated in excess of 500°F. for about 15 minutes. The Houghto-Safe 1010 fluid remains relatively stable with only slight color and acidity changes; whereas, Pydraul 150 decomposes leaving a dark material equivalent to one-third the original volume and containing solids having a very high acid number.

DSW 622049

A study of this thermal instability and the suggestion of a thermally stable Pydraul 150 supplement comprising Santicizer 140 and TCP is contained in a file note by R.E. Hatton dated May 6, 1958.

A mixture of Santicizer 140 and TCP turns yellow or light brown on heating for 15 minutes at 520°F. and has only a slight acid number increase. As stated above in the section on lubricating properties, considerable difficulty was encountered in finding lubricating additives which were effective and would stand up in the thermal stability test. The dioleyl malate-dilinoleic acid combination satisfies these requirements.

Two alternate blends which had acceptable lubricating properties and thermal stability contained Santolube 393 and Santolube 70, respectively. Oxidation-corrosion tests of these blends indicated excessive copper attack; therefore, they were abandoned. OS-104 oxidation-corrosion resistance is considered to be equivalent to that of Houghto-Safe 1010. Static thermal stability and oxidation-corrosion data for these fluids appear in Table II of the Appendix.

5. Fire Resistance

Fire resistance tests run on the OS-104 blend which include flash and fire points, a molten metal ignition test and high and low pressure spray flammability tests indicate this fluid to be superior to Pydraul 150 and equivalent to Houghto-Safe 1010 in fire resistance. Hot manifold tests run on the individual phosphate ester base stocks used in the OS-104 blend are also superior to Pydraul 150, and it is believed that the minor amounts of additive (total % 0.35%) will not decrease fire resistance in this test.

6. Miscellaneous Properties

a. Appearance

OS-104 is a slightly cloudy, almost colorless, oily liquid. The cloudiness is due to very small quantities of the antifoamant, DCF-200.

b. Foam

The use of dioleyl maleate in the OS-104 blend causes more foaming than is desirable. A concentration of 50 parts per million of Dow Corning Silicone DCF-200 (350 cs. at 25°C.) was used to inhibit foaming. This concentration is based on similar antifoamant concentrations used in some of the current Pydrauls. No work was done on the determination of minimum amounts of defoamant necessary.

DSW 622050

c. Rust

An attempt was made to include anti-rust additives in this formulation but most of those tried resulted in depreciation of lubricating properties. The dilinoleic acid used in the OS-104 blend has some utility as an anti-rust agent but is not present in sufficient quantity to give a completely rust-free D-665 specimen after 24 hours. The OS-104 formulation is considered to be equivalent to Houghto-Safe 1010 in rust resistance.

d. Neutralization Number

The acidity of OS-104 is slightly higher than the base component acidity due to the additives present. The initial total acid number of about 0.50 decreased to 0.35 in the oxidation corrosion test and increased to about 1.3 after the static heat test of 520°F.

7. Alternate Formulations

Before accepting OS-104, the Development Department requested additional information on two alternate blends. One of these was cresyl diphenyl phosphate (CDP) with Ucon 75, H-90,000 as a viscosity index improver and the other a similar viscosity index improved CDP with Santolube 70 added to improve antirust characteristics.

These formulations appeared to be equivalent to or better than Houghto-Safe 1010 in viscosity, 15 minute 520°F. static heat test, rust and foam. Lubricating properties and oxidation-corrosion resistance were not considered quite as good. Again water leaching of the Ucon additive and possible emulsion problems make this type of formulation less desirable. The requested information transferred to the Development Department for their decision appears in Table XI.

8. Toxicity

Toxicity of OS-104 is summarized as follows from a memo J.T. Garrett to R.E. Hatton, dated October 29, 1958:

"These data indicate that OS-104 may be classified as "relatively harmless" from a single oral ingestion standpoint, but must be classified as "practically non-toxic" from a skin absorption standpoint. It is important to note that the skin absorption toxicity indicates that OS-104 is somewhat more toxic by this route. This, of course, can indicate specie variation but the important observation is that by skin absorption in rabbits neurotoxic effects were noted that were not noted by any other route.

"Based on the above data, no particular hazard should be experienced in handling OS-104. However, excessive skin contact should be avoided."

9. Patent Status

A preliminary disclosure of invention has been prepared and submitted for the OS-104 formulation.

10. Cost Estimate

An economic evaluation of OS-104 was completed and reported in a memo M. Dmytryszyn to E.P. Cunningham, dated September 12, 1958.

E. Phosphate Ester Formulation

A project was initiated to provide a thickened, viscosity index improved, and shear stable TCP which would be competitive with a commercial fluid, Cellulube 220. Specific applications for this fluid were use as a chlorine-free fire resistant hydraulic fluid for the Navy and as a fire resistant and mineral oil insoluble fluid for an Atomic Energy Commission (AEC) installation. The oil insoluble properties were required where changeover was to be conducted without shutting down the system.

Due to the failure of Monsanto TCP and formulations containing this TCP to pass the Navy hydrolytic stability test and to the discontinuance of a need for the oil insoluble fluid for the AEC installation, this project was discontinued.

Physical properties and bench scale performance data for a TCP blend containing 1.55% (w) of Ucon 75-H-90,000 are presented in Tables XII, XIII and XIV, for record purposes. These data indicate the fluid to be equivalent to or superior to Cellulube 220 in viscosity, viscosity index, shear stability, pour point, antiwear properties and antirust properties. The fluid is relatively insoluble in both a paraffinic and a naphthenic base mineral oil with no significant V.I. improver removal by the oils. Although the TCP blend appears to be a little less soluble in the paraffinic than the naphthenic oil, a problem of separation from the paraffinic oil may be encountered due to emulsion formation.

Additional data indicate the Ucon type of V.I. improver creates a severe water emulsion which may be nearly eliminated by suitable demulsification additives.

F. Preservative Type Hydraulic Fluids

1. Pydraul 625, Pydraul F-9 Preservative Fluid

A project was initiated to provide a specific customer (Alcoa) with preservative type hydraulic fluid based on Pydraul F-9 and Pydraul 625 for equipment storage. Before this project was completed the need for such fluids ceased to exist and the work done to that time is reported for record purposes.

DSW 622052

STLCOPCB4096083

The Army-Navy Humidity Cabinet is used to evaluate fluid preservative properties. It was found that the steel test panels used for most of the screening work on this project did not give results consistent with other laboratories. Subsequent metallographic examination revealed that these panels were not cold rolled steel as specified for the humidity cabinet. Although these panels gave less severe results, the results are of value in that a separation of suitable and unsuitable rust inhibitors was made. Materials giving less than 50 hours protection were considered poor and those giving more than 50 hours protection were considered suitable for further study.

The inhibitors screened are listed in Tables XV & XVI. No specific protection times are shown since any future work must be done with suitable test panels which may give different results from those obtained.

Most of the screening was done with Pydraul 625 as the base fluid. In general, 3 to 5% of inhibitor is required for maximum protection. Materials exhibiting the best protective properties were a series of reaction products of tetrapropenylsuccinic anhydride with other materials, reaction products of maleic anhydride and various long chain fatty acids, dimer acids, and several commercial inhibitors.

2. OS-100 Preservative Type Hydraulic Fluid

The U.S. Navy Engineering Experiment Station outlined requirements for preservative type fluids compatible with or preferably based upon phosphate ester fluids and providing a minimum of 30 days protection in the humidity cabinet test. A fluid, designated OS-100, was formulated to meet these requirements. OS-100 consists of tricresyl phosphate (TCP) containing 2.37% (w) RT-67A (dilinoleic acid), 2.37% RT-53A (diamylphenylphosphoric acid), and 0.4% phenyl-alpha-naphthylamine. Formulation, physical properties, and bench scale performance of OS-100 are presented in a special report (St. Louis Research Report No. 2257, March 4, 1959).

G. Rust Inhibited Pydraul F-9 and Pydraul 625

1. Fluid Requirements and Final Blends

Sporadic "deposit" or rust problems have been experienced by various Pydraul F-9 customers and Alcoa, specifically, has indicated marked variance in rusting tendencies of the Pydrauls as judged by the ASTM D-665 Rust Test. For purposes of customer satisfaction and improved product quality, it was decided to include a rust inhibitor in the Pydraul F-9 and 625 formulations.

Previous laboratory work and limited field experience indicated the deposit problem is alleviated by Santolube 70 inhibition. On the basis of this lab and field experience it was decided to formulate Pydraul F-9 and Pydraul 625 with minimum quantities of Santolube 70 to give adequate rust protection. The

final blend designated OS-101 consists of 99.7% Pydraul F-9 and 0.3% Santolube 70 and the blend designated OS-102 consists of 99.7% Pydraul 625 plus 0.3% Santolube 70, and 50 ppm. DCF-200 by weight.

It was concluded from the results of bench scale performance tests and physical property measurements of OS-101 and OS-102 that these fluids have superior anti-rust properties and equivalent lubricating properties to the corresponding uninhibited Pydrauls. These tests indicate only slight deterioration in emulsion resistance and oxidation-corrosion resistance as exhibited by slightly higher copper attack.

OS-101 and OS-102 were transferred to the Development Department for limited field application. This qualified recommendation was given on the basis of the use of a surface active additive, Santolube 70. Many performance factors, including foaming, emulsion, lubricating properties and corrosion resistance may be affected by such materials and complete characterization of such fluids by bench scale tests is difficult.

It is recommended that any future work on the OS-101 and OS-102 type fluids should include the consideration of an additive other than Santolube 70 since this material imparts undesirable foaming and emulsion forming tendencies to the fluid and results in increased copper attack especially in systems where water is present.

2. Rust Inhibition

The minimum quantity of Santolube 70 required to give adequate rust protection to Pydraul F-9 and Pydraul 625 was determined to be 0.25% and 0.01% (wt.) respectively. These dosages were increased to 0.3% and 0.02% to give an additional margin of rust protection. Subsequent bench scale lubricating tests of the Pydraul 625 with 0.02% Santolube 70 indicated some deterioration in its load carrying properties. Increasing the Santolube 70 concentration to 0.3% restored the load carrying characteristics.

3. Lubricating Properties

As indicated in the section on rust protection, it was necessary to increase the concentration of Santolube 70 in Pydraul 625 in order to maintain the load carrying ability of this fluid. Almen and Timken values for 625 with 0.02% Santolube 70, and 625 with 0.3% Santolube 70 appearing below illustrate this.

	Almen Load	Timken Load
Pydraul 625	46 lb.-50 lb. torque	40 lb.-17,200 psi
Pydraul 625 + 0.02% Sant. 70	50 lb.-67 lb. torque	35 lb.-13,800 psi
Pydraul 625 + 0.3% Sant. 70	50 lb.-49 lb. torque	40 lb.-18,100 psi

DSW 622054

Shell 4-Ball Wear, Almen Wear, Almen Load and Timken Load values for OS-101 and OS-102 appear in Appendix Table IV. These data indicate some wear improvement and little change in load carrying properties upon addition of Santolube 70.

4. Fire Resistance

No fire resistance testing was done on OS-101 and OS-102. Since the quantity of additive is so low, it is believed that insignificant changes in fire resistance will occur.

5. Foam and Emulsion

Addition of 0.3% (wt.) Santolube 70 to Pydraul F-9 does not affect the foaming tendency or persistence; whereas, addition of the same amount to Pydraul 625 resulted in excessive foam which did not settle readily. The incorporation of 50 parts per million of Dow Corning Fluid 200 to the Pydraul 625-Santolube 70 mixture decreased the foam to an acceptable level.

The use of Dow Corning antifoam agent in the Pydraul 625 blend is necessary when Santolube 70 is used as the inhibitor. Pydraul F-9 already contains antifoamant and no further treatment is necessary upon Santolube 70 addition.

The use of 0.3% wt. Santolube 70 in Pydrauls F-9 and 625 results in only slight deterioration in emulsion forming tendencies. However, a definite emulsion layer does exist after the specified 30 minute settling period. It is not known if this amount of emulsification is enough to affect full scale operation.

Foam and emulsion values for OS-101 and OS-102 appear in Appendix Table IV.

6. Oxidation and Corrosion

Thermal and oxidative stability of OS-101 and OS-102 is similar to Pydrauls F-9 and 625. Copper attack is increased slightly and the inhibited fluid exhibits a small neutralization number decrease rather than the normal increase. The inclusion of Santolube 70 in the Pydrauls results in a higher initial total acid number. Fluid weight loss, and viscosity change are very low and no sludge is formed. OS-101 and OS-102 become brown on exposure to the 250°F. - 168 hour oxidation-corrosion conditions. These data appear in Appendix Table IV.

Although no specific tests were run on OS-101 and OS-102, it is emphasized that Santolube 70 containing fluids may result in greatly increased copper attack in systems containing water.

DSW 622055

7. Toxicity

Santolube 70 is reported by Scientific Associates (May 6, 1953) to be of relatively low order of toxicity when taken internally and to be only a mild-to-moderate eye and skin irritant. In handling OS-101 and OS-102, the same general precautions applicable to Pydraul F-9 and Pydraul 625 should be observed.

H. Steam Turbine Lubricant, OS-81

1. Rust Inhibited OS-81

Sporadic rusting has been observed on the steel catalyst coil from OS-81 steam turbine oil oxidation tests. Considerable work was done by J.D. Sullivan to find OS-81 rust inhibitors which were non-foaming and non-copper attacking (Job Numbers 2949 and 3334 and St. Louis Research Report No. 1779). Work done under this job number included oxidation-corrosion evaluation of OS-81 given various washing treatments, additive additions, and some tests to check test procedure variations. None of the washing treatments or additives tried completely eliminated rusting. Also, the minor test variations and refinements were unsuccessful in eliminating rusting or allowing reproducible tests.

The problem of test reproducibility is important since some tests with OS-81 give no rusting or only isolated rust spots; whereas, some duplicate tests resulted in considerable rusting. Further work will be done in the future to improve the test reproducibility and to find appropriate inhibitors. Although no conclusive results were obtained during the period covered by this report, the test results are presented in Table XVII.

2. OS-81 Resistant Coatings

Two coating materials were checked for resistance to OS-81. Both Nycote 4/30 Protective Coating (Nycote Laboratories, Van Nuys, California) and Steelcote 220-W-100 (Steelcote Manufacturing Co., St. Louis) appear to be resistant to OS-81. Panels coated with these materials were immersed in OS-81 at 150°F. for two months. The coatings were not loosened and exhibited no appreciable softening.

3. OS-81, Gas Turbine Lubricant Application

During the period of this report, OS-81 found application as an industrial gas turbine lubricant (Texas Eastern Transmission Corp.). Various information was supplied to facilitate this application.

Kinematic and Saybolt viscosities (calculated from centistoke values), determined over the range 50°F to 210°F., follow:

DSW 622056

<u>Temperature, °F.</u>	<u>K.V., centistokes</u>	<u>Saybolt Universal Seconds</u>
210	3.07	36.51
100	33.33	142.8
80	88.01	407.4
60	340.3	1573.
50	838.2	3872.

Fire resistance tests run on OS-81 are presented below:

Hot Manifold (1300°F.) - Does not flash or burn on tube or after leaving tube.

Low Pressure Spray - Very slight increase or brightening of flame.

High Pressure Spray - Will not ignite to three feet

Shell Four-Ball Wear values indicate OS-81 compares favorably with several commercial petroleum based fluids.

		<u>OS-81</u>	<u>Mobil DTE Medium</u>	<u>Sonoco Solnus 200</u>	<u>Sonoco Sunvis 921</u>	<u>Texaco Regal B R&O</u>
4-Ball Wear (167°F., 620 rpm, 1 hr.)						
Steel-on-Steel	4 Kg.	0.22	0.19	0.18	0.19	0.39
	10 Kg.	0.27	0.24	0.22	0.22	0.46
	40 Kg.	0.55	0.38	0.41	0.36	0.58
Steel-on-Bronze	4 Kg.	1.25	--	--	--	--
	10 Kg.	1.25	1.48	1.44	1.91	1.80
	40 Kg.	1.75	--	--	--	--

The gas turbine reservoirs were coated with a material not resistant to OS-81 (Rust-Ban 297, Esso Standard Oil Co.). It was found that OS-81 readily dissolved this coat except for the aluminum pigment. Some large soft flakes were removed which might have been difficult to filter. Sodium hydroxide solutions of 15 to 20% concentration removed the Rust-Ban readily. Higher caustic concentrations resulted in a gel-like coating which did not go into solution. OS-81 treated for two hours from 100°F. to 150°F. with 2% of the 20% caustic solution used for removing the Rust-Ban had a moderate increase in the 4-Ball Wear value (0.36 mm at 10 Kg. compared to the initial 0.27 mm value).

Stick lubricants for plug valves were checked for OS-81 resistance. Nordstrom Valve Lubricant #555 was dissolved; whereas Nordstrom Lubricant #P-55 appeared to be OS-81 resistant at 150°F. Valco #67 (Plug Valve Lubrication Co.) also appears to be OS-81 resistant.

DSW 622057

STLCOPCB4096088

This OS-81 application work was continued under a new job number (3766).

II. High Temperature and Fire Resistant Aircraft Type Fluids

A. High Temperature Fluid Screening and Formulation

1. General Screening

Preliminary screening tests were conducted on candidate fluids from Dayton, Lion Oil Division, St. Louis, and on competitive fluids. The fluids evaluated follow:

Mixed (o,m,p)bis(x-methylphenoxy)benzene, CP-30415
 1,1-Di-dodecyl 3,3-di-(2-ethylhexyl)urea
 2,2-Dimethyl-1,3-propyl dilaurate
 Bis(1-methylcyclohexylmethyl)hexahydroisophthalate, CP-30417
 Bis(1-methylcyclohexylmethyl)sebacate
 Santowax OM fraction liquid at room temperature
 Monoisopropyl biphenyl
 Diisopropyl biphenyl
 Ethylated Santowax R
 Isopropyl Santowax OM
 Tertiary butyl Santowax OM
 HB-40
 Ethylated HB-40
 Tert. butyl HB-40
 2-n-Butylamino-4-di-n-butylamino-6-piperidino-S-triazine
 2-N-n-propyl 4-N-n-butyl 6-N-n-hexyl melamine
 2-N-diethyl 4-N-di-phenyl 6-N-di-benzyl melamine
 m-Tolyl- α,α,α -trifluoro-m-tolyl sulfone
 o-Chlorophenyl p-nonylphenyl ether
 3,4-Diphenyl chlorobenzene
 Synjet Oil 15 (Texaco MIL-L-7808)
 General Electric Versilube F-50 silicone
 General Electric 81644 silicone
 General Electric 81717 silicone
 Fluorochemical FC-75

Preliminary evaluation of most of these fluids was done with the 347°F., 72-hour, MIL-L-7808C oxidation-corrosion test and by the Shell Four-Ball Wear Test. In most cases, viscosity and an estimate of the low temperature fluidity were determined. Physical properties appear in Table XVII, oxidation-corrosion values in Table XIX, and Shell Four-Ball Wear and Almen E.P. in Table XX.

After several of the above mentioned fluids had been evaluated, their performance qualities, usable maximum bulk temperature, and field of application (jet lube or hydraulic fluid) were reviewed. This information is summarized in Table XXI. None of these fluids appeared to be acceptable for specifications MIL-L-7808C, 9236A, or MIL-H-8446 without additive or structural modification. Three base stocks were chosen for formulation to meet the MIL-L-9236A jet engine lubricant specification. These included bis(1-methylcyclohexylmethyl)hexahydroisophthate, bis(1-methylcyclohexylmethyl)sebacate and the mixed (o,m,p)-bis(x-methylphenoxybenzene).

DSW 622058

STLCOPCB4096089

Two fluids, mixed (o,m,p)bis(x-methylphenoxy benzene) and 2,2-dimethyl 1,3-propyl dilaurate, were considered for formulation as high temperature hydraulic fluids.

2. High Temperature Jet Lubricant Formulation

Initial high temperature fluid formulation efforts were directed toward the jet engine lubricants. Oxidation-corrosion at 500°F. for 48 hours indicated the mixed (o,m,p)bis(x-methylphenoxy benzene) to be more stable than the sebacate and hexahydroisophthalate diesters. Both the diesters exhibit some response to antioxidant treatment although concentrations and additive types studied were not sufficient to hold acidity increases within the MIL-L-9236A limits. Oxidation inhibitors were chosen to include selenium and sulfur bearing compounds, a hindered phenol, and amine type inhibitors. These represented some of the more favorable compounds indicated in various government contract reports and include phenothiazine, phenyl mercapto benzothiazole, phenyl selenide, dilauryl selenide, 4,4'-methylene bis (2,6-di-tert.-butylphenol).

Due to the time and fluid consuming nature of the oxidation-corrosion test, a simplified static oven test was attempted. Copper and steel specimens were suspended by a bent stainless steel wire in a 50 ml. watch glass covered beaker of the fluid. Samples were removed for acidity determination at 8, 24, and 48 hours. Most of this test work was done with the dilaurate and hexahydroisophthalate ester since the supply of the sebacate ester and bis methylphenoxy benzene were currently depleted. Although these test conditions appeared to be too severe for the particular fluids tested, phenothiazine did exhibit some antioxidant activity.

At this point in the formulation program, emphasis was shifted to high temperature hydraulic fluid formulation. No further work was done on high temperature jet turbine lubricants utilizing the diesters or the mixed bis-methylphenoxybenzene during the report period covered by Job Number 3505. Some work done to find low melting polyphenyl and polyphenyl ether mixtures is presented in Section 4 which follows.

During the course of this formulation work the MIL-L-9236 specifications were changed to require a 3.0 cs. minimum 400°F viscosity rather than a 35 cs. minimum at 100°F. None of the candidate materials would pass this requirement with the exception of a few higher molecular weight polyphenyls or polyphenyl ethers which also have very high melting points. There was some question as to the low temperature limitations of the 9236 specification. An engine builder (Pratt and Whitney) indicated that they would be interested in a fluid with a relatively high pour point if it had sufficient high temperature stability and lubricating properties. In view of this, it was decided to work toward a fluid having the following characteristics.

a. Thermal and oxidative stability in the 500 to 600°F. range. This should be attained without the use of inhibitors.

b. As wide a liquid range as possible with liquid at room temperature being a must with a pour point of 0 to 30°F. acceptable. Maximum high temperature viscosity and fluidity down to -65°F. are desirable.

c. Lubricating ability to give adequate performance in a gas turbine engine. Additives may be necessary.

In order to accomplish the objective of obtaining an appropriate candidate fluid, the following steps were to be taken:

a. Select from fluids evaluated to date those which most nearly meet requirements of items 2-a and 2-b above.

b. Perform all 9236A specification tests on these fluids.

c. If bench scale tests indicate gross lubricating deficiencies, improve lubricating properties by limited formulation.

d. Send most promising fluids out for Ryder Gear and Bearing Fatigue testing.

e. Present results of tests b and d above to engine builder for selection of candidate fluid or comments on necessary improvements. Future work will be dictated by these results.

The polyphenyl ethers were chosen as the best fluid candidates for this application. Work on the evaluation and formulation of these fluids was done under Job Number 3767.

3. High Temperature Hydraulic Fluid Formulation

Formulation work on a 450°F. to 550°F. temperature hydraulic fluid was initiated to meet requirements outlined by a pump manufacturer's representatives (Vickers, Inc.). Two formulations of different base stocks, bis (1-methylcyclohexylmethyl)sebacate and 1,1-didodecyl 3,3-di(2-ethylhexyl)urea were prepared. Physical properties and bench scale performance of these fluids is presented in a special report, St. Louis Research Report No. 2335, Job No. 2-02-750.01-3505, 3767 of August 27, 1959.

4. Polyphenyl and Polyphenyl Ether Mixtures

A polyphenyl and polyphenyl ether blending study was initiated to obtain blends having improved low temperature properties for high temperature fluid and lubricant applications. This study indicated the most promising fluids to be the four or five-ring all-meta ethers alone or in combination with the three-ring meta ether. The low temperature properties of these fluids appear to be limited by viscosity rather than freezing point. Since viscosity is primarily an additive property, any further improvement would be by the addition of greater than additive quantities of a less viscous component. Currently available low viscosity materials were considered either less thermally stable or too volatile.

DSW 622060

STLCOPCB4096091

Binary mixtures of four unsubstituted polyphenyl ethers, biphenyl stripped Santowax OM, and three fractions of Montar 9, representing mixtures thought to be principally quaterphenyls, were examined for freezing point lowering by means of a hot stage microscope fusion technique (Walter C. McCrone, Jr., "Fusion Methods in Chemical Microscopy", Interscience Publishers, Inc., 1957). This work indicated that the only binary mixtures having liquid present at room temperature were combinations including fluids which already were liquid or had liquid present at room temperature. The viscous nature of these fluids at temperatures at or below room temperature resulted in supercooling to glasses and other difficulties encountered by the microscopic cold stage method necessitated a change of approach to the problem.

A plot of the viscosities of the 3, 4 and 5-ring unsubstituted polyphenyl ethers indicated the viscosities of these fluids below 400°F. are dependent upon the chain length. That is, all isomers; ortho, meta, para, or mixed; of equal molecular weights follow a single viscosity curve very closely. Previous experience has indicated that the "pour point" of fluids having no distinct pour point (fluids becoming glasses) as do paraffin-containing petroleum oils usually lies in a region bounded by 10,000 and 100,000 centistokes. It is also noted that this range includes the 13,000 cs. value which has been related to minimum starting ability of turbine engines. Of the polyphenyls and polyphenyl ethers, only the 4-ring and 3-ring ethers and the 3-ring polyphenyls have viscosities (extrapolated in some cases) in the 10,000 to 100,000 range below 30°F. The boiling point of biphenyl and diphenyl ether are considered too low for the engine lubricant application.

An 18% (w) mixture of the 3-ring meta ether in the 4-ring meta-meta ether remained a viscous fluid at 0°F. for three weeks. This blend was seeded with a few crystals of the 3-ring meta ether. During the three-week period the crystals appeared to neither grow nor decrease in size. This approach of mixing the 4- and 3-ring meta ethers is expected to yield a fluid with a pour point in the range of 0 to -20°F. The maximum allowable concentration of the 3-ring ether will be at the point that crystallization is not inhibited by the glass-like nature of the mixture.

The use of terphenyls as diluents for the 4-ring meta ether is an alternative. However, only slightly better lower temperature properties than the 4-ring ether alone would be expected since the terphenyls have only slightly better viscosity characteristics than the 4-ring ethers. The use of higher than the 4-ring ether or the 3-ring polyphenyl is warranted if higher boiling fluids are required. In this case the low temperature properties will also rise on the temperature scale.

An attempt was made to study the temperature-solubility characteristics of the three and four-ring meta polyphenyl ether mixture. The method used is ~~probably~~ worthy of further consideration to find the optimum ether blend, if such a blend is reconsidered for hydraulic or less stringent lubricant applications.

This method consisted of rapid agitation of the blend heavily seeded with the crystalline three-ring ether in centrifuge tubes. Periodically the tubes were removed from the constant temperature bath and were centrifuged to obtain the clear fluid. A refractive index of the fluid revealed the ether concentrations. At lower temperatures equilibrium appeared to be attained very slowly and heating during centrifuging remelted some crystals. This latter condition was improved by cooling the centrifuge holders prior to centrifuging.

Since it is believed that equilibrium conditions were not established, no specific values are reported and the method is given as a possible starting point for similar work in the future.

Work on polyphenyl ether formulation was conducted under the succeeding Job No. 2-02-750.01-3767.

5. Miscellaneous

Two samples of Santowax OM removed from Republic Aviation's high temperature hydraulic test systems exhibited only moderate change in viscosity from an unused sample. Sample No. 1 was taken from the test unit after being subjected to the following:

Room temperature to 310°F. - 30 minutes
310°F. to 700°F. - 50 minutes

Pressure: 2000-3000 psi

Linear O rings (Viton H)
Linear X way - 1 Back-up
Rings O Viton A - Asbestos

and Sample No. 2:

Room temperature to 350°F. - 30 minutes
350°F. to 700°F. - 50 minutes
700°F. to 780°F. - 15 minutes

Pressure: 1000-2000 psi

Linear Viton O - rings
PRP Viton O - rings
Duriod 5600 - Back-up

The viscosities determined for these samples are as follows:

	<u>100°F.</u>	<u>210°F.</u>
Santowax OM, No. 1	16.79 cs.	2.39 cs.
Santowax OM, No. 2	23.00	2.65
Santowax OM, S-55	18.03	2.41

DSW 622062

B. OS-45 Fluid Evaluation

1. Objective and Conclusions

The OS-45 high temperature hydraulic fluids, Type III (-65 to 400°F.) and Type IV (-65 to 550°F.), were introduced in 1951 and 1952, respectively. Since then, conflicting and inadequate information has been gained on the performance and service life of these fluids. For this reason, the project was initiated to augment current information on performance and service life. Similar competitive fluids, Oronite 8200 and Oronite 8515, were included as a basis for comparison.

These fluids were evaluated for static thermal stability, oxidation and corrosion characteristics, shear stability, and bench scale lubricating properties. Additionally, several samples of used OS-45 type fluids from high temperature pump tests were checked for lubricating properties. Chemical and physical properties were also determined in an attempt to correlate performance and service life factors. These included viscosity, acidity, specific gravity, refractive index, evaporation loss, foam, flash point, fire point, wear and load carrying ability, and attack of copper, steel, aluminum and silver. Conclusions drawn from this evaluation follow:

a. Lubricating Properties

OS-45 type fluids have better antiwear properties than the Oronite fluids, 8515 and 8200, as indicated by the Shell Four-Ball Wear test and the Almen Wear test. This statement pertains to both new fluids and fluids subjected to oxidative and thermal aging. All four fluids exhibit a slight increase in wear with aging. E.P. or load carrying properties of all four fluids, as measured by the Almen and Timken machines, are very low and approximately equal.

It is to be noted that the lubricating tests used in this evaluation represent a limited number of wearing surface configurations, do not take into account atmospheres other than air, and do not control temperature except in the Four-Ball test.

b. Thermal and Oxidative Stability

Thermal and oxidative treatment of OS-45 type fluids result in greater acidity increases than for the Oronite fluids, 8515 and 8200. Viscosity decrease was less for the OS-45 fluids than for the Oronite fluids in thermal and oxidative tests and about equal for all fluids in the shear test. Thermal and oxidative treatment of OS-45 Type IV results in the formation of a solid, believed to be selenium, which is very corrosive to silver and copper.

Evaporation loss at 400°F. for OS-45 Type III is excessive and loss for OS-45 Type IV approaches the same high value on thermal aging.

DSW 622063

STLCOPCB4096094

c. Service Life

Changes in acidity and viscosity of the OS-45 type fluids are probably the best indication of fluid condition for service life recommendations. No practical limits were established.

2. Discussion of Experimental Work

a. Lubricating Properties

Each of the unaged fluids, OS-45 Types III and IV, Oronite 8200, and Oronite 8515, were subjected to bench scale tests which included the Shell Four-Ball Wear test, Almen Wear, Almen Load, and Timken Passing Load. The fluids subjected to static thermal aging and the oxidation-corrosion test were checked for Four-Ball Wear values, as indicated in Section b below. In addition, Almen Wear and Load and Four-Ball Wear values were obtained on used OS-45 Type IV from Hamilton Standard and Vickers pump tests, and OS-45 Type III from an Eastern Industries system. Lubricating test data for the new and used fluids appear in Appendix Table III-A and values for the thermally aged and oxidation-corrosion samples appear in Appendix Tables III-B and III-C, respectively.

Standard accepted test procedures were used for each of the bench scale tests.

Determination of anti-wear properties by the Four-Ball Wear tester indicate that new OS-45 type fluids are somewhat better than the Oronite fluids, 8515 and 8200, in the steel-on-steel test. These values appearing in Appendix Table III-A also indicate that the Oronite fluids have better wear properties in the steel-on-bronze test. Wear values for two used OS-45 fluid samples from actual pump systems are nearly equivalent to those for new Oronite 8515. All four fluids subjected to the no air, no catalyst, 400°F. thermal aging exhibited only slight increase in steel-on-steel wear properties. This thermal aging improved the OS-45 steel-on-bronze values and depreciated the Oronite steel-on-bronze values.

Four-Ball Wear values on the fluid subjected to the MIL-H-8446 oxidation-corrosion test indicate a small deterioration in antiwear properties for all four fluids with the OS-45 fluids still equal or better than the Oronite fluids.

Wear tests conducted on the Almen machine gave relatively low wear values, but were variable. In general, OS-45 Type IV gave the best wear values at the highest loads with Oronite 8200, Oronite 8515, and OS-45 Type III following in the above order. It is significant that none of the fluids would complete the standard Almen Wear test of 4000 psi load for 20 minutes without seizure and welding of the specimens. In all cases, lower loads were required to complete the test. Values obtained for used OS-45 Type IV are considered to be equivalent to those for new fluid.

DSW 622064

Extreme pressure tests conducted on the Almen machine and the Timken machine indicate the OS-45 fluids to be somewhat superior to the Oronite fluids in load carrying ability. However, all values were low, indicating the fluids have only mild E.P. action. It is to be noted that all the bench scale lubricating tests used involve several configurations of sliding metal surfaces. The test which gave the most inconsistent and severe results was the Almen Wear test where the sliding surface is a relatively large area rather than a point or line contact. This type of configuration might lead to very thin films of a non-viscous lubricant or a lubricant starved condition where the abrasive or welding effect of very small wear particles would be more pronounced. Such a condition would be even more aggravated by lack of E.P. properties of the fluid.

A rolling type of test, such as would occur in a ball bearing, might give different results where fatigue failure resulting from low viscosity lubricants would be more pronounced. Another factor not studied is the effect of atmospheres other than air, such as might be encountered in an enclosed hydraulic system.

b. Thermal and Oxidative Stability

OS-45 Types III and IV, Oronite 8200 and Oronite 8515 were thermally aged for varying time intervals up to 480 hours at 400°F. in sealed glass tubes with a nitrogen atmosphere and no metal catalysts.

The glass containers consisted of 12-inch lengths of one-inch diameter pyrex gauge glass tubing sealed at one end and with a 5-inch length of one-half inch diameter gauge glass tubing on the other end. The volume of this container is approximately 90 cc, and only 70 cc of fluid was placed in each tube to allow sufficient vapor space for expansion. The tubes were cleaned with chromic acid, solvents, and distilled water and thoroughly dried before the fluid was introduced. The filled tubes were evacuated and then purged with nitrogen five to six times and then corked with a nitrogen atmosphere. After the tube was sealed off with a glass-blowing torch, it was placed into a length of 1-1/2 inch electrical conduit with a wire across each end to keep the tube from sliding out.

The schedule for placing the tubes into the oven was arranged so that each sample was introduced at such a time that all could be removed from the oven at one time. This was done so that they could be cooled in the oven to eliminate the shock of contacting room temperature and eliminate the handling of high temperature tubes which might have very high pressures. Each tube was placed in a 200°F. oven for about an hour before being placed in the 400°F. oven, a forced convection oven, to decrease the possibilities of thermal shock breakage. All heated tubes and tubes following thermal aging were handled within the metal conduit and with gloves and face shield until they were unsealed. Time intervals of 24, 48, 120, 192, 288 and 480 hours were chosen.

DSW 622065

The tubes were opened by applying a torch to the small sealed end. In all cases, there was a slight negative pressure in the tubes of the fluids tested.

Each fluid was evaluated at the successive aging interval for appearance, in specific gravity, refractive index, neutralization number, evaporation loss (ASTM D-972, 400°F., 6-1/2 hours), viscosity (100°F., 210°F.), foam (Microscale, 1/10 ASTM D-892), flash and fire points with a rerun of flash after the fire point, and Shell Four-Ball Wear. The values obtained appear in Table II.

The oxidation-corrosion test was conducted according to the procedure of military specification MIL-H-8446A. Apparatus for this test is described in Federal Test Method Standard No. 791, method 5308, and consists of a long glass tube fitted with an inlet tube and a reflux condenser. Temperature was maintained at 400°F. by means of a heated aluminum block bath, using Wood's metal as the heat transfer fluid. Dry air (water pumped breathing air) was passed into 100 ml. of the test fluid at a rate of 5 liters per hour for 72 hours at the test temperature of 400°F. The metal catalyst pieces of steel, aluminum, alloy and copper were tied in the form of a triangle and the silver specimen was suspended separately by means of a glass holder. After the test, the metal specimens were brushed and washed in solvents in a manner analogous to the procedure outlined in Federal Test Method Standard No. 791, method 3457.

In addition to the determination of the weight change and appearance of the metals, acidity change, viscosity change, and fluid appearance, aged fluids were checked for evaporation loss, foam, and Shell Four-Ball Wear. Values for these tests appear in Appendix Table III-C.

The most significant changes occurring from the static thermal aging were in acidity, evaporation loss, viscosity and, in the case of OS-45 Type IV, the appearance of a solid material. Only slight changes were noted in specific gravity, refractive index, foaming tendency, flash point, and fire point.

The total acid number of the OS-45 fluids increased from about 0.1 to 0.4 with the greatest increases occurring during the first 100 hours at 400°F. The Oronite fluids exhibited little acidity increase. Thermal aging had little effect on the evaporation loss for the Oronite fluids. OS-45 Type IV exhibited increased evaporation losses after about 100 hours of exposure, and OS-45 Type III had a very high evaporation loss of 40 to 45% at all stages of exposure.

This abnormally high loss as compared to previously published results (21.23%, WADC Technical Report 55-89, Part I) led to an evaporation loss check at several different temperatures. A plot of the evaporation loss versus temperature, Figure 1, indicates a break at about 365°F. Below this value the loss is

DSW 622066

probably due primarily to evaporation, and above is probably due to increased decomposition as well as evaporation. An extension of the lower portion to 400°F. would give an evaporation loss nearly equivalent to the WADC result. It is not known whether this difference in result is due to a difference in fluid stability or some characteristics of the respective evaporation loss equipment used by each laboratory.

The viscosity at 100°F and 210°F. of the OS-45 fluids decreased less than 10% and the Oronite fluids decreased from 30 to 50% in viscosity when subjected to the same 480 hour exposure at 400°F.

Static thermal aging of OS-45 Type IV resulted in the formation of dark sediment and grey-black, needle-like crystals one to three inches long. The solids began appearing at about 48 hours of exposure at 400°F. Similarly, the oxidation-corrosion test of OS-45 Type IV resulted in grey-black granular solids. In the first case the solids are believed to be selenium from the inhibitor, and in the second they are believed to be silver selenides since the silver catalyst was severely attacked.

In summary, these tests indicate that the oxidative and thermal decomposition products of the OS-45 type fluids are more volatile and have higher acidities than those from the Oronite fluids. The OS-45 fluids suffer less viscosity decrease than the Oronite fluids. The Four-Ball Wear of all fluids is increased only slightly from new fluid. The inhibitor used in OS-45 Type IV modifies the thermal and oxidative degradation, but results in a solid product and increased corrosion of copper and silver.

c. Service Life Limits

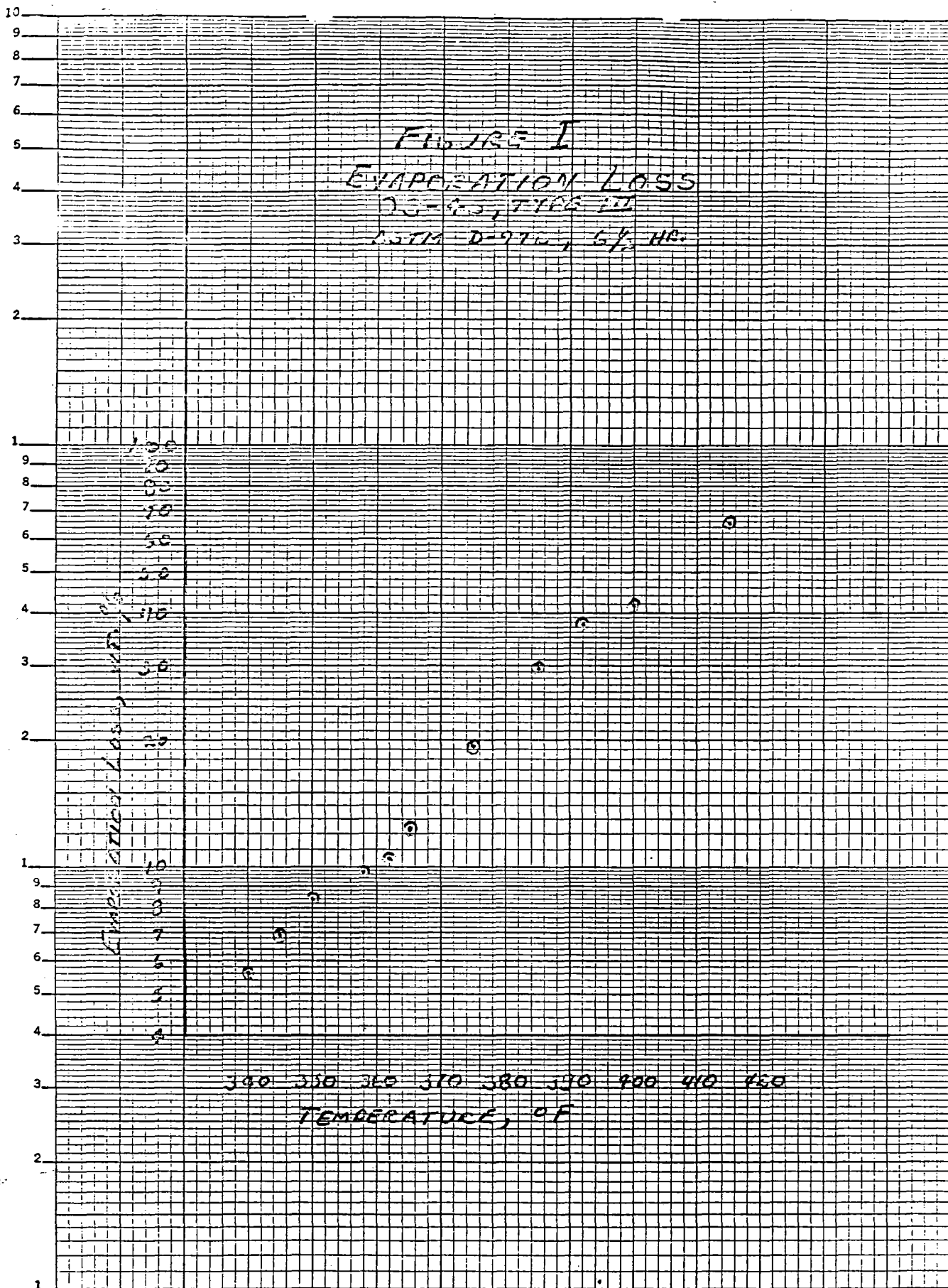
Several physical properties of the fluids were checked in an effort to find some property which would serve as a measure of the OS-45 type fluid useful life limit. Of these properties, acidity and viscosity are probably the most significant. The change in evaporation loss for OS-45 Type IV would indicate depletion of inhibitor. Changes in refractive index, specific gravity, foaming, wear and E.P. properties, flash points and fire points appear to be of little value due to the small amounts of change with thermal or oxidative exposure.

Viscosity changes for the OS-45 fluids were not excessive; however, any decrease of an already low viscosity fluid might result in lubrication and control difficulties in a hydraulic system. The shear tests illustrate that the fluids may decrease in viscosity without thermal or oxidative degradation.

Acidity increase of the OS-45 fluids is relatively large, especially under the oxidative conditions. This acidity, a total acid number of about six for the OS-45 fluids, does not result in excessive attack of catalyst metals used in the MIL-H-8446A oxidation-corrosion test.

DSW 622067

STLCOPCB4096098



Practical limits of viscosity and acidity were not established in this project and will be dependent on the requirements of an individual system.

d. Shear Stability

The two OS-45 type fluids and two Oronite fluids were subjected to ten passes through a diesel injector shear machine (General Motors Injector No. 5226710 operated at 1800 cycles per minute). Viscosities at 100°F. and 210°F. were measured on successive passes through the machine to determine the viscosity decrease profile for each fluid. Values for this test appear in Appendix Table III-D.

e. Miscellaneous

Viscosities at -65°F. and -40°F. for the OS-45 base stock, OS-59 (tetra(2-ethylhexyl)silicate), were run on two samples representing different sources of the OS-59 raw material, tetraethyl orthosilicate (TEOS). Cannon-Fensky viscosimeter tubes were used in a bath of controlled temperature. The low temperature viscosities for the two OS-59 samples prepared from John F. Queeny manufactured TEOS and Inorganic Division TEOS are listed below.

	<u>-40</u>	<u>-65</u>
OS-59 - John F. Queeny TEOS (A-189, Ba. #47)*	313.12 cs.	1383.3 cs.
OS-59 - Everett TEOS (NBP 194)	313.75 cs.	1322.7 cs.

* This material became slightly hazy on being cooled below +30°F.

Evaporation loss tests (ASTM D-972) were run on the OS-45 and Oronite fluids at 400°F. for 6-1/2 hours. Since values for OS-45 Type III differed considerably from results published by Wright Air Development Center (WADC Technical Report 55-89, Parts I, III and IV), additional tests were run at temperatures from 340°F. to 415°F. with the OS-45 Type III fluid and at 350°F. for the other fluids. These data are listed in Appendix Table III-E and OS-45 Type III data are illustrated graphically in Figure 1 of the discussion section.

Oxidation-corrosion tests were completed on samples of unused OS-45, Lot Z-163, and OS-45-1, Lot Z-487, which were returned from Wright Field where abnormally high copper attack by these two lots had been observed. Oxidation-corrosion results indicate that the copper weight loss of OS-45, Lot Z-163, is greater than for Lot A-414 which has been evaluated previously, but both are within the specification limits. Copper and silver weight loss for OS-45-1, Lot Z-487, are much greater than specification allowances but are equivalent to a previously evaluated lot, A-312.

DSW 622069

C. Skydrol 500

1. Reformulation to Eliminate Dermatitis

A severe dermatitis problem appeared with certain persons handling Skydrol 500. This was traced to the additive, phenyl glycidyl ether (PGE). Removal of PGE from the formulation resulted in pump corrosion. Both Douglas and Monsanto then embarked on an active program for screening replacement additives.

The 250°F., 168 hour oxidation-corrosion test was used in screening additives. Tests were run for purposes of correlation with Douglas, for determining the mechanism of the corrosion or the component responsible, and for general screening of additives. It was thought that the best indicators of additive activity were attack of copper, magnesium, and cadmium and changes in acidity. Viscosity and attack of aluminum and steel was not affected appreciably.

Correlation tests with Douglas stemmed from differences in results with a formulation containing the inhibitor, Ortholeum 300. These differences were never completely resolved for this formulation. Correlation work included the evaluation of a batch of FH-135 (base stock with VI improver) both at this laboratory and at Douglas. In addition, metal specimens were supplied by Douglas for our use. At the time this work was being done, we were reusing the best metal specimens. Recycle of the Douglas specimens indicated no appreciable changes on reusing. It was concluded that our oxidation-corrosion tests were essentially equivalent to Douglas' and that discrepancies with the Ortholeum formulation were due to the nature of this additive at the particular concentration used. Oxidation-corrosion test correlation results appear in Table XXII.

During this correlation work, it was found that differences existed between the two lots of FH-87 (Ba. 15-16 and Ba. 25-26) in the oxidation-corrosion test. This difference was traced to water-soluble salts. FH-87, Ba. 15-16, with 40 ppm water-soluble salts gave abnormally high metal attack; whereas, Ba. 25-26 with 8 ppm water-soluble salts gave normal results. Skydrol 500 prepared from either of these two FH-87 batches gave good oxidation-corrosion results with the Ba. 25-26 performing slightly better.

Several tests were run to determine the mechanism of, or the component responsible for, metal attack in the Skydrol 500 oxidation-corrosion tests. In addition to the corrosion contributed by FH-87 with high water-soluble salts indicated above, it was found that the Acryloid viscosity index improvers with higher than normal acidity contributed substantially to this attack. A run was made in which adsorption alumina was used with the poor FH-87 (Ba. 15-16) as well as high acidity Acryloid.

DSW 622070

TABLE XXII

SKYDROL 500

DACO-MCC TEST CORRELATION

Test No.	Fluid	Metal Attack, mg./cm. ²			Acidity, TAN	
		Cu	Mg	Cd/Fe	Initial	Final
DACO II-6	FH-135 (B-2069), 2% Ortholeum 300	0.34	0.03	0.01	---	---
DACO III-4	FH-135 (B-2069), 2% Ortholeum 300	+0.05	0.06	0	---	---
23-6	FH-135, 2% Ortholeum 300	0.79	0.03	0.01	0.124	0.884
DACO I-1	FH-135 (B-2069)	15.6	0.60	0.19	---	---
DACO II-1	FH-135 (B-2069)	---	0.63	0.17	---	---
20-1	FH-135 (B-2069)	17.9	0.467	0.059	0.06	3.53
DACO I	FH-87 (Ba 15-16)	6.94	0.071	0.09	0.06	2.27
DACO II	FH-87 (Ba 15-16)	5.91	0.057	0.07	0.06	1.96
15-1	FH-87 (Ba 15-16)	23.1	0.029	0.059	0.03	5.34
18-1	FH-87 (Ba 15-16)	16.2	0.089	0.044	0.03	3.36
20-2	FH-87 (Ba 25-26)	6.73	0.022	0.059	0.01	1.44
28-4	FH-87 (Ba 15-16) DACO metals	11.26	0.037	0.022	0.03	2.22
29-5	FH-87 (Ba 15-16) reused DACO metals	12.12	0.111	0.166	0.03	2.91
28-5	FH-87 (Ba 25-26) DACO metals	5.61	0.015	0.022	0.01	1.36
29-6	FH-87 (Ba 25-26) reused DACO metals	7.68	0.059	0.111	0.01	2.12

DSW 622071

Relatively low metal attacks and acidity increases were encountered indicating initial acidity or acidity contributed by base stock and viscosity index improver decomposition were the prime offenders. Acid scavenging type of inhibitors were thought to be desirable. These oxidation-corrosion test results appear in Table XXIII.

A large number of potential inhibitors were screened in FH-135. Those showing some promise included tetraphenyl tin, tetrabutyl tin, triphenylstibine, and epoxy 2-ethylhexyl tallate. This latter additive appeared to be the most effective both at a 1.0% concentration alone or in combination with 0.5% FH-132 (bis(1,2-phenylmercapto)ethane) which is normally used in the Skydrol 500 formulation. Several commercially available epoxidized fatty acid esters widely used as plasticizer stabilizers were evaluated with equivalent good results. Paraplex G-62 (Rohm and Haas) reported to be epoxidized soya oil was chosen for further evaluation based on its availability and reported low toxicity, being approved for use in food wrapping products.

Concentration studies with Paraplex G-62 indicate slightly increased copper attack with decreasing amounts below 1.0%. The use of FH-132 with the Paraplex G-62 had little effect except at lower concentrations of the epoxidized inhibitor. At 0.2% G-62 and 0.5% FH-132, magnesium is attacked more than with G-62 alone. Oxidation-corrosion test results for the promising inhibitors and the variations of the Paraplex G-62 formulation appear in Table XXIV and additives of little promise are tabulated in Table XXV.

Both the Ortholeum 300 (2.0%) containing formulation, OS-105, and the Paraplex G-62 (1.0%) version without FH-132 appeared to be non-irritating or skin sensitizing. On the basis of full scale pump tests at Douglas, it was decided to include 0.5% FH-132 in the formulation. This Paraplex G-62, FH-132 inhibited fluid was subsequently adopted as Skydrol 500A. Further work on this project was done under the new job number 3767.

2. Viscosity Index Improver

A proposed change in the Rohm and Haas Acryloid R-3876X viscosity index improver prompted a project for its evaluation. In the proposed Rohm and Haas VI improver process, the polymerization is conducted in Skydrol 500 base stock (FH-87) rather than in a solvent which is subsequently replaced by FH-87. Oxidation-corrosion tests on this new style Acryloid in the Skydrol 500 formulation and in FH-87 displayed increased metal attack over similar blends using the current VI improver.

To further clarify the cause of the increased metal attack, Rohm and Haas subjected FH-87 to their proposed polymerization procedure time and heat cycle, but without the presence of monomer or catalyst. A second sample was treated similarly, but with the polymerization catalyst present. These samples were subjected to the oxidation-corrosion test as is and at 12% concentration in untreated FH-87. These tests indicated increased metal attack from

the polymerization treatments and especially with the sample which included the catalyst. This information was returned to Rohm and Haas for further suggestions. Further work on this project was done under Job Number 3767.

Table XXVI presents oxidation-corrosion test results for the proposed Skydrol 500 viscosity index improver and for the various batches of treated FH-87.

D. Silicate Weapons Lubricant

A proposed silicate weapons lubricant, OS-94 (93.8% mixed tetra(2-ethylbutyl, 2-ethylhexyl)silicate, /FH-134/, 0.2% Phenyl alpha-naphthylamine, 6.0% Na-Sul BSN), failed to meet humidity cabinet rust tests specified by MIL-L-14107 at Rock Island Arsenal (R.I.A.). The qualification test resulted in failure at 264 hours, whereas, results at Douglas indicated the panels to be rust-free in excess of the minimum 400 hours for several batches of OS-94 and with several sets of metal specimens. Likewise, our humidity cabinet test results confirmed the results at Douglas.

After discussing the discrepancies with R.I.A., it was decided that they would repeat the test and provide us with some of their specimens for comparison with ours. Our test with their specimens confirmed their initial results. It was found that the steel panels which had been used were not cold rolled steel as specified for the humidity cabinet.

After humidity cabinet failure of OS-94 was confirmed, two alternate formulations prepared by L.W. Bannister were tested. One of these, OS-94 with an additional 0.3% phenyl alpha-naphthylamine (PAN), resulted in panel protection of 287 hours to 480 hours on three panels. The other formulation consisting of 92.0% FH-134 silicate base stock, 7.5% Bray's Synphonate 28, and 0.5% PAN gave protection for 552 to 672 hours. This latter formulation, designated OS-106, was subjected to the remaining MIL-L-14107 specification tests with acceptable results.

OS-106 was submitted for qualification testing but failed in the hydrolytic stability test. Since the market for lubricants of this type was diminished since the program was initiated, it was decided to terminate the project.

Several other preservative additives were screened in FH-134. Materials which provided more than 400 hours protection were 5% (w) N-7797 (R.P. of tetrapropenyl succinic anhydride (TPSA) and 2-methyl 2,4-pentanediol), 5% (w) Giegy NPA, and 5% Nirus N.P. (Synthetic Chemicals Corporation). Since this project was discontinued, no further work was done with these promising materials. Additives which did not give adequate protection in FH-134 follow:

DSW 622073

5% Santolube 203-C, 0.5% PAN
 5% Santolube 203-C
 5% Santolube 70, 0.5% PAN
 5% Santolube 70
 2-1/2% RT-53A, 2-1/2% RT-67A, 0.5% PAN
 2-1/2% RT-53A, 2-1/2% RT-67A
 5% Amine ODT, 0.5% PAN
 5% Amine ODT
 5% Alox 1724
 5% Alox 350 (insoluble)
 5% Alox 600 (insoluble)
 5% Priminox 43
 5% N,N,N',N'-tetrakis(2-hydroxypropyl)ethylenediamine
 (insoluble)

Table XXVII presents MIL-L-14107A specification test results run on OS-106.

E. Skydrol 7000 - Low Temperature Viscosity

Skydrol 7000 viscosities of production lots were reported to be near the mid-range at 210°F. and near the upper limit at 100°F. A question arose concerning the viscosity at -40°F. Viscosities for batches 44 and 45 of Skydrol 7000 indicated values 4 to 6% higher than the expected value of 7000 cs. This would indicate that low temperature viscosities as well as 100°F. viscosities are increased for batches with normal 210°F. viscosities.

	-40°F	Viscosity, cs. 100°F.	210°F.
Skydrol 7000, Ba 44	7302.	15.64	3.97
Skydrol 7000, Ba 45	7434.	16.17	4.01
Specification Limits	---	15.5-16.0	3.96-4.04

III. Miscellaneous

A. ASTM Cooperative Test Work

Laboratory tests were conducted on ASTM reference fluids supplied by Technical Committee N on Hydraulic Fluids of ASTM Committee D-2 for cooperative testing. These tests included Flash point, Fire point, High Pressure Spray Flammability, Low Pressure Spray Flammability, Hot Manifold Flammability, Four-Ball Wear, Almen E.P., D-943 Oxidation, and Oxidation-Corrosion tests. Fluids tested were designated as follows:

1-A-1	Petroleum oil
2-B-1	Water base
3-C-1	Phosphate ester
4-D-1	Halogenated base
5-D-1	Phosphate base
6-E-1	Oil emulsion
7-F-1	Silicate ester
8-G-1	Silicone

DSW 622074

Procedures and test results for these fluids appear in the Appendix.

B. Synthetic Lubricants for Refrigeration Units

A large potential use of synthetic lubricants in the refrigeration field prompted some preliminary work to establish the level of lubricating qualities desirable in such fluids.

A series of four-ball wear tests were run on Suniso 3-G, the refrigeration compressor fluid presently in use by Hussmann Refrigerator Company. Similar test data from Mobil D.T.E. fluid and Pydraul F-9 fluid is included in the chart below. The comparative data indicates that Suniso 3-G is inferior to both Pydraul F-9 and Mobil D.T.E. in lubricating qualities.

Test Conditions: 630 RPM

167°F.

1 hour duration

Fluid	Steel-on-steel			Steel-on-bronze
	4 kg	10 kg	40 kg	10 kg
Suniso 3-G	0.38	0.62	0.60	2.32
Pydraul F-9	0.22	0.28	0.63	1.18
Mobil D.T.E.	0.18	0.24	0.38	1.48

C. Low Temperature Viscosity Study

Samples of Skydrol 500 were checked for -40°F. and -65°F. viscosities for comparison with results obtained at Douglas Aircraft Company on the same samples. This was done to check our low temperature viscosity procedure.

Our results differed from the mean by about 0.75%, which is within the 1.0% reproducibility limit for temperatures below 60°F., as specified by ASTM D-445. These results are tabulated below:

	DACO	REL
	-65°F. Viscosity	-65°F. Viscosity
<u>Skydrol 500</u>		
Old Acryloid - Lot A-2762	2,470 cs.	2,489 cs.
New Acryloid - Lot A-2763	2,540 cs.	2,559 cs.

D. Lubricating Study - Paraffinic Petroleum Oil

A paraffinic petroleum oil base stock was treated with fine iron filings for 24 hours at room temperature to determine the effects of such treatment on the oil's wear properties. It was found that this did not change the wear properties. Four-ball wear tests were run with solvent washed balls and distilled water and jewelers rouge cleaned balls with no significant difference. The

DSW 622075

complete removal of all iron particles was found to be important since simple filtration by a filter paper left minute particles which led to gross wear.

Oil: Elk SAE 30 Base Stock, Inv. 2069.

Iron: Fisher Cat. No. 1-57, about 40 mesh filings.

Treatment: 250.7 gm. oil and 50.0 gm. iron stirred 24 hours at room temperature in a 1-liter round-bottom flask.

4-Ball Wear, 1250 rpm, 2 hours, 167°F., steel-on-steel, 40 kg.:

Inv. 2069-no treatment	0.91 mm.
Inv. 2069-Dicalite filtered	0.94
Inv. 2069-iron treated, filtered once through filter paper	1.05 (seizure in 5 seconds)
Inv. 2069-iron treated, filtered through Dicalite coated filter	0.93
Inv. 2069-iron treated, filtered through Dicalite coated filter	0.97*

* Steel balls used in this test cleaned with distilled water and jewelers rouge and others cleaned with Skellysolve B and acetone.

E. Compressor Lubricant - Four-Ball Wear

Shell Four-Ball Wear values were obtained on compressor lubricants to assist in a problem at the Texas City plant which involved lubrication of the seals and rods of reciprocating compressors. This information was used by Texas City to outline a lubricant test program. No further work was done on this project.

Four Ball Wear: 630 rpm, 167°F., 1 hour, steel-on-bronze.

	<u>4 kg.</u>	<u>10 kg.</u>	<u>40 kg.</u>
Coastal Pale Oil	0.90 mm.	1.49 mm.	2.17 mm.
Glycerine	0.48	0.75	2.48
Humble White Oil 335	0.61	1.34	2.80
Ramol	0.59	1.80	2.25

L. R. Stark

R. E. Hatton

de
9-23-60

DSW 622076

TABLE I

FIRE RESISTANT FLUID SCREENING
PHYSICAL AND FIRE RESISTANT PROPERTIES

Fluid	Viscosity, cs.					ASTM Slope						
	0°F.	20°F.	25°F.	40°F.	75°F.	100°F.	210°F.	20°F.- 100°F.	40°F.- 100°F.	75°F.- 100°F.	100°F.- 150°F.	150°F.- 210°F.
Chlorinated diphenyl ether	----	----	----	----	6730.	475.	6.31	----	----	1.57	1.37	1.19
Polybutylated Aroclor 1221	----	----	----	----	3150.	496.	10.1	----	----	1.05	1.14	1.02
Isopropyl Aroclor 1242	----	----	----	----	5098.	529.	8.06	----	----	1.32	1.24	1.12
n-Butyl Aroclor 1232	----	----	6050.	----	----	51.5	4.21	----	1.09	----	1.04	0.98
Chlorinated dodecylbenzene	4230.	----	----	----	----	20.9	3.11	0.99	----	----	0.95	0.92
Aroclor 1242	----	6790.	----	----	----	18.1	2.46	1.41	----	----	1.10	1.01
Aroclor 1248	----	----	----	6420.	----	58.4	5.48	----	1.33	----	0.93	0.87
Aroclor 1254	----	----	586.	----	6490.	421.	5.86	----	----	1.61	1.40	1.19
Dodecylbenzene	----	----	----	----	6.45	4.94	1.76	----	----	----	----	----

Fluid	Fire Point C.O.C. °F.	Flash Point C.O.C. °F.	Molten Metal Ignition (1)	
			Ignites spontaneously	Ignites with flame
Chlorinated diphenyl ether	525	>760	No	Slight Flash
Polybutylated Aroclor 1221	405	465	Yes - burns	Yes - burns
Isopropyl Aroclor 1242	435	540	No	Yes - burns
n-Butyl Aroclor 1232	400	465	No	Yes - burns
Chlorinated dodecylbenzene	350	370 (2)	Yes - burns	Yes - self extinguishing
Aroclor 1242	350	None	No	Slight Flash
Aroclor 1248	360 (2)	None (2)	No	Slight Flash
Aroclor 1254	None (2)	None (2)	No	Slight Flash
Dodecylbenzene	----	----	----	----

(1) aluminum alloy at 1200 to 1300°F.

(2) No flash or fire point up to boiling point of fluid (about 700-735°F.).

DSW 622077

TABLE II

FIRE RESISTANT FLUID SCREENING
OXIDATION-CORROSION, 250°F., 168 HOURS

	Isopropyl Triaryl Phosphate	Tert. Butyl Triaryl Phosphate	Tricresyl Phosphate
<u>Metal attack: mg./cm.²</u>			
Aluminum	0 Clear	0 Clear	0 Clear
Magnesium	0 Clear	+0.02 Lt. Grey St.	0 Lt. Grey St.
Copper	-0.51 Dk. Red-Green Stain	-0.04 Few Lt. Pit	0 Dull copper
Steel	0 Clear	-0.02 Clear	0 Clear
Cadmium Plate	0 Clear	-0.03 Clear	+0.01 Clear
<u>Acidity:</u>			
Initial, TAN	0.17	0.01	0.186
Final, TAN	3.45	0.01	0.215
Change			
<u>Viscosity:</u>			
100°F., Initial, cs.	55.99	49.3	34.8
Final, cs.	107.0	49.0	35.7
210°F., Initial, cs.	5.21	----	----
Final, cs.	7.42	----	----
<u>Fluid Condition:</u>			
	Yellow-brown opaque 1.3% wt. loss	Yellow, clear 2.0% wt. loss	Med. amber clear -1.0% wt. loss

DSW 622078

TABLE III

FIRE RESISTANT FLUID SCREENING
LUBRICATING PROPERTIES

Fluid	Four-Ball Wear, mm.					Almen EP		
	Steel-on-steel		Steel-on-bronze			Passing Load, lb.	Torque, lb.	
	4 Kg.	10 Kg.	40 Kg.	4 Kg.	10 Kg.			40 Kg.
Isopropyl triaryl phosphate	0.33	0.43	0.52	0.62	0.66	1.10	48	50
Tert. butyl triaryl phosphate	----	0.44	----	----	----	----	--	--
Tricresyl phosphate	0.32	0.38	0.56	----	----	----	52	54

DSW 622079

STLCOPCB4096110

TABLE IV

HIGH AROCLOR BASED FLUIDS
WITH ACRYLOID VISCOSITY INDEX IMPROVERS

Formulation No.	Fluid	Viscosity					4-Ball Wear, mm.**	
		Initial		Sheared*				
		100°F.	210°F.	V.I.	100°F.	210°F. V.I.		
---	Pydraul F-9	57.4	5.88	13.7	48.1	4.70	-106	0.28
---	Aroclor 1242	18.1	2.46		--	--	--	0.38
---	Aroclor 1248	43.0	3.20		--	--	--	0.39
---	Aroclor 1254	420.8	5.86		--	--	--	--
---	Cellulube 220	47.0	4.77		46.5	4.70	--	0.33
1	Aroclor 1242, Acryloid 710 (4%) ***	40.6	5.60	77	27.0	3.65	- 78	--
2	Aroclor 1242, Acryloid HF-855 (4%)	34.2	4.64	20.	29.7	3.97	- 51	--
3	Aroclor 1242 (40%), Aroclor 1248 (60%)	29.4	2.81	-82	--	--	--	--
4	Aroclor 1242 (40%), Aroclor 1248 (60%), Aroclor 1248 (60%), Acryloid 710 (4%)	62.4	6.49	38	--	--	--	0.40
5	Acryloid 1242 (40%), Aroclor 1248 (60%), Acryloid HF-855	53.3	5.52	-1	--	--	--	0.56
6	Aroclor 1248 (70%), Aroclor 1242 (30%)	32.4	2.90	--	--	--	--	--
7	Aroclor 1248 (67%), Aroclor 1242 (29%), Acryloid HF-855 (4%)	58.3	5.67	--	49.4	4.79	--	--
8	Aroclor 1242 (60%), Aroclor 1254 (40%)	40.9	3.02	--	--	--	--	--
9	Aroclor 1242 (59%), Aroclor 1254 (39%), Tricresyl phosphate (TCP) (2%)	41.3	3.05	--	--	--	--	0.30

* 12 passes through diesel injector
 ** 600 rpm, 1 hour, 10 KG, 167°F., 52-100 steel
 *** Formulation values are percent weight.

DSW 622080

TABLE IV (continued)

Formulation No.	Fluid	Viscosity				4-Ball Wear, mm. **
		Initial		Sheared*		
		100°F.	210°F.	V.I.	% Loss 100°F. 210°F. V.I.	
10	#9 (98.4%), Acryloid 710 (1.63%)	57.6	4.53			
11	#9 (97.1%), Acryloid 710 (2.9%)	66.3	5.75			
12	Aroclor 1248 (94%), TCP (2%), Acryloid 710 (2%), Acryloid HF-855 (2%)	75.2	5.83			
13	#3 (93.5%), TCP (3%), Acryloid 710 (3.5%)	57.07	5.88	--	--	0.26
14	Aroclor 1248 (76%), Aroclor 1242 (19%), Acryloid 710 (3%), Acryloid HF-855 (2%)	71.8 74.4	6.43 6.69			
15	#14 (98%), TCP (2%)	26.3	4.24			
16	Aroclor 1242 (96%), Acryloid HF-855 (4%)					
17	Aroclor 1248 (72%), Aroclor 1242 (24%), Acryloid 710 (2%), Acryloid HF-855 (2%)	65.95 65.8	6.06 6.05			
18	#17 (98%), TCP (2%)					
19	Aroclor 1248 (68%), Aroclor 1242 (27%), Acryloid 710 (2%), Acryloid HF-855 (2%), TCP (1%)	65.2	6.09	--	--	0.37
20	Aroclor 1248 (65%), Aroclor 1242 (30%), Acryloid 710 (2%), Acryloid HF-855 (2%), TCP (1%)	63.1	6.04			
21	Aroclor 1248 (60%), Aroclor 1242 (35%), Acryloid 710 (2%), Acryloid HF-855 (2%), TCP (1%)	60.1	5.88			
22	Aroclor 1248 (57.8%), Aroclor 1242 (36%), Acryloid 710 (2.2%), Acryloid HF-855 (2%), TCP (2%)	63.3	6.3			0.35

* See Footnotes on first page of table.

DSW 622081

TABLE IV (continued)

Formulation No.	Fluid	Viscosity				4-Ball Wear, mm. **
		Initial		Sheared*		
		100°F.	210°F.	V.I.	% Loss 100°F. 210°F. V.I.	
23	Aroclor 1242 (96%), Acryloid HF-825 (4%)					0.48
24	Aroclor 1248 (57%), Aroclor 1242 (37%), Acryloid 710 (2%), Acryloid HF-855 (2%), TCP (2%)	53.3	5.2			
25	Aroclor 1248 (56.9%), Aroclor 1242 (37%), Acryloid 710 (2.1%), Acryloid HF-855 (2%), TCP (2%), #25 (98%), Santolube 393 (2%)	58.9	5.89			
26	Aroclor 1248 (93.5%), Acryloid HF-825 (3.5%), TCP (3%)	64.1	4.75			0.26
27	Aroclor 1248 (55.9%), Aroclor 1242 (38%), Acryloid 710 (2.2%), Acryloid HF-855 (2%), TCP (2%)	60.2	6.01	11.2		0.29
28	Aroclor 1248 (55.4%), Aroclor 1242 (37.5%), Acryloid 710 (2.2%), Acryloid HF-855 (2%), TCP (3%)	60.1	6.01	11.5		0.27
29	Aroclor 1248 (64.7%), Aroclor 1242 (30.0%), Acryloid 710 (1.0%), Acryloid HF-855 (3.3%), Santolube 393 (1%)	55.7	5.57		43.8 4.32	0.58
30	Aroclor 1248 (65%), Aroclor 1242 (30%), Acryloid HF-860 (3%), Santolube 393 (1%)	49.4	4.81		37.95 3.82	
31						

* See Footnotes on first page of table.

DSW 622082

TABLE V

HIGH AROCLOR FLUID FORMULATION
UCON VISCOSITY INDEX IMPROVERS

Formulation No.	Formulation ***	Viscosity				
		Kinematic centistokes		Saybolt Seconds		
		100°F.	210°F.	V.I.	Kin. 100°F.	Abs.* 100°F.
1	Aroclor 1242 97.5%	34.2	3.99	neg.	160	219
2	Ucon 75-H-90,000 2.5%					
	Aroclor 1242 96%	30.98	3.67	neg.	146	199
3	Ucon 50-HB-5100 4%					
	Aroclor 1232 95%	17.8	3.48	68	88	120
4	Ucon 75-H-90,000 5%					
	Aroclor 1232 92%	18.6	3.6	73	92	---
5	Ucon 50-HB-5100 8%					
	Aroclor 1242 36%, Aroclor 1232, 60%	23.8	3.98	46	114	
6	Ucon 75-H-90,000 4%					
	Aroclor 1242 50%, Aroclor 1232 45%,	33.1	5.04	80	155	
7	Ucon 75-H-90,000 5%					
	Aroclor 1242 52%, Aroclor 1232 43.5%	30.7	4.69	62	144	198
8	Ucon 75-H-90,000 4.5%					
	Aroclor 1242 54%, Aroclor 1232 35.7%	31.6	4.72	58	149	
9	Ucon 75-H-90,000 4.3%					
	Aroclor 1242 54%, Aroclor 1232 41.6%	30.0	4.54	49	142	
10	Ucon 75-H-90,000 4.4%					
	Aroclor 1242 65%, Aroclor 1232 31%	31.3	4.48	25	147	194
10	Ucon 75-H-90,000 4%	31.6	4.49			
11	Sheared 12 times****					
	Aroclor 1242 63%, Aroclor 1232 30%	29.6	4.26	8	140	183
11	Ucon 50-HB-5100 7%	29.9	4.28			
	Sheared 12 times					

DSW 622083

TABLE V (continued)

Formulation No.	Formulation***	Viscosity			
		Kinematic		Saybolt Seconds	
		centistokes		Kin.	
		100°F.	210°F.	V.I.	Abs.*
12	Aroclor 1242 67%, Aroclor 1232 29%, Ucon 75-H-90,000 4%			100°F.	100°F.
13	Aroclor 1242 67%, Aroclor 1232 28.9%, Ucon 75-H-90,000 4% Santolube 393 0.1%			100°F.	100°F.

Refer to Table VI for continuation of the series of formulations based on formulation number 12.

* Determined by product of SUS at 100°F. and 100°F. density.

** 167°F., 620 rpm, 1 hour, 52-100 steel-on-steel

***Quantities are by weight

****12 passes through REL Diesel Injector Shear Machine.

DSW 622084

STLCOPCB4096115

TABLE VI

HIGH AROCLOR FLUID FORMULATION
UCON BLEND LUBRICATING PROPERTIES

Formulation based on approximately
Aroclor 1242 67%, Aroclor 1232 29%, and
Ucon 75-H-90,000 4%

Blend Number	Santolube 393 %	Other Additive %	Almen		4-Ball Wear *
			Load Pounds	Torque Pounds	
12	---	---	24	23	0.56
13	0.1	---	23	26	0.54
14	0.3	---	49	50	---
15	0.5	---	44	58	---
16	0.3	---	24	35	0.55
17	0.3	0.5% Santolube 70	24	28	0.40
18	0.3	1.0% Aloxx 350	40	60	0.53
19	0.3	0.2% Santolube 394-C	26	23	---
20(a)	0.3	0.2% Oleic Acid	46	60	---
20(b)	0.3	0.2% Dioctyl malate (DOM)	22	30	0.56
21(a)	0.3	0.3% DOM	40	40	---
21(b)	0.3	0.1% N-30	36	36	0.54
22	0.3	0.2% N-30	50	52	0.44
23	0.3	0.1% SL-5270 **	40	43	0.56
24	0.3	0.2% ST-35	44	41	---
25	---	0.2% p-nitrochlorobenzene (PNCB)	36	48	---
26	---	0.4% G58-797 **	28	32	---
27	0.3	0.3% G58-797	40	56	---
28	0.3	0.2% RI-113	28	32	---
29(a)	0.3	0.1% SL-5270; 0.05% Santolube 70	44	40	---
29(b)	0.4	0.1% SL-5270; 0.1% Aloxx 350	48	60	0.54
30	0.3	0.1% SL-5270; 0.1% Aloxx 350	36	40	---
31(a)	0.3	0.1% Oleic Acid	26	30	---
31(b)	0.4	0.1% Santolene C	44	60	0.57
32	0.3	0.1% Santolene C	20	24	---
33(a)	0.3	0.1% Aloxx 350	20	24	---
33(b)	0.5	0.5% tricresyl phosphate (TCP)	20	24	---
33(c)	0.6	0.1% Aloxx 350	46	43	---
		0.5% TCP, 0.1% Aloxx 350	50	40	0.57
		0.6% TCP; 0.1% Aloxx 350			

* 40 Kg., 1 hour, 167°F., steel-on-steel

** Experimental gear oil additive

*** Squealing during Almen E.P. test

DSW 622085

TABLE VI (continued)

Blend Number	Santolube 393 %	Other Additive %	Almen Load		4-Ball Wear *
			Load Pounds	Torque Pounds	
53(a)	0.5	0.1% SL-5266; 0.15% Sant. 394-C	46	52***	--
53(b)	0.6	0.1% SL-5266; 0.15% Sant. 394-C	44	55***	--
54(a)	0.4	0.1% Oleic Acid	44	60***	--
54(b)	0.5	0.1% Oleic Acid	44	56	--
55(a)	0.3	1.0% A-2290; 0.1% Sant. 394-C	46	40	--
55(b)	0.4	1.0% A-2290; 0.1% Sant. 394-C	30	27	--
56(a)	---	1.0% Alox 350	14	22	--
56(b)	0.2	1.0% Alox 350; 0.1% Sant. 394-C	18	27	--
56(c)	0.4	1.0% Alox 350; 0.1% Sant. 394-C	38	50	--
57(a)	0.5	1.0% Alox 350	36	52	--
57(b)	0.6	1.0% Alox 350	44	52***	--
57(c)	0.8	1.0% Alox 350	40	50***	--
58(a)	0.5	0.5% A-2290; 0.5% Alox 350	42	60***	--
58(b)	0.6	0.5% A-2290; 0.5% Alox 350	44	52***	--
58(c)	0.7	0.5% A-2290; 0.5% Alox 350	50	36	0.44
59	1.0	0.5% Alox 350	50	36	0.49

DSW 622086

TABLE VII

HIGH AROCLOR FLUID FORMULATION

Formulation Number	Formulation (% wt.)	Kinematic Viscosity Centistokes		Saybolt Seconds	
		100°F.	210°F.	Kin. 100°F.	Abs. 100°F.
1	55% Aroclor 1248, 45% Aroclor 1242	27.6	----	130	183
2	18% Aroclor 1254, 82% Aroclor 1242	24.7	----	117	166
3	78% Aroclor 1248, 22% Aroclor 1232	22.6	----	108	150
4	61% Aroclor 1248, 39% Aroclor 1242	29.0	----	---	193
5	20% Aroclor 1254, 80% Aroclor 1242	25.7	----	---	---
6	81% Aroclor 1248, 19% Aroclor 1232	24.30	----	---	---
7	65% Aroclor 1248, 35% Aroclor 1242	30.17	----	142	200
8	27% Aroclor 1254, 73% Aroclor 1242	29.6	----	---	---
9	88% Aroclor 1248, 12% Aroclor 1232	31.3	----	---	---
10	66% Aroclor 1248, 33% Aroclor 1242, 0.5% Santolube 393, 0.5% Santolube 70, 100 ppm. DCF-200	30.22	2.87	142	200

DSW 622087

STLCOPCB4096118

TABLE VIII.

OS-95 COMPATIBILITY LUBRICATING PROPERTIES

Fluid (% Vol.)	Align Load		Four-Ball Wear (620 rpm, 167°F, 1 hr.)		
	Pounds Load	Pounds Torque	Steel-on-Steel		Steel-on-Bronze 10 Kg.
			10 Kg.	40 Kg.	
100% OS-95	48	41	0.29 mm.	0.43 mm.	1.40
90% OS-95, 10% Pydraul F-9	47	41	0.23 mm.	0.50 mm.	----
75% OS-95, 25% Pydraul F-9	38	43	0.22 mm.	0.55 mm.	----
25% OS-95, 75% Pydraul F-9	50	45	0.22 mm.	0.63 mm.	----
10% OS-95, 90% Pydraul F-9	50	47	0.22 mm.	0.68 mm.	----
100% Pydraul F-9	50	44	0.28 mm.	0.67 mm.	1.18
100% OS-95	48	41	0.29 mm.	0.43 mm.	1.40
90% OS-95, 10% Cellulube 220	43	53	0.25 mm.	0.53 mm.	----
75% OS-95, 25% Cellulube 220	37	54	0.23 mm.	0.58 mm.	----
25% OS-95, 75% Cellulube 220	50	55	0.28 mm.	0.58 mm.	----
10% OS-95, 90% Cellulube 220	50	52	0.30 mm.	0.48 mm.	----
100% Cellulube 220	27	35	0.33 mm.	0.54 mm.	0.81
100% OS-95	48	41	0.29 mm.	0.43 mm.	1.40
90% OS-95, 10% Houghto-Safe 1120	40	44	0.24 mm.	0.50 mm.	1.57
75% OS-95, 25% Houghto-Safe 1120	36	56	0.22 mm.	0.55 mm.	1.47
25% OS-95, 75% Houghto-Safe 1120	49	57	0.31 mm.	0.57 mm.	1.12
10% OS-95, 90% Houghto-Safe 1120	50	47	0.37 mm.	0.51 mm.	0.98
100% Houghto-Safe 1120	50	51	0.34 mm.	0.56 mm.	0.90

DSW 622088

TABLE IX

EVALUATION OF USED OS-95

Chevrolet - Bay City Division - 260 Hrs. (14 hrs. at 190°F.)

	<u>OS-95 New</u>	<u>OS-95 Chev.-Bay City Div., 260 Hr.</u>
<u>Viscosity</u>		
100°F., cs.	30.63	31.8
210°F., cs.	2.89	3.05
<u>4-Ball Wear (630 RPM, 1 Hr., 167°F.)</u>		
Steel-on-Steel		
4 kg.	.21	.20
10 kg.	.29	.26
40 kg.	.43	.59
Steel-on-Bronze		
4 kg.	1.20	.95
10 kg.	1.40	1.48
40 kg.	1.70	1.69
<u>Neutralization Number</u>	0.82 TAN	0.82 TAN
<u>Rust Test (REL, F-101, 10.0=Perfect)</u>	10.0	10.0
<u>Appearance</u>	Yellow, slight cloudy	Reddish- brown-nearly opaque

DSW 622089

TABLE X

OS-104 FORMULATION, LUBRICATING PROPERTIES AND THERMAL STABILITY

Fluid	Four-Ball Wear (1)			Almen E.P.		Static Heat Test (2)
	4 kg.	10 kg.	40 Kg.	Load Lbs.	Torque Lbs.	
Triphenyl phosphate	0.40	0.44	0.74	--	--	----
Cresyl diphenyl phosphate (CDP)	0.38	0.42	0.65	2	8	----
Tricresyl phosphate (TCP)	0.32	0.38	0.56	50	54	----
2-Ethylhexyl diphenyl phosphate	0.30	0.31	0.55	50	48	Decomposes at 500°F.
Dibutyl phenyl phosphate	--	0.39	--	21	32	----
CDP/TCP: 70/30%	0.37	0.43	0.57	37	46	Turns light brown
CDP/TCP: 70/30% with 0.25% Diocetyl malate	--	0.41	--	35	44	----
CDP/TCP: 70/30% with 0.5% RT-67-A Dimer acid	--	0.31	--	10	22	----
OS-104	0.31	0.34	0.60	50	55	Turns light brown

(1) 630 rpm, steel-on-steel, 1 hour, 167°F.

(2) 520°F., 15 minutes

DSW 622090

TABLE XI

PYDRAUL 150, HOUGHTO-SAFE 1010, OS-104 AND VARIATIONS

	Pydraul 150	Houghto- Safe 1010	OS-104	CDF + (1) 0.5% Ucon (2) 0.05% Sant. 70	CDF + 0.5% Ucon	CDF
<u>Viscosity: os. at</u>						
210°F.	7.83	3.916	3.535	3.590	--	3.261
150°F.	14.6	8.37	7.34	7.20	--	6.562
100°F.	32.71	25.67	20.92	19.50	--	17.81
0°F.	906.5	6870.	4160.0	2560.	--	2102.
-10°F.	1982.	23,800.	13,620.	7800.	--	6096.
<u>Static Heat (520°F., 15 min.)</u>						
<u>Appearance</u>	Black	No change	amber	Dark amber	amber	amber
Initial Acidity, TAN		0.15	0.49	0.07	0.07	0.09
Final Acidity, TAN		0.20	1.33	0.54	0.76	0.76
N.N. Change	+123	+0.05	+0.84	+0.47	+0.69	+0.67
<u>Foam, Tendency at</u>						
75°F.	---	5 ml.	20 ml.	440, 60 ml.	20 ml.	---
200°F.	---	5 ml.	20 ml.	25, 25 ml.	15 ml.	---
75°F.	---	10 ml.	20 ml.	455, 30 ml.	30 ml.	---
<u>Collapse at</u>						
75°F.	---	5 sec.	30 sec.	7-1/2, 2-1/2 min.	15 sec.	---
200°F.	---	2 sec.	30 sec.	15, 15 sec.	5 sec.	---
75°F.	---	10 sec.	30 sec.	10 min., 35 sec.	35 sec.	---
<u>4-Ball Wear (167°F. 630 rpm, 1 hr.)</u>						
<u>Steel-on-Steel</u>						
4 Kg.	0.30	0.22	0.31	0.43	0.38	0.38
10 Kg.	0.36	0.33	0.34	0.40	0.42	0.42
40 Kg.	0.51	0.58	0.60	0.63	0.63	0.65
<u>Steel-on-Bronze</u>						
4 Kg.	0.58	0.56	0.56	0.56	0.53	0.53
10 Kg.	0.80	0.65	0.60	0.60	0.64	0.62
40 Kg.	1.58	1.10	1.21	1.60	1.68	1.57

(1) Cresyl diphenyl phosphate

(2) Ucon 75-H-90,000

(3) Second foam values are after adding 100 ppm. DCF-200.

(this table continued on next page)

DSW 622091

STLCOPCB4096122

TABLE XI (continued)

PYDRAUL 150, HOUGHTO-SAFE 1010, OS-104 AND VARIATIONS

	Pydraul 150	Houghto- Safe 1010	OS-104	CDP + (1) 0.5% Ucon (2) 0.05% Sant. 70	CDP + 0.5% Ucon 1/d/8	CDP ---
Rust, D-665, 24 hr., Distilled water (1)	.1/d/5 in. 1 hr.	.6/d/7	.4/d/6	9/s/8		
Oxidation-Corrosion (250°F., 168 hr.)						
Metal attack, mg./cm. ²						
Cu	-6.45	-0.17	-0.20	---	-0.39	
Mg	-0.51	0	0	---	-0.03	
Cd/Fe	-0.04	-0.12	0	---	+0.24	
Fe	-0.04	0	0	---	-0.04	
Al	0	0	+0.01	---	0	
Viscosity						
100°F. Initial, cs.	32.71	25.67	20.92	---	---	
Final, cs.	---	25.84	20.85	---	---	
Change	---	+0.66%	-0.33%	---	---	
210°F. Initial, cs.	7.83	3.916	3.535	---	---	
Final, cs.	---	3.946	3.568	---	---	
Change	---	+0.76%	+0.93%	---	---	
Neut. No.						
Initial, TAN	0.15	0.16	0.49	---	0.07	
Final, TAN	1.61	0.20	0.35	---	0.07	
Change	+1.46	+0.04	-0.14	---	0	
Appearance						

(1) Rating method of H.L. Faigen, ASTM Bulletin No. 154, October, 1948.
 First number is extent of rust 10 = perfect, 0 = complete rust.
 Other number and letter describe rust.

DSW 622092

STLCOPCB4096123

TABLE XII

TRICRESYL PHOSPHATE FORMULATION
PHYSICAL AND FUNCTIONAL PROPERTIES

	<u>TCP Blend</u> <u>(1)</u>	<u>Cellulube</u> <u>220, S-48</u>
Viscosity, cs.		
210°F.	5.38	4.77
100°F.	47.80	47.02
Viscosity Index, ASTM	+15.0	-8.1
Viscosity after shearing ⁽²⁾		
cs. 210°F.	5.376	4.70
100°F.	47.92	46.52
Pour Point, ASTM °F.	-15	-5
Shell 4-Ball Wear		
630RPM, 1 Hr., 167°F.		
Steel-on-Steel 4 Kg.	0.19 mm.	0.28 mm.
10 Kg.	0.23 mm.	0.33 mm.
40 Kg.	0.70 mm.	0.54 mm.
Almen Load or E.P.		
Load	14 lbs.	27 lbs. ⁽³⁾
Torque	27 lbs.	35 lbs.
Rust, ASTM D-665 ⁽⁴⁾		
1.0 hour	O.K.	O.K.
24 hours	6.75	5.5

Notes:

- (1) TCP containing 1.55% (w) Ucon 75-H-90,000
- (2) 12 passes through GM #5226710 Injector operated at 1800 CPM.
- (3) Average of several runs ranging from 8 lb. load - 16 lb. torque to 50# load - 56# torque.
- (4) REL - M-400 rating; 10.0 = Perfect

DSW 622093

STLCOPCB4096124

TABLE XIII

TRICRESYL PHOSPHATE FORMULATION
PETROLEUM OIL SOLUBILITY

Oil Solubility (1)	Viscosity		Layer Volumes		Layer Volumes	
	Before mixing cs.	after mixing cs. (2)	at 210°F.	at 100°F.	at 65°C. (3)	at 26°C. (4)
TCP Blend (5)	5.38	47.80	5.79	44.1	57 cc.	57 cc.
Naphthenic Oil (6)					No separation.	43 cc.
TCP Blend (7)	5.38	47.80	5.76	47.4	49 cc.	15 cc.
Paraffinic Oil (8)					51 cc.	20 cc.

(1) 50/50 volume of oil and TCP in 100 cc. stoppered graduate.

(2) Samples from final separation at 26°C. following heating to 65°C.

(3) Settled for 8 hours at 65°C.

(4) After removal from 65°C. oven. 16 hours for naphthenic oil and 40 hours for paraffinic oil.

(5) Tricresyl phosphate containing 1.55% Ucon 75-H-90,000.

(6) Naphthenic oil: Code AK oil, Union Carbide Nuclear Co., Paducah, Ky., Visc. at 100°F. - 155 SSU, non-inhibited.

(7) Paraffinic oil: Code B.S., Union Carbide Nuclear Co., Visc. at 100°F.-312 SSU, R&O inhibited

(8) Remainder of volume emulsion.

DSW 622094

TABLE XIV

TRICRESYL PHOSPHATE FORMULATION
DEMULSIFIER ADDITIVE STUDY

Additive in Tricresyl Phosphate Blend (1)	Emulsion Test (FS791-3201.3)		
	Fluid Layer, cc.	Emulsion Layer, cc.	Water Layer, cc.
TCP Blend, neat	0	56	24
Cellulube 220 (2)	39	0	41
Tricresyl Phosphate + 4% Rohm and Haas HF-825 (2)	39.5	0	40.5
Neutralized isoamyl octyl acid phosphite (RI-150), 200 ppm.	39	1	40
RI-150, 500 ppm.	40	1	39
RI-150, 0.1%	41	2	37
RI-150, 0.2%	41	2	37
Tret-o-Lite AD-9, 100 ppm.	40	6	34
Tret-o-Lite AD-9, 200 ppm.	41	2	37
Tret-o-Lite AD-9, 300 ppm.	43	2	35
Tret-o-Lite AD-9, 500 ppm.	42	1.5	37.5
Tret-o-Lite AD-5, 100 ppm.	39.5	3	37.5
Tret-o-Lite R-25, 100 ppm.	0	46.5	33.5
Tret-o-Lite L-16445, 100 ppm.	41	2	37
Dow Corning Fluid 200 (3)	--	Yes	--
Sterox 5 (3)	--	Yes	--
Sterox SE (3)	--	Yes	--
Sterox SK (3)	--	Yes	--
Sterox CD (3)	--	Yes	--
Sterox 110 (3)	--	Yes	--
Primene 81-R (3)	--	Yes	--
3-t-dodecylamine propionic acid (3)	--	Yes	--
Alkaterge T (Comm. Solv. Corp.) (3)	--	Yes	--
Isoamyl octyl acid phosphate (3)	--	Yes	--
Neutralized isoamyl diiso-octyl acid phosphate (3)	--	Yes	--
Neutralized isoamyl octyl acid phosphate (3)	--	Very little	--
Fluorochemical FC-75 (MMM) (3)	--	Yes	--
Fluorochemical FC-149 (MMM) (3)	--	Yes	--

(1) Tricresyl phosphate + 1.55% (wt.) Ucon 75-H-90,000.

(2) These fluids alone.

(3) One ounce distilled water and one ounce TCP blend with about 100 ppm. additive shaken in a four-ounce bottle as a screening test.

DSW 622095

STLCOPCB4096126

TABLE XV

SUITABLE* HUMIDITY CABINET RUST INHIBITORS
FOR PYDRAUL 625 BLENDS

Adol 14 ester/dimethylaminopropylimide, 66/34, lot R-677, RI-126**
Alkaterge T, Commercial Solvents Corp., RI-148
Diamyl-phenyl phosphoric acid, RT-53-A, RI-48
Dimer acid, RT-67-A, Emery M-461-R dimer acids, RI-24
Driacin, Universal Oil Products, RI-111
Emery dimer acid from tall oil, RI-10
Geigy NPA, Geigy Industrial Chemicals, RI-50
Gulf Agent 178, RI-125
Ionad 17, Shell, RI-121
Isoamyl-octyl acid phosphate, RI-149
Neutralized isoamyl-octyl acid phosphate, RI-150
Maleic anhydride/sperm oil R.P., CP-30294, RI-151
Maleic anhydride/butyl UFA R.P., CP-30295, RI-152
Maleic anhydride/oleic acid R.P. and 2-ethylhexanol, N-8412, RI-34
Na-Sul BSN, Vanderbilt Co. (barium dinonyl naphthalene sulfonate),
RI-128
Nirus N.P., Synthetic Chemicals, Inc., RI-119
Oleyl 2-ethylhexylmaleate, RD-18-588, RI-27
3-(Oleyloxy)propionic acid, RI-96
Paradyne HO 2, RI-123
Santolene C, RI-47
Santolene C, kerosene free, RI-124
Santolene C in Pydraul 150 carrier, RI-156
Santolube 70, RI-49
Santolube 203-C, RI-57
Santolube 333, RI-44

DSW 622096

See footnotes - next page

TABLE XV (continued)

Santolube 536-B, RI-46

Sarkosyl O, Geigy Chemical Corp., RI-51

Sodium Petroleum Sulfonate, Freedom Valvoline, RI-45

Tetrapropenylsuccinic anhydride/3-amino-1-propanol R.P. 2/1, RI-31
N-8364

Tetrapropenylsuccinic anhydride/1,4-butanediol R.P., N-7794A, RI-54

Tetrapropenylsuccinic anhydride/butyrolactone/water R.P., 1/1/1,
N-8366, RI-33

Tetrapropenylsuccinic anhydride/castor oil R.P. , 3/1, RI-30

TPSA/2,5-di-tert-amylhydroquinone, 2/1, N-8413, RI-35

TPSA/Duomeen T R.P., 2/1, RI-32 N-8365

TPSA/glycerine R.P., 2/1, N-8415, RI-37

TPSA/N-methyl pyrrolidine/water R.P., N-7796, RI-56

TPSA/2-methyl, 2,4-pentanediol R.P., N-7797, RI-52

TPSA/1,3-propanediol, 2/1, N-8414, RI-36

TPSA/triethanolamine R.P., 3/1, N-8087, RI-29

1-(triisobutenyl)diethylenetriamine, CP-12992, RI-131

x Providing more than 50 hours rust protection.

xx RI numbers are REL code numbers.

DSW 622097

STLCOPCB4096128

TABLE XVI

UNSUITABLE^x HUMIDITY CABINET RUST INHIBITORS
FOR PYDRAUL 625 BLENDS

Acridine, RI-13
Adol 14 ester/dimethylaminopropylimide 74/26, RI-161^{xx}
Adol 14 ester/dimethylaminopropylimide 82/18, RI-162
Adol 14 ester/dimethylaminopropylimide 91/9, RI-163
Alox 1724, Alox Corporation, RI-165
Amine ODT, Rohm and Haas, RI-89
Amine 9D-178, Rohm and Haas, RI-25
Ammonium oleate, RI-87
Ammonium Stearate, RI-86
Ar-dodecylphenyl melamine, RI-114
Armeen 160, RI-26
Armeen 2C, RI-94
Armeen 2C/di-tert.-butyl-p-cresol 50/50, RI-100
Armeen DM-18, RI-101
Armeen DMS, RI-90
Benzal SD, RI-63
Benzoic Acid, RI-78
Bis(1-2 phenylmercapto)ethane, RI-16
Calcium Petroleum Sulfonate, "Synfonate 28", RI-43
Chlorinated propylene polymer "B" and diethylenetriamine reaction product, CP-13021, RI-132
Chlorinated propylene polymer "B" and ethylenediamine reaction product, CP-13532, RI-135
Cyclohexylamine, RI-79
Cyclohexyl melamine, RI-115
Cyclohexylmethylaniline, RI-127

DSW 622098

TABLE XVI (continued)

UNSUITABLE^x HUMIDITY CABINET RUST INHIBITORS
FOR PYDRAUL 625 BLENDS

Dibenzyl disulfide, RI-70
Dicyclohexylamine, RI-80
1,7-Didodececenyl-diethylenetriamine, CP-12685, RI-130
Di-2-ethylhexylamine, RI-97
Di-hexyldithiophosphoric acid and cyclohexylamine salt, RI-113
Di-hydroabietyl malate, RT-52A, RI-28
2,2'-Dipyridylamine, RI-14
2,6-Di-tert.-butyl-4-methyl phenol, Shell Ionol, RI-39
Dodecoxyguanamine, RI-116
Dodecylamine, RI-91
Duomeen C, RI-92
Duomeen T, RI-93
Ethylene bis(tolyl sulfide), RI-38
Ferrocene, RI-7
Drew FOT-W, Drew and Co., RI-120
F.O.A., No. 7, DuPont, RI-110
Hexydecyl Ketene dimer, RI-88
Hydroxyphenyl stearic acid, RI-40
Kleery concentrate, Synthetic Chemicals Inc., RI-118
Maleic anhydride/safflower oil reaction product, 1/1, CP-30298, RI-155
Maleic anhydride/soybean oil R.P., 1/1, CP-30296, RI-153
Maleic anhydride/soybean oil R.P., 1/3.3, CP-30297, RI-154
Maleic anhydride/triethyl phosphite R.P., CP-14569, RI-141
Menthane diamine, RI-122
2-Mercapto Benzothiazole, RI-5

DSW 622099

TABLE XVI (continued)

UNSUITABLE^x HUMIDITY CABINET RUST INHIBITORS
FOR PYDRAUL 625 BLENDS

Mulsor No. 3CW, Synthetic Chemicals, Inc., RI-117
Nalcamine G13, National Aluminate Corp., RI-12
Nalco SR-155, RI-104
Nalco SR-158, RI-109
Nalco 158-D, RI-106
Nalco 301, RI-107
N(anilinomethyl)succinimide, CP-13812, RI-140
N-dodecenylethylenediamine, CP-13045, RI-133
Nicotinic Acid diethylamide, RI-18
Nicotinic Acid Hexyl ester, RI-19
N,N'-di-2-naphthyl-p-phenylenediamine, RI-1
N,N'-diphenylbenzidine, RI-17
N,N'-p-phenylene-bis-acetamide, RI-8
N,N'(1,1,3,3-tetramethylbutylimino)dimethylene diphthalimide,
CP-13891, RI-139
N-(1-tert-butyl-2-methylallyl)ethylenediamine, CP-11666, RI-129
N-triisobutenylethylenediamine, CP-13530, RI-134
o-Aminodiphenyl, RI-15
Oxodecyl EMA Methylimide, 85/15, lot R-577, RI-157
Oxodecyl EMA ester-imide, 15/15, lot R-580, RI-158
Oxodecyl EMA ester-imide, 90/10, lot R-581, RI-159
Oxotridecyl EMA ester-imide, 65/35, lot R-582, RI-160
p-Aminodiphenylamine, RI-11
Phenothiazine, RI-2
Phenyl-alpha-Naphthylamine, RI-3

DSW 622100

TABLE XVI (continued)

UNSUITABLE* HUMIDITY CABINET RUST INHIBITORS
FOR PYDRAUL 625 BLENDS

Phenyl glycidyl ether, RI-22
Phenyl mercapto benzothiazole, RI-4
Phenyl Selenide, RI-6
p,p'-dioctyl diphenylamine, RI-9
p-Phenoxyaniline, CP-14544, RI-142
Primene 81-R, Rohm and Haas, RI-64
RD-1944-P, Armour Chemical Div., RI-102
RD-2237-P, Armour Chemical Div., RI-98
RD-2322-P, Armour Chemical Div., RI-99
Santolene HLV, RI-42
Santolene J, RI-41
Santolube 394-C, RI-23
Stearamide, RI-20
Stearanillide, RI-21
1(1-tert-butyl-2-methylallyl)tetraethylenepentamine, CP-13536, RI-137
1(1-tert-butyl-2-methylallyl)triethylenetetramine, CP-13535, RI-136
3-tert-dodecylamino-propionic acid, RI-95
Tenameen 21, RI-108
Tenameen 22, RI-109
Triethylenetetramine/linoleic acid R.P., CP-15593-(2), RI-147
Triethylenetetramine/nonanoic acid R.P., CP-15592-(2), RI-146
Triethylenetetramine/stearic acid R.P., CP-15587-(2), RI-145
Triphenyl phosphite, RI-60
Unicor, Universal Oil Products, RI-103

DSW 622101

TABLE XVI (continued)

UNSUITABLE* HUMIDITY CABINET RUST INHIBITORS
FOR PYDRAUL 625 BLENDS

Universal Oil Products 176-5, RI-112

Vegetable Oleic acid monoglycerides, Distilled Products Industries,
RI-85

* Providing less than 50 hours rust protection.

** RI numbers are REL code numbers.

DSW 622102

TABLE XVII

STEAM TURBINE OIL OXIDATION ASTM D-943 (1)

Test No.	Purpose (2)	Fluid and Test Conditions	Days Exposure	Steel Condition	Copper Condition	Fluid Condition
1-1	P, M	OS-81, water washed, sintered glass disperser	15	Slight rusting	Bright	Clear
1-2	P, M	as 1-1 with straight sparger	15	Slight rusting	Bright	Clear
1-3	A, M	as 1-1 with 0.25% $\text{OSCH}_2\text{CH}_2\text{S}\phi$	15	Much rusting	Bright	Clear
1-4	A, M	as 1-2 with 0.25% $\text{OSCH}_2\text{CH}_2\text{S}\phi$	15	Much rusting	Bright	Clear
1-5	A	OS-81 water washed with 0.25% hydroxy-phenyl stearic acid	75	3 to 4 spots	Attacked	Dark with sludge
1-6	A	OS-81 water washed with 0.25% dipyrindyl amine	15	Black deposit	Clear	Black
2-1	P	OS-81, filtered through paper	51	few spots	few stained spots	Clear - some sludge
2-2	P	OS-81, washed Aroclor, unwashed TCP	51	Few rust spots	Clear	Slight yellow - slight sludge
2-3	P	OS-81, washed Aroclor, washed TCP	51	Few rust spots	Clear	Clear
2-4	P	OS-81, unwashed Aroclor, washed TCP	51	Few rust spots	Clear	Clear, sludge trace
3-1	A	OS-79 + 0.1% reduced Sant. 70	63	Clear	V. sl. darkening	Clear, yellow
3-2	A	OS-81 + 0.1% dicresyl phosphoric acid	63	Clear	Definite attack	Dark brown, sludge
3-3	P, M	OS-81, silver storage, wires washed before sanding, wiped dry after sanding	63	Few spots	Clear	Clear
3-4	P, M	as 3-3 with wires washed after sanding	63	Few spots	Clear	Clear, sl. sludge
3-5	A, M	as 3-3 with 0.25% $\text{OSCH}_2\text{CH}_2\text{S}\phi$	63	Several rust spots	Clear	Clear, sl. sludge
3-6	A, M	as 3-4 with 0.25% $\text{OSCH}_2\text{CH}_2\text{S}\phi$	63	Several rust spots	Clear	Clear, sl. sludge
4-1	A	OS-81 + 0.5% Ethyl AN-2	44	General rusting	Dull	Rusty, sludge
4-3	A	25 ppm diphenyl phosphoric acid	44	3-4 spots	Sl. dull	Trace of sludge
4-4	A	100 ppm diphenyl phosphoric acid	44	Bright with deposits	Bright with deposits	Trace of sludge
4-5	A	Lithium dichromate treated	44	1 spot	Sl. dull	Clear
4-6	P	Blank of 4-5	44	Bright	Sl. dull	Clear

(1) Oxygen entering tubes is bubbled through watersince water loss from tubes is excessive for heavier-than-water synthetics.

(2) P - process change or treatment to OS-81 or blank, A - additive, M - test method variation.

DSW 622103

TABLE XVIII

HIGH TEMPERATURE FLUID SCREENING

PHYSICAL PROPERTIES

Identity No.	Viscosity, cs.			Temperature, °F.		Pour Point, °F.	Boiling Point, °F.
	100°F.	210°F.	400°F.	at 10,000 cs.	at 100,000		
Mixed (o,m,p)bis(x-methylphenoxy)benzene (CP-30415)	21.3	3.23	--	+7	-7	-5	750
1,1-Didecyl 3,3-di-(2-ethylhexyl)urea	54.6	7.34	--*	-14	--	-5	615 at 2 mm.
2,2-Dimethyl-1,3-propyl dilaurate	16.2	3.77	1.2*	--	--	+57**	790
Bis(1-methylcyclohexylmethyl)hexahydro- isophthalate (CP-30417)	276.	13.5	2.0*	+40	+15	+39	820
Bis(1-methylcyclohexylmethyl)sebacate	47.2	7.04	1.7*	-22	-50	-49**	820
O, m-terphenyl and biphenyl mixture	18.0	2.41	--	--	--	+75**	491 to 684
Monoisopropylbiphenyl	4.38	1.39	--	-52	-68	--	563
Diisopropylbiphenyl	12.2	2.43	--	-20	-35	-23**	610
Ethylated Santowax R	510.	12.6	--	--	--	+75**	---
Isopropyl Santowax OM	278.	8.58	--	+55	+38	+30	710 to 790
Tertiary butyl Santowax OM	4780.	18.5	--	--	--	+75**	---
HB-40	31.8	4.02	--	+30*	--	--	600 to 750
Ethylated HB-40	277.	9.98	--	--	--	--	570 to 790
Tertiary butyl HB-40	80.95	5.96	--	--	--	--	---
2-n-Butylamino-4-di-n-butylamino-6-piperidino- S-triazine	614.	18.2	--	+50	+24	--	700
2-N-n-propyl-4-N-n-butyl-6-N-n-hexylmelamine	--	45.3	--	--	--	--	---
2-N-diethyl-4-N-diphenyl-6-N-dibenzylmelamine	--	--	--	--	--	+167**	---
m-Tolyl-, -trifluoro-m-tolyl sulfone	--	6.32	--	--	--	--	---
O-Chlorophenyl-p-nonylphenyl ether	--	--	--	--	--	--	---
3,4-Diphenyl chlorobenzene	--	--	--	--	--	--	---
Synjet Oil 15 (Texaco MIL-L-7808)	17.2	4.78	--	--	--	--	---
General Electric Versilube F-50 silicone	63.7	20.3	--	--	--	<-75	---
General Electric 81644 silicone	63.6	20.2	--	--	--	<-75	---
General Electric 81717 silicone	69.1	21.8	--	--	--	<-75	---
Fluorochemical FC-75	--	--	--	--	--	--	---

* Estimated value

** Crystals below this temperature.

DSW 622104

TABLE XIX

HIGH TEMPERATURE FLUID SCREENING

Oxidation-Corrosion, 347°F., 72 Hours

Identity No.	100°F. Viscosity Initial	Change %	Acidity		Fluid Weight Change %	Insolubles
			Initial, TAN	Change		
Mixed (o,m,p)bis(x-methylphenoxy)benzene (CP-30415)	21.3	+ 48	0.08	+ 3.34	+ 0.6	None
1,1-Didodecyl 3,3-di-(2-ethylhexyl)urea	46.2	+ 5	0.18	+ 1.63	- 2.0	Few
2,2-Dimethyl-1,3-propyl dilaurate	16.2	+67	0.24	+13.9	+ 0.8	Few
Bis(1-methylcyclohexylmethyl)hexahydro- isophthalate (CP-30417)	276.	+236	0.52	+ 4.2	+ 3.1	None
Bis(1-methylcyclohexylmethyl)sebacate	47.2	+155	0.01	+12.0	+ 0.5	Few
o,m-terphenyl and biphenyl mixture	18.0	+ 5	0.06	+ 0.32	+ 0.5	None
Monoisopropylbiphenyl	4.38	+196	0.05	+ 1.19	+ 3.0	Few
Diisopropylbiphenyl	12.2	+546	0.05	+ 2.00	+ 3.0	None
Ethylated Santowax R	510.	+258	0	+ 0.76	- 0.3	--
Isopropyl Santowax OM	278.	+1485	0.17	+ 1.30	+ 0.2	--
Tertiary butyl Santowax OM	4780.	+25.4	0.01	+ 0.14	- 0.06	--
HB-40	31.8	+3600.	0.01	+ 3.17	---	--
Ethylated HB-40	277.	-15.9	0	+16.08	---	--
Tertiary butyl HB-40	80.95	+236	0	+ 6.18	---	--
2-n-Butylamino-4-di-n-butylamino-6-piperidino- S-triazine	18.2*	+58*	7.12	- 2.55	-12.9	None
2-N-n-propyl-4-N-n-butyl-6-N-n-hexylmelamine	45.3*	+99*	11.8	- 3.0	- 3.6	--
2-N-diethyl-4-N-diphenyl-6-N-dibenzylmelamine	--	--	--	--	--	--
m-Tolyl- α,α,α -trifluoro-m-tolyl sulfone	6.39*	+196*	0.19	+15.1	+ 0.26	Few

* 210°F. viscosity

DSW 622105

TABLE XIX (continued)

HIGH TEMPERATURE FLUID SCREENING

Oxidation-Corrosion, 347°F., 72 Hours

Identity No.	100°F. Viscosity	Acidity		Fluid Weight Change %	Insolubles
		Initial	Change		
o-Chlorophenyl p-nonylphenyl ether	--	--	--	--	--
3,4-Diphenyl chlorobenzene	--	--	--	--	--
Synjet Oil 15 (Texaco MIL-L-7808)	S-31	17.2	+4	+1.16	Few
General Electric Versilube P-50 silicone	S-77	63.7	+1	-0.12	None
General Electric 81644 silicone	S-78	63.6	+5	-0.29	None
General Electric 81717 silicone	S-79	69.1	+5	-0.43	None
Fluorochemical FC-75	--	--	--	--	--
Texaco AM2 (MIL-O-5606 Fluid)	S-24	--	--	--	--

Identity No.	Metal Weight Change, mg./cm. ²				
	Copper	Steel	Aluminum	Magnesium	Silver
Mixed (o,m,p)bis(x-methylphenoxy)benzene (CP-30415)	S-45	- 0.53	+ 0.01	+ 0.01	+ 0.01
1,1-Didecyl 3,3-di-(2-ethylhexyl)urea	S-18	- 0.22	+ 0.01	0	+ 0.01
2,2-Dimethyl-1,3-propyl dilaurate	S-46	- 0.89	+ 0.02	+ 0.01	+ 0.02
Bis(1-methylcyclohexylmethyl)hexahydroisophthalate (CP-30417)	S-47	- 0.10	+ 0.01	0	+ 0.02
Bis(1-methylcyclohexylmethyl)sebacate	S-49	- 0.03	- 0.01	0	- 0.01
o,m-terphenyl and biphenyl mixture	S-55	- 0.01	- 0.02	- 0.01	- 0.02
Monoisopropylbiphenyl	S-39	- 0.02	- 0.02	+ 0.02	- 0.02
Diisopropylbiphenyl	S-42	- 0.04	- 0.01	+ 0.02	+ 0.02

DSW 622106

TABLE XIX (continued)

HIGH TEMPERATURE FLUID SCREENING

Oxidation-Corrosion, 347°F., 72 Hours

Identity No.	Metal Weight Change, mg./cm. 2				
	Copper	Steel	Aluminum	Magnesium	Silver
Ethylated Santowax R	- 0.06	- 0.02	- 0.02	- 0.02	- 0.03
Isopropyl Santowax OM	- 0.04	- 0.02	0	0	0
Tertiary butyl Santowax OM	- 0.04	- 0.01	- 0.01	- 0.01	- 0.01
HB-40	- 0.42	- 0.02	- 0.03	- 0.06	- 0.02
Ethylated HB-40	- 0.22	- 0.03	0	- 0.02	- 0.01
Tertiary butyl HB-40	- 0.04	- 0.02	- 0.01	- 0.07	- 0.02
2-n-Butylamino-4-di-n-butylamino-6-piperidino- 5-triazine	-66.2	-16.1	+ 0.02	- 0.35	-31.9
2-N-n-propyl-4-N-n-butyl-6-N-n-hexylmelamine	-14.3	- 8.5	0	- 0.08	- 9.5
2-N-diethyl-4-N-diphenyl-6-N-dibenzylmelamine	--	--	--	--	--
m-Tolyl- α,α,α -trifluoro-m-tolyl sulfone	- 0.65	+ 0.02	+ 0.01	+ 0.02	- 0.45
o-Chlorophenyl p-nonylphenyl ether	--	--	--	--	--
3,4-Diphenyl chlorobenzene	--	--	--	--	--
Synjet Oil 15 (Texaco MIL-L-7808)	- 0.20	- 0.05	+ 0.03	- 0.15	+ 0.02
General Electric Versilube P-50 silicone	- 0.05	+ 0.01	0	+ 0.02	0
General Electric 81644 silicone	- 0.31	+ 0.02	+ 0.02	+ 0.02	+ 0.01
General Electric 81717 silicone	+ 0.21	+ 0.02	0	+ 0.02	- 0.02
Fluorochemical FC-75	--	--	--	--	--
Texaco AM2 (MIL-O-5606 Fluid)	--	--	--	--	--

DSW 622107

TABLE XX

HIGH TEMPERATURE FLUID SCREENING

Lubricating Properties

Identity No.	Four-Ball Wear (1)				Almen E.P.	
	4 Kg. 167°F.	10 Kg.		40 Kg. 167°F.	Lb. Load	Lb. Torque
		167°F.	400°F.			
Mixed (o,m,p)bis(x-methylphenoxy)benzene (CP-30415)	S-45	0.55	0.65	0.92	0.94	2 7
1,1-Didodecyl 3,3-di-(2-ethylhexyl)urea	S-18	--	0.37	0.56	0.61	- -
2,2-Dimethyl-1,3-propyl dilaurate	S-46	0.50	0.53	1.01	0.61	9 15
Bis(1-methylcyclohexylmethyl)hexahydro- isophthalate (CP-30417)	S-47	0.21	0.73	1.12	0.98	5 13
Bis(1-methylcyclohexylmethyl)sebacate	S-49	0.37	0.75	1.11	1.13	7 12.5
o,m-terphenyl and biphenyl mixture	S-55	--	2.35(2)	--	--	0 4
Monoisopropylbiphenyl	S-39	--	0.51	1.13	1.04	3 7
Diisopropylbiphenyl	S-42	--	0.56	0.84	0.95	2 7
Ethylated Santowax R	--	--	0.70	--	1.5 (4)	- -
Isopropyl Santowax OM	--	--	0.58	--	1.24	- -
Tertiary butyl Santowax OM	--	--	0.54	0.75	1.21	- -
HB-40	--	--	0.52	seized	seized	- -
Ethylated HB-40	--	--	0.41	seized	seized	- -
Tertiary butyl HB-40	--	--	0.37	seized	seized	- -
2-n-Butylamino-4-di-n-butylamino-6-piperidino- 5-triazine	S-75	0.43	0.52	0.80	0.76	- -
2-N-n-propyl-4-N-n-butyl-6-N-n-hexylmelamine	S-99	--	0.43	0.92(5)	1.39(3)	- -
2-N-diethyl-4-N-diphenyl-6-N-dibenzylmelamine	--	--	0.38(3)	0.51(6)	0.59(6)	- -
(1) 1200 RPM, 2 hours, steel-on-steel	(3) 212°F.	(5) 1 hour run				
(2) 15 min. run	(4) 1 min. run	(6) 630 RPM				

DSW 622108

TABLE XX (continued)

HIGH TEMPERATURE FLUID SCREENING

Lubricating Properties

Identity No.		Pour-Ball Wear (1)				Almen E.P.	
		4 Kg.		10 Kg.		Lb. Load	Lb. Torque
		167°F.	400°F.	167°F.	400°F.		
S-76	m-Tolyl- α,α,α -trifluoro-m-tolyl sulfone	0.39(3)	0.50	0.63(3)	0.59 ^{xxx}	--	--
--	o-Chlorophenyl p-nonylphenyl ether	--	0.55	0.53	0.99	15	23
--	3,4-Diphenyl chlorobenzene	--	--	0.71	1.10	--	--
S-31	Synjet Oil 15 (Texaco MIL-L-7808)	0.27	0.31	0.27	0.47	--	--
S-77	General Electric Versilube P-50 silicone	0.21	0.66	0.56	1.00	--	--
S-78	General Electric 81644 silicone	0.33	0.65	0.47	1.24	--	--
S-79	General Electric 81717 silicone	0.32	0.70	0.48	0.76	--	--
--	Fluorochemical FC-75	--	--	2.47	--	--	--
S-24	Texaco AM2 (MIL-O-5606 Fluid)	--	0.41	0.30	0.51	--	--

(1) 1200 RPM, 2 hours, steel-on-steel

(2) 15 min. run

(3) 212°F.

(4) 1 min. run

(5) 1 hour run

(6) 630 RPM

DSW 622109

TABLE XXI

HIGH TEMPERATURE FLUID SCREENING

Summary of Fluid Performance

Fluid Identity No.		Performance Qualities (1)		Applicable Temperatures, °F.		Order of Preference	
		Viscosity	Oxidation-Corrosion	Lube	Jet Lube	Hydraulic Fluid	Jet Lube
	Mixed (o,m,p)bis(x-methylphenoxybenzene)	P(2)	P	P	600	700	3
S-45							1
S-45B	S-45 + 5% TCP, 0.5% Phenothiazine	P(2)	P	G	600	700	-
S-46	2,2-Dimethyl-1,3-propyl dilaurate	P(2)	P	P	500	600	4
S-46B	S-46 + 5% TCP, 0.5% Phenothiazine	P(2)	G	G	500	600	-
S-47	Bis(1-methylcyclohexylmethyl)hexahydroisophthalate	G	P	P	600	---	1
S-47B	S-47 + 5% TCP, 0.5% Phenothiazine	G	P-G	G	500	---	-
S-49	Bis(1-methylcyclohexylmethyl)sebacate	G	P	P	500	600	2
S-49B	S-49 + 5% TCP, 0.5% Phenothiazine	G	P-G	G	500	600	-
S-18	1,1-Didecyl 3,3-di(2-ethylhexyl)urea	G	P-G	P	500	600	6
S-39	Monoisopropylbiphenyl	P(2)	P-G	P	---	600	0
S-42	Diisopropylbiphenyl	P(2)	P-P	P	600	700	5
S-55	o, m-terphenyl and diphenyl mixture	P(2)	G	PP	400	700-800	0
S-75	2-n-Butylamino-4-di-n-butylamino-6-piperidino-5-triazine	G	PP	P	---	---	0
S-76	m-Tolyl-4,4'-trifluoro-m-tolyl sulfone	G	P	P	---	---	0

(1) P - Poor

F - Fair

G - Good

(2) Viscosity too low for MIL-L-9236A.

DSW 622110

TABLE XXIII

SKYDROL 500, BASE STOCKS, ADDITIVES

Test No.	Fluid	Metal Attack, mg./cm. ²		Acidity, TAN	
		Cu	Mg	Initial	Final
10-3	Skydrol 500 (typical lab prep.)	0.126	0	0.172	0.123
14-6	Skydrol 500 (typical lab prep.)	0.193	0.014	0.030	0.037
19-1	Skydrol 500 (1, (2)	0.141	0	0.031	0.062
20-4	Skydrol 500 (B-2069 FH-135)	0.193	0.037	0.049	0.345
20-5	Skydrol 500 (2), (3)	0.090	0.022	0.009	0.035
18-2	Skydrol 500 (1), (4)	0.385	0.165	0.230	1.414
10-1	FH-135 (typical lab prep.)	16.1	0.052	0.123	3.84
14-3	FH-135 (typical lab prep.)	16.7	0.22	0.037	6.912
15-2	FH-135 (1), (4)	34.9	8.84	0.327	5.436
19-2	FH-135 (1), (2)	9.4	0.044	0.027	2.050
20-3	FH-135 (3), (4)	8.4	0.045	0.009	1.838
15-1	FH-87 (Ba 15-16)	23.1	0.029	0.029	5.341
18-1	FH-87 (Ba 15-16)	16.2	0.089	0.029	3.358
20-2	FH-87 (Ba 25-26)	6.73	0.022	0.009	1.440
15-5	FH-87 (1), 0.5% FH-132	25.8	2.58	0.029	8.29
15-6	FH-87 (1), 1.0% PGE	0.20	0.007	0.017	0.400
15-3	FH-87 (1), 0.5% FH-132, 1.0% PGE	38.5	12.7	0.320	13.07
15-4	FH-87 (1), 1.0% PGE, high acid Acryloid	1.16	0.096	0.296	2.35

DSW 622111

TABLE XXIII (continued)

SKYDROL 500, BASE STOCKS, ADDITIVES

Test No.	Fluid	Metal Attack, mg./cm. ²			Acidity, TAN	
		Cu	Mg	Cd/Fe	Initial	Final
16-1	FH-135 (1), (4) 0.5% FH-132 + 25% adsorption alumina	1.85	0.014	0	0.244	0.261
(1)	Ba 15-16 FH-87.					
(2)	Ba 1853 Acryloid (low acid).					
(3)	Ba 25-26 FH-87.					
(4)	High Acid Acryloid.					

DSW 622112

TABLE XXIV

SKYDROL 500 REFORMULATION, PROMISING ADDITIVES

Test No.	Additive in FH-135 (1)	Metal Attack, mg./cm. ²			Acidity, TAN	
		Cu	Mg	Cd/Fe	Initial	Final
16-5	(1), (4)	0.41	0.30	0.92	0.217	1.32
16-6	(1), (4)	1.65	7.6	1.58	0.244	5.04
16-7	(1), (4)	4.5	12.9	0.96	0.226	7.76
21-4	(1), (3)	0.83	0.08	0.04	---	---
16-3	(1), (4)					
	0.5% FH-132, 1% terphenyl tin					
	0.5% FH-132, 1% tetrabutyl tin					
	0.5% FH-132, 1% triphenyl stibine					
	1% DuPont PL-297					
	0.5% FH-132, 1% Epoxy 2-ethylhexyl tallate					
19-3	(1), (3)	0.266	0.222	0.541	0.209	0.383
19-4	(1), (3)	0.133	0.007	0.037	0.018	0.027
	0.5% FH-132, 1% Epoxy 2-ethylhexyl tallate					
25-3	(2), (3)	0.037	+0.059	+0.081	0.018	0.035
25-4	(2), (3)	0.044	0.015	0	0.012	0.040
25-5	(2), (3)	0.037	0	0	0.012	0.027
25-6	(2), (3)	0.10	0.022	0.037	0.035	0.060
25-7	(2), (3)	0.074	0.037	0.015	0.022	0.055
25-8	(2), (3)	0.074	0.037	0.037	0.025	0.081
27-1	(2), (3)	0.11	0	0.015	0.017	0.064
27-2	(2), (3)	0.103	0	0	0	0.02
27-3	(2), (3)	0.562	0	0.03	0	0.45
	0.5% Rohm and Haas Paraplex G-62					
27-4	(2), (3)	0.052	0	0.022	0	0.02
	0.5% FH-132, 0.2% Rohm and Haas Paraplex G-62					
28-1	(2), (3)	0.68	0.56	0.03	0	0.688
	2% Ortholeum 300 + Perox Violet 3B dye					
		0.92	0.037	0.007	0.157	0.923

(1) Ba 15-16 FH-87.

(2) Ba 25-26 FH-87.

(3) Ba 1853 Acryloid (low acid).

(4) High acid Acryloid.

(continued -- next page)

DSW 622113

TABLE XXIV (continued)

SKYDROL 500 REFORMULATION, PROMISING ADDITIVES

Test No.	Additive in FH-135	Metal Attack, Mg./cm. ²			Acidity, TAN	
		Cu	Mg	Cd/Fe	Initial	Final
28-2	(2), (3) 2% Ortholeum 300 + Hytherm Purple KI dye	1.51	0.044	0.022	0.157	1.002
28-3	(2), (3) 2% Ortholeum 300 + Hytherm Purple KIF dye	0.54	0.037	0.030	0.157	0.981
29-1	(2), (3) 1% Rohm and Haas Paraplex G-62 + Hytherm Purple KIF dye	0.04	0	0.007	0.010	0.013
29-3	(2), (3) 1% Estanox 130	0.094	0	0.030	0.021	0.022
29-4	(2), (3) 1% Estanox 408	0.089	0.007	0.007	0.017	0.018

(2) Ba 25-26 FH-87.

(3) Ba 1853 Acryloid (low acid).

DSW 622114

TABLE XXV

SKYDROL 500 REFORMULATION, POOR ADDITIVES IN FH-135

<u>Test Number</u>	<u>Additive in FH-135</u>
16-2	0.5% FH-132; 1% Dipentene Oxide
16-4	0.5% FH-132, 1% 1,2-Epoxy ethylbenzene
16-8	0.5% FH-132, 1% Dibutyl tin Maleate
19-6	1% Nacalmine G-13
20-6	1% Mannich Base (SL-13562)
20-7	1% Mannich Base (SL-1353)
20-8	1% Succinic Acid Amine Ester (SL-13561)
21-1	1% Dibutyl Tin Dilamate
21-2	1% Morpholine
21-3	1% Ethyl Silicate
21-5	1% Kontrol 147
21-6	1% Thermolate 31
23-1	1% Kontrol 77
23-2	1% Amberlite LA-1
23-3	1% Amberlite LA-2
23-4	1% 1-o-Tolybiguanide
23-5	1% Butasan
23-7	0.5% FH-132, 1% Benzal SD
23-8	1% Tin Diethyldithiocarbonate (not very soluble)
24-1	0.5% FH-132, 1% PL-297
24-2	0.5% FH-132, 1% R-677
24-3	0.5% FH-132, 0.4% Ferrocene
24-5	0.5% FH-132, 1% Sarbosyl O
26-1	1% Citric Acid
26-2	1% N-Coco Morpholine
26-3	1% Dibutyl Fumarate
26-4	1% Dibutyl Maleate
26-5	1% Hydroxyquinaldine
26-6	1% Tartaric Acid
27-5	1% Methyl Salicylate
27-6	1% α -Methyl tetra Hydrofurane
28-6	1% Hexamethyl phosphianide

DSW 622115

TABLE XXVI

SKYDROL 500 - VISCOSITY INDEX IMPROVER

Fluid	Metal Weight Loss, mg./cm. ²					Acidity, TAN	
	Al	Mg	Cu	Fe	Cd/Fe	Initial	Final Change
Skydrol 500 (Acryloid Lot 2498)(1)	0.02	0.02	0.19	0.01	0.07	0.03	0.04 + 0.01
Skydrol 500 (Acryloid Lot K-6473) (1)	0.02	0.01	0.56	0	0.01	0.03	0.07 + 0.04
FH-135 (Acryloid Lot 2498) (1)	0	0.22	16.7	0	0.07	0.04	6.91 + 6.9
FH-135 (Acryloid Lot K-6473) (1)	0.22	13.0	34.9	0.10	3.6	0.03	11.35 +11.3
FH-87 (Ba 15-16, Salt treated)	0	0.09	16.2	0.02	0.04	0.03	3.36 + 3.3
FH-87 (Ba 25-26, typical)	0.02	0.02	6.73	0	0.06	0.01	1.44 + 1.43
FH-87 (K-17470) (2)	0.04	2.38	34.35	0.03	0.11	0.19	7.42 + 7.2
FH-87 (K-17471) (3)	0	0.08	15.95	0.01	0.08	0.05	3.04 + 2.99
FH-87 (12% K-17470 in Ba 15-16)	0	1.0	36.75	0	0.05	0.05	7.42 + 7.4
FH-87 (12% K-17471 in Ba 15-16)	0.02	0.10	17.79	0.05	0.09	0.03	3.53 + 3.5
FH-87 (12% K-17470 in Ba 25-26)	0	0.26	13.36	0	0.02	0.02	3.28 + 3.3
FH-87 (12% K-17471 in Ba 25-26)	0	0.12	5.64	0	0.02	0.01	1.83 + 1.82

(1) FH-87 used is typical of Ba 25-26.

(2) Rohm and Haas proposed process conditions with catalyst.

(3) Rohm and Haas proposed process conditions without catalyst.

DSW 622116

Procedures and test results for these fluids appear in the Appendix.

B. Synthetic Lubricants for Refrigeration Units

A large potential use of synthetic lubricants in the refrigeration field prompted some preliminary work to establish the level of lubricating qualities desirable in such fluids.

A series of four-ball wear tests were run on Suniso 3-G, the refrigeration compressor fluid presently in use by Hussmann Refrigerator Company. Similar test data from Mobil D.T.E. fluid and Pydraul F-9 fluid is included in the chart below. The comparative data indicates that Suniso 3-G is inferior to both Pydraul F-9 and Mobil D.T.E. in lubricating qualities.

Test Conditions: 630 RPM 167°F. 1 hour duration				
Fluid	Steel-on-steel			Steel-on-bronze
	4 kg	10 kg	40 kg	10 kg
Suniso 3-G	0.38	0.62	0.60	2.32
Pydraul F-9	0.22	0.28	0.63	1.18
Mobil D.T.E.	0.18	0.24	0.38	1.48

C. Low Temperature Viscosity Study

Samples of Skydrol 500 were checked for -40°F. and -65°F. viscosities for comparison with results obtained at Douglas Aircraft Company on the same samples. This was done to check our low temperature viscosity procedure.

Our results differed from the mean by about 0.75%, which is within the 1.0% reproducibility limit for temperatures below 60°F., as specified by ASTM D-445. These results are tabulated below:

	DACO -65°F. Viscosity	REL -65°F. Viscosity
<u>Skydrol 500</u>		
Old Acryloid - Lot A-2762	2,470 cs.	2,489 cs.
New Acryloid - Lot A-2763	2,540 cs.	2,559 cs.

D. Lubricating Study - Paraffinic Petroleum Oil

A paraffinic petroleum oil base stock was treated with fine iron filings for 24 hours at room temperature to determine the effects of such treatment on the oil's wear properties. It was found that this did not change the wear properties. Four-ball wear tests were run with solvent washed balls and distilled water and jewelers rouge cleaned balls with no significant difference. The

DSW 622117

complete removal of all iron particles was found to be important since simple filtration by a filter paper left minute particles which led to gross wear.

Oil: Elk SAE 30 Base Stock, Inv. 2069.

Iron: Fisher Cat. No. 1-57, about 40 mesh filings.

Treatment: 250.7 gm. oil and 50.0 gm. iron stirred 24 hours at room temperature in a 1-liter round-bottom flask.

4-Ball Wear, 1250 rpm, 2 hours, 167°F., steel-on-steel, 40 kg.:

Inv. 2069-no treatment	0.91 mm.
Inv. 2069-Dicalite filtered	0.94
Inv. 2069-iron treated, filtered once through filter paper	1.05 (seizure in 5 seconds)
Inv. 2069-iron treated, filtered through Dicalite coated filter	0.93
Inv. 2069-iron treated, filtered through Dicalite coated filter	0.97*

* Steel balls used in this test cleaned with distilled water and jewelers rouge and others cleaned with Skellysolve B and acetone.

E. Compressor Lubricant - Four-Ball Wear

Shell Four-Ball Wear values were obtained on compressor lubricants to assist in a problem at the Texas City plant which involved lubrication of the seals and rods of reciprocating compressors. This information was used by Texas City to outline a lubricant test program. No further work was done on this project.

Four Ball Wear: 630 rpm, 167°F., 1 hour, steel-on-bronze.

	<u>4 kg.</u>	<u>10 kg.</u>	<u>40 kg.</u>
Coastal Pale Oil	0.90 mm.	1.49 mm.	2.17 mm.
Glycerine	0.48	0.75	2.48
Humble White Oil 335	0.61	1.34	2.80
Ramol	0.59	1.80	2.25

L. R. Stark

R. E. Hatton

de
9-23-60

DSW 622117.01

TABLE XXVII
MIL-L-14107A
SILICATE WEAPONS LUBRICANT

	<u>MIL-L-14107A</u> <u>Specification</u>	<u>OS-106</u>
Flash Point, °F.	325°F., min.	386
Pour Point, °F.	-75°F., max.	Below -75°F.
Viscosity, cs. at 100°F.	5.8 cs., min.	6.297
cs. at -65°F.	900 cs., max.	852.4
Neutralization Number	1.0 max.	0.9
Evaporation Loss	3.0% max.	1.36%
Silica (SiO ₂ , %)	10.7-12.8	11.8%
Oxidation Stability:		
Viscosity at -65°F.	1200 cs., max.	821.8 cs.
Neut. No. Increase	0.5, max.	-0.9
Volatile acids	0.5 mg. KOH/gm. fluid	0.5
Copper Wt. Change mg./cm. ²	+0.2, max.	+0.02
Steel Wt. Change mg./cm. ²	+0.2, max.	0
Fluid Appearance	No gum or insol. sep.	O.K.
Humidity Cabinet Rust	400 hr., min.	552 hr.
Hydrolytic Stability		
Copper Wt. Change mg./cm. ²	+0.5, max.	-0.19
Acid No. Increase		
Oil Layer	0.5 mg. KOH, max.	0.03
Aqueous Layer	0.5 mg. KOH, max.	0
Visc. change at 210°F.	+20%, max.	-2.7%
Insolubles	0.5%, max.	0.3%
Storage Stability at -65°F.	No separation 168 hr.	No separation
at room temperature	No separation 6 mos.	No separation
Rubber Swell (Syn. L.)	15%, max.	7.8%, 9.0%, 14.2%

DSW 622118

STLCOPCB4096150

APPENDIX TABLE I

OS-95 PHYSICAL AND FUNCTIONAL PROPERTIES

Appearance: Slightly cloudy, light yellow, oily liquid.

<u>Viscosity:</u>	<u>Centistokes</u>	<u>Saybolt Universal Seconds</u>
at 210°F.	2.86	35.8
at 100°F.	30.14	142.
at 40°F.	1920.	8500.

Density:

at 100°F.	1.4161 gm./ml.
at 60°F.	1.4305 gm./ml.

Flash Point (COC): 380°F.

Fire Point (COC): 680°F.(1)

Hot Manifold (MIL-F-7100), 1300°F. - Does not flash or burn on tube or after leaving tube.

Low Pressure Spray Flammability (AMS 3150-B) - Flame brightened, but did not increase.

High Pressure Spray Flammability (AMS 3150-B) - Will not ignite.

Molten Metal Ignition (molten aluminum alloy at 1150 to 1250°F.) - Does not spontaneously ignite. Flashes by spark.

Pour Point: +50°F.

Neutralization No.

mg. KOH/gm. to	
pH 11:	0.83(2)

Steam Turbine Oil

Rust (ASTM D-665) 10.0 at 48 hours(3)

<u>Foam (ASTM D-892)</u>	<u>Foam Vol. ml.</u>	<u>Collapse Time Seconds</u>
at 75°F.	30	60
at 200°F.	8	60
at 75°F.	35	60

<u>Shell Four-Ball Wear:</u> 167°F., 630 RPM, 1 hr.	Steel-on-steel	4 Kg. - 0.21 mm.
		10 Kg. - 0.29
		40 Kg. - 0.43
	Steel-on-bronze	4 Kg. - 1.20
		10 Kg. - 1.40
		40 Kg. - 1.70

DSW 622119

APPENDIX TABLE I (continued)

OS-95 PHYSICAL AND FUNCTIONAL PROPERTIES

Almen E.P. Passing load - 48 lb. 24,000 psi.
 Passing torque - 41 lb.

Almen Wear
(wt. loss mg.): 1.3 mg.

Timken Load
(passing): 50 lb., 16,340 psi film strength

Steam Turbine Oil Oxidation (ASTM D-943): (1008 hours exposure)

Viscosity
 100°F. Initial 30.12 cs.
 Final 30.03 cs.
 210°F. Initial 2.87 cs.
 Final 2.86 cs.
Acidity Initial, TAN 0.82
 Final, TAN 0.21
Fluid Appearance: Greenish brown with greenish white deposit
 in tube bottom.
Steel Appearance: Soft black deposit on surface.
Copper Appearance: Black rough (etched) surface.
Four-Ball Wear: Initial 10 Kg.-0.29 mm., Final 10 Kg.-0.41 mm.

Oxidation-Corrosion: (Fed. Spec. 791 Method 530.8, 168 hrs. at 250°F.)

Metal Attack (wt. change mg/cm.²)

Copper - 0.05 no etch, greenish to straw stain.
Aluminum - +0.01 clear
Magnesium - 0 very slight trace surface pitting
Steel - +0.01 clear
Cadmium Plate - -0.01 clear

Viscosity
 100°F. Initial 30.1 cs.
 Final 31.8 cs.
 210°F. Initial 2.86
 Final 2.93
Acidity, TAN
 Initial 0.82
 Final 0.68

Evaporation Loss, %: 0.6

DSW 622120

Fluid Appearance: Clear, light amber, no deposits.

- (1) fluid boiled over onto heater.
- (2) Apparent high acidity due to nature of additives present.
- (3) Rust rating: REL M-400, 10.0 - no rusting.

APPENDIX TABLE I (continued)

OS-95 ADDITIVE STUDY

Aroclor 1242		Aroclor 1248		Sant. 393 %	Sant. 70 %	Other Additive %	4-Ball Wear (1)	Almen EP	
Lot	%	Lot	%					Load Pounds	Torque psi
Pydraul F-9									
Cellulube 220									
T-410	100	--	0	0	0	0	0.81	--	--
--	0	T-512	100	0	0	0	0.83	50	38
T-410	33	T-512	66	0.5	0	0	--	44(2)	39
T-410	33	T-512	65.95	1.0	0.05	0	--	40(2)	34
T-410	33.15	T-512	66.3	0.5	0.05	0	0.55	38(2)	46
T-412	33.15	T-523	66.3	0.5	0.05	0	0.53	34(2)	38
T-410	33	T-512	66	0.5	0.5	0	0.46	48	41
T-412	33	T-523	66	0.5	0.5	0	0.45	50	40
T-412	33.1	T-523	66.1	0.5	0.05	0.25(3)	--	38(2)	40
T-412	33.1	T-523	66.25	0.5	0.05	0.10(4)	--	--	--
T-412	33.0	T-523	66.15	0.5	0.05	0.20(4)	--	--	--
---	33.0	---	66	0.1	0	0	0.53	--	--

- (1) 1 hr., 40 kg., 630 rpm, steel-on-steel.
 (2) squealing during most of run.
 (3) No. 5 Process Oil (Santolube 70 inert).
 (4) Dioctylmaleate.

DSW 622121

APPENDIX TABLE II

OS-104 PHYSICAL AND FUNCTIONAL PROPERTIES

Property	Pydraul 150 Lot Z-465	Houghto-Safe 1010	OS-104
Appearance	----	Clear, green, oily liquid.	Slightly cloudy, almost colorless, oily liquid.
Viscosity, cs. at 210°F.	7.83	3.92	3.54
150°F.	----	8.32	7.34
100°F.	32.7	25.7	20.9
0°F.	907.	6870.	4160.
-10°F.	1980.	23,800.	13,620.
Pour Point, °F.	----	-30	-40
Density: gm./ml. at 100°F.	----	----	1.172
at 60°F.	----	----	1.190
Acidity, TAN mg. KOH/gm.	----	0.16	0.49
Flash Point, COC, °F.	420	525	500
Fire Point, COC, °F.	470	575	600
Molten Metal Ignition (aluminum alloy at 1200-1300°F.)			
Spontaneous: Spark:	No Burns to completion	No Burns - self-extinguishing	No Burns - self-extinguishing
High Pressure Spray Flammability:	Flashed 2-1/2 ft. from orifice-self extinguishing.	Will not ignite.	Will not ignite.
Low Pressure Spray Flammability:	Slight increase of flame.	Slight increase of flame.	Slightly greater increase of flame than for 1010

DSW 622122

APPENDIX TABLE II (continued)

OS-104 PHYSICAL AND FUNCTIONAL PROPERTIES

Property	Pydraul 150 Lot Z-465	Houghto-Safe 1010	OS-104
Hot Manifold	Flashing and burning on tube-occasional burning in pan.	Does not flash or burn on tube or after leaving tube.	Does not flash or burn on tube or after leaving tube.
Foam (ASTM D-892)	----	5 ml.	20 ml.
75°F. foam collapse	----	5 sec.	30 sec.
200°F. foam	----	5 ml.	20 ml.
collapse	----	5 sec.	30 sec.
75°F. foam	----	10 ml.	20 ml.
collapse	----	10 sec.	20 sec.
Emulsion (FS-791, 3201.5) (1% NaCl solution)			
Fluid Layer	----	43 ml.	42 ml.
Emulsion	----	---	---
Water layer	----	37 ml.	38 ml.
Four-Ball Wear (1 hour, 167°F., 630 RPM)			
Steel-on-steel	0.30	0.22	0.31
4 Kg.	0.36	0.33	0.34
10 Kg.	0.51	0.58	0.60
40 Kg.	0.58	0.56	0.56
Steel-on-bronze	0.80	0.65	0.60
4 Kg.	1.58	1.10	1.21
10 Kg.			
40 Kg.			
Almen Load			
Passing, lb.	46	50	50
Torque, lb.	46	51	55
Timken Load			
OK load, lb.	40	15	10
Film Strength, psi	18,200	6,250	5,000
Steam Turbine Oil Rust (D-665) (distilled water)			
1 hour	----	10.0	10.0
24 hours	----	0.6/d/7 (1)	0.4/d/6

DSW 622123

APPENDIX TABLE II (continued)

OS-104 PHYSICAL AND FUNCTIONAL PROPERTIES

Property	Pydraul 150 Lot Z-465	Houghto-Safe 1010	OS-104
Static Heat Test 520°F., 15 min.			
Appearance	Black	No change.	Amber
NN change (TAN)	---	0.15 to 0.20	0.49 to 1.33
Oxidation-Corrosion (FS-791, 5308.4- 250°F., 168 hr.)	---	---	---
Viscosity			
100°F. Initial		25.7	20.9
100°F. Final		25.8	20.8
% Change		+0.7	-0.3
210°F. Initial		3.92	3.54
210°F. Final		3.95	3.57
% Change		+0.8	+0.9
Acidity			
Initial, TAN		0.16	0.49
Final, TAN		0.20	0.35
Change		+0.04	-0.14
Fluid Wt. Loss, %		-0.4	-0.5
Fluid Appearance		green-clear	yellow-clear
Metal Attack, mg./cm. ²			
Copper		-0.17	-0.20
Magnesium		0	0
Steel		0	0
Aluminum		0	-0.01
Cadmium Plate		-0.12	0

(1) M-403 rating system, 10 = perfect.

DSW 622124

APPENDIX TABLE III-A

OS-45 EVALUATION - LUBRICATING PROPERTIES

Shell Four-Ball Wear

Fluid	Test Conditions				Wear Scar Diameter		
	Ball Material (1)	RPM	Hrs.	Temp.	4 Kg.	10 Kg.	40 Kg.
Oronite 8515 (S-19)	S-S	1250	2	167°F.	0.45	0.66	1.15
	S-S	1250	2	400	--	0.99	--
	S-S	630	1	167	0.36	0.50	0.75
	S-B	630	1	167	1.83	1.56	2.12
Oronite 8200 (S-20)	S-S	1250	2	167	0.82	0.95	1.35
	S-S	1250	2	400	--	0.97	--
	S-S	630	1	167	0.60	0.65	0.82
	S-B	630	1	167	1.62	1.60	1.86
OS-45, Type III (S-14)	S-S	1250	2	167	0.41	0.59	1.07
	S-S	1250	2	400	--	0.79	--
	S-S	630	1	167	0.30	0.39	0.69
	S-B	630	1	167	1.91	1.96	2.68
OS-45, Type IV (S-15)	S-S	1250	2	167	0.42	0.64	1.03
	S-S	1250	2	400	--	0.87	--
	S-S	630	1	167	--	0.39	0.64
	S-B	630	1	167	--	1.91	2.00
OS-45, Type IV Hamilton Std. (2)	S-S	1250	2	167	0.49	0.66	1.09
	S-S	1250	2	400	--	0.97	--
OS-45, III Eastern Industries (3)	S-S	1250	2	167	--	0.60	--

See footnotes - next page.

APPENDIX TABLE III-A (continued)
OS-45 EVALUATION - LUBRICATING PROPERTIES

Almen Seizure Load

<u>Fluid</u>	<u>Load, psi⁽⁴⁾</u>	<u>Torque, psi</u>	<u>Remarks</u>
OS-45, III (S-14)	4000	17	
OS-45, IV (S-15)	5000	20	
OS-45, IV, Ba. 2A, Feb., 1957	5000	19	
OS-45, IV, Ba. 6, Feb., 1957	5000	21	
Oronite 8200 (S-20)	3500	13	
Oronite 8515 (S-19)	3000	16	
Oronite 8515, Lot L-620-2	3000	15	
OS-45-III, Eastern Industries ⁽³⁾	----	--	
OS-45-IV, Hamilton Standard ⁽²⁾	7000	22	
OS-45-IV, Vickers test-1	5000	19	From Vickers 4-12-57 use unknown.
OS-45-IV, Vickers test-2	4000	13	4-12-57, 60 hrs., 95% at 400°F.
OS-45-IV, Vickers test-3	6000	21	4-12-57, use unknown.

- (1) S-S is 52100 steel-on-steel; S-B is 52100 steel-on-bronze.
 (2) Hamilton Standard sample used 60 hrs., 95% of time at 400°F.
 (3) From system with pump failure. Time and temperature unknown.
 (4) Almen loads are psi on projected bearing area.

DSW 622126

APPENDIX TABLE III-A (continued)

OS-45 EVALUATION - LUBRICATING PROPERTIES

	Almen Wear ⁽¹⁾					
	4000 psi		3000 psi		2000 psi	
	Wt. Loss	Torque psi	Wt. Loss	Torque psi	Wt. Loss	Torque psi
OS-45, III (S-14)	seized		seized		seized	
OS-45, IV (S-15)	seized		3.1 mg. 16		--	
OS-45, IV Ba. 6, Feb., 1957	seized		seized		1.2 mg. 11	
OS-45, IV Ba. 2A, Feb., 1957	seized		2.1 mg. 12.5		--	
Oronite 8200 (S-20)	seized		seized		0.35 mg. 6	
Oronite 8515 (S-19)	seized		--		seized	
Oronite 8515, Lot L-620-2	seized		seized		seized	
OS-45, IV Vickers test-1	seized		1.5 mg. 13		--	
OS-45, IV Vickers test-2	seized		seized		seized	
OS-45, IV Vickers test-3	seized		3.3 mg. 12.5		--	
OS-45, IV Hamilton Standard test	seized		seized		2.7 mg. 11	
						1.2 mg. 7
						--
						--
						--
						0.6 mg. 5
						--
						--
						--

Timken Passing Load

	Passing Load pounds	Bearing Pressure psi
OS-45, III (S-14)	9	7400
OS-45, IV (S-15)	8	8000
Oronite 8515 (S-19)	10	6800
Oronite 8200 (S-20)	8	6400

(1) Standard wear test is 20 minutes at 4000 psi load. All wear values are average of two successive tests at indicated load.

APPENDIX TABLE III-B

OS-45 EVALUATION - STATIC THERMAL AGING

(400°F., no metal, N₂ atmosphere, glass container)

Appearance

OS-45, Type III (S-14)

All samples from 0 to 480 hours exposure were sediment-free with the ones exposed longest being slightly lighter in color than the initial material.

OS-45, Type IV (S-15)

Samples with 480, 288, and 192 hours exposure had dark sediment and grey-black, needle-like crystals 1 to 3 inches long. Fluid slightly darker than new. Sample with 120 hours exposure had dark sediment with no crystals and was darker colored than new. Exposure for 48 hours resulted in a very small amount of sediment in the slightly darkened fluid and 24 hours exposure the same appearance as new fluid.

Oronite 8515 (S-19)

When material was first removed from sample tubes, the fluid aged 120 to 480 hours had a bloom; whereas, that aged 0 to 48 hours had no bloom. After standing 24 hours, none of the samples had a bloom.

No sediment was present and the samples were progressively darker in color from 0 to 192 hours with the 288 and 480 hour samples appearing slightly lighter.

Oronite 8200 (S-20)

Appearance of O-8200 samples were the same as for Oronite 8515 above.

DSW 622128

STLCOPCB4096160

APPENDIX TABLE III-B (continued)

OS-45 EVALUATION - STATIC THERMAL AGING

(400°F , no metal, N₂ atmosphere, glass container)

Specific Gravity 60/60°F.:

<u>Hours Exposure</u>	<u>OS-45, III (S-14)</u>	<u>OS-45, IV (S-15)</u>	<u>Oronite 8515 (S-19)</u>	<u>Oronite 8200 (S-20)</u>
0	0.887	0.891	---	0.935
24	0.887	0.893	0.932	0.935
48	0.886	0.892	0.932	0.934
120	0.886	0.892	0.931	---
192	0.886	0.892	0.932	---
288	0.886	0.893	0.932	0.932
480	0.886	0.893	0.932	---

Refractive Index -25°C.:

<u>Hours Exposure</u>	<u>OS-45, III (S-14)</u>	<u>OS-45, IV (S-15)</u>	<u>Oronite 8515 (S-19)</u>	<u>Oronite 8200 (S-20)</u>
0	1.4389	1.4392	1.4361	1.4337
24	1.4390	---	---	---
48	1.4390	---	---	---
120	1.4390	1.4392	1.4362	1.4337
192	1.4391	---	---	---
288	1.4391	---	---	---
480	1.4391	1.4390	1.4364	1.4335

Neutralization Number (potentiometric TAN):

<u>Hours Exposure</u>	<u>OS-45, III (S-14)</u>	<u>OS-45, IV (S-15)</u>	<u>Oronite 8515 (S-19)</u>	<u>Oronite 8200 (S-20)</u>
0	0.081	0.104	0.049	0.076
24	0.134	0.155	0.057	0.088
48	0.144	0.233	0.049	0.072
120	0.158	0.326	0.061	0.077
192	0.291	0.375	0.059	0.077
288	0.333	0.442	0.065	0.107
480	0.363	0.465	0.068	0.119

Evaporation Loss, % (ASTM D-972, 400°F., 6-1/2 hr.):

<u>Hours Exposure</u>	<u>OS-45, III (S-14)</u>	<u>OS-45, IV (S-15)</u>	<u>Oronite 8515 (S-19)</u>	<u>Oronite 8200 (S-20)</u>
0	41.5%	29.5%	21.0%	21.1%
24	44.6	30.5	---	---
48	44.99	30.7	---	---
120	45.3	34.2	20.4	20.7
192	41.6	43.3	---	---
288	40.5	39.6	---	---
480	41.6	39.8	21.0	21.2

PENDIX

TABLE III-B (continued)

OS-45 EVALUATION - STATIC THERMAL AGING

(400°F., no metal, N₂ atmosphere, glass container)

Viscosity, cs.:

Hours Exposed	OS-45, III (S-14)		OS-45, IV (S-15)		Oronite 8515 (S-19)		Oronite 8200 (S-20)	
	100°F.	210°F.	100°F.	210°F.	100°F.	210°F.	100°F.	210°F.
0	11.81	3.98	12.14	3.93	24.27	8.12	32.60	11.29
24	11.77	3.89	12.00	3.87	23.02	7.69	30.82	10.62
48	11.60	3.81	11.87	3.83	22.10	7.37	29.10	10.01
120	11.32	3.74	11.47	3.72	19.68	6.51	25.50	8.75
192	11.14	3.66	11.22	3.66	18.05	6.05	23.34	8.00
288	11.06	3.63	11.04	3.60	16.92	5.61	21.46	7.33
480	10.87	3.60	10.86	3.54	15.73	5.23	19.42	6.63

Foam (Microscale 1/10 ASTM D-892):

Fluid	Hours Exposure	Sequence			Collapse	5 min. foam	5 min. foam	Collapse	5 min. foam	Collapse
		75°F.	210°F.	75°F.						
OS-45, III (S-14)	0	11cc	7 min.	7cc	130 sec.	11cc	11cc	6 min.	6 min.	
	120	11	3-1/2	6	90	11	11	3-3/4	3-3/4	
	480	8	3-1/2	6	90	8	8	4	4	
OS-45, IV (S-15)	0	11cc	5 min.	7cc	120 sec.	12cc	12cc	6 min.	6 min.	
	120	11	4-1/2	6	90	11	11	5	5	
	480	10	4	7	90	11	11	4-1/2	4-1/2	
Oronite 8515 (S-19)	0	79cc	2cc-10 min.	45cc	7-1/2 min.	66cc	66cc	3cc-10 min.	3cc-10 min.	
	120	78	3cc-10 min.	30	6	78	78	3cc-10 min.	3cc-10 min.	
	480	71	1cc-10 min.	17	4	68	68	4cc-10 min.	4cc-10 min.	
Oronite 8200 (S-20)	0	79cc	18cc-10 min.	61cc	1cc-10 min.	83cc	83cc	-----	-----	
	120	82	25cc-10 min.	33cc	1cc-10 min.	81	81	10cc-10 min.	10cc-10 min.	
	480	75	-----	12cc	5 min.	78	78	2cc-10 min.	2cc-10 min.	

DSW 622130

APPENDIX

TABLE III-B (continued)

OS-45 EVALUATION - STATIC THERMAL AGING

(400°F., no metal, N₂ atmosphere, glass container)

Flash and Fire Points, C.O.C.:

<u>Fluid</u>	<u>Hours Exposure</u>	<u>Flash, °F.</u>	<u>Fire, °F.</u>	<u>Reflash after Fire, °F.</u>
OS-45, III (S-14)	0	370	455	---
	192	340	455	415
	480	340	455	410
OS-45, IV (S-15)	0	390	455	---
	192	310	435	415
	480	305	430	380
Oronite 8515 (S-19)	0	395	455	---
	192	415	455	420
	480	345	455	---
Oronite 8200 (S-20)	0	405	450	410
	192	410	450	---
	480	405	450	415

Shell Four-Ball Wear

<u>Fluid</u>	<u>Hours Exposed</u>	<u>Test Conditions</u>				<u>Wear Scar Diameter</u>		
		<u>Ball Matl. (1)</u>	<u>RPM</u>	<u>Hrs.</u>	<u>Temp.</u>	<u>4 Kg.</u>	<u>10 Kg</u>	<u>40 Kg</u>
OS-45, III (S-14)	0	S-S	1200	2	167°F	0.41	0.59	1.07
	96	S-S	1200	2	167	--	0.56	1.11
	480	S-S	1200	2	167	--	0.56	1.08
	0	S-S	1200	2	400	--	0.79	--
	96	S-S	1200	2	400	--	0.75	--
	480	S-S	1200	2	400	--	0.80	--
	0	S-B	630	2	167	2.37*	--	--
	96	S-B	630	2	167	2.85*	--	--
	480	S-B	630	2	167	7.80*	--	--
OS-45, IV (S-15)	0	S-S	1200	2	167	0.42	0.64	1.03
	96	S-S	1200	2	167	--	0.66	1.29
	480	S-S	1200	2	167	--	0.62	1.15
	0	S-S	1200	2	400	--	0.87	--
	96	S-S	1200	2	400	--	1.05	--
	480	S-S	1200	2	400	--	1.00	--

DSW 622131

APPENDIX TABLE III-B (continued)

OS-45 EVALUATION - STATIC THERMAL AGING

(400°F., no metal, N₂ atmosphere, glass container)

Shell Four-Ball Wear:

Fluid	Hours Exposed	Test Conditions				Wear Scar Diameter		
		Ball Matl. (1)	RPM	Hrs.	Temp.	4 Kg.	10 Kg	40 Kg
OS-45, IV (S-15) (continued)	0	S-B	630	2	167°F	2.19*	--	--
	96	S-B	630	2	167	1.40*	--	--
	480	S-B	630	2	167	1.16*	--	--
Oronite 8515 (S-19)	0	S-S	1200	2	167	0.45	0.66	1.15
	96	S-S	1200	2	167	--	0.64	1.14
	480	S-S	1200	2	167	--	0.68	1.13
	0	S-S	1200	2	400	--	0.99	--
	96	S-S	1200	2	400	--	0.99	--
	480	S-S	1200	2	400	--	0.99	--
	0	S-B	630	2	167	--	--	--
	96	S-B	630	2	167	2.30*	--	--
	480	S-B	630	2	167	2.29*	--	--
	0	S-S	1200	2	167	0.82	0.95	1.35
	96	S-S	1200	2	167	--	0.87	1.03
	480	S-S	1200	2	167	--	0.96	1.23
Oronite 8200 (S-20)	0	S-S	1200	2	400	--	0.97	--
	96	S-S	1200	2	400	--	0.95	--
	480	S-S	1200	2	400	--	0.99	--
	0	S-B	630	2	167	1.62*	--	--
	96	S-B	630	2	167	1.87*	--	--
	480	S-B	630	2	167	1.85*	--	--

* 5 Kg.

DSW 622132

APPENDIX TABLE III-C

OS-45 EVALUATION - OXIDATION-CORROSION (MIL-H-8446A)

Oxidation-Corrosion Test Results

	<u>OS-45, III</u> <u>(S-14)</u>	<u>OS-45, IV</u> <u>(S-15)</u>	<u>Oronite</u> <u>8515</u> <u>(S-19)</u>	<u>Oronite</u> <u>8200</u> <u>(S-20)</u>
Metal wt. change, mg./cm. ²				
Copper	-0.11	-1.20	-0.29	-0.04
Steel	-0.20	-0.01	+0.06	-0.01
Aluminum	0	0	0	-0.01
Silver	0	-6.28	-0.01	-0.03
Acidity, mg. KOH/gm., TAN				
Initial	0.08	0.104	0.049	0.076
Final	5.9	6.3	0.86	0.49
Change	+5.8	+6.2	+0.81	+0.42
Viscosity, cs.				
100°F. Initial	11.81	12.14	24.24	32.57
100°F. Final	14.36	15.21	19.27	25.10
% Change at 100°F.	+21.6	+25.3	-20.5	-21.0
210°F. Initial	3.98	3.93	8.14	11.26
210°F. Final	3.24	3.54	5.55	7.40
% Change at 210°F.	-18.6	-9.9	-31.8	-34.2
Fluid Evap. Loss, %	8.6	8.0	4.3	3.96
Fluid Condition	dark sl. sediment	dark solids	dark no solids	dark no solids
Foam (Micro Scale 1/10 ASTM D-892)				
75°F. Sequence				
New	11cc	11cc	79cc	79cc
After O&C	20cc	23cc	68cc	90cc
210°F. Sequence				
New	12cc	7cc	45cc	61cc
After O&C	15cc	8cc	25cc	30cc
75°F. Sequence				
New	11cc	12cc	66cc	83cc
After O&C	15cc	15cc	88cc	100cc

DSW 622133

APPENDIX TABLE III-C (continued)

OS-45 EVALUATION.- OXIDATION-CORROSION (MIL-H-8446A)

Oxidation-Corrosion Test Results

	<u>OS-45, III</u> <u>(S-14)</u>	<u>OS-45, IV</u> <u>(S-15)</u>	<u>Oronite</u> <u>8515</u> <u>(S-19)</u>	<u>Oronite</u> <u>8200</u> <u>(S-20)</u>
Four-Ball Wear (1250 RPM, 2 hr., (steel- on-steel)				
New, 167°F., 10 Kg.	0.59	0.64	0.78	0.63
40 Kg.	1.07	1.03	1.14	1.18
O-C, 167°F., 10 Kg.	0.78	0.75	0.63	0.84
40 Kg.	1.14	0.96	1.18	1.17
New, 400°F., 10 Kg.	0.79	0.87	0.71	0.99
O-C, 400°F., 10 Kg.	0.71	0.74	0.99	1.06

DSW 622134

APPENDIX TABLE III-D

OS-45 EVALUATION - SHEAR STABILITY

Diesel Injector Shear Machine

Fluid	Passes Through Shear Machine	Viscosity			
		at 100°F.		at 210°F.	
		cs.	%, Decrease	cs.	%, Decrease
OS-45, III (S-14)	0	12.17	0	4.00	0
	1	11.95	1.81	3.92	2.00
	2	11.80	3.04	3.91	2.25
	4	11.66	4.19	3.87	3.25
	6	11.56	5.01	3.80	5.0
	8	11.53	5.26	3.76	6.0
	10	11.43	6.08	3.72	7.0
	12	11.36	6.66	3.69	7.75
OS-45, IV (S-15)	0	12.14	0	3.97	0
	1	11.93	1.73	3.93	1.01
	2	11.81	2.72	3.86	2.77
	4	11.65	4.04	3.76	5.29
	6	11.57	4.70	--	--
	8	--	--	3.72	6.30
	10	11.43	5.85	3.67	7.56
	12	--	--	--	--
Oronite 8515 (S-19)	0	24.24	0	8.14	0
	1	23.39	3.51	7.86	3.44
	2	23.06	4.87	7.71	5.28
	4	22.75	6.15	7.63	6.27
	6	22.50	7.18	7.52	7.62
	8	22.35	7.80	7.47	8.23
	10	22.30	8.00	7.42	8.85
	12	--	--	--	--
Oronite 8200 (S-20)	0	32.57	0	11.26	0
	1	31.65	2.82	10.93	2.93
	2	31.24	4.08	10.79	4.17
	4	30.93	5.04	10.64	5.51
	6	30.58	6.11	10.56	6.22
	8	30.36	6.79	10.48	6.93
	10	30.28	7.03	10.43	7.37
	12	30.23	7.18	10.42	7.46

DSW 622135

APPENDIX TABLE III-E

OS-45 EVALUATION - EVAPORATION LOSS

(ASTM D-972, 6-1/2 hours)

Temperature, °F.	Weight Loss, %			
	OS-45, III (S-14)	OS-45, IV (S-15)	Oronite 8515 (S-19)	Oronite 8200 (S-20)
340	5.6	--	--	--
345	6.9	--	--	--
350	8.4	8.5	8.5	7.9
358	9.7	--	--	--
362	10.3	--	--	--
365	12.2	--	--	--
375	19.2	--	--	--
385	29.5	--	--	--
392	37.7	--	-- (2)	--
400	41.5 (1)	29.5	21.1	21.0 (3)
415	65.8	--	--	--

(1) 21.23% WADC Technical Report 55-89, Part I.

(2) 22.3% WADC Technical Report 55-89, Part IV.

(3) 7.9% WADC Technical Report 55-89, Part III.

DSW 622136

APPENDIX TABLE IV

RUST INHIBITED PYDRAUL F-9 AND PYDRAUL 625

Physical and Functional Properties

Property	Pydraul F-9 Lot Z-81	Pydraul F-9 with 0.3% Santolube 70 (OS-101)	Pydraul 625 Lot A-1505	Pydraul 625 with 0.3% Santolube 70 (OS-102)(1)
Four-Ball Wear (1 hr., 630 rpm, 167°F.)				
Steel-on-steel	4 Kg. 10 Kg. 40 Kg.	0.21 mm 0.28 0.67	0.20 mm 0.26 0.51	0.20 mm 0.26 0.55
Steel-on-bronze	4 Kg. 10 Kg. 40 Kg.	-- 1.18 1.28	-- 0.75 0.84 1.42	-- 0.69 0.77 1.03
Almen Wear				
Wt. Loss, mg.	2.0	3.5	2.2	2.0
Torque, lb.	15	17	16	16
Almen Load				
Passing Load, lb.	50	45	46	50
Torque, lb.	44	49	50	49
Timken Load				
Passing Load, lb.	45	45	40	40
Film Strength, psi.	19,000	19,600	17,200	18,100
Steam Turbine Oil Rust (D-665, distilled water)				
1 hour (2)	10.0	10.0	10.0	10.0
24 hours	5.5	10.0	8.0	10.0

DSW 622137

APPENDIX TABLE IV (continued)

RUST INHIBITED PYDRAUL F-9 AND PYDRAUL 625

Physical and Functional Properties

Property	Pydraul F-9 Lot Z-81	Pydraul F-9 Santolube 70 (OS-101)	Pydraul 625 Lot A-1505	Pydraul 625 with 0.3% Santolube (OS-102) (1)
Oxidation-Corrosion (250°F., 168 hr.)				(1) 50 ppm DCF-200, 350 cs. included in OS-102
Metal attack, mg./cm. ²				
Copper	-0.16	-0.24	-0.13	-0.17
Magnesium	0	0	0	0
Steel	0	0	0	-0.01
Aluminum	0	0	0	+0.01
Cadmium Plate	0	-0.07	0	-0.01
Acidity, TAN, mg. KOH/gm.				
Initial				
Final				
Change				
Viscosity				
100°F. Initial	56.7	57.0	137.	136.
100°F. Final	58.6	58.6	143.	140.
% Change	+3.4	+2.8	+4.2	+3.1
210°F. Initial	5.51	5.54	5.73	5.70
210°F. Final	5.61	5.67	5.81	5.79
% Change	+1.8	+2.3	+1.4	+1.6
Fluid Weight Loss, %	0.47	0.55	0.64	0.48
Fluid Condition	Yellow-clear	brown-clear	yellow-clear	brown-clear
4-Ball Wear (1 hr., 630 rpm, 167°F.)				
New Steel-on-steel 10 Kg.	0.28	0.26	0.28	---
40 Kg.	0.63	0.51	0.61	---
O&C Steel-on-steel 10 Kg.	0.30	0.29	0.30	---
40 Kg.	0.76	0.91	0.78	---
New Steel-on-bronze 10 Kg.	1.18	0.84	0.98	---
40 Kg.	1.28	1.42	1.07	---
O&C Steel-on-bronze 10 Kg.	1.08	1.00	0.86	---
40 Kg.	1.50	1.39	1.12	---

DSW 622138

APPENDIX TABLE IV (cont'd)

RUST INHIBITED PYDRAUL F-9 AND PYDRAUL 625

Physical and Functional Properties

Property	Pydraul F-9 Lot Z-81	Pydraul F-9 with 0.3% Santolube 70 (OS-101)	Pydraul 625 Lot A-1505	Pydraul 625 with 0.3% Santolube 70 (OS-102)
Foam (D-892) at 75°F.	4 ml.	45 ml.	10 ml.	24 ml.
200°F.	8	12	14	15
75°F.	40	48	10	20
Collapse (all collapsed in less than 10 minutes)				
Emulsion (FS-791, 3201.5, 1% NaCl solution)				
Fluid layer	40	43	42	45
Water layer	40	37	38	35

(1) 50 ppm. DCF-200, 350 cs. included in OS-102.

(2) REL: M-400 rating, 10.0 = No rusting.

DSW 622139

APPENDIX

ASTM COOPERATIVE TESTS

Section VI - Fire Resistance

A. Flash and Fire Points

1. Procedure and Apparatus

The Cleveland Open Cup method was conducted according to the procedure outlined in ASTM Standard Method D-92-52. The apparatus used was a Cleveland Open Cup, Fisher/Tag Improved Model, ASTM, electrically heated, Fisher Scientific Company No. 13-538.

2. Test Results

<u>ASTM Reference Fluid</u>	<u>Flash Point, °F.</u>	<u>Fire Point, °F.</u>
1-A-1	430	465
2-B-1	no result -- boiling and frothing	
3-C-1	510	620
4-D-1	395	665
5-D-1	455	630
6-E-1	no result -- boiling and frothing	

B. High Pressure Spray

1. Procedure and Apparatus

The procedure used is outlined in Military Specification MIL-F-7100 (December, 1950) Section 4.2.2.8.1 as follows:

"Assemble equipment for applying 1,000 psi to the test fluid. A suitable arrangement shown in Figure 1 (of the specification) consists of a nitrogen bottle, a hydraulic cylinder and necessary lines, valves, and gauges. The orifice nozzle shall incorporate a clean, sharp-edged orifice approximately 0.0145 inch in diameter in metal plate or disc 0.064 inch thick and with a flat area of at least 0.38 inch in diameter on the fluid side of the orifice. The cylinder shall be charged with the test fluid at the temperature of 15.6° to 37.8°C. (60° to 100°F.). Apply nitrogen pressure so that the pressure on the fluid side is 1,000 psi, and open the valve at the orifice and attempt to ignite the spray at the orifice with an oxy-acetylene torch adjusted to a neutral flame and using a No. 3 Purox tip or equivalent, while maintaining the fluid pressure at 1,000 + 50 psi. If the fluid cannot be ignited at the orifice, the test shall be repeated by applying the flame at increasing distances from the orifice up to 18 inches from the orifice. The results shall be recorded as follows: 'Will not ignite', 'Flashes with difficulty', or 'Flashes readily'. Also, the distance

from the orifice at which ignition or flashing is carried downstream from the test flame area and whether any flashing is self-extinguishing or results in a sustained fire shall be recorded."

An aircraft type hydraulic accumulator with no diaphragm was used by this laboratory instead of the hydraulic cylinder reservoir specified. The apparatus was cleaned after each test by circulating first kerosene and then acetone, followed by air and nitrogen blowing to remove the solvents. Attempts to ignite the spray stream were made up to distances of 3 feet from the orifice instead of the specified 18 inches.

2. Test Results

<u>ASTM Reference Fluid</u>	<u>Result</u>
1-A-1	Flashes and burns readily.
2-B-1	Will not ignite.
3-C-1	Flashes with difficulty only when the torch was about 2 feet from orifice. Self-extinguishing.
4-D-1	Will not ignite.
5-D-1	Will not ignite.
6-E-1	Will not ignite.

C. Low Pressure Spray Flammability

1. Procedure and Apparatus

The procedure used is outlined in Aeronautical Material Specification SMS 3150B (Society of Automotive Engineers, Inc.), Revision of February 15, 1953, Section 4.9.2 as follows:

"Prepare a shallow metal pan approximately 1 ft. square with sides not over 2 inches high, into which is placed a platform or grid made from 1/8 to 1/4 inch mesh wire and spaced 1/2 inch from the bottom of the pan. Saturate 10 gm. of cotton waste with 20 gm. of Grade 1120 engine oil, roll the waste into a ball and place in the center of the grid.

"Place a quantity of the test fluid in a Binks Thor No. 7 paint spray gun (0.070 inch orifice). Set line pressure to 40 psi, and adjust gun to produce a dense cone-shaped spray.

"Ignite the oil soaked waste and allow the fire to reach maximum intensity. Aim spray gun on the fire as shown in Figure 6 (center of spray 1-1/2 to 3 inches above burning waste and 16 to 20 inches away) and intensify fire

DSW 622141

by depressing trigger slightly (air jet only). Introduce fluid mist by fully depressing trigger in bursts. Record the results as 'increase', 'no increase', or 'decrease' in fire when the fluid mist is introduced into fire. Spray several ounces of liquid to be certain of results.

"Use new waste for each test and insure that the pan is approximately at room temperature."

2. Test Results

<u>ASTM Reference Fluid</u>	<u>Result</u>
1-A-1	Greatly increased flame
2-B-1	Decrease in flame
3-C-1	No increase in flame
4-D-1	No increase in flame
5-D-1	No increase in flame
6-E-1	No increase in flame

D. Hot Manifold

1. Procedure and Apparatus

The simulated exhaust stack section and test procedure described in Military Specification MIL-F-7100, Section 4.2.2.8.2 was followed. Two 10 ml. portions were dropped at the specified rate of 40 to 60 seconds per 10 ml. for each fluid tested. The manifold was heated to 1300°F. and the pan and shield around the manifold was cooled nearly to room temperature between each test.

2. Test Results

<u>ASTM Reference Fluid</u>	<u>Hot Manifold Results</u>
1-A-1	Flashing and burning on tube (mostly along bead). Does not flash or burn after leaving tube.
2-B-1	Does not burn on tube. Only occasional flashing along bead (very small flames). Does not flash or burn after leaving tube.
3-C-1	Does not flash or burn on tube or after leaving tube.
4-D-1	Does not flash or burn on tube or after leaving tube.
5-D-1	Does not flash or burn on tube or after leaving tube.

APPENDIX - ASTM COOPERATIVE TESTS
Section VI

Page 4

<u>ASTM Reference Fluid</u>	<u>Hot Manifold Results</u>
6-E-1	Flashes along bead. Does not flash or burn after leaving tube.
7-F-1	Burns on tube. Droplets burn while falling to pan. Does not flash or burn in pan.
8-G-1	Burns on tube and falling droplets. Does not flash or burn in pan.

DSW 622143

STLCOPCB4096175

APPENDIX

ASTM COOPERATIVE TESTS

Section VII - Lubricating Properties

A. Four-Ball Wear

1. Procedure and Apparatus

The apparatus used was a Shell Four-Ball Wear Machine manufactured by Roxana Machine Works, St. Louis, Missouri, which has appropriate devices for rotating ball speed control, ball loading, temperature control and test time control. Test balls used were SKF Industries 0.5 inch, grade 1, EP, Atlas Ball Bearings of SAE 52-100 steel and Hartford Steel on bronze at a spindle speed of 635 rpm, a temperature of 167°F., and loads of 4, 10 and 40 kilograms, for one hour.

The test balls were cleaned with Skellysolve B and acetone prior to the test. The cleaned balls were chucked in the machine and the fluid introduced, the load was applied, and the fluid and balls heated to 167°F. At the test temperature, the machine was started and run for 1 hour. The wear spot diameter on the three lower stationary balls was measured, averaged, and reported.

2. Test Results

ASTM Reference Fluid	Wear Scar Diameter, mm, 167°F., 1 hour 635 rpm					
	Steel-on-Steel			Steel-on-Bronze		
	4 Kg	10 Kg	40 Kg	4 Kg	10 Kg	40 Kg
1-A-1	0.21	0.24	0.57	1.52	2.11	2.69
2-B-1	0.49	0.63	1.05	1.13	1.17	1.65
3-C-1	0.27	0.37	0.55	0.60	0.82	1.05
4-D-1	0.32	0.32	0.79	0.81	0.79	1.03
5-D-1	0.21	0.24	0.66	0.79	1.03	1.17
6-E-1	0.52	0.59	0.70	0.80	0.85	0.98

B. Almen Extreme Pressure Test

1. Procedure and Apparatus

An Almen EP testing machine was used in which a 1/4 inch diameter drill rod journal is rotated at 600 rpm between the halves of a split bushing, the complete assembly being inversed in the test oil. After a 30 second no load break-in period, two pound weights are added to the loading system at 10-second intervals until seizure occurs or 50 pounds have been added. The loading system is such that the load is applied to the bushing halves

APPENDIX - ASTM COOPERATIVE TESTS
Section VII

Page 2

with each two-pound weight equivalent to 1,000 lbs. per sq. in. on the projected bearing area. The passing load for a completed 10-second interval is recorded along with the torque in pounds at the end of the interval. Duplicate runs were made and the average is reported.

The test specimens (rod and split bushings) were obtained from Laboratory Equipment Company, Mooresville, Indiana. Prior to the test they are washed with Skellysolve B and acetone.

2. Test Results

<u>ASTM Reference Fluid</u>	<u>Almen Passing Load Pounds</u>	<u>Almen Passing Torque Pounds</u>
1-A-1	14	21
2-B-1	8	12
3-C-1	50	50
4-D-1	50	38
5-D-1	50	47
6-E-1	3	9

DSW 622145

STLCOPCB4096177

APPENDIX

ASTM COOPERATIVE TESTS

Section VIII - Stability

A. Oxidation - D-943

1. Procedure and Apparatus

The Oxidation Characteristics of Inhibited Steam-Turbine Oils method was conducted according to the procedure outlined in ASTM Standard Method: D-943-54.

A 250-ml. filter flask fitted with a stopper and glass tube extending down into the flask was added to the oxygen inlet system between the flow meters and oxidation tubes. Distilled water was maintained in this flask to a depth of about 3/4 inch on the center inlet tube. Oxygen bubbled through this water and passed on to the oxidation tubes resulted in slower removal of water from the oxidation tubes in the cases where the water layer is lighter than the oil layer. Neutralization values were run by a potentiometric method, ASTM Designation: D-664-54, with results reported as total acid number (TAN) or total base number (TBN), mg. KOH per gm.

2. Test Results

Refer to Table 1 following.

B. Oxidation and Corrosion Test

1. Procedure and Apparatus

The procedure and apparatus used in this test is outlined and described in Federal Test Method Standard No. 791, Method 5308.4. Test conditions used were 250°F., 168 hours, and an air flow of 5 ± 0.5 liters per hour to each tube.

2. Test Results

See Table 2 following.

DSW 622146

STLCOPCB4096178

TABLE 1

ASTM REFERENCE FLUIDS NEUTRALIZATION VALUE
IN ASTM D-943 OXIDATION TEST

ASTM Reference Fluid	0 Hours	168 Hours	336 Hours	504 Hours	1008 Hours	Final Catalyst Coil Appearance	Final Fluid Appearance
1-A-1 TAN	0.127	0.131	0.041	neg.	0.095	steel-bright	light orange-yellow and cloudy
TBN	0.029	0.004	0.004	0.004		copper-dark	no sludge
2-B-1 TAN	.318	0.554	4.56	12.599		steel-severely attacked	sludge at 336 hr. fluid-
TBN	7.78	2.997	2.997	3.546		copper-dark etched	dark reddish brown
3-C-1 TAN	0.023	0.454	1.486	2.624		steel-bright	sludge at 168 hr. fluid-
TBN						copper-dark etched	very dark brown
4-D-1 TAN	0.0002	neg.	0.0025	neg.	0	steel and copper both	no sludge - clear
TBN	0.0003	0.0029	0.0029	0.0014		bright with dark spots on about 25% of surface	
5-D-1 TAN	0.063	0.116	0.173	0.224	0.329	steel-bright	sludge at 336 hr. fluid-
TBN						copper-reddish brown	dark brown
6-E-1 TAN	0.264	0.196	0.296	0.562	12.672	steel-etched	sludge at 336 hr. fluid-
TBN	1.17	0.704	0.704	0.661		copper-etched	very dark brown

DSW 622147

TABLE 2

ASTM Reference Fluid	Metal Weight Loss, mg./cm. ²					Neutralization		Evap. Loss %	Viscosity, cs.			
	Cadmium Plate	Aluminum	Copper	Magnesium	Steel	Initial TAN	NN Change		Initial 100°F.	% Change 100°F.	Initial 210°F.	% Change 210°F.
1-A-1	0 Clear	0 Clear	-0.014 Very slight etch-- slight brown stain	0 Clear	0 Clear	0.127	-0.043	0	32.33	+0.23	4.944	+1.27
3-C-1	-0.007 Clear	-0.007 Clear	-0.037 Slight brown stain	-0.007 Clear	0 Clear	0.023	+0.461	0.17	45.92	+9.95	4.756	+4.96
4-D-1	-0.022 Clear	0 Clear	-0.007 light green to red stain	0 Clear	-0.007 Clear	0.0002	0	0.35	41.77	+4.24	3.007	+0.73
5-D-1	-0.022 Clear	-0.007 Clear	-0.145 red- brown to purple stain	-0.022 Clear	0 Clear	0.063	+0.013	0	54.04	+3.20	5.436	+1.97
7-F-1	-0.014 Clear	-0.007 Clear	0 bright orange- red stain	0 Clear	-0.014 Clear	0.070	-0.006	1.93	24.32	+7.23	7.681	+6.69
8-G-1	-0.022 Clear	0 Clear	+0.007 v. sl. etch lt. green to red stain	-0.007 no stain trace of surface pitting	0 Clear	0.093	+0.012	0.29	64.67	+0.40	20.78	+0.53

DSW 622148



DSW 622148.01