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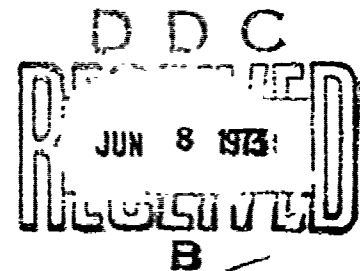
**CHEMICAL, PHYSICAL AND MECHANICAL
PROPERTIES OF LOW DENSITY PHOSPHATE
ESTER HYDRAULIC FLUIDS**

F. BROOKS

H. SCHWENKER

TECHNICAL REPORT AFML-TR-73-78

APRIL 1973



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**AIR FORCE MATERIALS LABORATORY
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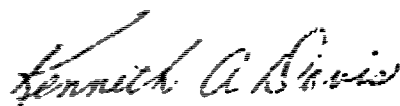
FOREWORD

This report was prepared by the Lubricants and Tribology Branch of the Nonmetallic Materials Division, Air Force Materials Laboratory, Air Force Systems Command, Wright-Patterson Air Force Base, Ohio. The work was conducted under Project 7340, "Nonmetallic and Composite Materials", Task No. 734008, "Energy Transfer Fluids". Herbert Schwenker and F. C. Brooks were the Project Engineers.

The purpose and results of the effort expended between March 1970 and June 1972 are presented.

The manuscript was submitted for publication by the authors in August 1972.

This technical report has been reviewed and is approved.



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ABSTRACT

Three low density phosphate ester fluid candidates, MLO-70-32, MLO-70-62 and MLO-71-37, were characterized as to their physical and chemical properties. MLO-71-37 which exhibited the most acceptable characteristics was further evaluated for its reactions in simulated functional and system environments. MLO-71-37 was found to possess the most satisfactory overall properties and exhibited potential operational capability over a temperature range of -65 to 275 F. All three candidate fluids displayed a sensitivity to elastomeric materials, with specific manufacturer and compound designations required for satisfactory performance.

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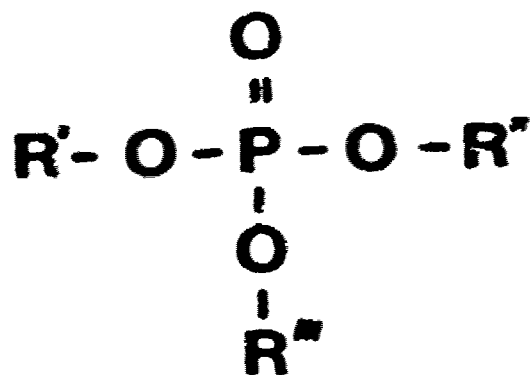
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SECTION I
INTRODUCTION

Tertiary phosphate esters (Fig. 1) have found wide usage as fire-resistant hydraulic fluids in commercial aircraft and their ground support equipment. These materials have not been widely used in current operational military aircraft and support equipment because of incompatibility with currently used MIL-H-5606(C) hydraulic fluid, elastomers, paints and electrical insulation. Their limited high temperature capability of +225°F also prevents their use in most of the newer military aircraft. General characteristics of the tertiary phosphate esters are well known and have been covered in great detail in the literature (1)(2). A recent advance in the state-of-the-art within this area has been the development of the so called, "Low Density Phosphate Esters". These low density phosphate esters have found wide acceptance and usage in hydraulic systems of commercial aircraft. The exact chemical composition of these low density phosphate esters is proprietary knowledge of their producers (of which there are several), however, they are probably primarily trialkyl phosphate esters. These low density phosphate esters have better low temperature properties, somewhat lower densities and are significantly lower in cost than the previously used alkyl-aryl phosphate esters. Fire resistance of the low density phosphate esters is good but somewhat lower than that of the alkyl-aryl phosphate esters. Phosphate ester hydraulic fluids are covered under Boeing Aircraft Company Material Specification BMS-311-C Hydraulic Fluid, Fire Resistant. This specification is used by industry and the commercial airlines. It covers three fluid types. Type III deals with the low density phosphate esters.

TERTIARY PHOSPHATE ESTERS



WHERE R', R'' & R''' ARE ARYL
OR
ALKYL GROUPS

FIGURE 1 STRUCTURE, TERTIARY PHOSPHATE ESTERS

Although the low density phosphate ester hydraulic fluids are not suitable for use in most current operational military aircraft without extreme and costly retrofit or risking operational failure by accidental mixing of the phosphate esters with currently used petroleum hydraulic fluids (they are absolutely non-compatible), they are of concern to the Air Force because they are used in commercial aircraft purchased and used by the Air Force. For example, it is used in Boeing 707 (VC-137) aircraft used for transport of personnel and cargo.

Military Specification MIL-H-83306 (USAF) Hydraulic Fluid, Fire Resistant, Phosphate Ester Base, Aircraft covers low density fire resistant hydraulic fluids for use over the temperature range of -55 to +225°F in Air Force owned and operated commercial aircraft (designed to use these materials in their hydraulic systems). This specification was issued to insure quality control and fluid performance of the low density phosphate esters used by the Air Force.

Data supplied by the various producers of the low density phosphate esters indicated their operational capabilities were considerably improved over the previously used alkyl-aryl phosphate esters. This investigation was concerned with an assessment of their potential capabilities as fire resistant aerospace hydraulic fluids for advanced system use, since there are compatible seals, paints and wiring which can be used with them in new designs. It is the purpose of this document to provide the results of the characterizations conducted on the candidate low density phosphate esters.

SECTION II

MATERIALS SELECTION

Low density fire resistant phosphate ester hydraulic fluids were obtained from the manufacturers of the three materials being used by commercial airlines. These materials were given laboratory code numbers MLO-71-37, MLO-70-62 and MLO-70-32.

SECTION III

PHYSICAL AND CHEMICAL CHARACTERIZATION

A. Thermal Stability

Thermal stability of the three low density phosphate esters was determined by the Penn State Bomb Test described in MIL-B-27601 with the exception that the test temperatures were 350, 400, 450 and 500°F, rather than the specified temperature of 700°F. All three of the low density phosphate ester hydraulic fluids (MLO-71-37, MLO-70-62 and MLO-70-32) had good thermal stability at 350°F (see Table I). Thermal stability tests were run at 400, 450 and 500°F on the MLO-71-37 phosphate ester hydraulic fluid which appeared to have the best overall properties based on other physical and chemical characterization results. The thermal stability characteristics of MLO-71-37 were unsatisfactory at 400°F, and rapidly deteriorated at 450 and 500°F (Table II). Thermal stability of the phosphate ester MLO-71-37 is estimated to be about 375°F on the basis of these tests.

TABLE I

Low Density Phosphate Esters
Thermal Stability Tests
(Per MIL-H-27601*)
6 Hours at 350°F

	<u>MLO-71-37</u>	<u>MLO-70-62</u>	<u>MLO-70-32</u>
Viscosity @ 100°F, (cs)			
Original	9.93	10.19	11.19
After Exposure	9.94	10.46	11.24
Increase %	0.1	2.6	0.4
Neutralization No. mgKOH/gm			
Original	0.02	0.02	0.04
After Exposure	0.06	0.07	0.05
Increase %	0.04	0.05	0.01
Appearance After Exposure	Clear	Little Change	Unchanged
Corrosion Specimen Weight Changes, mg/sq. cm			
M-10 Steel	0.00	0.04	0.00
Bronze	0.00	0.04	0.01
52100 Steel	0.00	0.04	0.01

* Except for temperature change as noted.

TABLE II

Thermal Stability Test of MLO-71-37
(Per MIL-H-27601) (1)

6 Hours @ 350°, 400°, 450°, and 500°F

	Temperature			
	350°F	400°F	450°F	500°F
Viscosity @ 100°F cs				
Original	9.93	9.93	9.93	9.93
After Exposure	9.94	10.31	11.29	410
Increase %	0.1	3.8	13.7	4.028
Neutralization No. mgKOH/g				
Original	0.02	0.02	0.02	0.02
After Exposure	0.06	8.91	19.93	76.42
Increase	0.04	8.89	19.91	76.40
Appearance				
After Exposure	Clear	Clear	Blue to Green, Clear	Blue to Dark Brown Dark Brown Precipitate
Change in Weight of Metals				
mg/sq. cm				
M-10 Steel	-0.00	-0.00	-0.01	+0.01 (4)
Bronze	-0.00	-0.02	-0.02 (2)	-0.46 (5)
52100 Steel	-0.00	-0.00	-2.38 (3)	-5.1 (6)

- (1) Except for temperature changes as noted.
- (2) Some area of gray discoloration.
- (3) Etched: gray discoloration.
- (4) Dark amber discoloration.
- (5) Coppertone discoloration.
- (6) Corrosion, etched, white gray discoloration.

B. Shear Stability

Shear stability of the low density phosphate ester hydraulic fluids was determined by the Sonic Shear Method as specified in MIL-H-5606(C) (Table III). Two of the phosphate ester fluids (MLO-70-62 and MLO-71-37) exhibited satisfactory shear resistance (conformed to MIL-H-5606(B)); however, the other phosphate ester fluid (MLO-70-32) had much poorer shear resistance and failed to meet MIL-H-5606(C) requirements. All three fluids however, conform to MIL-H-83306 (USAF) in all other respects.

C. Lubricity

Lubricity of the low density phosphate ester fluids was determined by the Shell Four-Ball Wear Test Method. The results of the tests, shown in Table IV, reveal that the wear characteristics of three candidate fluids are within the requirements of specifications MIL-H-5606(C) and MIL-H-83306 (USAF). Following these tests a more extensive low/temperature wear profile was conducted with fluid MLO-71-37. The results of this effort are presented in Table V. Some unusual occurrences were noted on the four-ball specimens which were run at 350°F and 40 kilogram loads. There was an unusual accumulation of a very viscous soap-like material at the edge of the wear scar, as shown in Figs. 2 and 3. Infrared spectrum and emission analysis of this material has identified it as an iron phosphate formed as a decomposition product of the organic phosphate ester fluid. Also, immediately outside of the wear scars are areas from which ball material has been removed (Figs. 4 and 5). Although the exact mechanism of this metal removal process has not been determined, the possibility exists that it results from the attack of acids formed from fluid degraded in the high temperature scar areas.

TABLE III

Shear Stability
(Sonic Shear Per MIL-H-5606B)

	<u>Reference Fluid</u>	<u>MLO- 71-37</u>	<u>MLC- 70-32</u>	<u>MLO- 70-62</u>
Viscosity @ 130°F, cs:				
a. Before Irradiation	10.16	6.79	7.87	7.16
b. After Irradiation 30 ml fluid for 5 min	8.66	6.33	6.62	6.70
c. % Change	-14.76	-6.77	-15.92	-6.42
Viscosity @ -40°F, cs:				
a. Before Irradiation	385.9	414.2	319.2	303.4
b. After Irradiation 30 ml fluid for 5 min	341.4	376.8	270.9	285.2
c. % Change	-11.52	-9.0	-15.12*	-6.01
Neutralization No. mgKOH/g				
a. Before Irradiation	---	0.02	0.04	0.02
b. After Irradiation 30 ml sample for 30 min	---	0.22	0.34	0.24
c. Change	---	+0.20	+0.30	+0.22

* Failed

TABLE IV

Shell Four-Ball Wear Data

(167°F, 600 rpm, 40 kg Load, 2 Hours
Using 52100 Steel Balls)

<u>MLO-Number</u> <u>Fluid</u>	<u>Average Wear Scar Diameter (mm)</u>
MLO-70-32	0.556
MLO-70-62	0.469
MLO-71-37	0.567

TABLE V

Four-Ball Wear Test Results
MLO-71-37

SPEED: 600 rpm
TIME: 1 Hour
Ball Dia: 0.500 in.

Avg Scar Dias in mm

Temperature °F	Material	Load, Kilograms				
		1	4	10	20	40
167	52100	.21	.27	.33	.40	.49
	M-10	.17	.23	.28	.37	.49
200	52100					.60
	M-10					.55
250	52100					.64
	M-10					.56
300	52100					.71
	M-10					.55
350	52100					.73
	M-10					.55



FIGURE 2 WEAR SCAR WITH MATERIAL DEPOSIT



FIGURE 3 WEAR SCAR WITH MATERIAL DEPOSIT

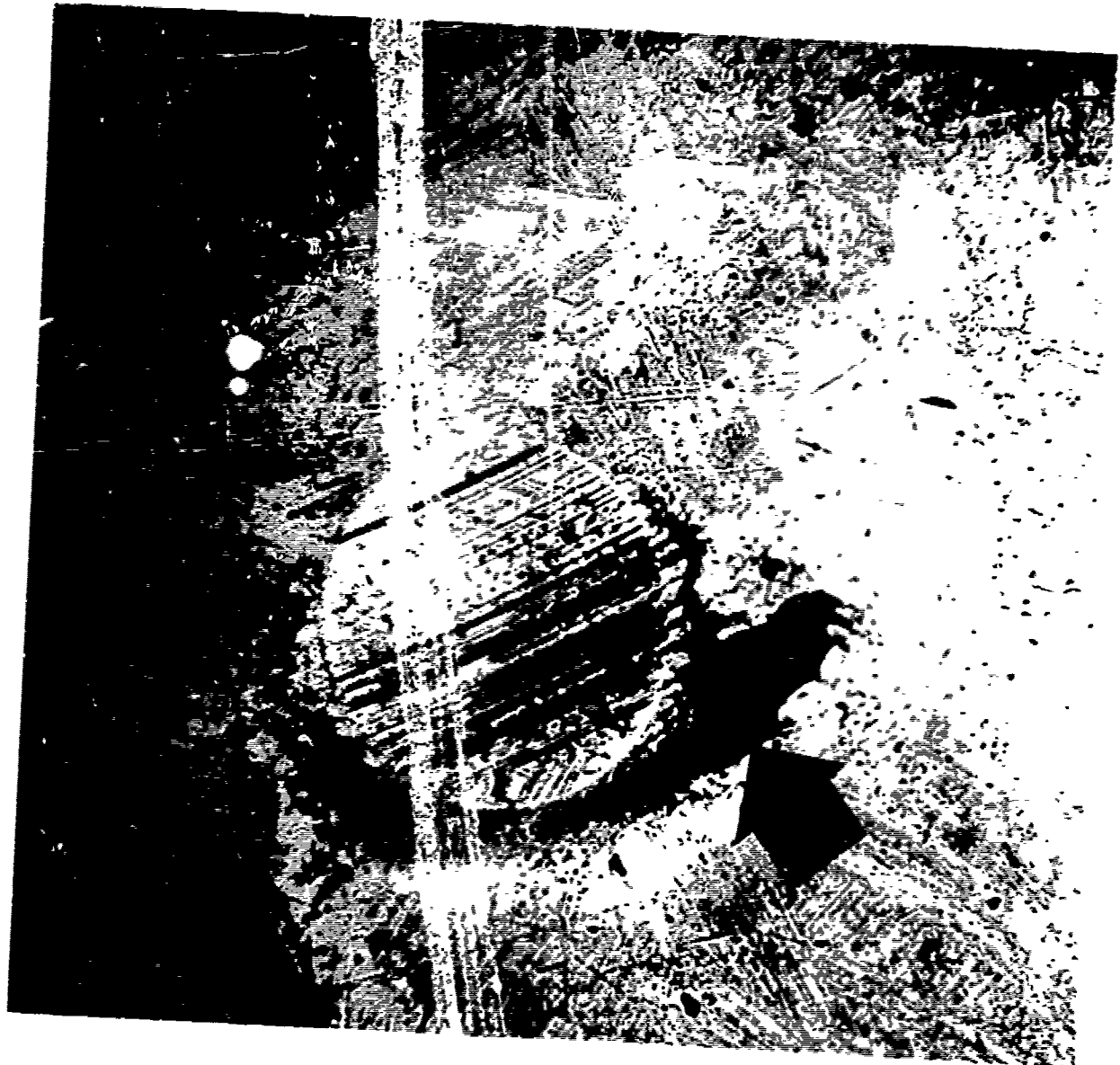


FIGURE 4 WEAR SCAR WITH UNUSUAL METAL REMOVAL



FIGURE 5 WEAR SCAR WITH UNUSUAL METAL REMOVAL

TABLE VI

Hydrolytic Stability Tests

168 Hours @ 225°F
MIL-H-5606(B) Amendment III

(0.87% by Weight of Distilled Water)
(99.13% by Weight of Oil Sample)

Tests On The Original Oil

	MLC-70-32	MLO-71-37	MLO-70-62
Viscosity @ 130°F, cs	7.64	6.62	6.76
Neutralization No. mgKOH/g	0.04	0.06	0.03

Tests On The Hydrolyzed Oil

Viscosity @ 130°F, cs	8.02	7.01	6.70
Neutralization No. mgKOH/g	0.01	0.01	0.03
Evaporation Loss, %	1.3	2.4	1.75
Appearance After Hydrolysis	Give green No Ppt.	Greenish Blue/ No Ppt.	No change No Ppt.
Increase In Viscosity, %	5.0	5.9	0.9
Decrease In Neutralization No. mgKOH/g	0.03	0.05	0.00

Weight Change Of Metals, mg/sq.cm

Magnesium	+0.01 (1)	0.01	0.00
Aluminum	0.00	0.00	0.00
Copper	-0.04 (2)	0.01 (4)	0.02 (2)
Cadmium	0.00 (3)	0.03	0.01 (2)
Steel	0.00 (1)	0.00	0.00 (3)

- (1) Brown Spots
(2) Moderate Tarnish - 2C
(3) Light Bronze and Brown Spots
(4) Moderate Tarnish - 2A

TABLE VII

Hydrolytic Stability Tests

168 Hours @ 275°F

(0.87% by Weight of Distilled Water)
(99.13% by Weight of Oil Sample)Tests On The Original Oil

	<u>MLO-70-62</u>	<u>MLO-71-37</u>	<u>MLO-70-32</u>
Viscosity @ 130°F, cs	6.76	5.62	7.64
Neutralization No., mgKOH/g	0.03	0.06	0.03

Tests On The Hydrolyzed Oil

Viscosity @ 130°F, cs	11.58	7.21	8.41
Neutralization No., mgKOH/g	25.29	0.01	0.09
Evaporation Loss %	5.5	1.4	3.4
Appearance After Hydrolysis	Dark Brown	Light Brown	Brown
	No Ppt	No Ppt	No Ppt
Increase In Viscosity %	7.13	8.9	10.1
Increase In Neutralization No.,	25.26	0.05	0.06

Weight Change Of Metals mg/sq.cm

Magnesium	4.91(1)	0.01	+0.02 (5)
Aluminum	0.00	0.00	0.00
Copper	32.35(2)	0.04 (4)	-0.13 (6)
Cadmium	40.97(1)	0.02	0.00 (7)
Steel	0.00	0.00	0.00 (8)

- (1) Pitted and gray discoloration.
- (2) Etched, pitted.
- (3) Pitted.
- (4) Moderate tarnish - 2B.
- (5) Gray discoloration.
- (6) Moderate Tarnish - 2C.
- (7) Light brown discoloration.
- (8) Light brown discoloration and spots.

TABLE VIII

Corrosion and Oxidation Stability Tests
(Per MIL-H-5606)

168 Hours At 275°F

Tests On The Original Oil

	<u>MLO-70-32</u>	<u>MLO-70-62</u>	<u>MLO-71-32</u>
Viscosity @ 130°F, cs	7.87	7.15	6.79
Neutralization No., mgKOH/g	0.04	0.02	0.02

Tests On The Oxidized Oil

Viscosity @ 130°F, cs	8.19	9.28	7.12
Neutralization No., mgKOH/g	0.04	6.50	0.02
Evaporation Loss %	1.3	7.1	0.96
Appearance After Oxidation	Brown No Ppt	Dark Brown No Ppt	Light Brown No Ppt
Increase In Viscosity %	4.1	29.6	4.9
Change in Neutralization No., mgKOH/g	0.00	6.48	0.00

Weight Change Of Metals, mg/sq. cm

Magnesium	0.00	0.56 (1)	0.00
Aluminum	0.00	0.00	0.00
Copper	0.01 (1)	3.31 (2)	0.02 (3)
Steel	0.00	0.00	0.00
Cadmium	0.01 (2)	15.70 (1)	0.02

- (1) Moderate tarnish - 2C.
(2) Light bronze discoloration.
(3) Moderate tarnish - 2B.

TABLE II

Fluid Viscosities (cs)

<u>Temperature</u>	<u>MLO-71-37</u>	<u>MLO-70-32</u>	<u>MLO-70-62</u>
-65°F	1691	1130	1130
-40°F	414.2	319.2	303.4
100°F	9.93	11.19	10.19
210°F	3.27	3.93	3.55

D. Hydrolytic, Stability Test

The hydrolytic stability of the three low density phosphate ester hydraulic fluids was determined by evaluating them in a modified oxidation corrosion test. The test method and procedures of MIL-H-5606(B) were used with the following modifications:

(a) 0.87% by weight of distilled water was combined with 99.13% by weight of oil sample:

(b) separate tests were run at 225°F for 168 hours and at 275°F for 168 hours.

All three of the low density phosphate ester hydraulic fluids displayed adequate hydrolytic stability at 225°F (see Table VI). However, at 275°F, one of the low density phosphate ester fluids (MLO-70-62) degraded severely while the other two phosphate ester fluids still gave satisfactory results (Table VII).

E. Oxidation and Corrosion Stability

The corrosion and oxidation stability of these low density phosphate ester hydraulic fluids was determined by evaluation using the Oxidation and Corrosion test specified in MIL-H-5606(B). Test temperature was 275°F.

Two of the low density phosphate ester hydraulic fluids, MLO-70-32 and MLO-71-37, gave satisfactory results while the MLO-70-62 low density phosphate ester fluid was severely degraded (Table VIII).

F. Viscosity

The viscosities of the low density phosphate ester hydraulic fluids were measured at -65, -40, 100 and 210°F (Table IX). The viscosities of all the fluids met the viscosity requirements of MIL-H-83306 (USAF). The low density phosphate esters have excellent low temperature viscosities equal to or better than other currently used aircraft hydraulic fluids.

G. Volatility

The volatility of the low density phosphate ester MLO-71-37 was determined by the Isoteniscope Method. Results are shown in Table X. A comparison is made with the volatility of MIL-H-5606(B).

H. Fire Resistance

Assessment of the fire resistance of the low density phosphate esters was not the purpose of this project; however, a few typical flammability tests were made. Test results are shown in Table XI.

I. Elastomer Compatibility

Elastomer compatibility was investigated with ethylene propylene (ERP) "O" rings immersed in the low density phosphate ester fluids at 275°F for 72 hours. The compatibility of the low density phosphate esters with ethylene propylene elastomers varies greatly, depending upon the particular ethylene propylene elastomer used. Results range from satisfactory to disastrous (Table XII). EPR "O" ring B completely deteriorated in all three of the low density phosphate ester fluids in static evaluation tests at 275°F with low density phosphate ester MLO-70-62. Manufacturer's data on EPR compound A show satisfactory compatibility with all three low density phosphate ester candidates at 275°F, as shown in Table XIII. Consequently, from these elastomer-fluid compatibility studies, O-ring packings of elastomer compound A were selected for dynamic packing tests with candidate fluid MLO-71-37.

J. Dynamic Packing Tests

Dynamic packing tests were conducted with candidate fluid MLO-71-37 and O-rings packings of EPR compound A, according to the procedures set forth in Military Specification MIL-P-25732B, paragraphs 4.6.6 and 4.6.7.1, with modifications to permit elevated temperatures and changes in fluid

TABLE X

Fluid Volatility
 Vapor Pressure (mm Hg)
 Isoteniscope Method

Temperature (°F)	MLO-71-37	MIL-H-5606(B) (MLO-69-58)
100	---	0.2
150	0.25	1.1
200	1.10	4.7
250	3.70	15.5
300	11.30	46
350	39.0	120
400	71.0	270
450	175	530
500	390	---

TABLE XI

Fire Resistance

	<u>MLO-71-37</u>	<u>MLO-70-62</u>	<u>MLO-70-33</u>	<u>MLO-70-74*</u>
Flash Point (°F) (Cleveland Open Cups) ASTM D-92	345	340	335	355
Fire Point (°F) ASTM D-92	400	390	380	410
Spontaneous Ignition Temperature (°F)				
ASTM D-286	1000+	1000+	1000+	1000+
ASTM D-2155	725	740	765	940
Flame Propagation Test Propagation Rate (cm/sec)	No Flame Advance	No Flame Advance	---	---

* Alkyl-aryl phosphate ester hydraulic fluid type used by airlines before introduction of low density phosphate esters

TABLE XII

Rubber Compatibility
72 Hours @ 275°F
(Fluid MLO-70-62)

	<u>EPR "O" Ring A</u>	<u>EPR "O" Ring B</u>
% Swell	17.29	125.3
Durometer Hardness		
Before Test	70	
After Test	66	
Elongation (inches)		
Before Test	1.8	Gross Elastomer Degradation - No Evaluation Possible
After Test	1.3	
Tensile Strength (psi)		
Before Test	167	
After Test	86	

Condition of MLO-70-62 Fluid

Viscosity @ 100°F cs		
Before Test	10.19	10.19
After Test	10.44	10.99
Increase %	2.4	7.8
Neutralization No. mgKOH/g		
Before Test	0.02	0.03
After Test	0.16	0.08
Increase	0.14	0.05

TABLE XIII

Rubber Compatibility
 EPR "O" Ring A
 72 Hours @ 275°F

Fluid	Swell %	Tensile Strength (psi)	Hardness Shore "A"	Elongation %
MLO-71-37	+16	1886	70	236
MLO-70-62	+14	1925	70	224
MLO-70-32	+13	1829	72	220
Original "O" Ring Properties	---	1925	80	200

and elastomer types. The results of tests at 275, 300 and 350°F are presented in Table XIV. Although packing tests results were satisfactory at 300°F, fluid degradation at this temperature limits systems use to 275°F.

K. Hydraulic Pump Circuit

The ultimate judgment of a hydraulic fluid candidate must be made on its ability to properly function in the environment of an aircraft hydraulic system. Consequently, from its performance in the chemical and physical bench tests, low density phosphate ester candidate MLO-71-37 was selected for evaluation in the hydraulic pump circuit.

The hydraulic pump circuit, shown schematically in Fig. 6, has demonstrated the ability to simulate the physical (thermal and mechanical stresses) and chemical (materials) environment of aircraft hydraulic systems with controlled operating parameters.

The aircraft type hydraulic pump, a high temperature check valve design, was retrofitted with new elastomeric components made from EPR compound A.

Fluid evaluations were made at temperatures of 275, 300, and 350°F for periods of 50 hours, or until either the pump or fluid exhibited signs of degradation or failure. Operating conditions for these evaluations are shown in Table XV.

Operational problems were few and of little significance. The 275°F test was interrupted at 15 hours due to a power outage. This test was terminated at 32.5 hours due to fluid loss from an instrument line failure. This termination was not considered significant since there were no signs of fluid or pump degradation. The 300°F fluid evaluation was interrupted at 16.2 hours due to a pump drive control malfunction.

Fluid property changes exhibited by the low density phosphate ester fluid MLO-71-37 during the three hydraulic pump circuit tests are shown in Tables XVI, XVII and XVIII.

TABLE XIV

Dynamic Packing Test Results

FLUID: MLO-71-37
 PACKING: Elastomer Compound "A"

<u>275°F Test</u>	<u>Leakage (ml)</u>	
1	3.0	All Packings Excellent
2	5.8	" " "
3	6.5	" " "
4	8.0	" " "
5	8.5	Very slight nibbling on drag ring O.D.
6	Endurance	Packing failed at 78,000 cycles. Total leakage 38.0 ml at 66,000 cycles.
<u>300°F</u>		
1	6.0	All Packings Excellent
2	6.0	" " "
3	8.0	Very slight nibbling & scuffing on drag ring.
4	5.5	All Packings Excellent
5	6.5	" " "
6	Endurance	Packing failed at 57,100 cycles. Total leakage 34.0 ml at 36,000 cycles.
<u>350°F</u>		
1	Failed	Rod & cylinder packing
2	Failed	failed from combined rolling,
3	Failed	nibbling & cutting. Condition
Test series terminated		very bad.

Evaluations conducted to MIL-P-25732B with modifications for elevated temperatures & specimens.

TABLE XV

Operating Data
For
Hydraulic Pump Circuit
MIL-37 at 275^o, 300^o, and 350^oF

Nominal Fluid Temperature	^o F	275	300	350
Duration of Experiment	hrs	32.5	50.0	13.1
Nominal Pump Speed	rpm	3750	3750	3750
Pump Flow Rate	gpm			
Maximum Flow		8.39	8.49	8.29
Minimum Flow		0.96	1.02	1.24
Shear Cycles		5065	7925	2080
Pump Discharge Pressure	psig			
Maximum Flow		2681	2637	2624
Minimum Flow		2950	2992	3020
Inlet Filter P at max flow	psig			
Initial		16	17	15
Final		20	15	15
Temperatures	^o F			
Pump Case		253	271	315
Pump Inlet		258	281	333
Pump Discharge		272	295	345
After Throttling Valve		278	302	351
Chamber Atmosphere		88	98	90
Leakage, Shaft Seal	ml	75	42	30
Remaining Initial Fluid	%	30.4	82.9	87.4

TABLE XVI

**MLO-71-37 Property History
275°F Hydraulic Pump Circuit Test**

Sample Time Hrs	Viscosity(1) cs		Flash(2) Point °F	Fire(2) Point °F	Acid(3) No. mgKOH/gm	Insolubles
	100°F	210°F				
New	10.26	3.27	330	370	0.08	
0	9.73	3.12	310	380	0.10	
2	9.50	3.12	335	385	0.10	
4	9.36	3.01	325	380	0.14	
6	9.22	2.95	330	360	0.15	
8	9.17	2.93	310	380	0.17	
10	9.04	2.88	350	375	0.14	
15	8.76	2.81	330	370	0.15	
25	8.14	2.76	305	375	0.16	
32.5	9.25	2.98	335	370	0.21	None

- (1) ASTM Method D445
(2) ASTM Method D92
(3) ASTM Method D974

TABLE XVII

MLO-71-37 Property History
300°F Hydraulic Pump Circuit Test

Sample Time Hrs	Viscosity(1) cs		Flash(2) Point °F	Fire(2) Point °F	Acid(3) No. mgKOH/gm	Insolubles
	100°F	210°F				
0	9.34	2.97	355	370	0.12	
2	9.30	2.95	340	375	0.13	
4	9.20	2.96	325	380	0.15	
6	9.23	2.90	330	380	0.16	
8	9.18	2.87	325	385	0.17	
10	8.91	2.84	345	390	0.19	
16.2	9.31	2.97	350	390	0.11	None
16.2	9.24	2.95	350	385	0.16	
25	8.63	2.88	335	380	0.32	
50	8.63	2.71	355	385	8.22	Trace

(1) ASTM Method D445

(2) ASTM Method D92

(3) ASTM Method D974

TABLE XVIII

MLO-71-37 Property History
350°F Hydraulic Pump Circuit Test

Sample Time Hrs	Viscosity(1) cs		Flash(2) Point °F	Fire(2) Point °F	Acid(3) No. mgKOH/gm	Insolubles
	100°F	210°F				
0	9.16	2.85	340	380	1.10	
2	9.20	2.91	330	375	10.18	
4	9.29	2.93	315	365	8.66	
6	9.45	2.95	310	365	11.76	Trace
8	9.69	3.07	325	360	19.17	Trace
10	10.00	3.17	345	350	21.88	Trace
13.1	9.89	3.14	325	375	16.01	Trace

- (1) ASTM Method D445
(2) ASTM Method D92
(3) ASTM Method D974

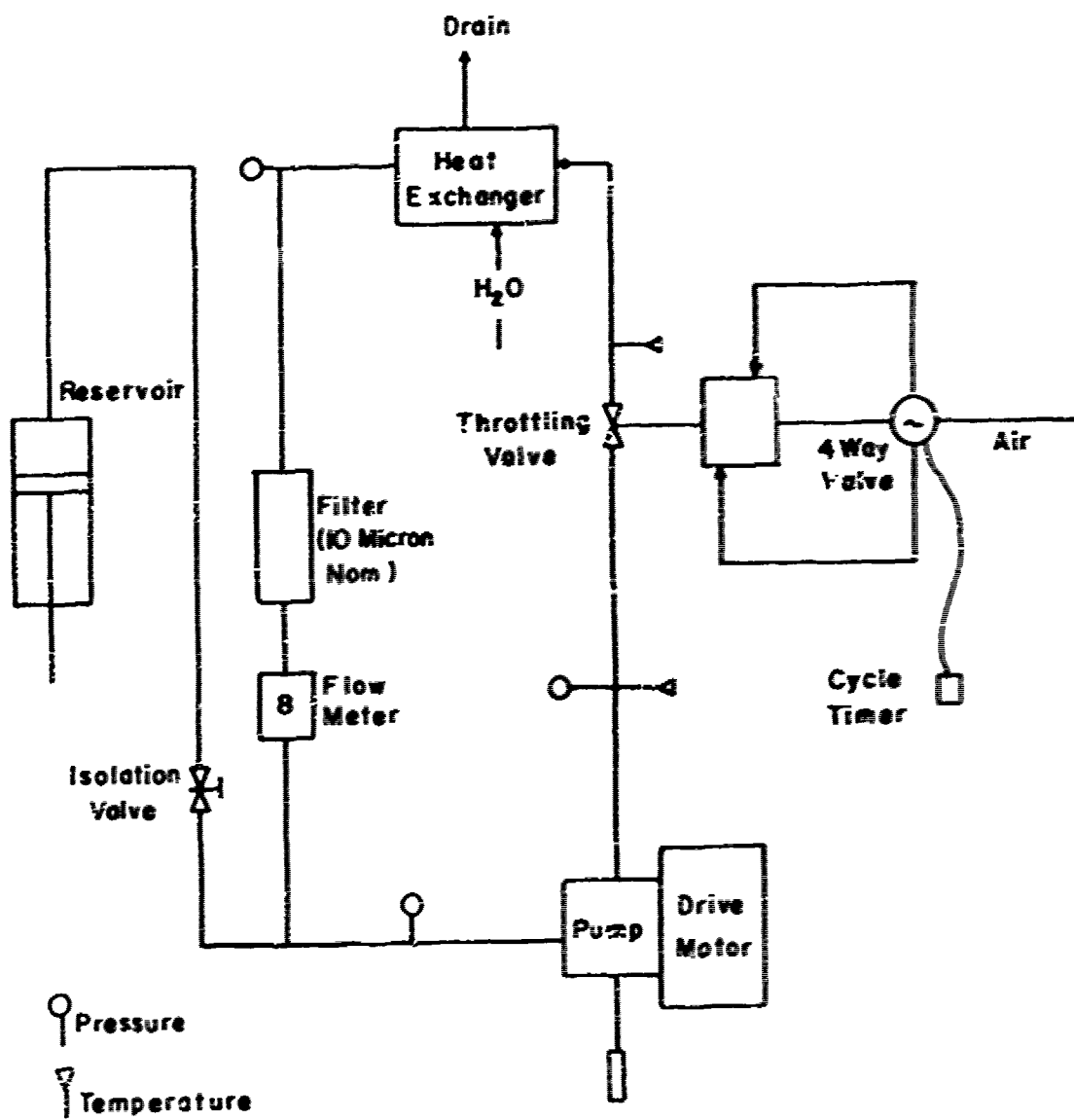


FIGURE 6 HYDRAULIC PUMP CIRCUIT SCHEMATIC

There were no fluid property changes during the 275°F evaluation which could be considered as indicative of gross fluid degradation. During the 300°F evaluation, a sharp increase in acid number, accompanied by a trace of insolubles, was produced at between 25 and 50 hours. These changes were viewed as signs of incipient fluid degradation. The fluid evaluation at 350°F produced signs of gross fluid degradation at 2 test hours with a sharp increase in acid number and confirmed the suspected incipient fluid breakdown during the 300°F test. A precipitate, produced during the 300°F evaluation, was separated and washed with solvents to a pure white powder. This powder was subjected to spectral analysis and identified as an iron phosphate. It was hypothesized that the strong acids produced by the fluid degradation reacted with the iron containing system components to produce the iron phosphate. The color of the fluid samples from the 350°F evaluation changed from the original clear medium blue to blue-green to yellow-orange.

The stainless steel element filters contained a small amount of light grey gelatinous residue and some fine copper colored particles after the 275°F evaluation. After the 350°F test, the pump discharge filter and the disc elements contained a considerable quantity of copper colored material from the piston slippers and the pump thrust bearing. Also, the filter elements retained a thin but apparently porous film of gelatinous or grease-like material. From the residue on the four-ball specimens tested at 350°F and 40 kilograms and the material in the filter, it appears that the thermal degradation of MIL-71-37 produces a gelatinous by-product.

The aircraft type hydraulic pump was disassembled for a pre-test inspection and after each of the fluid evaluations. There was no evidence of pump degradation from either visual examination or changes in the weights of the pump friction elements following the 275 and 300°F fluid evaluations.

However, after the 350°F test, pump elements exhibited signs of gross wear from lubrication failure to the point that a pump overhaul would be required to restore it to operating condition. The primary areas of wear were the copper alloy piston heads and thrust bearing, which exhibited degradation severe enough to render them unserviceable. (The degree of wear, as indicated by element weight loss for various pump parts, is presented in Table XIX.) Although the pump was in excellent condition, the incipient fluid degradation at the end of the 300°F test limits the use of the candidate fluid to a maximum temperature of 275°F.

TABLE XIX

Hydraulic Pump Friction Element Weight Changes
 Low Density Hydraulic Fluid MLO-71-37

	Change After 275°F Test	Change After 300°F Test	Change After 350°F Test	Total Change
Pump Piston Assemblies*	-.0285	-.0079	-.1896	-.2260
Pump Collars*	-.0005	-.0007	-.0824	-.0836
⊗ Pump Sleeves*	-.0143	-.0003	-.0103	-.0250
Nutation Plate	-.0020	-.0043	-.4624	-.4687

All weights in grams.

* Average of seven elements.

SECTION IV

CONCLUSIONS

The three low density phosphate ester fluids evaluated were suitable for use under Military Specification MIL-H-83306 (USAF) Hydraulic Fluid, Fire Resistant, Phosphate Ester Base; and Boeing Aircraft Company Material Specification BMS-311-C Hydraulic Fluid, Fire Resistant. These specifications cover operational ranges of -65 to +225°F and -65 to +225/250°F, respectively. There are, however, significant differences in the physical and chemical characteristics of the candidate fluids. The low density phosphate ester MLO-70-32, exhibited greater susceptibility to shearing action than the other two fluids (MLO-70-32 failed the shear resistant requirement of MIL-H-5606(B)). The low density phosphate ester MLO-70-62 was hydrolytically and oxidatively unstable and corrosive to metals at 275°F, whereas the other two fluids, MLO-70-32 and MLO-71-37, were satisfactory.

The low density phosphate ester with the best overall properties, as determined by the chemical tests, exhibited very good characteristics in simulated hydraulic pump and actuator systems to 275°F. This temperature limit was established by incipient fluid degradation at 300°F in the hydraulic pump circuit at 50 test hours.

The low density phosphate ester, MLO-71-37, which had shown the best overall properties in this evaluation, exhibited potential operational capability over the -65 to 275°F temperature range. Extreme care is required in the selection of elastomeric materials for use with the low density phosphate ester fluids. The use of properly specified elastomers must be complied with for satisfactory results.

REFERENCES

1. Gunderson, R. C. and Hart, A. W., "Synthetic Lubricants", Reinhold Publishing Corp., New York City, 1st ed., 1962, pp 103-150.
2. Hatton, R. E., "Introduction to Hydraulic Fluids", Reinhold Publishing Corp., New York City, 1st ed., 1962, pp. 190-211.

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Three low density phosphate ester fluid candidates, MLO-70-32, MLO-70-62 and MLO-71-37, were characterized as to their physical and chemical properties. MLO-71-37 which exhibited the most acceptable characteristics was further evaluated for its reactions in simulated functional and system environments. MLO-71-37 was found to possess the most satisfactory overall properties and exhibited potential operational capability over a temperature range of -65 to 275 F. All three candidate fluids displayed a sensitivity to elastomeric materials with specific man- ufacturer and compound designations required for satisfactory performance.			

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KEY WORDS	GROUP 1		GROUP 2		GROUP 3	
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