AD-787 191

AIRCRAFT TURBINE ENGINE FUEL CORROSION INHIBITORS AND THEIR EFFECTS ON FUEL PROPERTIES

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Air Force Aero Propulsion Laboratory
Wright-Patterson Air Force Base, Ohio

July 1974

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AIR FORCE/56780/24 September 1974 — 250

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REPORT DOCUMENTATION PAGE

READ INSTRUCTIONS BEFORE COMPLETING FORM

1. REPORT NUMBER AFAPL-TR-74-20	2. GOVT ACCESSION NO.
3. RECIPIENT'S CATALOG NUMBER AD-787191	
4. TITLE (and Subtitle) AIRCRAFT TURBINE ENGINE FUEL CORROSION INHIBITORS AND THEIR EFFECTS ON FUEL PROPERTIES	5. TYPE OF REPORT & PERIOD COVERED Final Technical Report
	6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s) Charles R. Martel Royce P. Bradley James R. McCoy Joseph Petrarca	8. CONTRACT OR GRANT NUMBER(s)
9. PERFORMING ORGANIZATION NAME AND ADDRESS Air Force Aero Propulsion Laboratory Wright-Patterson AFB, Ohio 45433	10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS 30480523 30480546 304805FL
11. CONTROLLING OFFICE NAME AND ADDRESS Air Force Aero Propulsion Laboratory Wright-Patterson AFB, Ohio 45433	12. REPORT DATE July 1974
	13. NUMBER OF PAGES 42
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)	15. SECURITY CLASS. (of this report) UNCLASSIFIED
	15a. DECLASSIFICATION DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

Approved for Public Release; Distribution Unlimited

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18. SUPPLEMENTARY NOTES

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

Corrosion Inhibitors
Jet Fuel Thermal Stability
Jet Fuel Filterability
Jet Fuel Lubricity
Fuel System Icing Inhibitor
Jet Fuel Additives

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

This report discusses the effects of corrosion inhibitors on the thermal stability, filterability, and lubricity of aircraft turbine engine fuels.

The corrosion inhibitors currently qualified to MIL-I-25017, QPL-25017-9, were found to affect the thermal stability, filterability, and lubricity of fuels differently. For example, some of the corrosion inhibitors gave no measurable improvement in the lubricity of the fuel while others were quite effective, using the Furey ball-on-cylinder test. Similarly, most of the

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U.S.Government Printing Office: 1974 657-013/102

AFAPL-TR-74-20

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FOREWORD

This report was prepared by personnel of the Fuels Branch, Fuels and Lubrication Division, Air Force Aero Propulsion Laboratory. The report discusses data generated through in-house programs by the Fuels Branch.

The data discussed herein was generated during the summer and fall of 1973 as part of Work Units 30480523, 30480546, and 304805FL. A single report was prepared to incorporate all of the data as it pertained to corrosion inhibitor and their effects on fuel properties.

Special recognition is hereby given to Mr. Paul C. Hayes, Jr. who did most of the filterability laboratory work.

This technical report has been reviewed and is approved for publication

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ABSTRACT

This report discusses the effects of corrosion inhibitors on the thermal stability, filterability, and lubricity of aircraft turbine engine fuels.

The corrosion inhibitors currently qualified to MIL-I-25017, QPL-25017-9, were found to affect the thermal stability, filterability, and lubricity of fuels differently. For example, some of the corrosion inhibitors gave no measurable improvement in the lubricity of the fuel while others were quite effective, using the Furey ball-on-cylinder test device. Similarly, most of the corrosion inhibitors caused no measurable degradation of the thermal stability of the fuels while others did. All of the corrosion inhibitors were found to decrease the filterability of the fuel when sea water and a bare steel surface were simultaneously exposed to the fuel containing the corrosion inhibitors. However, the severity of the filtration problem varied among the corrosion inhibitors.

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SECTION I

INTRODUCTION

Fuel-soluble corrosion inhibitor additives are an effective means for reducing the corrosion that may occur within fuel handling and storage systems. More recently, some corrosion inhibitors have proven to be effective fuel lubricity additives and are often used primarily for this reason. Unfortunately, corrosion inhibitors also have disadvantages. Previous work has shown that corrosion inhibitors can degrade fuel filtration, water separation characteristics, and the thermal stability of fuels.

This report is a compilation of recent in-house work concerning the effects of corrosion inhibitors on fuel thermal stability, filterability, and lubricity.

SECTION II

BACKGROUND

1. GENERAL

With the introduction of the jet aircraft and kerosine type jet fuels in the middle 1940's, the Air Force began to have fuel contamination problems which were more severe than those experienced with aviation gasolines. The greater viscosity and density of jet fuels resulted in the entrainment of water and solid matter that often carried over into aircraft fuel systems.

The addition of corrosion inhibitors to jet fuels was begun in the early 1950's to combat excessive corrosion in the ground fuel systems and the subsequent carry-over of the corrosion products into the aircraft.

Corrosion inhibitors were first required to be added to JP-4 fuels by Amendment 1 (18 March 1954) to the jet fuel specification MIL-F-5624B (7 December 1953). The corrosion inhibitor specification itself first appeared as MIL-I-25017 (ASF), issued 20 October 1954 and entitled "Inhibitor, Corrosion, for Aircraft Engine Fuels." To determine the effective level of inhibitor required in the fuel, a corrosion test was required to be performed according to VV-L-791, Method 4011, Procedure B. This 20 hour test conducted at a bath temperature of 100°F defined the minimum effective concentration required for each corrosion inhibitor which qualified to specification properties.

The first Qualified Products List (QPL) for corrosion inhibitors was issued as QPL-25017-1 on 15 September 1955. Table I shows the dates of all QPL's, the number of inhibitors qualified, and the range of minimum effective concentrations (expressed in pounds per 1,000 barrels of fuel) for each QPL. The preparing activity for the first corrosion inhibitor specification and QPL was the Navy Bureau of Aeronautics.

TABLE I

CHRONOLOGICAL LISTING OF QPL'S

QPL Issue	Date	Inhibitors Qualified	Range of Minimum Effective Concentration (lb/1,000 bbl)
QPL-25017-1	15 Sep 55	3	4-7
QPL-25017-2	12 Oct 55	3	4-7
QPL-25017-3	15 May 56	6	4-14.5
QPL-25017-4	27 Sep 57	8	4-15
QPL-25017-5	23 Sep 59	7	4-17
QPL-25017-6	26 Apr 6	6	4-17.5
QPL-25017-7	26 Aug 63	7	4-17.5
QPL-25017-8	30 Apr 71	10	3-7.5
QPL-25017-9	3 Nov 72	12	3-7.5

The "A" revision of MIL-I-25017 was issued on 23 September 1959. Although the corrosion test designation was changed to ASTM Method D 665, Procedure B, this procedure was essentially the same as the VV-L-791 method.

The "C" and "D" revisions of MIL-F-5624 (dated 18 May 1955 and 24 December 1957, respectively) stated that a corrosion inhibitor "shall be" added to JP-4 and JP-5 fuels, but the "E" revision of 23 March 1960 changed this wording to "may be" added. Subsequently, the "F" revision of MIL-J-5624, dated 25 September 1962, indicated that a corrosion inhibitor "shall be added to JP-4," but "shall not be added to JP-5 unless approval is obtained."

A month later MIL-I-25017B was published (22 October 1962) by the Bureau of Naval Weapons. This revision stated that the corrosion test must be performed in accordance with "Method 4011 of FTMS No. 791

(ASTM Method D 665, Procedure B)." The reference to both test methods indicates that they are essentially identical. A 20-hour corrosion period was still required.

In the early 1960's, a jet fuel filtration problem was first associated with the use of corrosion inhibitors. A chemical reaction involving undissolved water, metal (aluminum, steel, magnesium, or zinc), and a corrosion inhibitor was found to result in the formation of a gelatinous material which would rapidly plug filters.

Also, in the late 1950's some corrosion inhibitors were found to cause severe fuel/water separation problems. In particular, fuel filter/water-coalescer units would not effectively remove undissolved water from the fuel when particular corrosion inhibitors were present in the fuel. This led to the removal of several of the corrosion inhibitors from the QPL and later to the use of the Water Separometer Index (WSI) to insure acceptable fuel/water separation characteristics with the additive present. The WSI was later modified and is still in use in JP-4 and JP-5 fuel specifications as the Water Separation Index, Modified (WSIM).

The requirement for a corrosion inhibitor to be added to JP-4 fuel was deleted when MIL-T-5624G appeared on 5 November 1965. The specification stated that a corrosion inhibitor shall not be added to grade JP-4 or JP-5 fuel unless prior approval is obtained. Almost immediately, several specific models of jet engines started to have fuel control malfunctions, especially sticking of servo valves. This problem was soon traced to the removal of the corrosion inhibitor and the value of the corrosion inhibitor as a lubricity agent for JP-4 fuel.

Because of the severity of this problem, the Air Force issued an operational Technical Order on 9 March 1966 to blend corrosion inhibitor into all JP-4 fuel at the base level. This was followed by a purchase exhibit requirement for the addition of corrosion inhibitors in all JP-4 procured during the July-December contract period. Finally, Amendment 1 to MIL-T-5624G was issued on 21 November 1966 reinstating the requirement for corrosion inhibitor conforming to MIL-I-25017 to be blended into

JP-4 fuel by the supplier. However, the use of inhibitor in JP-5 was still excluded. The same policy has continued through the "H" (30 October 1970) and "J" (30 October 1973) revisions of MIL-T-5624.

Subsequently, fuel lubricity problems in other countries have also been resolved by the mandatory use of a fuel corrosion inhibitor. In most cases the use of Hitec E-515 (formerly Santolene C) has been specified, as it is the standard NATO corrosion inhibitor, and most European countries have considerable experience with it. In the USAF, however, 12 corrosion inhibitors (Table II) are presently qualified to QPL-25017-9 and any one of these inhibitors may be used in JP-4 fuel.

The effectiveness of corrosion inhibitors in preventing fuel system corrosion has not been questioned. When used consistently and in the proper concentrations, the corrosion inhibitors keep a fuel system essentially free of corrosion and corrosion products. Several large pipeline operators require the use of a corrosion inhibitor in products transferred through their pipelines for corrosion protection.

TABLE II

CORROSION INHIBITORS QUALIFIED TO QPL-25017-9

ADDITIVE	CONCENTRATIONS (Lbs/1000 Bbl) REL. EFF.(1)	MIN. EFF.(2)	MAX. ALLOW.
Lubrizol 541	2	3	6
Tretolite Tolad 244	3	4.5	6
Tretolite Tolad 245	5	7.5	12
Apollo PRI-19	2	3	8
Edwin Cooper Hitec E-515*	5	7.5	16
Edwin Cooper Hitec E-534*	3	4.5	8
DuPont AFA-1*	3	4.5	12
DuPont DCI-4A	2	3	8
Nalco 5400-A*	2	3	8
Nalco 5402	2	3	8
UOP Unicor J	2	3	8
Conoco T-60	4	6	16

*Contain phosphorus

(1) The relative effective concentration is the minimum concentration which would pass the corrosion test of MIL-I-25017.

(2) The minimum effective concentration is the minimum quantity that may be added to JP-4 fuel.

2. THERMAL STABILITY

The effect of jet fuel corrosion inhibitors on the thermal stability of jet fuels has not been well established. Previous work by Shamblin and Johnston (Reference 1), using a CRC-ASTM Standard Coker, indicated that a corrosion inhibitor may reduce filter-plugging deposits but may also increase tube deposits. Similar tests by Johnston, Monita, et al (Reference 2), gave similar results. More recently, Shell Research Limited, using a special high temperature fuel test rig with a maximum fuel temperature of 180°C (355°F), found that Hitec E-515 caused a significant increase in filter-plugging deposits and reduced heat exchanger performance (References 3 and 4).

Subsequent to the above referenced investigations, several new corrosion inhibitors have been qualified to MIL-I-25017 and some of the older additives are no longer on the QPL. Also, some new aircraft under development will thermally stress fuels to temperatures approaching the specified thermal stability limits for conventional fuels. Thus, the possibility that some of the approved fuel additives might degrade the thermal stability of the fuel needed to be investigated. This led to the decision to evaluate all corrosion inhibitors qualified to QPL-25017-9.

Oleic acid, a fatty acid believed to be similar in chemical structure to many of the corrosion inhibitors and known to be a good fuel lubricity additive, was tested for its effect on thermal stability. Also, the fuel system icing inhibitor (ethylene glycol monomethyl ether) conforming to MIL-I-27686E, was examined for its effect on jet fuel thermal stability as it is normally added to all JP-4 and JP-5 fuel.

3. FUEL FILTERABILITY

In recent years, the premature plugging of ground fuel handling filter-separator units has cost the Air Force tens of thousands of dollars for replacement filters. The premature plugging problem is believed to be primarily caused by a gelatinous precipitate formed by the interaction of a corrosion inhibitor, water, and metal. McLaren, Krynitsky, and Hazlett demonstrated the formation of precipitates and their filter-plugging properties in 1965 (Reference 5). Their work was initiated following filtration problems at Ramey Air Force Base, Puerto Rico, in the early 1960's. At that time the role of a corrosion inhibitor in causing the premature plugging of a filter-separator was first documented.

Subsequently, the Air Force tried to eliminate the use of corrosion inhibitors in 1965, but their value as a lubricity agent was discovered and they were put back into JP-4 in 1966. The possible use of the Silting Index as a method for determining fuel filterability was investigated in the late 1960's, but never adopted due to test method deficiencies.

In 1972, the Air Force Directorate of Aerospace Fuels developed the Fuel Filtration-Time test. Subsequently, the Fuels Branch of the Air Force Aero Propulsion Laboratory refined the Fuel Filtration-Time test and incorporated it into specification MIL-T-5624J dated 30 October 1973 (see Appendix). This test consists of measuring the time required to filter a given volume of fuel through a standard filter under prescribed conditions. The Filtration-Time test has been difficult for some fuel suppliers to consistently pass; especially those who ship their fuel via ocean-going barge or tanker prior to delivery. This problem has been traced back to the formation of the gelatinous precipitates formed by the reaction of the corrosion inhibitor with sea water and a reactive metal such as steel.

As one approach to solving this fuel filterability problem a decision was made to reexamine the formation of the gelatinous precipitate and the variables affecting the reaction. For example, the importance of sea water as opposed to fresh water, the choice of corrosion inhibitor, the concentration of the corrosion inhibitor, and the reaction time were variables of interest.

4. FUEL LUBRICITY

Since the reintroduction of the corrosion inhibitors into JP-4 fuel in 1966, the Air Force has not had any fuel lubricity problems of consequence. However, operators of commercial and military aircraft using Lucas fuel pumps have had numerous pump failures traced to a lack of fuel lubricity. Similarly, the JT9D engines used on the Boeing 747 have had fuel pump problems when operated on widecut fuels (i.e., Jet B types) which do not contain a corrosion inhibitor or a lubricity additive. As mentioned previously, many of these problems have been solved by the mandatory use of Hitec E-515 as a fuel lubricity additive.

From 1965 to 1968 the Air Force Aero Propulsion Laboratory contracted with the ESSO Research and Engineering Company to investigate fuel lubricity and fuel lubricity test methods. One of the results of this contract was the delivery of a Furey Ball-on-Cylinder fuel lubricity test device to the Air Force. The ESSO program demonstrated the sensitivity of this device to differences in fuel lubricity (Reference 6). Subsequently, work by the Air Force and others has demonstrated that this device is one of the best fuel lubricity test methods presently available. Preliminary data indicates a good correlation exists between the Ball-on-Cylinder rig and the Lucas Dwell Meter, a fuel lubricity device developed by the Lucas Aerospace Ltd. Also, a correlation exists between the Ball-on-Cylinder and the CRC-Bendix Fuel System Simulator, a fuel lubricity test device simulating a fuel control servo valve (Reference 7).

SECTION III

TEST PROGRAM

1. EFFECT OF ADDITIVES ON JET FUEL THERMAL STABILITY

The Jet Fuel Thermal Oxidation Tester (JFTOT) was selected for evaluating the effects of corrosion inhibitors on jet fuel thermal stability. The JFTOT is believed to provide the most realistic data of any of the currently available thermal stability test devices. The ASTM-CRC Fuel Coker does not operate at sufficiently high temperatures and the operating pressures of both the Fuel Coker and the Research Coker are too low to prevent nucleate boiling of the JP-4 fuels.

The first series of tests was conducted using a specially processed, low aromatic JP-4 fuel (Reference 8). The baseline fuel, without additives, was evaluated at maximum metal temperatures above 700°F at 500 psig in the JFTOT without failing (Table III). The additives were added to the test fuel at their maximum allowable concentrations. Fuel samples containing each of the additives, including oleic acid and fuel system icing inhibitor (FSII), were first evaluated on the JFTOT at a maximum heater tube metal temperature of 690°F to determine which additives significantly reduced the thermal stability of the fuel. All tests were conducted for 2.5 hours at 500 psig. FSII, oleic acid, and eight of the corrosion inhibitors successfully passed the tests, as shown in Table III.

Additional tests were conducted on the three additives that failed during the 690° test, i.e., AFA-1, Hitec E-515 and Nalco 5400-A, to determine the reduction in breakpoint due to each additive. The visual tube rating shown in Table III for these three samples were plotted in Figure 1. The plots show that the resulting visual rating breakpoints are 565°F, 575°F, and 535°F for the AFA-1, Hitec E-515, and Nalco 5400-A additives, respectively.

TABLE III

JFTOT RESULTS USING FUEL JP-4 (SPECIAL)

ADDITIVE DESIGNATION	DATE OF TEST	TEST TEMP (°F)	MAX TUBE RATING VISUAL	MAX TUBE RATING TDR (SPUN)	FILTER PRESS. DROP (IN./HG)
BASELINE	27 Sep 73	720	3	35.5	0
	20 Oct 73	710	2	15.8	0
	4 Oct 73	710	2	10.3	0
	5 Oct 73	720	2	26.5	0
	9 Oct 73	730	2+	18.1	0
	11 Oct 73	730	2+	18.0	0
	12 Oct 73	750	2	15.1	0
	15 Oct 73	780	4	50.0	0.2
	16 Oct 73	770	4	50.0	0.2
AFA-1	2 Oct 73	690	4P+	43.9	0
	4 Oct 73	640	4P	38.1	0
	5 Oct 73	590	4	17.5	0
	9 Oct 73	570	4	18.0	0
	10 Oct 73	500	2	4.8	0.2
	23 Oct 73	535	2	1.7	0
	24 Oct 73	550	1	6.4	0
CONOCO T-60	10 Oct 73	690	2	7.8	0
DCI-4-A	10 Oct 73	690	2	6.3	0
HITEC E-515	1 Oct 73	690	4+	44.0	0
	4 Oct 73	640	4P	39.0	0
	4 Oct 73	590	3	17.4	0
	5 Oct 73	570	3+	16.3	0
	9 Oct 73	550	3	10.9	0
	11 Oct 73	525	1	5.7	0
	12 Oct 73	535	2	4.2	0
LUBRIZOL 541	1 Oct 73	690	1+	9.0	0
NALCO 5400-A	5 Oct 73	690	4P	50.0+	0
	9 Oct 73	590	4P	23.3	0
	10 Oct 73	540	4	18.0	0
	11 Oct 73	525	1	6.0	0
NALCO 5402	5 Oct 73	690	1	13.0	0
[illegible]	3 Oct 73	690	1	9.0	0
TOLAD 244	2 Oct 73	690	1	10.0	0
TOLAD 245	3 Oct 73	690	1	7.7	0
UNICOR J	10 Oct 73	690	1	7.0	0.2
OLEIC ACID	16 Oct 73	690	2	11.2	0
[illegible]	10 Oct 73	690	2	9.0	0

The JFTOT tubes were also rated using the ALCOR Tube Deposit Rater (TDR). These ratings are tabulated in Table III and plotted in Figure 2. Assuming that the TDR ratings of 18 or above indicate a failure, the breakpoints of the fuel samples containing AFA-1, Hitec E-515, and Nalco 5400-A are 580°F, 580°F, and 555°F, respectively.

The additives were then evaluated in a Jet A fuel (AFFB-14-70) with a breakpoint of 560°F. The results of these tests are tabulated in Table IV. Tests were first run at a temperature below the breakpoint of the fuel to determine if the additives degraded the fuel. There was no evidence of significant degradation caused by any of the additives (Table IV). This result is as would be expected from the low aromatic JP-4 test data.

There has been some evidence that additives may actually increase thermal stability. A second set of tests was conducted at a temperature slightly above the breakpoint of fuel AFFB-14-70. The results of these tests (Table IV) show that none of the additives improve the thermal stability of the base fuel.

General agreement is evident between the visual and TDR tube ratings. However, wide discrepancies do exist, e.g., the Hitec E-515 test on 23 October 1973 with fuel AFFB-14-70 where a visual rating of 4+ and a TDR rating 16.3 was obtained.

No significant increase in pressure drop was measured for any of the fuels, including those tests involving the three additives that failed at temperatures of 580°F or below. These additives were run at temperatures at least 110°F above the breakpoint and only the deposits on the test section were affected. This indicates that the presence of the additive would affect the heat transfer of a fuel/oil cooler but would not cause an increase in filter pressure drop.

It should be pointed out that the data indicates that none of the additives reduce the thermal stability of the fuel below the current specification limit of 500°F.

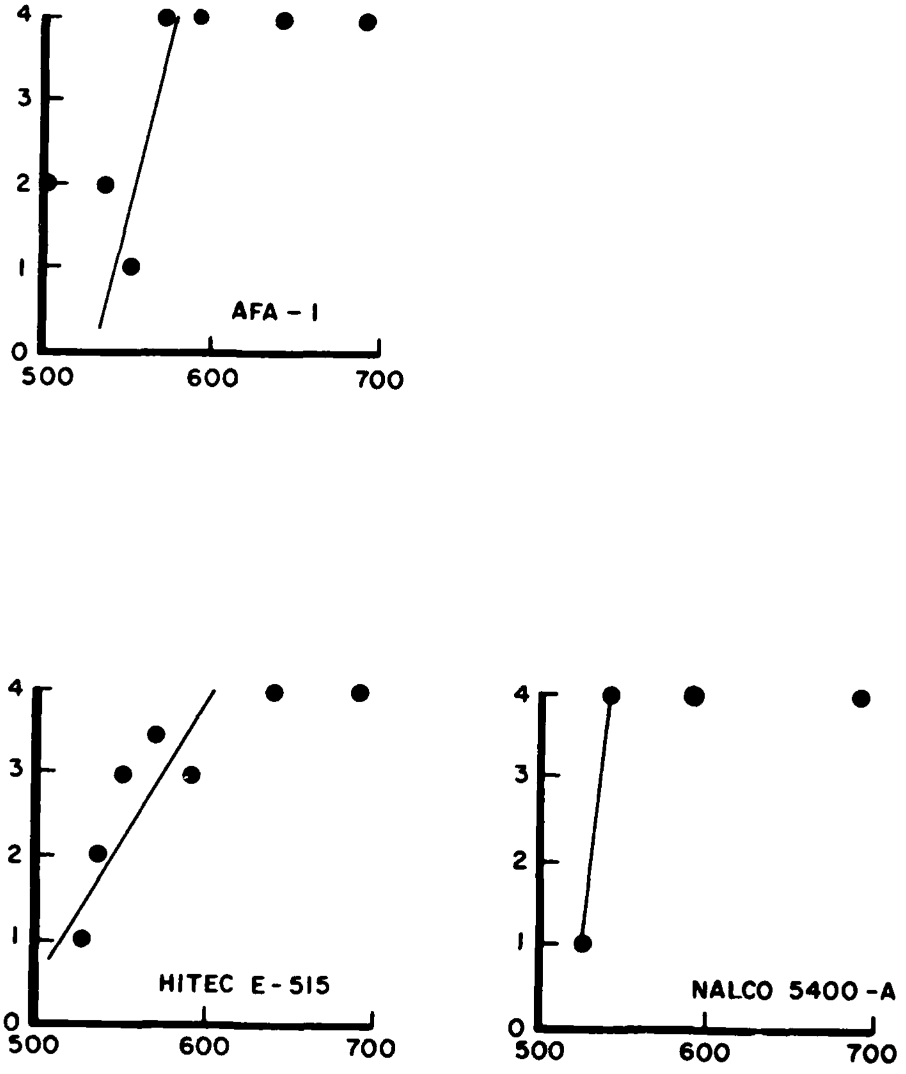

Figure 1. JFTOT Visual Breakpoints

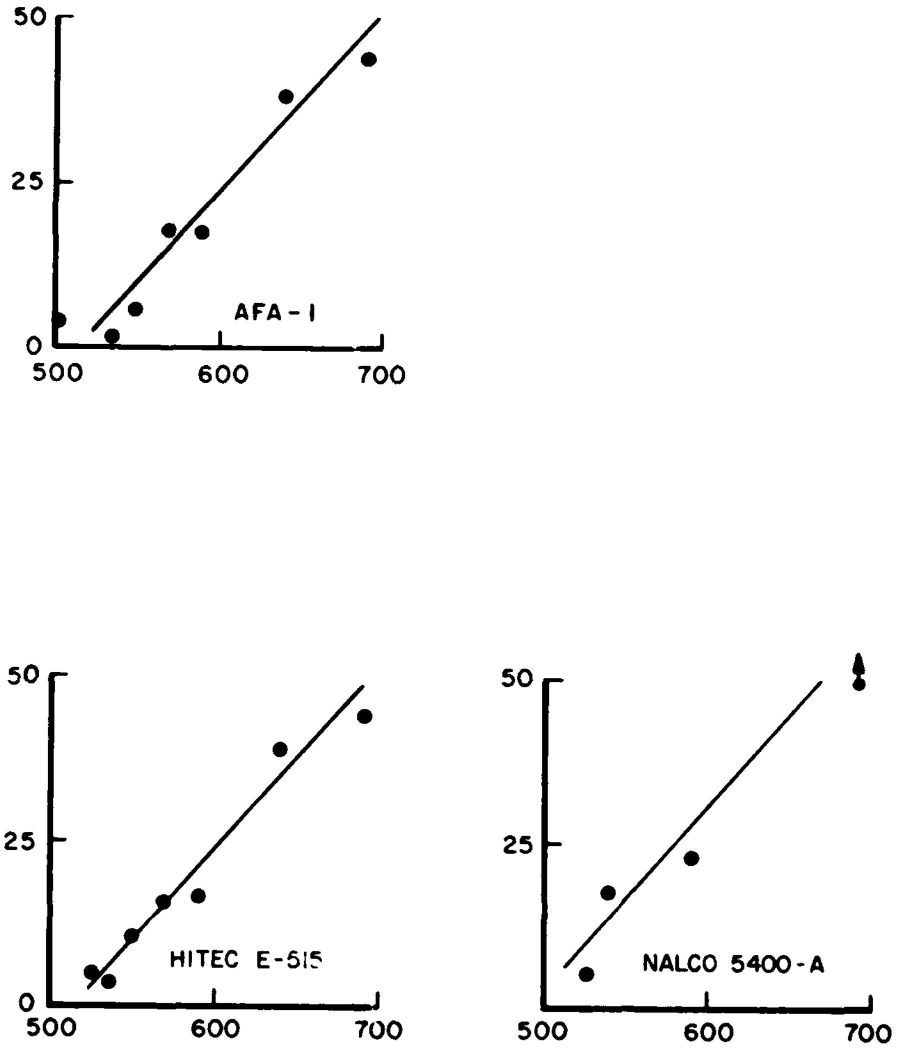

Figure 2. JFTOT TDR Breakpoints

TABLE IV

JFTOT RESULTS USING FUEL AFFB-14-70

ADDITIVE DESIGNATION	DATE OF TEST	TEST TEMP (°F)	MAX TUBE RATING VISUAL	TDR (SPUN)	FILTER PRESS. DROP (IN./HG)
BASELINE	15 Oct 73	570	3	18.0	0
	16 Oct 73	550	1	9.0	0
	21 Oct 73	560	3	22.2	0
AFA-1	16 Oct 73	540	1	5.2	0
	25 Oct 73	570	4	21.0	0
CONOCO T-60	18 Oct 73	540	1+	4.1	0
	1 Nov 73	570	3+	23.8	0
DCI-4A	17 Oct 73	540	2	10.0	0
	31 Oct 73	570	4P	22.2	0
HITEC E-515	19 Oct 73	540	2+	9.0	0.2
	23 Oct 73	555	4+	16.3	0
LUBRIZOL 541	17 Oct 73	540	2	7.5	0.2
	31 Oct 73	570	4	24.8	0
NALCO 5400-A	17 Oct 73	540	2	4.0	0
	24 Oct 73	555	3	18.5	0
NALCO 5402	19 Oct 73	540	2	4.0	0
	2 Nov 73	570	3+	21.2	0
PRI-19	17 Oct 73	540	2	11.0	0.2
	1 Nov 73	570	3+	25.7	0
TOLAD 244	18 Oct 73	540	2	13.8	0
	31 Oct 73	570	3	22.5	0
TOLAD 245	18 Oct 73	540	1	5.3	0
	1 Nov 73	570	4	24.5	0
UNICOR J	19 Oct 73	540	2	6.5	0
	2 Nov 73	570	3	26.4	0
OLEIC ACID	2 Nov 73	550	2	8.9	0
	5 Nov 73	570	3	23.0	0
FSII	19 Oct 73	540	1	7.0	0
	1 Nov 73	570	4	20.0	0

It is interesting to note that only the three additives that result in breakpoints below 690°F contain phosphorus. Thus, caution should be exercised in the use of additives containing phosphorus if thermal stability is of concern. This conclusion is in agreement with the results of work done by Shell Research Limited (Reference 9). Shell ran tests on two special fuel samples (M6068 and M6069); one containing half the normal amount of phosphorus and the other similar to Hitec E-515 but with the phosphorus omitted. The data indicates that the adverse effect on thermal stability in the JFTOT was due to the presence of the phosphorus component in the additive.

Tests by Shell Research Limited in a high temperature fuel system rig (References 3 and 4) also revealed the deleterious effect of Hitec E-515 on fuel thermal stability. The addition of Hitec E-515 to a hydrotreated fuel and an acid treated fuel had a detrimental effect on both the filter pressure drop and the heat exchanger performance of the fuel at a temperature of 356°F. The effect on the acid treated fuel was much more pronounced than was the effect on the hydrotreated fuel.

Shell also found (Reference 4) that the presence of Hitec E-515 encouraged solids transmission through the filters and even with reduced velocities the performance of the filters was below normal.

Standard Coker tests were conducted by Shell on a fuel containing Hitec E-515 (References 3 and 4). The threshold temperature of the base fuel was 385°F. There was some indication that the addition of Hitec E-515 to the fuel lowered the threshold temperature, however, the evidence was inconsistent.

2. EFFECTS OF FUEL CORROSION INHIBITORS ON FUEL FILTERABILITY

Small scale tests were devised to simulate the environment that JP-4 fuels would likely encounter during barge or tanker shipment on fresh and salt waterways. The effects of known variables on the filtration-time of the fuel were then examined by varying one condition at a time. Three sets of the test were conducted.

a. Test Set A

Twelve liters of clay-treated JP-4 fuel, containing 0.15% FSII, and the corrosion inhibitor under test, were added to a clean, 5-gallon, epoxy-lined steel can. Polished, soft-steel shim stock, having a steel-surface/fuel-volume ratio of 0.03 sq in/cu in, was then placed in the test container. Also 240 ml of artificial sea water, conforming to ASTM test method D-665, was then added to the test container. The test can was then stored at ambient conditions for 72 hours with periodic agitation. After a 40 hour settling period, two 1-gallon fuel samples (samples #1 and #2) were poured or siphoned from the can with minimum agitation. The filtration-time for each of these two samples was then measured using the procedure described in the Appendix.

Variations to this series of tests included: (a) variation of the fuel corrosion inhibitor type and concentration. (b) total absence of FSII, (c) absence of the bare steel surface, and (d) substitution of distilled water or a hard (Wright-Patterson Air Force Base, Ohio) tap water for the artificial sea water. All possible combinations were not run due to the large number of tests that would have been required. Rather, two of the additives were selected to evaluate the effects of these test variations.

Eleven of the 12 corrosion inhibitors presently qualified to MIL-I-25017 and listed on QPL-25017-9 were tested using the Test Set A method. (The other additive, Hitec E-534, was not available.) The results of these tests are listed in Table V. Without a corrosion inhibitor present, the filtration-times ranged from 3.3 to 4.0 minutes (see Control Test, Table V); the presence or absence of the bare steel surface or the sea water had no effect.

With a corrosion inhibitor in the fuel, the filtration-times were always significantly higher than without a corrosion inhibitor, provided that both the bare steel and the sea water were present. At the minimum effective concentration, most of the corrosion inhibitors caused the filtration-time to exceed 20 minutes when tested with the bare steel and

TABLE V

SUMMARY OF TEST DATA FOR TEST SET A

ADDITIVE	CONCENTRATION (Lb/1000 Bbl)	FILTRATION TIMES (Minutes)		REMARKS
		Sample Nr. 1	Sample Nr. 2	
AFA-1	4.5	24.6	26.5	Duplicate(1)
		40+	40+	
Lubrizol 541	3.0	38.8	37.1	
		80+	80+	Duplicate(1)
Tolad 244	4.5	27.3	30.8	
		100+	100+	Duplicate(1)
Apollo PRI-19	3.0	35.0	34.4	
		50+	55+	Duplicate(1)
Hitec E-515	7.5	11.9	23.3	
		7.0	14.7	Duplicate
		4.6	12.4	W/O FSII
		4.1	9.7	W/O Steel
		4.3	3.7	Distilled H_2O
		3.9	3.7	Hard Tap H_2O (1)
	16.0(2)	5.7	20.7	
DuPont DCI-4A	3.0	24.4	24.4	
		8.0	7.6	Duplicate(1)
		5.2	5.1	Triplicate (1)
Nalco 5400-A	3.0	5.9	6.3	
		18.8	18.7	Duplicate
		12.7	21.7	W/O FSII
		4.3	4.9	W/O Steel
		3.8	4.3	Distilled H_2O
		3.9	4.0	Hard Tap H_2O(1)
	8.0(2)	39.6	36.6	
Nalco 5402	3.0	10.2	11.5	
Unicor J	3.0	30.1	19.4	
Tolad 245	7.5	180+	177.5	
Conoco T-60	6.0	22.9	30.3	
Control	None	4.0	3.9	
		3.8	3.3	Duplicate
		4.0	4.0	W/O Steel
		3.5	3.2	Distilled H_2O

(1) Steel Shim Stock from different batch and samples withdrawn by siphoning.

(2) Maximum allowable concentration rather than minimum effective concentration.

the sea water. Tolad 245 gave the highest filtration-times, about 180 minutes, while Hitec E-515, DuPont DCI-4A, Nalco 5400-A, and Nalco 5402 gave the lowest filtration times when both the bare steel and sea water were present.

The duplicate and one triplicate tests indicated poor test repeatability. Thus, some of the additives which gave high filtration-times under the sea water and bare steel test conditions may be the victims of statistics; i.e., additional tests might show these additives to be less severe, on the average, then indicated in Table V. For example, additive DCI-4A appeared to be a relative bad-actor in terms of filtration-time effects based on the first test; yet the repeat tests gave much lower filtration-times than the first test. The poor test repeatability may be caused by entrained water in the fuel, as suspended water droplets might also affect the filtration rate as well as the filter-plugging precipitate.

The tests conducted without FSII present are inconclusive. The absence of FSII appeared to slightly decrease the filtration-times when tested with Hitec E-515 and had no apparent effect when tested with Nalco 5400-A. However, earlier work by McLaren, et al (Reference 5) showed that FSII was a major variable. The tests conducted by McLaren used FSII that contained 0.4% glycerol by volume. Their tests were much more severe (and more realistic) than the Test Set A test conditions. They added sufficient FSII to the water bottoms to give a 20% concentration. This ensured that a significant quantity of the FSII would remain in the fuel phase during the test. Test Set A conditions probably resulted in almost all of the FSII leaving the fuel phase and entering the water phase.

The absence of the bare steel in contact with the fuel and water greatly reduced the deleterious effects of the corrosion inhibitors on the filtration-times. Again referring to Table V, the absence of the bare steel gave filtration-times of 4.1 to 9.7 minutes for corrosion inhibitors Hitec E-515 and Nalco 5400-A. Based on these results, it appears that the epoxy-coating used on the inside of the steel test cans was relatively effective in preventing the metal walls of the test container from reacting with the sea water and the corrosion inhibitor.

The substitution of distilled water and hard tap water for the artificial sea water also greatly decreased the deleterious effects of the corrosion inhibitors. With distilled water the filtration-times ranged from 3.7 to 4.3 and with the hard water, 3.7 to 4.0 minutes. It was anticipated that the hard tap water would be significantly more severe than the distilled water. Earlier work (Reference 10) showed that the hard tap water significantly degraded filter-separator coalescence with a corrosion inhibitor in the fuel while the distilled water had little effect.

The results of Test Set A indicated that: (1) there appears to be significant differences among the corrosion inhibitors as to the effect on fuel filterability, (2) the corrosion inhibitors have little effect on fuel filterability unless both a bare steel surface and sea water are also present (previous work by McLaren (Reference 5), showed that zinc, magnesium, and aluminum also react with corrosion inhibitors and sea water to form filter-plugging precipitates), and (3) the tests gave very poor repeatability.

b. Test Sets B and C

Results from Test Set A raised questions as to the effects of reaction time and settling time on the measured filtration-time results. All Test Set A samples had been drawn after the fuel had settled for 40 hours. Test Set B and C were designed to examine the effects of settling and the reaction time required for the corrosion inhibitor, sea water, and bare steel to form the filter-plugging precipitate.

Test Set B used the same basic test set-up as Test Set A except that the fuel volume was increased from 12 liters to 15 liters with appropriate increases in the corrosion inhibitor, FSII, sea water, and bare steel. After the test container had been prepared (with the test fuel, corrosion inhibitor, steel, water, etc.), it was stored at ambient temperature for

48 hours with periodic agitation. After 48 hours the test container was well agitated (by shaking) and a 1-gallon fuel sample was immediately drawn (Sample Nr. 1). The test container was then left undisturbed for 24 hours and another 1-gallon sample was drawn without agitation (Sample Nr. 2). Immediately afterward the container was agitated and Sample Nr. 3 was drawn. This resulted in three samples: Sample Nr. 1, an "agitated-sample" after 48 hours; Sample Nr. 2, a "settled-sample" after 72 hours; and Sample Nr. 3, an "agitated-sample" after 72 hours. Filtration-time measurements were run on each sample immediately after they were drawn.

Test Set C was identical to Set B except for the reaction time. Sample Nr. 1 was drawn 144 hours (6-days) after test start, and Samples Nr. 2 and Nr. 3 were drawn 168 hours (7-days) after test start.

Table VI lists the results of the filtration-time measurements for Samples Nr. 1, 2, and 3 for Test Sets B and C. Four additives were tested, Hitec E-515, Nalco 5400-A, Nalco 5402, and Tolad 245. Three conclusions can be drawn from the results of Test Sets B and C. First, Test Set B results confirm the Test Set A results that Tolad 245 is a "bad actor" insofar as filtration-times are concerned when sea water and bare steel are present. Its use in JP-4 that is to be shipped by barge or tanker over salt water should be avoided.

The second conclusion is that two or three days is more than sufficient reaction time for the corrosion inhibitor, bare steel, and salt water to react to form the filter-plugging precipitate. The longer reaction times of Test Set C as compared to Test Set B resulted in no significantly greater filtration-times in two of our three cases.

The third conclusion is that the test repeatability is poor. Further testing, with additional refinement of the test procedure, would be required to obtain a valid ranking of corrosion inhibitors, to determine the true significance of settling, and to determine minimum reaction time, for example.

TABLE VI

SUMMARY OF TEST DATA FOR TEST SETS B AND C

ADDITIVE	CONCENTRATION (Lbs/1000 Bbl	FILTRATION TIMES (Seconds)					
		Test Set B			Test Set C		
		Sample Nr. 1,	Nr. 2,	Nr. 3	Sample Nr. 1,	Nr. 2	Nr. 3
Hitec E-515	7.5	25.9	11.5	47.0	6.9	7.9	32.7
Nalco 5400-A	3.0	6.3	6.4	15.2	17.9	17.3	30.7
Nalco 5402	3.0	9.2	8.3	16.7	10.2	7.4	15.6
Tolad 245	7.5	90	90	90+	---	---	---

The effect of settling on filtration-times could not be determined. Comparing Samples Nr. 1 and 2 from Test Sets B and C; settling appears to have no significant effect on filtration-times. Yet, comparing Samples Nr. 2 and 3; settling does appear to be worthwhile. Thus, no conclusion concerning settling was obtained.

c. Visual Observations

A 5-gallon glass jar was used as a container into which JP-4 containing Hitec E-515, artificial sea water, and soft steel shim stock were placed. A flocculent or spongy appearing precipitate was seen to form at the fuel/water interface. Any agitation of the jar resulted in the precipitate entering the fuel phase where it floated with little apparent settling.

The precipitate was similar to rust in color; i.e., an orange-brown color. It is believed that this precipitate is the primary filter-plugging material formed when a corrosion inhibitor, sea water, and bare steel interact. Analysis of the precipitate identified iron, magnesium, and sodium as the major metals present; lead, calcium, maganese, and tin were present in minor quantities; and silicon, aluminum, copper, and silver were present in trace quantities. Also, carbonyl functional groups were identified using infrared spectrographic analysis after treating the precipitate with hydrochloric acid and extracting it with ethyl ether. This indicates that a soap was present in the precipitate (Reference 11).

It is interesting to note that phosphorus was not found in the precipitate although Hitec E-515 contains phosphorus. This would indicate that the phosphorus containing ingredient of Hitec E-515 is not contributing to the filterability problem.

3. DETERMINATION OF EFFECTIVENESS OF CORROSION INHIBITORS AS FUEL LUBRICITY ADDITIVES

The Air Force is currently investigating the effectiveness of corrosion inhibitors as lubricity agents with the Furey Ball-on-Cylinder apparatus. This device described by Reference 6 consists of a stationary steel ball which rides on a rotating steel cylinder. The bottom portion of the cylinder is immersed in the test fuel. The force of the ball on the cylinder and the rotational speed of the cylinder can be adjusted. Also, the temperature and atmosphere of the environment surrounding the test ball, cylinder, and fuel are controlled.

The output of the test is the wear scar diameter on the ball. The actual wear scar has an elliptical pattern. The wear scar diameter reported is the average of the minimum and maximum wear scar diameters. The coefficient of friction is also calculated but this has previously been found (Reference 6) to be a much poorer indicator of fuel lubricity than the wear scar diameter.

In the test program, mixtures of each corrosion inhibitor at its relative effective and maximum allowable concentrations in the base fuel were tested with the ball-on-cylinder apparatus. The base fuel was a JP-4 which had been clay-treated to remove any existing fuel additives or polar compounds which might have affected its lubricity. The operating conditions for the test include:

	Ball	Cylinder
Material	52100 AISI Steel	52100 AISI Steel
Hardness	61 Rockwell C	22.5 Rockwell C
Surface Finish	2 Micro inches CLA	6 to 10 micro inches CLA

Cylinder Rotation Speed -- 240 rpm
Loading on Ball -- 1000 grams
Length of Test -- 32 minutes
Controlled Atmosphere -- 0.5 cfm dry air at 75°F, indirect flow

The mean wear scar diameters, based on three trials per mixture of each corrosion inhibitor at its relative effective and maximum allowable concentrations are shown in Table VII.

TABLE VII

THE EFFECT OF CORROSION INHIBITOR ADDITIVES ON THE LUBRICITY OF JP-4

	RELATIVE EFFECTIVE CONCENTRATION		MAXIMUM ALLOWABLE CONCENTRATION	
Corrosion Inhibitor	Concentration lbs/1000 BBL	Wear Scar Diameter (mm)	Concentration lbs/1000 BBL	Wear Scar Diameter (mm)
None	0	.52	0	.52
AFA-1	3	.50	12	.525
LUBRIZOL 541	2	.51	6	.42
TOLAD 244	3	.52	6	.45
PRI-19	2	.52	8	.39
HITEC E-515	5	.46	16	.34
DCI-4A	2	.52	8	.34
NALCO 5400-A	2	.53	8	.50
UNICOR-J	2	.56	8	.38
TOLAD 245	5	.49	12	.37
CONOCO T-60	4	.50	16	.31
NALCO 5402	2	.52	8	.38

At the relative effective concentration of the corrosion inhibitors in the base fuel, only HITEC E-515 noticeably improved the lubricity of the base fuel as measured in the Ball-on-Cylinder rig. The wear scar diameter was .46 mm for the HITEC E-515 mixture as compared to .52 mm for the base fuel.

At the maximum allowable concentration of the corrosion inhibitors in the base fuel, the inhibitors which improved the lubricity of the clay-treated JP-4 the greatest were CONOCO T-60, HITEC E-515, and DCI-4A. The corresponding wear scar diameters for the additive mixtures were .31, .34 and .34 mm. These three additives are approximately equally effective lubricity agents. However, the maximum allowable concentration of DCI-4A was 8 lbs/1000 bbl. which is half that of CONOCO T-60 or HITEC E-515. On a concentration basis, the DCI-4A is the superior lubricity agent.

Two of the inhibitors at the maximum allowable concentration produced no improvement in the lubricity of the base fuel. These additives, AFA-1 and NALCO 5400-A, produced wear scar diameters of .525 and .50 mm, respectively.

The remaining corrosion inhibitors at their maximum allowable concentrations were found to produce an intermediate improvement in the lubricity of the base fuel.

Additional work with corrosion inhibitors in other base fuels indicates the same relationships established with the inhibitors at their maximum allowable concentration in the clay-treated JP-4 fuel hold true. This work, upon completion, will be presented in a future report.

SECTION IV

CONCLUSIONS

1. THERMAL STABILITY EFFECTS

a. None of the additives lowered the thermal stability of fuels below the current specifications limit.

b. Additives containing phosphorus drastically reduce the thermal stability of the fuel.

c. The additives did not effect filter pressure drop.

d. None of the additives improved the thermal stability of the fuel.

2 FUEL FILTERABILITY

a. Corrosion inhibitors will react with bare metal surfaces and sea water to form a precipitate which will rapidly plug filters. This precipitate is believed to be a soap formed by the reaction of organic acids (from the corrosion inhibitor or fuel) and metal hydroxides.

b. Some corrosion inhibitors are much worse than others in forming the filter-plugging precipitate.

c. Two or three days are more than ample time for a corrosion inhibitor to react with sea water and steel to form a filter-plugging precipitate.

d. Settling will tend to remove the precipitate if sufficient time is available. However, agitation of the fuel/water interface will reintroduce the precipitate into the fuel phase.

e. Sea water is much more reactive with the metal surface and corrosion inhibitors than is either distilled water or a hard tap water.

f. The filter-plugging precipitate can be prevented or reduced dramatically from forming by: removing the corrosion inhibitor, coating the bare metal surface, or preventing the contamination of the fuel with sea water.

g. Epoxy-coated steel is relatively unreactive with the corrosion inhibitor and sea water.

3. FUEL LUBRICITY

a. Preliminary results using the Ball-on-Cylinder lubricity test device reveal significant differences among the qualified corrosion inhibitors in regards to their effectiveness as fuel lubricity additives.

b. Two inhibitors, DuPont AFA-1 and Nalco 5400-A appear to have no significant beneficial effect on the lubricity of a clay-treated JP-4 fuel at their relative effective or maximum allowable concentrations.

c. The other nine inhibitors, Lubrizol 541, Tolad 244, Tolad 245, Apollo PRI-19, Unicor J, Nalco 5402, Conoco T-60, Hitec E-515, and DuPont DCI-4A improved the lubricity of the fuel to varying degrees when tested at their maximum allowable concentration.

d. Only Hitec E-515 appears to impart a measurable improvement in the lubricity of the clay-treated JP-4 fuel at its relative effective concentration.

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APPENDIX

METHOD FOR DETERMINATION OF JP-4 FILTRATION TIME & TOTAL SOLIDS

1. Scope. This method describes a procedure for determining simultaneously the filterability characteristics and solids contamination of jet fuel. Purpose is to detect and prevent contaminants in jet fuel which can plug ground system as well as aircraft filtration equipment.

2. Summary of Method. One gallon of jet fuel is filtered through a membrane filter in the laboratory. The time required to filter this volume is measured in minutes and solids content is determined gravimetrically.

3. Apparatus:

a. Membrane Filter - plain 47 mm diameter, nominal pore size 0.8 micron, Millipore part number AAWP04700.

b. Filtration Apparatus - of the type shown in Figure A3-ASTM Method D 2276. It consists of a funnel and funnel base with a filter support such that a membrane filter can be securely locked or clamped between the sealing surfaces of the funnel and its base. The funnel and funnel base can be of glass or stainless steel construction. If a fritted glass funnel base is used, it should periodically be checked for plugging.

c. Insert Ring - a 47 mm diameter stainless steel insert ring with dimensions to give a filtering area of 4.8 cm^2.*

d. Vacuum Flask - a minimum of four liters.

e. Vacuum System - that preferably develops a minimum of 20 inches of mercury vacuum.

f. Oven - of the static type (without fan assisted circulation) controlling to 90 ± 5°C.

g. Forceps - flat-bladed with unserrated non-pointed tips.

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* A suitable insert ring is available from the Millipore Corporation, Part Number XX10 04707.

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h. Solvent Filtering Dispenser - containing a minimum pore size of 1.2 micron filter in the delivery line.

i. Glass Petri Dish - approximately 125 mm in diameter with removable cover.

j. Analytical Balance - single or double pan, the precision standard deviation of which must be 0.07 mg or better.

4. Preparation of Apparatus and Sample Containers. All components of the filtration apparatus (except the vacuum flask), sample containers and their caps must be cleaned as described in A2.6.1.1 through A2.6.1.7 of AST, D 2276.

5. Sampling. Obtain a representative 1-gallon sample as directed in A2.7 of ASTM D 2276.

6. Test Procedure:

a. Membrane filters will be removed from the package and placed in a dessicator for a minimum of 24 hours prior to use in the test. Filters will not be preheated.

b. Weigh one membrane filter. Should the filter weigh in excess of 90 mg, it should not be used as excessive filtration times may result.

c. Center the stainless steel insert ring on the filter base. Place the membrane filter directly over the stainless steel insert ring. Lock or clamp top funnel into place.

d. Immediately prior to filtering the fuel, shake the sample to obtain a homogenous mix and assure that fuel temperature does not exceed 85°F. Clean the exterior top portion of the sample container to ensure that no contaminants are introduced.

e. With the vacuum off, pour approximately 200 ml of fuel into the funnel.

f. Turn vacuum on and record starting time. Continue filtration of the 1-gallon sample. Record the vacuum in inches of mercury one minute after start and again immediately prior to completion of filtration. Throughout filtration, maintain a sufficient quantity of fuel in the funnel so that the membrane filter is always covered.

g. Report the filtration time in minutes expressed to the nearest whole number. If filtration of the one gallon is not completed within 30 minutes, the test will be stopped and the volume of fuel remaining to be filtered will be measured. In these cases, results will be reported as 30+min/Volume of fuel remaining.

h. Report the vacuum in inches of mercury as determined from the average of the two readings taken in Paragraph 6f.

i. After recording the filtration time, shut off vacuum and rinse the sample container with two 50 ml portions of filtered petroleum ether and dispense into filtration funnel. Allow the 100 ml of petroleum ether to soak the filter for approximately 30 seconds. Turn vacuum on and filter the 100 ml of rinse. Turn vacuum off and rinse the inside of the funnel with approximately 25 ml of petroleum ether. Turn vacuum on. Carefully remove the top funnel and rinse the periphery of the membrane filter by directing a gentle stream of petroleum ether from the edge to the center, taking care not to wash contaminants off the filter. Maintain vacuum after final rinse for a few seconds to remove the excess petroleum ether from the filter.

j. Using forceps, carefully remove the membrane filter from the filter base and place in a clean Petri dish. Dry in an oven at 90°C for 30 minutes with the cover on the Petri dish slightly ajar. Place dish in a desiccator and allow to cool for 15 minutes. Reweigh the filter.

k. Report the total solids content in mg/liter by using the following formula:

$$\frac{\text{Weight gain of filter in mgs}}{3.785} = \text{mg/liter}$$

l. Should the sample exceed the 30 minutes filtration time and a portion of the fuel is not filtered, the solids content in mg/liter will be figured as follows: Determine the volume of fuel filtered by subtracting the ml of fuel remaining from 3785.

$$\frac{\text{Weight gain of filter in mgs}}{\text{ml of fuel filtered X 0.001}} = \text{mg/liter}$$

7. Test Limits:

a. Filtration Time:

(1) Maximum allowable filtration time is 15 minutes.

(2) Average vacuum of the two readings must not exceed 28 inches of mercury.

b. Total Solids - As required by specification or applicable field guidance documents.

NOTES: 1. If it is desired to determine the filtration time and not the total solids content, perform the test by omitting steps 72, 7b, 7c 7d.

2. If it is desired to determine the total solids content and not the filtration time, use of the stainless steel insert ring may be omitted.

3. If total solids is determined and not filtration time, it is permissible, but not required, to use a control filter for a specific analyses or a series of analyses. When this is accomplished the procedures specified in A.2 of ASTM Method D 2276 applies.