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FINAL REPORT ON FULLY FUELED TACOM VEHICLE STORAGE TEST PROGRAM

INTERIM REPORT
AFLRL No. 154

By

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Under Contract to

U.S. Army Mobility Equipment Research
and Development Command
Energy and Water Resources Laboratory
Fort Belvoir, Virginia

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This report discusses the results of a program to monitor the quality of fuel stored at ambient conditions in newly built M60-A3 battle tanks. The vehicles were in storage for up to two years. The fuel tanks in the vehicles were filled with diesel fuel to reduce fuel system deterioration and to allow the vehicles to be exercised at regular intervals. The fuel was treated with a stabilizer-additive package to reduce fuel degradation, and with a biocide to

20. ABSTRACT (Cont'd)

prevent any microbiological growth in the fuel system. Selected vehicles were sampled at 3-month intervals, and the fuel samples were analyzed at AFLRL. Test results showed that the fuel remained clean and stable throughout the storage period. No fuel-related problems were encountered with operation of the vehicles either at TACOM or at the user's location upon receipt.

FOREWORD

The work presented herein was conducted at the U.S. Army Fuels and Lubricants Research Laboratory (USAFLRL), Southwest Research Institute, San Antonio, TX, under Contracts DAAK70-80-C-0001 and DAAK70-82-C-0001. Work was funded by the U.S. Army Tank Automotive Command DRSTA-GSP/DRCPM-M60-TDT and U.S. Army Mobility Equipment Research and Development Command (USAMERADCOM), Ft. Belvoir, VA, with Mr. F.W. Schaekel (DRDME-GL) serving as contract monitor. Project technical monitor was Dr. J.V. Mengenhauser, USAMERADCOM (DRDME-GL).

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TABLE OF CONTENTS

LIST OF TABLES

LIST OF ILLUSTRATIONS

I. INTRODUCTION

Over a period of approximately 24 months, beginning in early 1979, 450 to 470 newly built M60-A3 battletanks were placed in storage at the Chrysler tank plant in Warren, MI. The vehicles were being stored pending receipt of vital components that were on backorder from the manufacturer. They were kept outside as pictured in Figure 1; and were stored in a fully fueled configuration in an attempt to reduce fuel system deterioration (rusting, corrosion, etc.) associated with prolonged exposure to the elements. Keeping fuel in the vehicles also allowed them to be exercised at regular intervals. However, fuel that is in prolonged storage, especially storage in vehicle fuel tanks, has been known to deteriorate and to possibly lead to fuel filter plugging or even fuel system damage. If water bottoms accumulate in the fuel tank, there is also the possibility of microbiological growth at the fuel/water interface. Several examples of such fuel deterioration and microbiological growth were cited in a recent report.(1)* The fuel in the vehicles in this program was treated with an additive package to prevent fuel deterioration and microbiological growth. This report summarizes the laboratory and field experience gained with fully fueled vehicles stored at the U.S. Army Tank Automotive Command (TACOM).

II. EXPERIMENTAL PROCEDURE

The stabilizer additive package consisted of fuel additive FOA-15,+ which functions as a dispersant, antioxidant, metal deactivator, rust inhibitor, and color stabilizer; and a biocide, BIOBOR-JF.** More detailed information about the additive package may be found in Appendix A. A purchase description (PD ME-103) was prepared in December 1980 to allow procurement of the additive package. A Department of Defense specification is being coordinated which, when issued, will replace PD ME-103. The FOA-15 was added at a concentration of 25 pounds per 1000 barrels (71.3 mg/liter), and the BIOBOR-JF was added at 270 ppm by volume (22.7 mg/liter). The same additive package was also used

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* Underscored numbers in parentheses refer to the list of references at the end of this report.

\+ Registered trademark of E.I. du Pont de Nemours Inc.

**Registered trademark of United States Borax and Chemical Corporation.

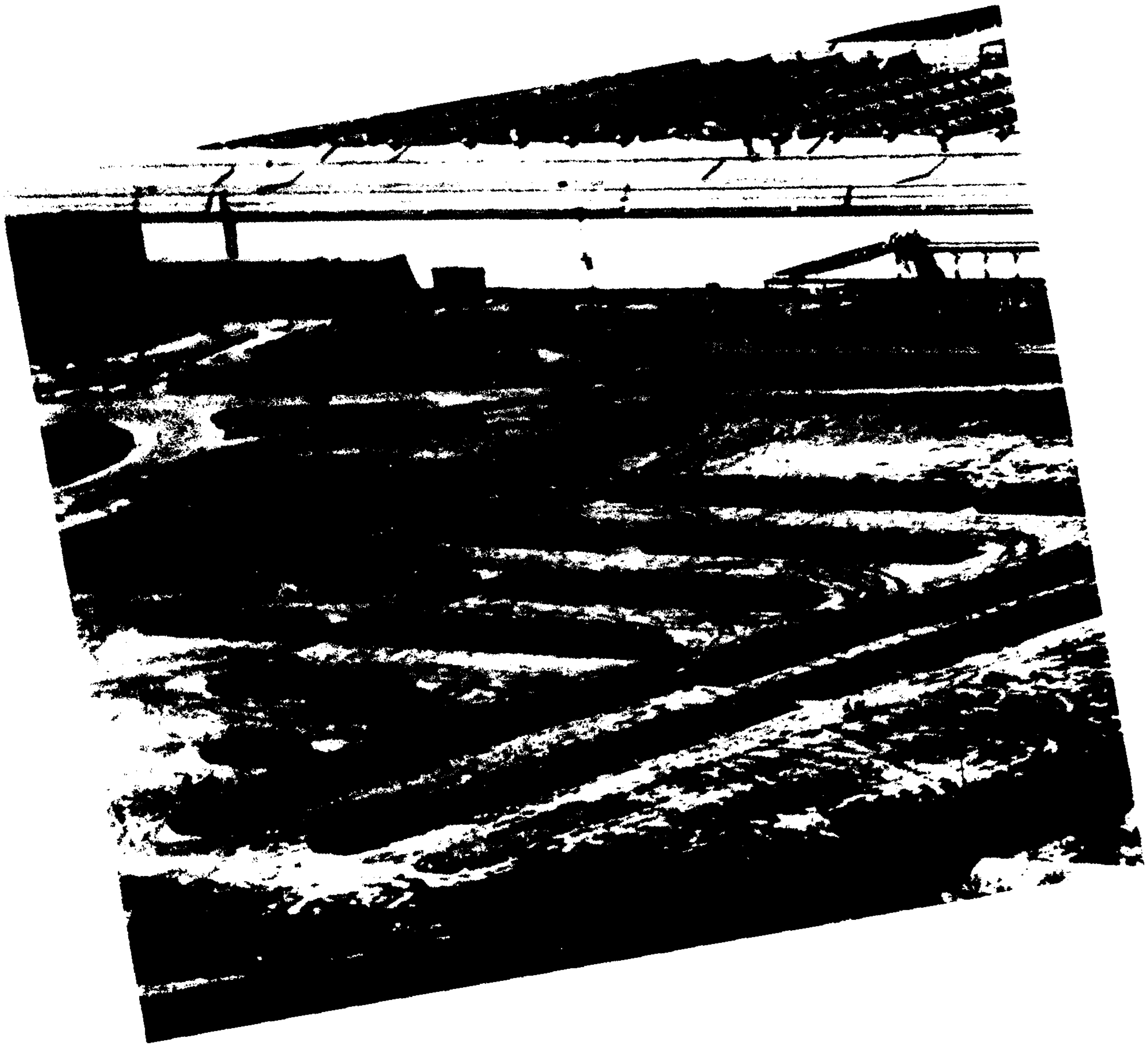

FIGURE 1. FULLY FUELED M-60 TANKS IN STORAGE
AT CHRYSLER TANK PLANT IN WARREN, MI

successfully in a two-year storage program involving POMCUS (Prepositioned Overseas Material Configured to Unit Sets) vehicles.(2)

Although all vehicles contained additive-treated fuel, only 21 vehicles were selected as test vehicles. Table 1 lists the vehicles used during the program by the vehicle identification number, the storage date of each vehicle, and the amount of time in storage for each vehicle. The J/N number of each vehicle is used as the vehicle code throughout this report to simplify discussions. Although the test program began with 10 test vehicles, it was expanded to 20 test vehicles at the request of U.S. Army Fuels and Lubricants Research Laboratory (AFLRL) to give a more representative sampling of the 450 to 470 vehicles in storage. Table 2 summarizes the sampling of each vehicle.

TABLE 1. VEHICLES USED IN TACOM STORAGE PROGRAM

Vehicle Identification			*Time in
J/N Number	S/N Number	Storage Date	Storage, Months
A3-20	0316	28 August 1979	24
A3-17	0313	31 August 1979	24
A3-26	0322	4 September 1979	23
A3-111	0407	18 September 1979	23
A3-177	0473	9 November 1979	20
T-72	0907	2 January 1980	17
A3-393	0689	18 January 1980	17
T-85	0920	14 February 1980	16
T-158	0993	22 March 1980	15
T-137	0972	24 March 1980	15
A3-90	0386	18 September 1979	23
A3-105	0401	18 September 1979	23
A3-132	0428	22 September 1979	23
A3-57	0353	24 September 1979	23
A3-144	0440	25 September 1979	23
A3-151	0447	25 September 1979	23
T-76	0911	2 January 1980	17
T-77	0912	2 January 1980	17
T-78	0913	3 January 1980	17
T-64	1149	2 July 1980	13
T-41	1126	8 July 1980	13

*Approximate time elapsed from the date the vehicle was placed into storage until the final fuel samples were taken.

TABLE 2. SUMMARY OF SAMPLING OF TACOM VEHICLES

Vehicle Code	Sample Set 1	Sample Set 2	Sample Set 3	Sample Set 4	Sample Set 5	Sample Set 6
A3-20	X	--	--	--	--	--
A3-17	X	X	X	X	X	X
A3-26	X	X	X	X	X	X
A3-111	X	X	X	X	X	X
A3-177	X	X	X	X	X	X
T-72	X	X	X	X	X	X
A3-393	X	X	X	X	X	X
T-85	X	X	X	X	X	X
T-158	X	X	X	X	X	X
T-137	X	X	X	X	X	X
A3-90	--	--	--	--	X	--
A3-105	--	--	--	--	X	X
A3-132	--	--	--	--	X	X
A3-57	--	--	--	--	X	X
A3-144	--	--	--	--	X	X
A3-151	--	--	--	--	X	X
T-76	--	--	--	--	X	X
T-77	--	--	--	--	X	X
T-78	--	--	--	--	X	X
T-64	--	--	--	--	X	X
T-41	--	--	--	--	X	X
Date Sample Taken	3-25-80	7-3-80	10-6-80	1-20-81	4-22-81	8-19-81

X = sampled
-- = not sampled

All the vehicles in storage were newly built M60-A3 battletanks equipped with new aluminum fuel tanks. The fuel tanks were cleaned by the manufacturer and shipped to the tank plant with all openings sealed to prevent contamination from foreign debris. The volume of the fuel tank in each vehicle is 385 gallons, each vehicle was fueled with approximately 375 gallons of additive-treated fuel.

The vehicles were exercised every 120 days, and fuel samples were taken every 3 months. The fuel samples were returned to AFLRL and evaluated using the test methods listed in Table 3. Only Methods D 130, D 2274, and D 2276 are required by the federal specification for diesel fuel, VV-F-800.

TABLE 3. TEST METHODS USED TO EVALUATE FUEL SAMPLES

• ASTM D 130	Test for Detection of Copper Corrosion From Petroleum Products by the Copper Strip Tarnish Test.
• ASTM D 664	Test for Neutralization Number by Potentiometric Titration.
• ASTM D 381	Test for Existent Gum in Fuels by Jet Evaporation
• ASTM D 873 (Modified)	Test for Oxidation Stability of Aviation Fuels (Potential Residue Method)
• ASTM D 1500	Test for ASTM Color of Petroleum Products (ASTM Color Scale)
• ASTM D 2274	Test for Oxidation Stability of Distillate Fuel Oil (Accelerated Method)
• ASTM D 2276 (Modified)	Test for Particulate Contaminant in Aviation Turbine Fuels
• NACE TM-01-72	Antirust Properties of Petroleum Products Pipeline Cargoes
• Appendix B	Proposed Test for Distillate Fuel Storage Stability at 43°C
• Appendix B	Test Method for High-Temperature Stability of Distillate Fuels
• Appendix C	Test for Growth Potential of Microorganisms

III. DISCUSSION

Table 4 is a compilation of data for the base (bulk) fuel and the base fuel plus additives. As can be seen from the data in Table 4, the fuel is within Federal Specifications VV-F-800B for each parameter. Addition of the additive package to the base fuel lowered the amount of total insolubles in the ASTM Method D 2274 Accelerated Stability Test from 1.2 to 0.4 mg/100 ml. However, the additive package did increase the amount of soluble gum (ASTM Method D 381, Steam Jet Gum) present in the fuel from 8.0 to 10.2 mg/100 ml. This

TABLE 4. DATA FOR THE BASE FUEL, NEAT AND ADDITIVE-TREATED

AFLRL Code No.	9174 (neat)	9174 (Additive-treated)	VV-F-800B Specification
Sampling Date	2-11-80	--	--
Fuel Type	DF-2	DF-2	DF-2
Time in Storage Prior to Sampling	2 months	--	--
Particulates, D 2276, (Mod.), 1.2 μm, mg/1000 ml	1.3	1.1	8.0*
Color, D 1500	1.0	1.0	--
Accelerated Stability, D 2274, Total Insolubles, mg/100 ml	1.2	0.4	1.5
Steam Jet Gum, D 381, mg/100 ml	8.0	10.2	--
Total Acid Number, D 664, mg KOH/g sample	0.20	--	--
Light Absorbance @			
650 nm	0.009	--	--
575 nm	0.024	--	--
540 nm	0.042	--	--
500 nm	0.081	--	--
NACE, TM-01-72, mild steel corrosion	B+	A	--
Copper Strip Corrosion, D 130	1a	1a	3
150°C Accelerated Stability			
Filterable Insolubles, 1.2 μm, mg/100 ml (1.5/3.0 hr)	3.6/5.5	2.5/5.5	--
Adherent Insolubles, mg/100 ml (1.5/3.0 hr)	0.7/1.1	0.8/0.9	--
Total Insolubles, mg/100 ml (1.5/3.0 hr)	4.3/6.6	3.2/6.4	--
Soluble Gum, mg/100 ml (1.5/3.0 hr)	7.3/8.4	7.6/8.5	--
Total Potential Residue, D 873, mg/100 ml	19.4	19.9	--

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*"C" revision of VV-F-800 allows 10 mg/liter for 0.8 μm filter.

increase in gum is probably due to the polymers present in the additives and has been previously observed in similar tests. These data indicate the additive package had little or no effect on either the amount of material formed in the 150°C accelerated stability test (as outlined in Appendix B) or the total potential residue by ASTM Method D 873. However, the data from these last two procedures (150°C and D 873) are not as widely accepted as is data from D 2274 as indicators of fuel stability.

The base fuel was also tested for stability using the 43°C storage test outlined in Appendix B. Samples of the fuel, both treated and untreated, were stored in vented, borosilicate glass bottles for 4, 8, and 12 weeks. It is generally accepted that storage at 43°C for 13 weeks is approximately equivalent to storage at ambient temperatures for 12 months.(3) The results of this test are given in Table 5. Examination of the data in Table 5, as well as the data in Table 4, shows that the base fuel was very stable in storage.

TABLE 5. DATA FOR STORAGE OF BASE FUEL (AL-9174-F) AT 43°C

	Storage Time					
	4 Weeks		8 Weeks		12 Weeks	
Parameter	Untreated	Treated	Untreated	Treated	Untreated	Treated
Filterable Insolubles, mg/100 ml	0.1	0.1	0.1	0.1	0.1	0.2
Adherent Insolubles, mg/100 ml	0.2	0.1	0.1	0.2	0.1	0.1
Total Insolubles, mg/100 ml	0.3	0.2	0.2	0.3	0.2	0.3
Steam Jet Gum, D 381, mg/100 ml	11.8	11.6	12.0	12.1	14.6	14.9
Total Gum, mg/100 ml	12.1	11.8	12.2	12.4	14.8	15.2

Table 6 is a summary of the D 2274 accelerated stability test results for the six sets of samples taken from the test vehicles at TACOM. The results are well within the federal specification limit of 1.5 mg/100 ml, with the exception of the sample set 6 fuel sample from Vehicle T-64. It is believed that this unusually high value of 21.1 mg/100 ml indicates contamination of the sample by an external source. Overall, the very low values of the fuel samples indicate that the fuel remained stable throughout the time in storage.

TABLE 6. SUMMARY OF ASTM METHOD D 2274
ACCELERATED STABILITY TEST VALUES FOR TACOM FUEL SAMPLES

Vehicle Code	D 2274, Total Insolubles, mg/100 ml					
	Sample Set 1	Sample Set 2	Sample Set 3	Sample Set 4	Sample Set 5	Sample Set 6
A3-20	0.3	*	*	*	*	*
A3-17	0.3	0.3	0.3	0.2	0.5	0.2
A3-26	0.3	0.3	0.1	0.3	0.4	0.3
A3-111	0.3	0.4	0.3	0.1	0.2	1.0
A3-177	0.4	0.3	0.3	0.2	0.5	0.1
T-72	0.9	0.7	0.4	0.2	0.3	0.2
A3-393	0.9	0.3	1.2	0.2	0.2	0.1
T-85	0.3	0.2	0.3	0.1	0.3	0.3
T-158	0.2	0.3	0.1	0.1	0.2	0.2
T-137	0.2	0.3	1.1	0.2	0.3	0.2
A3-90	*	*	*	*	0.5	*
A3-105	*	*	*	*	0.3	0.1
A3-132	*	*	*	*	0.4	0.8
A3-57	*	*	*	*	0.4	0.1
A3-144	*	*	*	*	0.3	0.1
A3-151	*	*	*	*	0.3	0.2
T-76	*	*	*	*	0.4	0.2
T-77	*	*	*	*	0.2	0.4
T-78	*	*	*	*	0.3	0.3
T-64	*	*	*	*	1.0	21.1**
T-41	*	*	*	*	0.9	1.3
Mean	0.4	0.3	0.5	0.2	0.4	0.4+
Std Dev.	±0.3	±0.1	±0.4	±0.1	±0.2	±0.3

* = Vehicle not sampled.
** = A duplicate check on the same sample confirmed this result.
\+ = Omitted vehicle T-64 for inconsistency.

Table 7 is a summary of the D 2276 particulates data for the six sample sets. During the course of the testing, revision "C" of VV-F-800 was published. However, since the program was started under VV-F-800B, which requires the use of D 2276 modified to use a 1.2-micrometer pore size size filter, this procedure was continued until completion of the testing. The high value of 8.5 mg/100 ml from Vehicle A3-26 in sample set 4 is probably due to contamination of the fuel sample from external sources. Later samples from the same vehicle gave much lower particulate levels. Overall, however, the fuel in the vehicles remained very clean.

TABLE 7. SUMMARY OF PARTICULATES FOR TACOM FUEL SAMPLES

	Particulates, D 2276 (Mod.) 1.2 µm, mg/1000 ml					
Vehicle Code	Sample Set 1	Sample Set 2	Sample Set 3	Sample Set 4	Sample Set 5	Sample Set 6
A3-20	1.4	*	*	*	*	*
A3-17	1.4	0.6	0.9	5.8	0.4	0.3
A3-26	2.2	1.5	3.1	8.5	0.2	0.9
A3-111	1.8	1.9	3.2	2.1	0.7	0.3
A3-177	2.7	1.7	1.0	0.4	0.9	0.4
T-72	2.1	5.4	5.1	0.9	0.3	0.3
A3-393	2.3	1.6	3.5	5.1	1.2	0.3
T-85	3.9	1.0	4.3	0.6	0.2	0.5
T-158	0.8	1.4	0.8	0.1	0.7	0.8
T-137	0.8	1.1	2.5	2.7	1.5	0.8
A3-90	*	*	*	*	3.0	*
A3-105	*	*	*	*	0.2	0.4
A3-132	*	*	*	*	0.7	0.8
A3-57	*	*	*	*	0.6	0.2
A3-144	*	*	*	*	0.8	0.4
A3-151	*	*	*	*	0.9	0.6
T-76	*	*	*	*	0.3	0.4
T-77	*	*	*	*	0.5	0.4
T-78	*	*	*	*	1.1	0.3
T-64	*	*	*	*	1.3	3.6
T-41	*	*	*	*	0.5	1.0
Mean	1.9	1.8	2.7	2.9	0.8	0.7
Std Dev.	±0.9	±1.4	±1.5	±2.9	±0.6	±0.7

---

* = Vehicle not sampled.

Table 8 summarizes the steam jet gum results for the six sample sets. In most cases, the amount of soluble gum (steam jet gum) continues to increase with each set of samples. This may be of importance since soluble gum may be a precursor to insolubles formation during extended storage. There was not,

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TABLE 8. SUMMARY OF STEAM JET GUM DATA FOR TACOM FUEL SAMPLES

	ASTM Method D 381, Steam Jet Gum, mg/100 ml					
Vehicle Code	Sample Set 1	Sample Set 2	Sample Set 3	Sample Set 4	Sample Set 5	Sample Set 6
A3-20	6.2	*	*	*	*	*
A3-17	6.6	17.5	22.1	15.3	22.6	9.9
A3-26	8.2	23.1	16.2	28.4	31.6	12.3
A3-111	16.9	24.4	21.5	23.2	28.5	18.9
A3-177	19.2	42.2	25.3	29.1	40.0	16.2
T-72	8.4	12.8	18.9	15.2	19.0	16.2
A3-393	14.3	19.0	16.3	20.1	23.0	11.9
T-85	5.4	5.5	9.0	5.4	8.1	6.7
T-158	4.8	8.9	8.0	20.0	12.1	15.0
T-137	5.0	9.1	6.0	6.7	8.4	15.0
A3-90	*	*	*	*	25.0	*
A3-105	*	*	*	*	12.6	9.1
A3-132	*	*	*	*	44.2	19.7
A3-57	*	*	*	*	26.0	11.9
A3-144	*	*	*	*	51.0	19.4
A3-151	*	*	*	*	37.7	21.9
T-76	*	*	*	*	15.4	14.1
T-77	*	*	*	*	21.8	15.3
T-78	*	*	*	*	11.8	10.4
T-64	*	*	*	*	24.4	29.9
T-41	*	*	*	*	68.0	26.3
Mean	9.5	18.1	15.9	18.2	26.6	15.8
Std Dev.	±5.3	±11.2	± 6.9	± 8.4	±15.4	± 5.9

* = Vehicle not sampled.

---

however, a significant increase in insolubles throughout the duration of the test.

Table 9 is a summary of the total insolubles data for 1.5 and 3 hours at 150°C for the six sample sets. According to previously published data,(4) a fuel

TABLE 9. SUMMARY OF 150°C ACCELERATED STABILITY TEST VALUES FOR TACOM FUEL SAMPLES

	150°C Test, Total Insolubles, 1.5 Hr/3.0 Hr, mg/100 ml					
Vehicle Code	Sample Set 1	Sample Set 2	Sample Set 3	Sample Set 4	Sample Set 5	Sample Set 6
A3-20	2.7/5.2	*	*	*	*	*
A3-17	2.3/8.1	1.5/7.3	2.7/18.0	2.2/17.1	1.4/3.0	1.0/7.1
A3-26	0.6/3.1	1.5/5.0	0.2/2.5	2.9/6.2	1.1/3.7	0.3/5.9
A3-111	3.7/5.9	2.4/14.3	0.2/0.9	2.0/5.1	1.5/3.1	1.2/1.3
A3-177	0.3/3.5	0.1/6.4	0.9/2.0	1.4/3.7	1.7/1.6	0.3/4.7
T-72	3.0/10.6	2.2/3.8	2.2/14.7	1.8/5.8	2.7/5.5	0.6/6.9
A3-393	0.4/0.3	0.8/7.9	0.8/2.0	1.7/4.5	1.9/2.9	2.7/7.6
T-85	0.7/1.7	0.2/1.8	3.5/9.5	1.1/5.9	2.3/5.1	0.6/6.8
T-158	0.1/0.3	3.2/8.7	3.5/0.5	6.2/13.2	4.7/6.3	8.7/10.1
T-137	0.2/0.1	3.2/9.0	2.3/0.5	5.2/6.9	5.1/7.7	1.5/9.3
A3-90	*	*	*	*	2.3/2.1	*
A3-105	*	*	*	*	2.5/4.1	1.5/6.2
A3-132	*	*	*	*	1.0/2.9	0.1/5.1
A3-57	*	*	*	*	2.3/4.8	0.5/5.9
A3-144	*	*	*	*	0.1/2.0	4.3/3.8
A3-151	*	*	*	*	2.1/2.1	4.6/6.5
T-76	*	*	*	*	1.8/6.1	0.5/5.3
T-77	*	*	*	*	2.3/5.1	0.6/1.4
T-78	*	*	*	*	3.6/5.9	0.7/6.5
T-64	*	*	*	*	0.1/5.4	0.7/0.3
T-41	*	*	*	*	0.1/0.3	0.7/8.9

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* = Vehicle not sampled.

could, in most cases, be considered good if the total insolubles for the 1.5-hour 150°C test are less than 4 mg/100 ml. Total insolubles between 4 and 6 mg/100 ml indicate a fuel of marginal stability and greater than 6 mg/100 ml indicate a fuel of poor stability. Judging from this information, the majority of the samples would be rated as having good stability throughout the storage period.

Each fuel sample was also tested for water and sediment (ASTM Method D 1796). All the samples in the first five sample sets showed a trace of sediment and no water. When sample set six was evaluated, the majority of samples showed approximately 0.01 percent water, but still only a trace of sediment. This does not, however, indicate the amount of water that might possibly be present as water bottoms in the fuel tank.

Table 10 lists the laboratory data for the final sample set as an indication of the quality of the fuel. Again, note the high value of total insolubles (ASTM Method D 2274) for the fuel in Vehicle T-64. The unusually high value of 21.1 mg/100 ml probably indicates contamination of the sample source by external sources. Examination of the other data in Table 10 indicates that the fuel quality was still good after as long as two years in storage.

As a measure of the effectiveness of the biocide, six of the fuel samples were selected at random from the final sample set and were tested with a commercially available (Boron Oil Company) test kit for the growth potential of microorganisms. Appendix C gives the test procedure for this test kit. Samples from a control fuel sample at AFLRL with a water bottom were tested as control samples. This fuel sample had been previously innoculated with a culture of Cladosporium resinae and was known to contain living organisms. Aliquots were taken from the fuel layer and the water layer of the control sample. The control sample was then shaken and allowed to stand for 24 hours and another aliquot was taken from the fuel layer. This last step was performed as a precaution in case there were only a small amount of viable organisms in the fuel layer and the sensitivity of the test kit was insufficient to register their presence. Table 11 gives the results of the microorganism testing. The tests showed essentially no viable organisms present in the fuel

TABLE 10. LABORATORY DATA FOR SIXTH SET OF FUEL SAMPLES FROM FULLY FUELED M60 TANKS IN WARREN, MI

J/N Number	T-85	A3-111	A3-17	T-72	A3-393	A3-177	A3-26	T-137	T-158	T-77
Accelerated Stability, D 2274, Total Insolubles, mg/100 ml	0.3	1.0	0.2	0.2	0.1	0.1	0.3	0.2	0.2	0.4
Particulates, D 2276, 1.2 μm, mg/1000 ml	0.5	0.3	0.3	0.3	0.5	0.4	0.9	0.8	0.8	0.4
Steam Jet Gum, D 381, mg/100 ml	6.7	18.9	9.9	16.2	11.9	16.2	12.3	15.0	15.0	15.3
Color, D 1500	0.5	3.5	2.5	0.5	2.0	2.5	2.5	0.5	0.5	1.5
Visual Inspection	C&B*	C&B	C&B	C&B	C&B	C&B	C&B	C&B	C&B	C&B
150°C Accelerated Stability Test, Total Insolubles, mg/100 ml										
1.5 hr	0.6	1.2	1.0	0.6	2.7	0.3	0.3	1.5	8.7	0.6
3.0 hr	6.8	1.3	7.1	6.9	7.6	4.7	5.9	9.3	10.1	1.4
Sediment	Trace	Trace	Trace	Trace	Trace	Trace	Trace	Trace	Trace	Trace
Vol% Water	0.0	0.01	0.01	0.01	0.02	0.01	0.01	0.0	0.0	0.0

---

* Clear and bright

TABLE 10. LABORATORY DATA FOR SIXTH SET OF FUEL SAMPLES FROM FULLY FUELED M60 TANKS IN WARREN, MI (Cont'd)

J/N Number	A3-57	A3-105	A3-132	A3-144	A3-151	T-41	T-64	T-76	T-78
Accelerated Stability, D 2274, Total Insolubles, mg/100 ml	0.1	0.1	0.8	0.1	0.2	1.3	21.1+	0.2	0.3
Particulates, D 2276, 1.2 μm, mg/1000 ml	0.2	0.4	0.8	0.4	0.6	1.0	3.6	0.4	0.3
Steam Jet Gum, D 381, mg/100 ml	11.9	9.1	19.7	19.4	21.9	26.3	29.9	14.1	10.4
Color, D 1500	2.5	2.0	3.0	3.0	3.0	3.0	3.5	1.0	1.0
Visual Inspection	C&B*	C&B	C&B	C&B	C&B	C&B	C&B	C&B	C&B
150°C Accelerated Stability Test, Total Insolubles, mg/100 ml									
1.5 hr	0.5	1.5	0.1	4.3	4.6	0.7	0.7	0.5	0.7
3.0 hr	5.9	6.2	5.1	3.8	6.5	8.9	0.3	5.3	6.5
Sediment	Trace	Trace	Trace	Trace	Trace	Trace	Trace	Trace	Trace
Vol% Water	0.01	0.01	0.02	0.01	0.02	0.0	0.01	0.01	0.0

---

* C&B = Clear and bright.

\+ A duplicate test on the same sample confirmed the high value.

TABLE 11. RESULTS OF TEST FOR THE GROWTH POTENTIAL OF MICROORGANISMS (APPENDIX C)

Sample I.D.	Test Result (Two Week)
T-85	No visible signs of growth
A3-111	No visible signs of growth
T-72	No visible signs of growth
A3-177	No visible signs of growth
T-137	No visible signs of growth
T-64	No visible signs of growth
Control Sample*	
Water Layer	Large mat of growth at interface, dark pink color
Fuel Layer	Some growth at interface, water turbid, light pink color
Fuel Layer**	Some growth at interface, water turbid, light pink color

---

* Containing active growth of Cladosporium resinae

** Sample was shaken and allowed to stand for 24 hours prior to obtaining aliquot.

samples. All of the control samples gave good growth with the most growth from the water layer aliquot as would be expected.

In a subsequent letter (5), the Project Manager, M60 Tanks, reported no fuel-related problems during removal of the tanks from storage. Additionally, there were no problem reports from any user upon receipt of the vehicles.

IV. CONCLUSIONS AND RECOMMENDATIONS

- Under the conditions of the TACOM storage, it was shown to be feasible to store vehicles fully fueled for periods of at least 24 months. These conditions included a stable base fuel, clean fuel system, and the use of fuel stabilizer additives.

- There were no fuel-related problems during removal of the vehicles from storage.

- There were no problem reports from any user upon receipt of the vehicles.

- A test for the presence of viable microorganisms (having growth potential) in the final sample set was negative.

- The fuel in the test vehicles remained in good quality throughout the test, as indicated by several test methods.

- Phased implementation of fully fueled diesel vehicle storage is recommended, provided (1) proper use of additives, (2) fuel quality is initially established, (3) subsequent "spot" sampling of stored vehicles for fuel quality monitoring is conducted, and (4) sufficient justification for this type of storage rather than "Level A" preservation is established.

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3. "Navy-CRC Barge Storage Program," Coordinating Research Council (CRC) Report, New York, July 1957.

4. Stavinoha, L.L. and Westbrook, S.R., "Optimization of Accelerated Stability Test Techniques for Diesel Fuels," Department of Energy Report No. DOE/BC/10043-25, prepared by Southwest Research Institute, June 1981.

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VI. BIBLIOGRAPHY

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2. Letter from AFLRL to TACOM (DRSTA-GSP) dated 13 December 1979 with the subject "Monitoring of Additive Treated Fuel in Fully Fueled M60 Vehicles in Storage."

3. Trip report of S.R. Westbrook for the period 21-22 April 1980.

4. Letter from AFLRL to MERADCOM (DRDME-GL) dated 10 July 1980 with the subject "Storage of Fully Fueled M60 Battle Tanks at TACO, in Warren, MI."

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8. Letter from AFLRL to MERADCOM (DRDME-GL) dated 27 July 1981 with the subject "Storage of Fully Fueled M60 Battle Tanks at TACOM in Warren, MI; Laboratory Data for the Fifth Set of Fuel Samples."

9. Letter from TACOM (DRSTA-GSP) to AFLRL dated 13 May 1981 with the subject "Status on Fuel Sampling."

APPENDIX A

INFORMATION PAPER ON DIESEL FUEL STABILIZER ADDITIVE PACKAGE

INFORMATION PAPER ON DIESEL FUEL STABILIZER ADDITIVE PACKAGE

1. Background

The concept of a stabilizer additive for diesel fuel originated in 1973 as a result of fuel-related problems which were occuring in Army depot operations. During re-build operations and in temporary storage, vehicles were experiencing severe fuel system malfunctioning (i.e., excessive plugging of fuel filters) and fuel cell and filler pipe corrosion. Analyses of fuels obtained from vehicle fuel cells and fuel filter reservoirs revealed the samples were oxidized and corrosive to ferrous materials. Based upon these analyses and a background knowledge of mobility fuel requirements and the fuel deterioration processes, a need surfaced for a multi-functional additive. The additive requirements were to provide an antioxidant/stabilizer, metal deactivator, dispersant, corrosion inhibitor, and a biocide. The purpose of each function is summarized as follows:

Antioxidant--This retards the tendency for autooxidation to occur. Diesel fuels will normally undergo autooxidation and deterioration which causes formation of particulates, gums, and acid complexes.

Metal deactivator--This removes free copper and other trace metal from reacting in the autooxidation process and allows the antioxidant to function effectively. Without the metal deactivator, the effectiveness of the antioxidant would be reduced significantly.

Dispersant--This causes any sediment or agglomerates present to be maintained in a finely dispersed state. It prevents the formation of large particulates and reduces the tendencies for deposits to occur on critical fuel system components.

Corrosion inhibitor--This reduces the tendency for corrosion of ferrous or metallic surfaces by forming a protective barrier over the surface. It is designed to protect fuel-wetted surfaces from corrosion, but will not function in environments of heavy water contamination (i.e., water bottoms or sump regions).

Biocide--This functions to kill any microorganisms that originate at the fuel-water interface in fuel storage/tank environments.

2. Stabilizer Additive Characteristics

Additive suppliers were solicited to provide a material that met the requirements outlined above. The multi-functional approach was requested to facilitate use of the additive when fielded. Several companies responded to the request and provided materials which fulfilled four of the five requirements. The biocide development was subsequently developed through a joint effort with the U.S. Army Natick Research and Development Command (NARADCOM) who have the lead responsibility for microbiological deterioration RDTE. The stabilizer additive subsequently selected was based upon its effectiveness in a wide range of diesel/distillate fuels tested.

The stabilizer additive is a two-component package which provides the five functions specified above. One component, identified as FOA-15, which is manufactured by E.I. du Pont and Company, provides four of the five requirements; namely, antioxidant, metal deactivator, dispersant, and corrosion inhibitor. The other component, identified as BIOBOR JF, which is manufactured by U.S. Borax and Chemical Corporation, provides the biocide.

FOA-15 is an ashless organic complex composed of a mixture of amines, polymers, alkyl ammonium alkyl phosphate, and azo dye. It has an amine odor and its characteristics are as follows:

Density, g/ml	0.9
Flash point, °F	140
Fire point, °F	145
Pour point, °F	-50
Neutralization No.,	
TAN	25
TBN	130

Viscosity, cSt, @	
32°F	270
100°F	64
210°F	18
Ash, wt%	0.000

FOA-15 is completely miscible with diesel and distillate fuels and may be added directly to fuels or in a prediluted form.

BIOBOR JF is an organo-boron complex of which 95 percent is composed of 2,2'-oxybis (4,4,6-trimethyl-1,3,2-dioxaborinane) and 2,2'-(1-methyltrimethylene dioxy) bis-(4-methyl-1,2,2-dioxaborinane). The remaining 5 percent is petroleum naphtha. BIOBOR JF has the following characteristics:

Density, g/ml	1.05
Flash point, °F	144
Pour point, °F	-27.5
Viscosity, cSt, @ 70°F	29
Boron content	7.4%
Water (free hydroxyl)	0.4%

BIOBOR JF is an EPA registered additive which is soluble in both fuel and water, partitioning between fuel and water bottoms. The active ingredients in BIOBOR JF will readily hydrolyze upon direct contact with water. BIOBOR JF is completely miscible with fuel oils and may be added directly to the fuel.

3. Stability Additive Development

In the initial solicitation to additive suppliers, information was requested on recommended treatment levels and supporting test data. To meet the requirements for insuring adequate storage stability of diesel fuel, it was recommended that the following treatment levels be adhered to:

FOA-15 - 25 pounds per 1000 bbls (71.3 mg/liter)
BIOBOR JF - 270 parts per million by volume (227 mg/liter)

The data supplied by the manufacturer of FOA-15 stated that this product has been a refinery processing additive for the past fifteen years, and as such has been used by many commercial diesel-powered fleets. The components of FOA-15 were reported to have been extensively fleet tested and there have been no detrimental effects ever evidenced. Laboratory tests were conducted to assess whether addition of FOA-15 to diesel fuel would (1) alter the limiting inspection requirements, and (2) create any adverse side effects (i.e., water emulsification, injector fouling, etc.) The tests revealed that FOA-15 added at the recommended treatment was satisfactory and did not alter any of the inspection property requirements.

With respect to the BIOBOR JF, considerable research was conducted by NARADCOM to define its effectiveness regarding microbiological activity. These tests and other laboratory performance tests including a 531-hour Detroit Diesel 6V-71N engine endurance test are documented in AFLRL Report No. 61, "Evaluation of Biocides' Effects on Diesel Fuel Under Prolonged Storage (AD No. A012779) which is available from the Defense Technical Information Center. The presence of Boron was cause for concern, and data were subsequently requested by the manufacturer. Recent correspondence from U.S. Borax has provided the following listing of major engine/airframe manufacturers which have approved the use of BIOBOR JF or have published service bulletins on its use:

Waukesha Engine
Cummins Engine Company
Detroit Diesel Allison
United Airlines
American Airlines
McDonnell Douglas
Rolls Royce
Pratt and Whitney

Laboratory tests were also conducted to confirm the absence of any adverse side effects. These tests as well as those mentioned previously revealed the treatment level of 270 parts per million (ppm) for BIOBOR JF to be satisfactory and not contributing to any changes in fuel property characteristics. The addition of the stabilizer additive formulation actually improves many of the fuel property characteristics. To illustrate this point, Table A-1 provides a tabulation of analyses data for an untreated versus a treated reference diesel fuel. As is noted, the addition of the stabilizer additive generally causes a decrease in Carbon Residue on 10% Bottoms, Accelerated Stability, and Particulate Contamination, all of which are reflective of enhanced storage stability and improved product quality.

4. Field Tests Involving the Stabilizer Additive

The stabilizer additive was initially tested in M109 and M110 self-propelled artillery vehicles at Yuma Proving Grounds. The test program was initiated in 1978 and terminated in mid-1979 because of non-availability of vehicles. This test involved operational vehicles which had been experiencing fuel filter malfunctioning because of the unusual operating requirements (i.e., long engine idle periods) which enhanced fuel deterioration. The stabilizer additive did reduce the occurrence of filter plugging and no problems were evidenced relative to its use during this one-year period.

The stabilizer additive was also tested at Miseau, Germany, in conjunction with the concept to store POMCUS equipment in a fully fueled condition. The test involved twenty-five vehicles which included M109, M578, M577, and M35 equipment. The test was initiated in 1978 and recently terminated after approximately a 2½ year storage interval. All vehicles operated satisfactorily and the presence of the additive did maintain the overall quality of the fuel during the storage period.

The additive is currently being tested in vehicles on board a USMC RDF float located at Diego Garcia. The vehicles under test include twenty vehicles; namely, M60 tanks and LVTP7 personnel carriers. In this test program, the stabilizer additive is being evaluated in both diesel fuel and JP-5 aviation turbine fuel. The vehicles, stored partially fueled, are to remain in this equipment float for a four-year period. This cooperative test program was initiated in June 1980.

The additive is being used to reduce fuel-related problems which have recently surfaced at Army facilities. These problems (i.e., excessive replacement of vehicle fuel filters, etc.) have occurred because of the quality of the procured diesel fuel (VV-F-800), the environmental use pattern (i.e., high ambient temperatures and excessive engine idle periods), and the slow turnover rate. At Ft. Irwin, CA, 200,000 gallons of fuel in their bulk underground fuel supply tanks were treated with the additive.

5. Cost and Intended Use

The total cost of the additive package for treatment of fuel is approximately 2 to 3¢ per gallon. The treatment rates are:

FOA-15 @ 25 pounds per 1000 barrels
BIOBOR JF @ 270 parts per million

The additive is designed to enhance product stability by preventing fuel deterioration, corrosion and microbiological growth. Although this additive has proven effective in reducing corrosion, fuel degradation, and microbiological growth with proper use, it should not be thought of as a substitute for proper fuel system cleaning and maintenance. In addition, this stabilizer additive will not rectify an already existing problem with unusable fuel; it is a preventive-type additive only.

U.S. Army Mobility Equipment
 Research and Development Command
DRDME-GL, M.E. LePera
Fort Belvoir, VA 22060

Prepared: 19 December 1980

TABLE A-1. EFFECTS OF STABILIZER ADDITIVE ON FUEL PROPERTIES

Fuel Property	Reference Diesel Fuel*	Reference Diesel Fuel Plus Additive Stabilizer	Test Methods
Distillation, °F:			D 86
Initial Boiling Point	400	403	
5% evap.	443	441	
10% evap.	462	461	
50% evap.	521	522	
70% evap.	550	550	
90% evap.	605	605	
End Point	671	672	
Cloud Point, °F	+30	+32	D 2500
Pour Point, °F	15	15	D 97
Existent Gum**, mg/100 mL	71	41	D 381
Carbon Residue on 10% Btms, wt%	0.17	0.13	D 524
Ash, wt%	<0.01	<0.01	D 482
Accelerated Stability, mg/100 ml	2.71	0.53	D 2274
Particulate Contamination,			
1.2 μm membrane, mg/L	9.2	3.12	D 2276
Filterability Ratio	1.1	0.50	--
Flash Point, °F	142	140	D 93
Corrosiveness, Copper Strip Rating	2B	2C	D 130
Jet Fuel Thermal Oxidation Test			D 3241
Heater Tube Temperature, °C	245	245	
ΔFilter Pressure, mm Hg	0	0	
Heater Tube Deposit:			
ASTM Code No.	2	1	
TDR Spun Rating	50	52	
TDR Spot Rating	42	43	

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*Cat 1-H reference diesel fuel conforming to specification in Federal Test Method Standard 791, Method 341.

**Air-Jet Evaporation @ 450°F Used

APPENDIX B

EXPERIMENTAL TEST PROCEDURES FOR DISTILLATE FUELS

APPENDIX B
DESCRIPTION OF TEST METHODS

Method 1. Storage Test at 43°C

Storage at 43°C was performed as per the following Draft ASTM (Tech Division E-V) method.

Proposed Test for Distillate Fuel Storage Stability at 43°C

1. Scope

1.1 This method covers the measurement of the inherent stability (as defined in paragraph 4.1 below) of a distillate fuel. The fuel is stored at a slightly elevated temperature over an extended period to give an indication of the fuel's potential long-term storage when stored at lower temperatures.

> Note 1: This method is intended for use with high flash point (>38.7°C) distillate-type fuels under ASTM specifications D 2880, D 396 and D 974 for turbine, burner and diesel fuels, respectively.

2. Applicable Documents

2.1 ASTM Standards:

D 270	Sampling Petroleum and Petroleum Products
D 2274	Test for Oxidation Stability of Distillate Fuel Oil (Accelerated Method)
D 381	Test for Existent Gum in Fuels by Jet Evaporation
D 1500	ASTM Color of Petroluem Products

3. Summary of Method

3.1 A measured volume of filtered fuel in several glass containers is stored at 43°C for up to 24 weeks. After aging for various time intervals, one set of samples is removed from storage, cooled to room temperature, and analyzed for particulates by filtration. Adherent gum on the wall of the aging container is also measured. Color and steam jet gum tests may also be performed on the fuel if desired.

4. Significance

4.1 Past research has indicated that storage at 43°C for 1 week is approximately equivalent to storage at average ambient (21°C) conditions for 4 weeks, assuming all other factors are equivalent. This test does not account for any environmental effects or container effects that may be encountered under a given field storage condition.

5. Apparatus

5.1 Sample containers are borosilicate glass bottles. The containers should preferably have a lid or cover with a Teflon insert and a hole for a borosilicate glass vent. The total capacity of the container is 500 ml.

5.2 Storage oven should be large enough to contain all sample bottles. It should be thermostatically controlled to maintain a temperature of 43° ±1°C. It should be as dark as possible to prevent degradation due to photolytic reactions and shall also be "explosion proof."

5.3 Drying oven, maintained at 99°±1°C.

> Warning! There are exposed hot surfaces on the drying oven. Avoid contact with exposed skin by use of protective equipment as required.

5.4 Gooch crucible, porcelain, No. 4.

5.5 Filter flask assembly, as shown in Figure B-1.

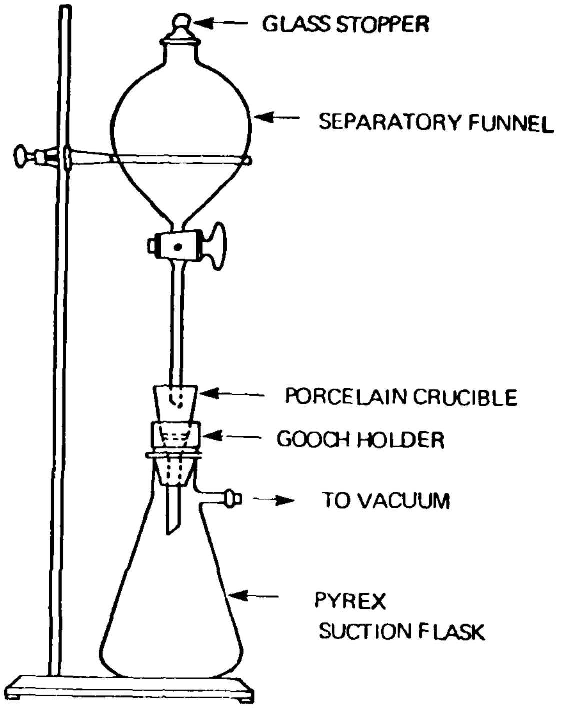

FIGURE B-1. SELF-FEEDING FILTERING ASSEMBLY

6. Reagents and Materials

6.1 Glass fiber filter, 2.4-cm circle*

6.2 Hydrocarbon solvent, isooctane, ASTM knock test reference fuel grade, filtered through two glass-fiber filter papers.

Warning! Flammable. Harmful if inhaled. See Table B-1.

6.3 Adherent insolubles solvent--Mix equal parts of reagent grade acetone, methanol, and toluene.

Danger! Extremely Flammable. Vapors harmful. Vapors may cause flash fire. See Table B-1.

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*The glass fiber paper, available from H. Reeve Angel Co., New York, NY as Catalog No. X-934-AH, has been found satisfactory for this purpose.

TABLE B-1. PRECAUTIONARY STATEMENTS

Adherent Insolubles Solvent

Danger! Extremely flammable. May cause flash fire.
Inhalation of vapors harmful.
Keep away from heat, sparks, and open flame.
Keep container closed.
Use with adequate ventilation.
Vapors may spread long distances and ignite explosively.
Avoid build-up of vapors and eliminate all sources of ignition, especially non-explosion-proof electrical apparatus and heaters.
Avoid prolonged breathing of vapor or spray mist.
Avoid contact with eyes and skin.
In case of spillage, absorb in clay or diatomaceous earth and flush with large volumes of water.
In case of fire, use dry chemical, CO_2 or foam.

Distillate Fuel

Caution! Combustible. Vapor harmful.
Keep away from heat, sparks and open flame.
Keep container closed.
Use with adequate ventilation.
Avoid breathing vapor or spray mist.
Avoid prolonged or repeated contact with skin.
In case of spillage, soak up with clay, diatomaceous earth, or similar materials.
In case of fire, use foam, dry chemical, or CO_2.

Isooctane

Danger! Extremely flammable.
Harmful if inhaled. Vapors may cause flash fire.
Keep away from heat, sparks, and open flame.
Keep container closed.
Use with adequate ventilation.
Avoid build-up of vapors and eliminate all sources of ignition, especially non-explosion-proof electrical apparatus and heaters.
Avoid prolonged breathing of vapor or spray mist.
Avoid prolonged or repeated skin contact.
In case of spillage, soak up with sand or diatomaceous earth.
In case of fire, use foam, dry chemical or CO_2.

Acetone

Danger! Extremely flammable. Vapors may cause flash fire.
Keep away from heat, sparks, and open flame.
Keep container closed.
Use with adequate ventilation.
Vapors may spread long distances and ignite explosively.
Avoid build-up of vapors and eliminate all sources of ignition, especially non-explosion-proof electrical apparatus and heaters.
Avoid prolonged breathing of vapor or spray mist.
Avoid contact with eyes and skin.
In case of spillage, absorb and flush with large volumes of water.
In case of fire, use water, dry chemical, CO_2 or foam.

6.4 All equipment required to determine color by ASTM Method D 1500 (if desired).

6.5 All equipment required to perform steam jet method described in ASTM Method D 381 (if desired).

7. Sampling Procedure

7.1 Samples for testing shall be procured by the method outlined in ASTM Method D 270. Sample containers should preferably be 1 gallon or larger epoxy-lined cans. These containers should be filled almost to the top to avoid a significant air space, and then the ullage should be purged with nitrogen. Where possible, samples should be stored at reduced temperature, -7° to 4°C, prior to use.

8. Preparation of Apparatus

8.1 Sample storage bottles--Scrub each bottle with a detergent solution, and rinse it with water. Soak the bottle overnight in an alkaline laboratory glassware cleaning solution.* Rinse the bottle with tap water, then invert and flush with a stream of distilled water. Allow the bottles to dry, and rinse the bottles with 50 ml of the fuel sample. Bottles are vented during storage, preferably by a Teflon insert and a glass tube bent in a u-shape to prevent contamination of the sample from airborne particulates (See Figure B-2).

8.2 Insert two glass-fiber filter disks into each of three clean Gooch crucibles (Note 2). Wash the filters by pouring 200 ml of isooctane through the Gooch crucible. Dry the crucible and filters for 1 hr in an oven maintained at 99°±1°C. After drying, place the crucible and filters in a dessicator (without dessicant) for at least 30 min (Note 3). Weigh to the nearest 0.1 mg and retain for sample analysis. This procedure must be followed for

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*A 2-percent solution of RBS-35, manufactured by Pierce Chemical Company, has been found suitable. An alternate procedure is to use a chromium-free cleaning solution, "Nochromix," available from Godax Labs, 6 Varick Street, New York, NY 10013.

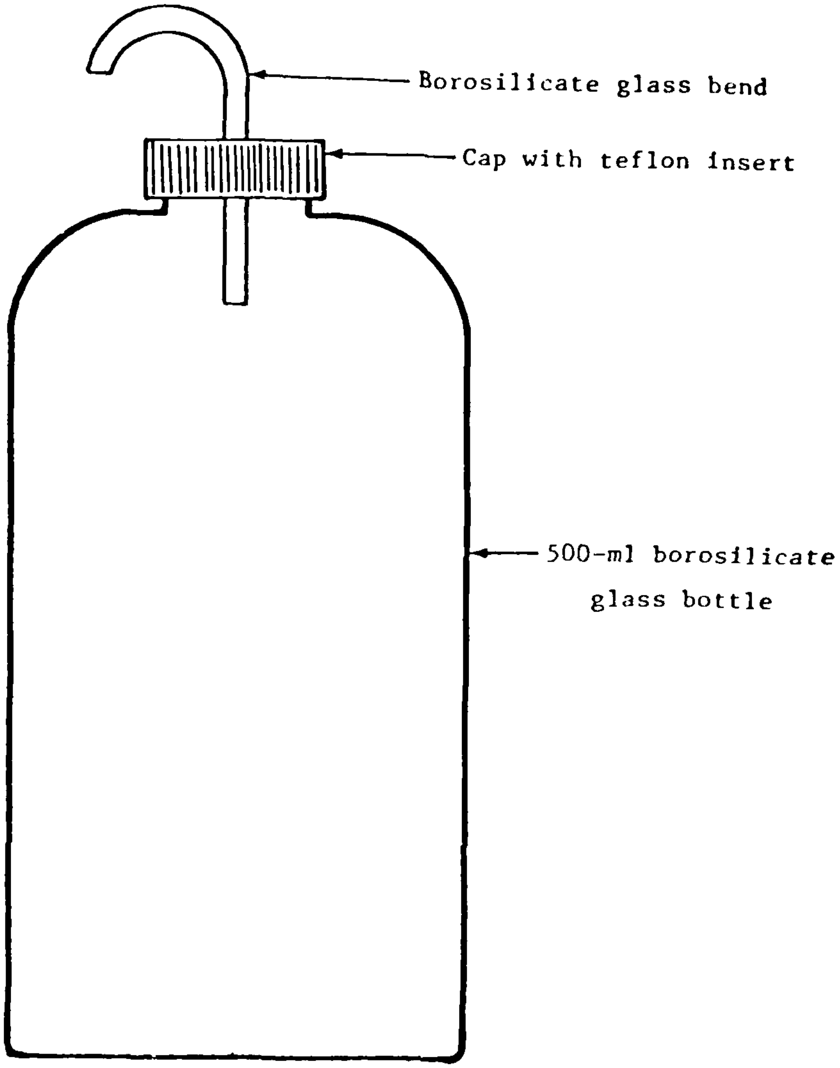

FIGURE B-2. SAMPLE STORAGE CONTAINER

the original sample and for each test sample interval. Prepare one crucible with filters for each bottle to be analyzed at each test sample interval and one crucible with filters to be used as a moisture correction. When a set of bottles is being analyzed, only one "moisture blank" is required. The "moisture blank" crucible should be weighed whenever the other crucibles are weighed and its change in weight subtracted from the final sample weight to give a sample weight that is corrected for atmospheric moisture.

Note 2: A single paper will not prove satisfactory since it may rupture under strong vacuum.

Note 3: A dessicator is used to protect the crucible and filters from airborne particulates. The dessicant is removed from the dessicator to allow the crucible to cool in an environment similar to the surrounding environment and thereby prevent possible errors due to sudden absorption of moisture from the atmosphere when the crucible is removed from the dessicator. The crucible used as a "moisture blank" will correct for the atmospheric moisture.

9. Preparation of Sample

9.1 Prior to storage, the fuel oil shall be filtered through two glass fiber filters. It is suggested that a filtration system as shown in Figure B-1 be used to filter the fuel (Note 4).

Note 4: The receiving flask, separatory funnel, and glass funnel should be as clean as the storage bottles (Sec 8.1). These items should also be rinsed with filtered isooctane.

10. Procedure

10.1 Adjust the storage oven for sample storage to a temperature of 43°±1°C.

Warning! Avoid contact with exposed hot surfaces on the drying oven by use of protective equipment as required.

10.2 Prepare the fuel for storage (Sec 9.1).

10.3 Place 400 ml of filtered fuel into each bottle. Use two bottles for each sampling period. (The suggested sampling periods are 4, 8, 12, 18, and 24 weeks). Also, fill two bottles with fuel to be used as extras. These extra bottles may be used in the case of accidents, if extra fuel is needed for further tests, or if desired to extend the overall storage test duration. Also filter enough fuel to be analyzed for zero-week data. These analyses should be performed on the same day that the other samples are placed in storage. Label each storage bottle completely.

10.4 At the end of each prescribed period, remove two bottles and allow them to cool to 21°-27°C for 4-24 hr in a dark environment.

10.5 Analysis of the fuel, after it has cooled, should be performed in the following manner. Pour the sample from one bottle into a clean separatory funnel, using a glass funnel (Note 4), and filter through a tared Gooch crucible containing two glass-fiber filter papers (Note 5). Use a self-feeding system as illustrated in Figure B-1. Repeat this procedure with the other bottle of fuel using a different crucible and filters (Note 6). Rinse the bottles used during aging with filtered hydrocarbon solvent (use three rinsings of approximately 50 ml each) to remove all traces of fuel from the bottle.

Warning! Flammable. Harmful if inhaled. See Table B-1.

Filter these rinsings through the Gooch crucible. After the crucible is washed oil-free on the inside, turn off the suction and remove the crucible. Then rinse the outside of the crucible (using a wash bottle) with additional hydrocarbon solvent until its surface is oil-free. The filtrate can be discarded.

Note 5: If severe filter plugging is encountered so that filtration is not complete in 3 hr, discontinue the test with the notation that filter plugging occurred.

Note 6: ASTM D 1500 color and D 381 Steam Jet Gum can be determined on the filtered fuel if desired. If these analyses are to be carried out, do not add rinsings or contaminate the filtered fuel in any manner, and protect the sample from light until these tests are completed.

10.6 Place the crucibles, including the "moisture blank" in the drying oven for 4 to 6 hr. Then place the crucibles in a dessicator without dessicant and allow to cool to room temperature [required at least 1 hr (Note 3)]. Weigh to nearest 0.1 mg.

10.7 After the final washing with the hydrocarbon solvent, dissolve any adherent gum on the sample container walls with two washings of 30 to 35 ml of the adherent insolubles solvent.

> Danger! Extremely flammable. Vapors harmful. Vapors may cause flash fire. See Table B-1.

Determine the adherent insolubles content by evaporating the solvent at 160°C by the air jet method described in ASTM Method D 381, using 1 or 2 gum beakers as required for the amount of solvent used.

> Warning! There are exposed hot surfaces on the evaporation bath. Avoid contact with exposed skin by use of protective equipment as required.
>
> Warning! The sample beakers and conical jets are extremely hot after completion of evaporation of solvent. Handle with care and use protective equipment or forceps as required.
>
> Danger! The solvent vapors evaporated during this procedure may be extremely flammable or combustible and hazardous to inhale. The evaporation bath must be provided with an effective exhaust hood to control such vapors.

11. Calculation

11.1 Calculate the total insolubles after aging, in milligrams per 100 ml, as follows:

$$A = B + C/4.0$$

where:

A = Total insolubles, mg/100 ml,
B = Weight of filterable insolubles (Note 7), mg, and
C = Weight of adjerent insolubles (Note 7), mg, as determined in 10.7 above.

Note 7: In this calculation, B and C must be corrected for moisture using the moisture blank.

12. Report

12.1 For unaged, filtered fuel, report results from 11.1 including A and B in appropriate units, and color and steam jet gum if these tests have been performed.

12.2 For the aged fuel, report A, B, and C from 11.1 for both bottles to show repeatability of tests at each aging period. Also, report color and steam jet gum if these tests have been performed.

Method 2. Storage Test at 150°C

Storage at 150°C was performed as per the proposed ASTM Standard Practice with the following exceptions: (1) The amount of material present on the filter was determined gravimetrically rather than by visual rating or reflectance. (2) Glass fiber filters were used in place of the Whatman No. 1 filters.

Proposed Standard Practice (Test Method) for High-Temperature Stability of Distillate Fuels

1. Scope

1.1 This method determines relative stability of distillate fuels under high temperature aging conditions with limited air exposure.

2. Applicable Documents

2.1 D 270 Sampling Petroleum and Petroleum Products
2.2 D 1500 ASTM Color of Petroleum Products

3. Summary of Method

3.1 A measured volume of distillate fuel in an open tube is aged 90 minutes or three hours at 150°C (302°F) with air exposure. After aging and cooling, fuel is filtered and the amount of insoluble residue formed is estimated by measuring the light reflection of the filter pad. Alternatively, the filter pad is compared with reference blotters and assigned a rating number corresponding to the reference blotter which matches its appearance.

4. Significance

4.1 The test provides an indication of thermal oxidative stability of distillate fuels when heated to high temperatures before combustion. Results have not been substantially correlated to burner or engine operation.

4.2 When the test is used to monitor manufacture of storage of fuels, changes in filter rating values may indicate a relative change in inherent storage stability. Storage stability predictions are more reliable when correlated to long-term storage tests, or when used in combination with other test.

4.3 Additives may have positive or negative effects in this test which may or may not be observed in actual storage or use.

5. Definitions

5.1 Thermal Stability: The tendency of thermally stressed fuel to form degradation products which may cause operating problems.

5.2 Storage Stability: The tendency of fuel to form degradation products during storage at ambient conditions.

5.3 Inherent Stability: The stability of fuel in the absence of environmental contaminants and effects encountered during actual field storage.

6. Apparatus

6.1 Aging tubes, 2.5 x 20 cm, heavy wall test tubes made of borosilicate glass.

6.2 Filter paper, Whatman No. 1, 4.25 cm or equivalent.

6.3 Membrane filter holder, to fit 47 mm membrane filters, vacuum source, and filtration flasks.

6.4 Heating bath, with liquid heating medium, thermostatically controlled and stirred to maintain the oil sample in the aging tube within 1.5°C (3°F) of 150°C (302°F). It shall be large enough to hold aging tubes immersed in the heating liquid to a depth above the level of samples in the tubes. The bath and its location shall enable shielding of the samples from direct light during aging. The volume of oil in the bath and its heat recovery rate shall be such that the temperature of the heating medium does not drop more than 5°C (9°F) when the maximum number of aging tubes are inserted, and recovery to 150°C (302°F) does not require more than 15 minutes.

> Warning! - The flash point of the liquid heating medium must be at least 180°C (350°F). Both vapors and oil sample vapors must be properly vented to prevent their entering the work atmosphere. The bath should be located in an area free of sparks and open flame.
>
> Warning! - There are exposed hot surfaces on the apparatus and the hot heating medium can cause severe burns. Avoid contact with exposed skin by use of protective equipment as required.

6.5 Paper reflection meter*, black glass surface for calibration, and opaque white surface at least 10 x 20 cm in size.

6.6 Reference blotters, du Pont F21-61 or equivalent.

7. Reagents

7.1 Hydrocarbon solvent, isooctane, ASTM knock test reference fuel grade.

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*A Photovolt Paper Brightness Meter 670, complete with search unit W and black glass calibration standard, Catalog No. 00-572-32, is suitable and is available from Photovolt, 1115 Broadway, New York, NY 10010. Other meters may be satisfactory, provided the measured area on a filter pad is greater than 1 cm and less than 3 cm in diameter.

7.2 Adherent insolubles solvent, equal parts of reagent grade toluene, methanol and acetone.

> Danger! Extremely flammable. Harmful if inhaled. Vapors may cause flash fire. Keep away from heat, sparks and open flame. See Table B-1.

8. Sampling

8.1 Samples for stability testing should be all-level samples obtained according to methods outlined in D 270, Sampling Petroleum and Petroleum Products. Samples should be stored in metal cans, preferably with epoxy lining. Clear glass bottles are not acceptable as sample containers. If a sample cannot be tested immediately, it should be stored in the dark and under nitrogen at a temperature not higher than 10°C (50°F). If samples are stored longer than one week, the date of sampling and date of testing shall be reported.

9. Preparation of Apparatus

9.1 Cleaning aging tubes - Clean new tubes by filling with a cleaning solution and allow to soak at least 2 hours. Rinse each scrupulously with tap water to remove all traces of acid, then with distilled water, followed by acetone, then dry. If compressed air is used, it must be oil-free. Used tubes should be rinsed with adherent insolubles solvent, then with detergent, tap water; distilled water followed by acetone, then air-dried.

9.2 Cleaning filter assembly - Membrane filter holders for which the filter rests on a sintered glass surface must be periodically cleaned with adherent insolubles solvent or cleaning solution followed by rinses as above (Note 1).

> Adherent insoluble solvent
> Danger! - Extremely flammable. May cause flash fire. Inhalation of vapors harmful. Keep away from heat, sparks and open flame. See Table B-1.

Acetone
Danger! - Extremely flammable. Vapors may cause flash fire. Keep away from heat, sparks and open flame. See Table B-1.

Note 1 - Partial blockage of the sintered glass surface can lead to uneven deposition of insoluble residues on the filter surface and reduce reliability of results. Contaminants may also be introduced during pre-filtration of fuel to give erratic results.

10. Procedure

10.1 Adjust the heating bath to a temperature high enough to maintain the oil in the aging tubes at 150 ± 1.5°C (302 ± 3°F).

Warning! There are exposed hot surfaces on the apparatus and the hot heating medium can cause severe burns. Avoid contact with exposed skin by use of protective equipment as required.

10.2 Assemble the filtration assembly with a new filter paper. Filter at least 50 ml of the sample through the paper. Measure a 50 ml portion of the filtered oil and decant into the aging tube (Note 2).

Distillate Fuel
Caution! - Combustible. Vapor harmful. See Table B-1.
Note 2 - The ASTM D 1500 color ("initial" color) of the fuel may be determined after filtration.

10.3 Place the uncapped sample tube in the heating bath for 90 minutes ± 3 minutes, or 3 hours ± 5 minutes. Samples should be shielded from direct light during aging (Note 3).

Warning! Fuels will be heated above their flash points. Fuels with initial boiling points below that of No. 2 fuel (kerosene, Jet A) may boil under the conditions of the test. Fuel samples which may contain gasoline or other volatile materials may vaporize excessively and should not be tested. The bath must be located in a ventilated area (hood) and kept away from sparks and open flame.

Note 3 - The severity of the test is increased by aging for longer times at 150°C (302°C). The selection of the aging time depends on the applications and should be established by correlation with other tests or field experience.

10.4 Remove the sample from the bath and allow to cool gradually to 21 to 26°C (70 to 80°F) over a period of 1½ to 4 hours in a location shielded from light (Note 4).

> Caution! - The hot samples can cause severe burns. Use protective equipment.
>
> Note 4 - If samples are allowed to stand more than 4 hours, insoluble gum may adhere to the aging tubes, resulting in erratic results.

10.5 Prepare a filtration assembly with a new filter paper and filter the fuel sample (Note 5). Wash the aging tube with three small portions of isooctane and filter through the filter paper. Wash the filter assembly with isooctane and remove the funnel portion of the assembly. Wash the filter pad with several very small portions of isooctane and allow to dry (Notes 6, 7).

> Isooctane
> Danger! - Extremely flammable. Harmful if inhaled. Vapors may cause flash fire. Keep away from heat, sparks, and open flame. See Table B-1.
>
> Filtration
> Caution! - Use a properly grounded system to avoid static discharge and ignition.
>
> Note 5 - Filtrate may be decanted for measurement of D 1500 color (test color), before use of isooctane.
>
> Note 6 - Some laboratories may contain sufficient airborne contamination to change results if the air drying period is prolonged.
>
> Note 7 - If the deposit on the filter pad is not evenly distributed, the pad should be rejected and the test re-run after cleaning the filtration apparatus.

10.6 If filter pads are to be rated by light reflection, proceed as follows in steps 10.6 to 10.9. If reference blotters are used, go to step 10.10. Turn on the reflection meter and allow at least 30 minutes for warm-up. The "suppression" knob should be in the "off" position.

10.7 Place a new filter paper on the opaque white surface, place the search unit in the center of the filter and adjust the meter reading to 100% using the "sensitivity" knob.

10.8 Place the search unit in the center of the black glass standard and adjust the meter reading to 0% using the "zero" knob. Recheck the 100% adjustment against the new filter pad resting on the white surface and readjust if necessary.

10.9 Place the test filter pad on the white surface, center the unit on the filter, and record the meter value as "percent reflection after test," rounding off to the nearest whole percentage point (Note 8).

> Note 8 - Dark fuels may stain the filter pads, resulting in lower reflectance ratings than due to insoluble residue alone.

10.10 To rate filter pads by visual comparison, first select the reference set (grey or brown tone) which gives the better color match with the test pad. If neither reference set gives a distinctly better match, use the grey-tone reference blotters.

10.11 Rate each filter pad by choosing a reference blotter which give the best visual match. Record the number of the matching blotter and its color (G = grey or B = brown) (Note 9).

> Note 9 - If more than one filter pad is being rated at the same time, they should first be rated independently. Then ratings and relative appearance should be compared to assure consistency within the set of filter pads.

11. Report

11.1 Report the aging time at 150°C (302°F).

11.2 If filter pad light reflection is measured, report the "reflection pad rating".

11.3 If the filter pad is rated by comparison to reference blotters, report "visual pad rating" and the rating scale used for comparison (grey or brown).

11.4 If the test was not run within one week of fuel sampling, report sampling date and date of test.

12. Precision

12.1 The repeatability and reproducibility of this test have not been determined. The following factors have been reported to affect results in some laboratories.

12.2 Sample Storage Time - During storage some fuels form degradation precursors which markedly affect results, even when fuel appearance has not changed.

12.3 Filter Paper Porosity - The sensitivity of the method is changed when filters with different porosity or surface roughness are used.

12.4 Additive Adsorption - Some additives are tenaciously adsorbed on the aging tubes and may affect results.

12.5 Heating Bath Design and Location - Exposure to light during the aging step may affect results. Air flow rates across the open tubes during aging may affect the severity of the test.

APPENDIX C

TEST FOR THE GROWTH POTENTIAL OF MICROORGANISMS

MICROB MONITOR® TEST KIT INSTRUCTIONS

1. PURPOSE:

Detects the presence of live microorganisms in a sample from a petroleum product system: (1) in tank water bottoms, (2) in fuel-water interface, or (3) in the fuel.

2. SCOPE:

Equal parts of the sample from (1), (2), or (3) above in that order of preference or availability are injected into each of two bottles labeled A and B. Both bottles contain water, oil, air, and other elements known to promote rapid growth of microorganisms such as bacteria and fungi. Bottle B also contains the recommended dosage of Biobor JF, the trade name of a biocide from the U.S. Borax & Chemical Corporation. After a short incubation period Bottle A will detect microbial growth if live organisms were present in the sample. Bottle B is the control which remains unchanged by killing the live organisms with Biobor JF.

3. PROCEDURE:

a. Draw a sample from the tank using as aseptic procedure as is possible.
b. Carefully peel off metal protectors on the pair of bottles A and B— **DO NOT TOUCH THE RUBBER CAPS.**
c. Take the syringe from its wrapping; directions for this are attached. **DO NOT TOUCH THE NEEDLE.**
d. Stick the needle into the sample and pull in more than 2 ml of sample. Invert the syringe so that the needle is pointing up and push the plunger in to expel air bubbles; push plunger to the 2.0 ml mark.
e. Carefully insert the needle into the rubber cap of Bottle A and push plunger to the 1.0 ml mark.
f. Withdraw needle. Re-insert into Bottle B. Empty the other 1.0 ml into bottle.
g. Shake bottles and place in dark, at room temperature.
h. **BEND OR BREAK NEEDLE SO THAT IT CANNOT BE REUSED. DISCARD BOTH NEEDLE AND SYRINGE.**

4. OBSERVATIONS:

a. **If test fuel is microoganism contaminated, Bottle A may turn pink to red within 24 to 48 hours. However, observations should continue for one week. Slight pink may be visible in a few hours and deepen.**
b. **Bottles should be shaken at least once a day. Results will be obtained sooner and more dramatically the more the bottles are shaken.**
c **Microbial slimes and growth will develop within one week. The rate of growth will depend on the degree of contamination, type of microorganisms present and sample representation. (Microbial population is greater in the water bottoms and fuel-water interface.)**
d. **In all cases the development of color, turbidity, slimes and/or heavy growths in Bottle A are readily compared with Control Bottle B.**
e. **It is suggested that each sample injection be recorded and each pair of test bottles identified with the date and sample source. Also record the chronological observations, i.e., color change, development of slimes, in the space provided on the box.**

"November, 1966"

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ATTN CODE 473 1
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ROBINS AFB GA 31098

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2100 2ND ST SW
WASHINGTON DC 20590

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DIV OF TRANS ENERGY CONSERV 2
ALTERNATIVE FUELS UTILIZATION BRANCH
20 MASSACHUSETTS AVENUE
WASHINGTON DC 20545

DIRECTOR
NATL MAINTENANCE TECH SUPPORT CTR 2
US POSTAL SERVICE
NORMAN OK 73069

US DEPARTMENT OF ENERGY
BARTLESVILLE ENERGY RSCH CTR
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