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IMPROVED SILICONE FLUIDS AS CANDIDATE GAS TURBINE  
ENGINE OILS FOR -40°F TO 465°F TEMPERATURE RANGE

AIR FORCE MATERIALS LABORATORY (MBT)

JULY 1973

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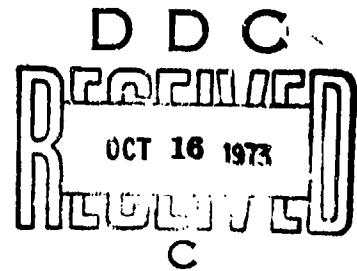
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*GEORGE J. MORRIS*

TECHNICAL REPORT AFML-TR-73-72

JULY 1973



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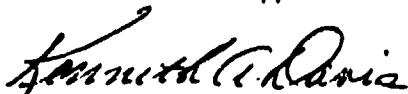
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FOREWORD

This report was prepared by the Lubricants and Tribology Branch, Nonmetallic Materials Division, Air Force Materials Laboratory, Air Force Systems Command, Wright-Patterson Air Force Base, Ohio, with George J. Morris as Project Engineer. Work was initiated under Project No. 7343, "Aerospace Lubricants," Task No. 734303, "Fluid Lubricant Materials." The report covers work accomplished from November 1971 to July 1972. It was submitted by the author in August 1972.

This technical report has been reviewed and is approved.



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## ABSTRACT

A physical and chemical laboratory study of several silicone fluids that represent some of the latest technology in the area has been conducted. These fluids were an alkyl methyl silicone, a trifluoropropyl methyl silicone and a methyl silicone improved by the use of additive formulation. These fluids were investigated for possible use as candidate gas turbine engine oils in the temperature range of  $-40^{\circ}\text{F}$  to  $+465^{\circ}\text{F}$ . The viscosity-temperature characteristics, volatility, oxidative stability, corrosion reactivity towards selected metals and lubrication capabilities were assessed for conformance with the recently established specification, MIL-L-27502, covering the aforementioned temperature range. The alkyl methyl silicone, although having favorable rheological and lubrication behavior, was so oxidatively unstable and corrosion prone that further study is not recommended. Both trifluoropropyl methyl silicone and improved methyl silicone have demonstrated sufficient oxidation and corrosion stability to warrant further study.



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## SECTION I

### INTRODUCTION

Current operational gas turbine engine oils conforming to specifications MIL-L-7808 (-65 to 325°F) and MIL-L-23699 (-40 to 350°F) will not meet the oxidative and thermal stability requirements imposed by future generation Air Force gas turbines. These new gas turbine engines will operate with greatly increased turbine inlet air temperatures, and are of reduced size and weight and higher thrust potential to provide the desired aircraft performance. These design improvements thereby place greater operational demands on the lubricant such as higher temperature capability, better load carrying ability, and reduced volatility (Reference 1).

Coincident with the engine developments was the better definition of desirable lubricant properties into firm specification requirements by the Lubrication Branch of the Air Force Aero Propulsion Laboratory. This upgrading of specification requirements progressed from MIL-L-7808 through revision "G" and to the currently sought "H" (previously referred to as "Head & Shoulders" quality). Concurrent, but separate from the early revisions of MIL-L-7808, target requirements for the initial 500°F gas turbine engine oil were proposed as a tentative specification, MIL-L-27502, back in the early 1960's. However, these high temperature requirements and other desirable lubricating properties proved exceedingly restrictive and very difficult to meet. In fact, no fluid was ever found to satisfactorily meet all the requirements of this specification. Within this same time frame, the specification, MIL-L-9236, for the gas turbine engine oil used in the B-70 was believed to be adequate up to 425°F. However, subsequent laboratory experience with it indicated marginal performance even at 400°F. The current issue of MIL-L-27502, dated 25 January 1972, superseded MIL-L-9236, which was cancelled on 1 February 1972. It is contemplated that the requirements contained in MIL-L-27502 will serve as a bridge between existing fluids and the emergence of gas turbine engine oils for use at temperatures in excess of 500°F (Reference 2).

At the 1970 Air Force Materials Laboratory Symposium, the Fluid and Lubricant Materials Branch expressed the philosophy that compromises with respect to low-temperature flow would have to be made in the continuing search for fluids with high-temperature capabilities. That is, the scale would have to be shifted upward from the traditional  $-65^{\circ}\text{F}$  to perhaps  $-40^{\circ}\text{F}$  or even as high as  $-20^{\circ}\text{F}$ . This is not due to the relaxation of equipment operational limits or to a change in design philosophies, but merely recognizes the low-temperature rheology of the high-temperature fluids which would provide candidates for the wide-temperature coverage (Reference 3). The one possible exception to this generalization is the extremely versatile silicone class of lubricants which are unsurpassed in certain areas, such as rheology. Their inherently high thermal stability, good viscometric properties, and wide liquid ranges make them excellent candidates for the  $425$  to  $465^{\circ}\text{F}$  temperatures and oxidizing environments being considered for gas turbine engine oils. Despite the previous experiences of silicone incompatibility with carbon seals and the companion tendency to gel at high temperatures in oxidizing environments, it was felt that sufficient advances have been made in current silicone technology to significantly improve the fluids to be considered as candidates in the gas turbine engine oil area (Reference 4). The results of preliminary investigations into some currently available silicone fluids are covered in this report.

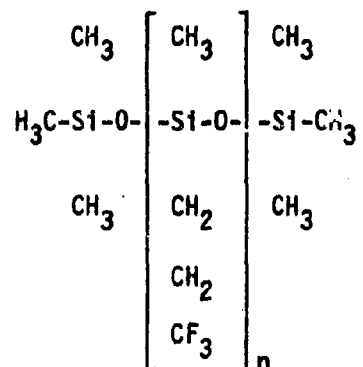
## SECTION II

## SILICONE FLUID DEVELOPMENT BACKGROUND

The first commercially prepared silicone fluids became available as specialty lubricants with the chemical orientation of dimethyl polysiloxanes. They exhibited the characteristic extremely flat viscosity-temperature curves but were found to be lacking as steel on steel lubricants, nor did they possess particularly good thermal or oxidative stability in the temperature range of our current consideration.

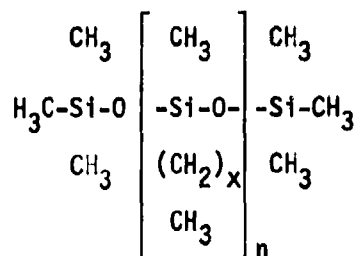
An improvement in silicone technology was made by the commercial availability of methyl phenyl polysiloxanes that could be prepared with various ratios of phenyl to methyl substituents. By increasing the phenyl to methyl ratio, both thermal and oxidative resistance were improved, but at the expense of the low temperature properties and the viscosity-temperature coefficient. These silicones did not provide any improvement in lubricating properties over the dimethyl polysiloxanes. It was also this chemical configuration that was involved in the carbon seal studies where it was determined that degradation resulting in sludge formation caused by gelation of the silicone oil made the seal malfunction, which in turn resulted in high oil loss, clogged shaft-seal interface, and extremely high local temperatures (Reference 5). A significant improvement in the lubricating properties of methyl phenyl polysiloxanes was obtained by partially chlorinating the phenyl group. However, the normally low vapor pressure and high thermal stability were adversely affected. A further improvement in fluid quality was achieved when the oxidation resistance was enhanced by coupling an inhibitor on the methyl phenyl polysiloxane structure (Reference 6).

More recently the silicone fluids mentioned above were structurally modified to include fluorine as in the trifluoropropylmethylsiloxane:



Early test data on this type of fluid showed it to be a better lubricant for ferrous metals than previous silicones and to approach 500°F oxidative stability under certain conditions. Certain additives that enhance antiwear and extreme pressure properties as well as high temperature oxidation resistance were shown to be miscible in the fluid (Reference 7). A typical fluid representative of this class of trifluoropropylmethylpolysiloxanes has been evaluated as a gas turbine engine oil and the data reported herein.

A latter development in silicone technology was the introduction of methyl alkyl polysiloxanes. They comprised those siloxanes with a single carbon methyl unit and a multi-carbon alkyl unit attached to the same silicone atom. The general structure appears as follows:



The alkyl side chain can vary in length from an ethyl group to a tetradecyl group and still remain fluid at room temperature. These structures showed improved mechanical properties, namely wear resistance, frictional

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behavior and surface characteristics over other silicones at an optimum octyl side chain length (Reference 8). The eight carbon analogue was characterized as a gas turbine engine oil and the results appear in this report.

The latest improvement in silicone fluid technology made available to this laboratory was the synthetic lubricating oils based on dimethyl polysiloxane polymers. This base stock has been modified by additive formulation to provide improved lubricating characteristics and better corrosion protection compared to unmodified dimethyl silicone fluids. They are available in several viscosity grades and were also characterized for potential gas turbine engine oils in this investigation.

### SECTION III

#### DISCUSSION OF CHARACTERIZATION TECHNIQUES

Since high temperature oxidation and corrosion inhibition is still considered to be of primary importance for candidate Air Force gas turbine engine oils, the oxidation-corrosion (O-C) test was used for screening. These evaluations were directed toward the O-C requirements of MIL-L-27502 (Reference 9) with certain procedural modifications. The test methods used were as follows:

- a. Method 5307 from Federal Test Method Standard 791b
- b. AFML Improved Micro-O-C Test Procedure

Test Method 5307 was altered to exclude the interim oil sampling because of limitations on the glassware configuration (and sample size). To provide for some information on progressive fluid deteriorations during the course of the test, duplicate samples were used. One was terminated at 24 hours, and the remaining sample was continued through the 48 hour period stipulated by the specification. The AFML improved micro-O-C procedure, described in Reference 10, was changed by reducing the air flow from 20 to 1 liter per hour and by increasing the test time on one of the duplicate samples to 48 hours. By using the micro procedure, the sample size and air flow were reduced by a factor of 10 from the method 5307. This allowed for greater conservation of fluid sample. It was felt that data obtained from the micro O-C test could be adequately correlated with that obtained from method 5307 for these fluid evaluation purposes. This was based on the experience acquired in the development work that led to the adoption of the micro O-C test as our internal fluid screening tool. It was demonstrated by Wyandotte (Reference 10) that data obtained in the Wyandotte miniature oxidation test was comparable to data from the micro O-C test through a range of fluid chemical types including a silicone where the fluid sample size and air flow were also reduced by a factor of 10 to 1.



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All the fluids were first tested for oxidation stability without metal coupons and then with the metal coupons to study fluid reactivity with the metals or the effect of metal catalysis on fluid degradation. The initial test temperature was 428°F and where the data appeared promising, the temperature was raised to 464°F.

## SECTION IV

## LABORATORY EVALUATIONS

Preliminary characterization of each silicone type was conducted in the laboratory and compared to the requirements of MIL-L-27502. The data which appears in Table I shows that the viscosity-temperature relationship of all the fluids is in agreement with the requirements through the temperature range of -40 to 500°F with one exception: the 500°F viscosity of the trifluoropropyl methyl silicone is slightly below (0.04 cs) the minimum of 1.0. However, the other rheological properties of that particular fluid appear to be sufficiently below their maximum values to allow for upgrading of the high temperature viscosity to the 1.0 cs minimum. All the fluids have acceptable low temperature flow properties of -65°F or lower, as indicated by the pour point, and have exceptionally low viscosities at -40°F. Their flash points, with the exception of that for trifluoropropyl methyl silicone, are above the minimum of 475°F. The neutralization numbers of all the fluids range from 0.01 to 0.09, which is well below the maximum requirement of 0.5. The evaporation loss at 400°F for 6-1/2 hours of the alkylmethyl silicone and the improved low viscosity dimethyl silicone exceeded the 5.0 percent maximum with 12.8 and 5.5 percent values, respectively. Since the improvement in performance of this particular dimethyl silicone is obtained through additive compounding, the higher evaporation rate could be due to the higher volatility of these additive materials. Evaporation for the other improved dimethyl silicones is well below the maximum at 3.9 percent with the trifluoropropyl methyl silicone having a minimal evaporation level of 0.7 percent. Where determined, the autogeneous ignition temperatures were well above the 770°F minimum except that for the high viscosity dimethyl silicone, which fell below the requirement by 20°F.

The lubrication potential of these same silicones, as demonstrated by their behavior under 4-ball test conditions, appears on Table II. Improvements over the old methyl silicones were evident. The trifluoropropyl methyl silicone showed the lowest wear scars of all the fluids tested - even below the additive paraffinic oil and a former gas turbine engine lubricant. As has been discussed in previous studies of silicones

as gas turbine engine oils, there is no direct relationship between 4-ball wear data and lubrication of a gas turbine engine (Reference 5). However, it was still considered desirable to have lubricating properties of any candidate fluid within the range of currently used ester containing lubricants. These performed satisfactorily in engine components where sliding friction predominated and prevented severe wear; thus, equivalent or better 4-ball performance is still a practical goal in the absence of an actual engine test.

#### 1. ALKYL METHYL SILICONES

Oxidation-corrosion studies were performed on these silicone fluids in accordance with the procedures outlined in the previous section. The results from the micro oxidation tests at 428°F on the alkylmethyl silicone appear in Table III and show excessive viscosity increase and high acid numbers. When using metal coupons at this same temperature, fluid deterioration was not as great but was still unacceptable, as shown by a slightly high viscosity increase and three times the allowable 2.0 acid number. Possibly the metals had some inhibiting effect on the fluid. The fluid attack on magnesium was greater than the specification limit, but was minimal on the other six metals. This fluid was also evaluated in the modified macro apparatus described in Federal Test Method No. 5307. The magnesium was again the only metal attacked to the degree that it failed the specification limit. Fluid viscosity increase and acid number were higher than the values obtained on the micro O-C test and the / failed the specification requirement. The data showed that micro O-C test would give comparable data to the Federal Test Method at the 428°F test temperature. That is, it still showed failure but not to the same degree. Because of the excessive fluid deterioration of the alkylmethyl silicone at 428°F and high magnesium corrosion, we felt that further evaluation at increased temperature was not warranted.

TABLE I  
SUMMARY OF LABORATORY CHARACTERISTICS OF SILICONE FLUIDS

Fluid Properties	Alkyl Methyl	TriFluoro-propyl methyl	Improved Dimethyl			MIL-L-27502 Requirements
	71-16*	71-26*	71-100*	71-101*	71-102*	
Viscosity, cs at 500°F	1.4	0.96	3.4	3.4	6.1	1.0 Min. Report
at 210°F	7.9	6.2	15.8	15.7	28.8	Report
at 100°F	34	29	40	39	73	Report
at -40°F	5231	5000	355	350	530	17,000 Max.
Pour Point, °F	-65	-65	-70	-70	-70	-65 Max.
Flash Point, °F	505	470	525	540	520	475 Min.
Neutralization No. (D664)	0.02	0.01	0.09	0.09	0.09	0.5 Max.
Evaporation Loss, % 400°F (D972)	12.8	0.7	5.5	3.9	3.9	5.0 Max.
Autogenous Ignition Temp., °F	820	970	-----	-----	750	770 Min.

\*Sample No. MLO-

TABLE II

## SILICONE FLUID LUBRICATION BEHAVIOR

(4-Ball Wear Data)\*

Fluid	Sample No. MLO-	Temperature (°F)	Load (kg)	Wear Scar (mm)		
Alkyl Methyl	71-16	350	4	0.44		
			10	0.58		
Trifluoropropyl Methyl	71-26	167	4	0.18		
			10	0.32		
			20	0.52		
		400	4	0.57		
			10	0.77		
			20	0.87		
Improved Dimethyl	71-100	167	10	0.42		
			20	0.57		
			40	0.91		
		450	40	1.40		
			71-101	167	10	0.50
					20	0.65
40	1.00					
450	40	1.20				
		Paraffinic Compounded Oil	---	167	10	0.50
					20	0.70
40	0.66					
450	40	1.90				
		Unimproved Dimethyl Silicone	---	167	10	0.45
					20	0.74
40	2.30					
450	40	2.80				
		MIL-L-9236** (Ester Base)	---	167	10	0.60
					40	0.90
400	1.00					

\*Test Conditions: Ball Metal - 52100 Steel  
Speed - 1200 rpm (except MIL-L-9236)  
Test Duration - 1 hour

\*\*Test run at 600 rpm

TABLE III

## OXIDATION-CORROSION RESULTS ON ALKYL METHYL SILICONE

Temperature, °F	428	428	428
Type Test OX or O-	OX	O-C	O-C Macro*
Test Duration, Hrs.	24	48	48
<u>Laboratory Tests</u>			
Metals, Weight Change, mg/cm <sup>2</sup>	NONE	NONE	
Aluminum		+0.01	0.00
Silver		+0.03	0.01
Bronze, AMS 4616		+0.01	0.00
Mild Steel		+0.04	0.01
M-50		+0.04	0.00
Magnesium		-5.92	-1.02
Titanium		+0.05	-0.03
<u>Used Oil Data</u>			
Oil Loss, Evaporation %	0.5	1.6	2.6
Viscosity Change at 100°F, %	+55	+95	+31
Acid Number	2.4	5.1	6.1

\* Fed. Test Method No. 5307 Modified

## 2. TRIFLUOROPROPYL METHYL SILICONES

Micro oxidation stability tests conducted on the trifluoropropylmethyl silicone at 428°F showed a moderate viscosity increase and progressively higher acid numbers from 24 through 48 hours. These results appear on Table IV. In the presence of metals under O-C conditions, the fluid did not show as much deterioration by viscosity increase as without metals, but did show a very high acid number (five times the maximum 2.0 specification requirement). Excessive metal attack was confined to the magnesium in duplicate tests and to the mild steel in one instance. This evaluation was also performed on a macro scale, modified Federal Test Method No. 5307, as described in the previous section. Very similar data was obtained for viscosity increase and acid number increase as under the micro conditions. Minimal metal corrosion occurred on six of the coupons. In the macro test, unacceptable corrosion was measured on the mild steel instead of the magnesium in the micro test. Apparently the attack on the mild steel is on the threshold at the 428°F temperature level, as evidenced by its intermittent appearance and data obtained later at 464°F. Duplicate micro O-C tests conducted on the trifluoropropylmethyl silicone at 464°F showed no greater fluid deterioration than at 428°F as demonstrated by viscosity and acid number increases. Attack on five of the metal coupons was at a minimum. Mueller's bronze and the mild steel showed heavy corrosion, the latter seemingly reinforcing the pattern of corrosion at the lower temperature of testing.

## 3. IMPROVED METHYL SILICONES

Micro oxidation tests were conducted at 428°F on the three improved dimethyl silicones identified below. The data appears in Table V.

MLO-71-100 (50 cs) - antiwear, inhibitor, metal activator  
additive combination

MLO-71-101 (50 cs) - antiwear, EP additive only

MLO-71-102 (100 cs) - no additive.

TABLE IV  
OXIDATION-CORROSION RESULTS ON TRIFLUOROPROPYL METHYL SILICONE

Temperature, °F Type Test, OX or O-C Test Duration, Hrs.	428 OX		428 O-C		428 Macro*		464 O-C	
	24	48	48	48	48	48	48	48
<u>Laboratory Tests</u>								
Metals, Weight Change, mg/cm <sup>2</sup>								
Aluminum	None	None	0.00	+0.01	-0.01	+0.06	+0.05	
Silver			-0.03	-0.02	-0.06	+0.02	+0.03	
Bronze, AMS 4616			-0.07	-0.01	-0.02	---	---	
Bronze, Muellers			---	---	---	-1.7	-1.9	
Mild Steel			-0.08	-1.2	+0.53	-1.5	-1.9	
M-50			-0.05	-0.01	0.00	+0.02	+0.01	
Magnesium			-1.63	-1.67	0.00	---	---	
Nispatloy			---	---	---	-0.02	-0.03	
Titanium			0.03	0.00	0.04	-0.04	-0.02	
<u>Used Oil Data</u>								
Oil Loss,	0.7	1.8	7.3	3.6	---	2.0	2.4	
Evaporator, %	+38	+45	+28	+30	+37	+37	+39	
Viscosity Change at 100°F, %	6.9	---	9.5	10.8	9.9	8.7	10.5	
Acid Number								

\*Fed. Test Method No. 5307 Modified



The results on MLO-71-100 were very encouraging as evidenced by viscosity increases of 2.0 and 3.2 percent and acid numbers of 0.20 and less through the 24 and 48 hour time periods. Fluids MLO-71-101 and MLO-71-102 transformed into solid gel-like materials after 24 hours testing; no further evaluation of these two fluids was conducted. Under micro O-C conditions with metals at 428°F, the MLO-71-100 fluid condition remained unchanged to the same degree as in the test without metals. On duplicate specimens, corrosion was acceptable on five of the different metals but excessive on the silver and on the AMS 4616 silicone bronze in one instance. Although fluid deterioration in these tests was very slight, apparently the acidic products were sufficient to attack the susceptible metals. Progressing to the higher temperature level of 464°F, micro oxidation evaluation of MLO-71-100 after 24 hours resulted in the formation of the characteristic gel so often associated with complete silicone fluid breakdown. No further evaluations were conducted on this fluid.

In order to establish a practical stable temperature level for the alkyl methyl and trifluoropropylmethyl silicones, micro tests were conducted at a reduced level of 400°F. The results appear in Table VI. Oxidation stability of the alkyl methyl silicone was no better than at 428°F in that it still exceeded the 25 percent viscosity change allowed by the specification, but at a lesser magnitude, (i.e., 95 percent versus 65 percent after 48 hours). The acid number showed the same trend of exceeding the specification requirement. When the metal catalysts were introduced, improvement in viscosity change was noted but the acid number was equivalent to the 428°F data. Both again failed the specification. As before, attack was excessive on the magnesium, but minimal and passing on the remaining six metal coupons.

Oxidation tests on the trifluoropropylmethyl silicone at 400°F failed badly with 76 percent viscosity change and 10.2 acid number. Micro O-C evaluation showed little improvement on the oil condition over the 428°F test. Viscosity change and acid number were reduced to some extent, but remained outside specification limits. Corrosion was excessive on the silver and bronze (a new combination) and passing on the four remaining metal specimens.

TABLE V  
OXIDATION-CORROSION RESULTS ON IMPROVED METHYL SILICONES

	MLO-71-100 (50 cs)				MLO-71-101 (50 cs)		MLO-71-101 (100 cs)	
	428		428		428		428	
	OX	48	0-C	48	OX	24	OX	24
Temperature, °F	24	48	24	48	428	428	428	428
Type Test, OX or 0-C					0-C		OX	OX
Test Duration, hrs.					24	48	24	24
<u>Laboratory Tests</u>	None							
<u>Metals, Weight Change, mg/cm<sup>2</sup></u>	None							
Aluminum			+0.05	+0.03				
Silver			+0.56	+0.49				
Bronze, AMS 4616			-0.12	-1.63				
Bronze, Muellers			---	---				
Mild Steel			+0.09	+0.07				
M-50			+0.10	+0.07				
Magnesium			+0.02	+0.02				
Naspalloy			---	---				
Titanium			+0.03	+0.02				
<u>Used Oil Data</u>								
Oil Loss, Evaporation, %	0.1	0.1	1.0	1.0	1.0	1.0	Fluid	Fluid
Viscosity Change at 100°F, %	+2.0	+3.2	+2.8	+3.8	+2.8	+3.8	Gelled	Gelled
Acid Number	0.15	0.20	0.2	0.15	0.2	0.15		

TABLE VI

400°F OXIDATION-CORROSION RESULTS

Laboratory Tests	Alky! Methy!		Trifluoropropyl Methy!		MIL-L-27502 Requirements at 428°F
	No Metals	48 Hrs	No Metals	48 Hrs	
	24 Hrs	48 Hrs	24 Hrs	48 Hrs	
Metals, Weight Change, mg/cm <sup>2</sup>					
Aluminum	+0.03	+0.02	-0.03	+0.01	±0.2
Silver	+0.06	+0.01	+0.07	+0.97	±0.2
Bronze AMS 4616	-0.03	-0.05	-0.05	-1.13	±0.4
Mild Steel	+0.04	+0.03	-0.06	+0.01	±0.2
M-50	+0.05	+0.06	-0.02	-0.03	±0.2
Magnesium	-0.47	-3.55	+0.03	-0.05	±0.2
Titanium	+0.01	+0.02	0.00	+0.03	±0.2
Used Oil Data	24 Hrs	48 Hrs			
Oil Loss, Evaporation, %	0.5	1.6	0.88	2.67	---
Viscosity Change at 100°F, %	71	65	22	52	±25
Acid Number	0.87	3.1	8.1	8.6	2.0
			48 Hrs		
			1.8, 2.2		
			69,83		
			10.3,10.1		

SECTION V

CONCLUSIONS

The physical characteristics of the alkyl methyl silicones, particularly their rheological properties, appear to make them promising candidates as lubricants for -40 to +465°F gas turbine engines. However, results of this evaluation indicated that their excessive volatility and poor oxidation/corrosion characteristics down to 400°F rule them out of contention in this area. The trifluoropropyl methyl silicones demonstrated superiority in lubricity over other types of silicone fluids. Other characteristics such as viscosity, flash point, and oxidation/corrosion stability fall short of the MIL-L-27502 goals; however, these deficiencies appear correctable by improved fractionation and/or improved additive packages.

The improved methyl silicones showed promise in physical characteristics and lubricity when fully formulated. Those with only an antiwear additive or no additive at all were unstable from an oxidative standpoint. The fully formulated fluid, on the other hand, was oxidatively and O-C stable at the lower temperature, as shown by extremely low viscosity change and acidity. However, metal attack was evident to a moderate degree, which could probably be handled by further additive treatment. These improved silicones appear to show promise for the requirements for future gas turbine engine oils.

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