



IEP ABOA BY TON FI BY

Report DTRC/SME-CR-10-89

'AD-A224 868

AN EVALUATION OF COMMERCIAL ADDITIVES FOR FUEL STABILITY ENHANCEMENT; BOTTLE TESTS AT 65°C

by

Keith W. Flohr **ARTECH Corporation** 14554 Lee Road Chantilly, Virginia 22021

Contract N00167-83-D-0012

April 1990

Approved for public release; distribution unlimited.



Prepared for

Dr. E.W. White David Taylor Research Center Bethesda, Maryland 20084

ATT TIAL	31 -17-17 A 17-17 1	OF THE PAGE

REPORT DOCUMENTATION PAGE					
MEPORT SECURITY CLASSIFICATION		TO RESTRICTIVE	MARKINGS		
UNCLASSIFIED					
SECURITY CLASSIFICATION AUTHORITY		3 DISTRIBUTION: AVAILABILITY OF REPORT Approved for public release; distribution			
DECLASSIFICATION / DOWNGRADING SCHEDU	Approved funlimited.	or public re	elease; dist	ribution	
PERFORMING ORGANIZATION REPORT NUMBE	5 MONITORING	ORGANIZATION R	EPORT NUMBER	5)	
J8350.48	DTRC/SHE-C				
NAME OF PERFORMING ORGANIZATION	60 OFFICE SYMBOL (14 applicable)	78 NAME OF MC	MITORING ORGA	NIZATION	
RTECH Corporation			or Research		le 2759)
ADDRESS (City, State, and 21P Code)		76 ADDRESS (CA	y. State, and ZIP (Code)	
14554 Lee Road	i	Betheeds	Maryland 20	MR/5000	
Chantilly, Virginia 22021		pechesez, .		004-2000	
NAME OF FUNDING / SPONSORING ORGANIZATION	86 OFFICE SYMBOL (If applicable)	9 PROCUREMENT	INSTRUMENT ID	ENTIFICATION NU	MBER
Office of Naval Research	Code 123		00167-83-D-0		
ADDRESS (City, State, and ZIP Code)			UNDING NUMBER		
A=14==== V4==4=4= 22217=500	V	PROGRAM ELEMENT NO	PROJECT NO	TASK NO	WORK UNIT
Arlington, Virginia 22217-500	~	63724N	<u> </u>	R0838	DN 578153
TITLE (Include Security Classification)		27.241	<u> </u>	1 VOOD	1 20 2/0133
TYPE OF REPORT SEARCH & Development FROM SUPPLEMENTARY NOTATION	LO LO	14 DATE OF REPO	April	Day) IS PAGE	35
COSATI CODES	18 SUBJECT TERMS (
FIELD GROUP SUB-GROUP	Fuel Diesel Fuel	ASTM D46	bility 25	Bottle Te	
	Distillate Fuel		=		
ABSTRACT (Continue on reverse if necessary			, Madicives	<u> </u>	<u></u>
In a joint program with the Naval Research Laboratory, the Center has been evaluating the effectiveness of using 10 commercial additives (and two Army additives) to suppress the formation of insolubles in mid-distillate fuels from different fuel sources and processes. The Center contracted ARTECH Corporation to evaluate the 10 additives at two concentration levels in each of four base fuels, using a bottle test similar to ASTM D4625, but run at 55°C rather than 43°C. Four of the additives were found to be effective, and six were slightly effective or innocuous. ARTECH Corporation also conducted two special studies. One evaluated the effect that					
reaged fuel had on insolubles	formation in 6	SOC bottle to	ests; the ot	her examine	d the effect
f the condition of sampling a			_		-
ersus unlined storage cans, and wet versus dry storage. It was found that air blown or					
				on reverse	side.)
DISTRIBUTION/AVAILABILITY OF ABSTRACT UNICLASSIFIED/UNLIMITED SAME AS	RPT DTIC USERS	21 ABSTRACT SE	CURITY CLASSIFIC	ATION UNCLASSIFIE	3D
a NAME OF RESPONSIBLE INDIVIDUAL	C DIK OSEKS	225 TELEPHONE	Include Area Code		
r. E.W. White		(301) 26			2832
D FORM 1473, 84 MAR 83 A	PR edition may be used un All other editions are of			CLASSIFICATION	OF THIS PAGE

BLOCK 19 (Continued)

air blanketed fuel stored in unlined cans in the presence of moisture produced appreciably more insolubles than the same fuels under an argon blanket in a lined can in the absence of moisture.

Accession For	
NTIS GRA&I	
DTIC TAB	1
Una mounced 🔲	1
Justilication	\dashv
	7
P.	4
Dysamitudien/	\dashv
Ampiloh lity Codes	
Averl and/or	
Pant Special	İ
·	
H-1	



CONTENTS

	Page
ABBREVIATIONS	v
ABSTRACT	1
ADMINISTRATIVE INFORMATION	1
ACKNOWLEDGMENTS	2
INTRODUCTION	2
MATERIALS AND EQUIPMENT	3
FUEL SAMPLES	3
STABILITY ADDITIVES	5
SOLVENTS	6
Hydrocarbon Solvent	6
Adherent Insoluble Solvent	6
Solvent Qualification	6
TEST PROCEDURES	7
OVERVIEW	7
PRESTRESS PROCEDURES	7
<u>Cleaning</u>	7
Bottle Venting	8
Time and Temperature	8
Sample Preparation	8
Oven Loading	8
POSTSTRESS PROCEDURES	9
Filterable Insolubles Determination	9
Adherent Insolubles Determination	9

CONTENTS (Continued)

	Page
DISCUSSION OF RESULTS	. 10
EXPERIMENTS 8541, 8542, AND 8544	10
EXPERIMENTS 8645m AND 8647	14
CONCLUSIONS	. 19
APPENDIX A - RAW DATA	. 21
REFERENCES	. 33
TABLES	
1. Physical properties of neat fuels	4
2. Description of fuel additives	. ه
3. Summary of stress periods used	. 7
4. Insolubles from Experiment 8541, Week 8	. 11
5. Insolubles from Experiment 8542, Week 8	. 12
6. Insolubles from Experiment 8544, Week 12	. 13
7. Average apparent improvement percentages of additives	. 14
8. Insolubles from Experiment 8645m, Week 8	. 15
9. Insolubles from Experiment 8647 Week 8	17

ABBREVIATIONS

AI Adherent insolubles

AIP Apparent improvement percentage

ASTM American Society for Testing and Materials

Avg Average

OC Degrees Celsius

cm Centimeters

cSt Centistokes

OF Degrees Fahrenheit

FI Filterable insolubles

HPLC High performance liquid chromatography

LCO Light cycle oil

mg/100 mL Milligrams per 100 milliliters

mL Milliliters

NA Not available

NIPER National Institute for Petroleum and Energy Research

NRL Naval Research Laboratory

ppm Parts per million

Std Dev Standard deviation

SR Straight run stock

TAM An equal volume mixture of toluene, acetone, and methanol

TI Total insolubles

ABSTRACT

In a joint program with the Naval Research Laboratory, the Center has been evaluating the effectiveness of using 10 commercial additives (and two Army additives) to suppress the formation of insolubles in mid-distillate fuels from different fuel sources and processes. The Center contracted with ARTECH Corporation to evaluate the 10 additives at two concentration levels in each of four base fuels, using a bottle test similar to ASTM D4625, but run at 65°C rather than 43°C. Four of the additives were found to be effective, and six were slightly effective or innocuous.

ARTECH Corporation also conducted two special studies. One evaluated the effect that preaged fuel had on insolubles formation in 65°C bottle tests; the other examined the effect of the condition of sampling and storage, e.g., air versus argon blanketing, epoxy-lined versus unlined storage cans, and wet versus dry storage. It was found in this study that air blown or air blanketed fuel stored in unlined cans in the presence of moisture produced appreciably more insolubles than the same fuels under an argon blanket in a lined can in the absence of moisture.

ADMINISTRATIVE INFORMATION

This task was conducted under Contract N00167-83-D-0012-0048. The original delivery order of 28 December 1984, required completion by 31 October 1985. Delays in the availability of fuel samples necessitated an extension to 31 July 1986.

The work was funded by the Office of Naval Research (Dr. Alan Roberts and Mr. Wayne Vreatt, Code 123) under Program Element 63724N, Task Area R0838, and Center Work Unit 2759-313. The program was block-funded to the David Taylor Research Center where Mr. W.H. Stoffel of the Shipboard Energy R&D Office (Code 2759) was the block program manager and Mr. R. Strucko (Code 2759) was the project engineer. The technical representative at the David Taylor Research Center was Dr. D.R. Ventriglio (Code 2801); Dr. E.W. White (Code 2832) was the technical manager; and Ms. C. White (Code 5323) was the contracting officer. Mr. K.W. Flohr served as the ARTECH project manager.

All opinions expressed and trade names or specific product identifications used are the sole responsibility of ARTECH Corporation and should not be construed as opinions or endorsements by any U.S. government agency or employee.

ACKNOWLEDGMENTS

The laboratory work at ARTECH was performed by Eric Snyder and Greg Hamm, whose assistance and knowledge are greatly appreciated. The editorial assistance and advice provided by Dr. E.W. White are appreciated.

INTRODUCTION

In a joint program with the Naval Research Laboratory (NRL), the Center has been evaluating the effectiveness of using commercial stability additives to suppress the formation of insolubles in mid-distillate fuels. NRL has been conducting bottle storage tests (similar to ASTM D4625) at 43°C and 80°C, while the Center has been conducting accelerated stability tests similar to ASTM D2274. The Center contracted ARTECH Corporation to conduct bottle tests similar to ASTM D4625, but at 65°C rather than 43°C. All of the fuels and fuel/additive blends were specified by NRL, the lead laboratory. Under contract to NRL, the National Institute for Petroleum and Energy Research (NIPER) collected fuel stocks and blended then with the additives.

In response to contract requirements, ARTECH conducted experiments on five lots of fuel. In three of the experiments (8541, 8542, and 8544), various blends of straight-run and light cycle oil (LCO) stocks were used neat (without additive) and dosed with each of the 12 additives at high and low dosage levels. In the fourth experiment (8645m), one lot of fuel was preaged by air sparging and storing for 1 month at ambient laboratory temperatures before subjecting the fuel/additive blends to 65°C storage. In the fifth experiment (8647), various sampling and

storage conditions were used to determine their effects on insolubles formation.

In this experiment, only three additives were used and only at the high dosage level.

The contract required that ARTECH supply data on filterable, adherent, and total insolubles, and on the relative effectiveness of various additives in reducing the amount of insolubles. Data presented in this report have been distributed to participating laboratories at meetings held to compare and discuss results. This is a final report to summarize our test methods and results. Herein, we describe the work performed, present summaries of the data collected, and provide an evaluation of the relative effectiveness of the various additives. While all data are included in this report, our discussion is generally limited to the 10 commercial additives. The two Army additive blends are considered unsuitable for shipboard use because of their water dispersant properties.

MATERIALS AND EQUIPMENT

FUEL SAMPLES

NIPER obtained the samples used in Experiments 8541 and 8542 from a Gulf Coast refinery in 1985. Experiments 8544 and 8645m used samples obtained from a West Coast refinery during a 1985 visit; the fuels for Experiment 8647 were obtained from the same West Coast refinery in 1986. All fuels were treated at the refinery with stability-enhancing additives as would be the case during production runs.

NIPER assigned a four-digit identifying number to each fuel stock and each blend at the time the fuel was collected and blended. (Those numbers are retained in this report.) The fuels were shipped in 5-gallon cans to the Center or NRL where they were subdivided into 1-gallon, epoxy-lined cans for distribution to the participating laboratories. The source refinery location for each lot of fuel and selected properties of the neat straight-run/LCO blends are shown in Table 1.

Table 1. Physical properties of neat fuels.*

ID No.	LCO (1)	Refinery Location	Gravity (°API)	Viscosity 40°C (cSt)	Bromine No.	Acid No. mg KOH/g		Cetane Index D976	Flash Point (°C)
1773	30	Gulf Coast	34.6	3.36	0.23	0.19	0.25	49	81
1774	40	Gulf Coast		3.05	0.24	0.15	0.22	48	83
1880	40	West Coast	25.5	3.15	0.51	0.04	0.86	36	88
1879	30	West Coast	27.5	3.09	0.43	0.04	0.67	39	87
2065**	30	West Coast	29.1	3.15	NA	0.07	NA	NA	102

*Refer to Reference 1.

**Refer to Reference 2.

Samples used in Experiments 8645m and 8647 were specially treated before testing. DTRC treated samples used in 8645m by air sparging at a rate of 4 L/hr of room temperature air for approximately 3 hr. The samples then were sealed and retained by ARTECH for 4 weeks at ambient temperature prior to testing.

The samples for Experiment 8647 were collected at the West Coast refinery under a number of conditions to determine the effects of collection, storage, and transport of the fuels; three additives, rather than 12, were used in this experiment. Previous samples had been collected in 5-gallon, epoxy-lined cans, with argon in the can ullage. The samples for Experiment 8647 were collected in epoxy-lined cans or in unlined metal cans; air contact was allowed in some cans and in others an argon blanket prevented any air contact; some cans were kept as dry as possible and others had water added. These conditions were employed to determine what effect the variations in sample collection and handling may have on the production of insoluble products produced by accelerated storage stability tests.

STABILITY ADDITIVES

Of the 12 stability additives, 9 were standard commercial products, one was a blend of two of the commercial additives, and the two remaining were Army additive blends included for testing in this program. (The Navy cannot use the Army blends due to their water dispersant properties.) The additives were supplied on a condition of nondisclosure. Therefore, this report uses the additive codes supplied by NRL. Table 2 indicates the general nature of the 10 Navy additives, 3 as supplied by the manufacturers. The chemical nature of the two Army additives was not supplied.

Table 2. Description of fuel additives.

Code	Chemical Description	Active Ingredient (%)
1	Unknown	70
2	Tertiary amine	100
3	Amine (proprietary)	80
4	Amine + dispersant + corrosion inhibitor	70
5	Tertiary amine + metal deactivator + dispersant	15
6	Secondary amine + metal deactivator	82
7	Unknown + dispersant + corrosion inhibitor	46
8	Unknown	50
9	Secondary amine	70
10	Mixture of 2 and 6	90

Each of the first three neat fuels was dosed with two different concentrations of each of the 12 additives, producing a total of 27 separate samples per test condition (straight run stock, LCO stock, the neat blended stock, and 12 each high-and low-dose additives). The low additive dosage level was 12 ppm for the 10 Navy commercial additives; the high dosage level was 24 ppm. For the two Army additives, the higher dosage level was triple the lower level treatment, but the concentrations of active ingredients in the liquid additive package is unknown. Experiment 8645m was run with the higher concentration of additives only. The fuel samples for

Experiment 8647 were treated with only three of the additives (1, 2, and 9) at the higher dosage, because the experiment was designed to evaluate the effects of sampling and storage conditions rather than the additive concentration or the 10 commercial additives.

SOLVENTS

Hydrocarbon Solvent

The hydrocarbon solvent specified in ASTM D4625 is ASTM knock-grade fuel standard iso-octane which dissolves only the liquid fuel and will not effect the filterable or adherent insolubles. ARTECH used high performance liquid chromatography (HPLC) grade iso-octane in lieu of the specified ASTM grade.

Adherent Insoluble Solvent

ARTECH used the adherent insoluble solvent specified in ASTM D4625, an equal volume mixture of filtered ACS grade toluene, acetone, and methanol (TAM). TAM dissolves the adherent insolubles, so they can be transferred to a beaker for weight determination after drying.

Solvent Qualification

The solvents used in these tests were purchased from Fisher Scientific and Aldrich Chemical Companies. The HPLC grade iso-octane has no detectable residue after evaporation; therefore, it does not affect the final mass of filterable insolubles produced. Solvent purities and solid residues were determined for the iso-octane and the mixed TAM by solvent blank runs for each new lot of solvent. We found no appreciable residual mass from any of the solvents used.

TEST PROCEDURES

OVERVIEW

ASTM D4625 specifies that 400-mL samples of filtered fuel be stored in 500-mL borosilicate bottles at 43°C (110°F) for 0, 4, 8, 12, 18, and 24 weeks. After aging, the fuels are cooled to room temperature and analyzed for filterable and adherent insolubles.

ARTECH tests were conducted in triplicate using a modified version of the ASTM D4625 procedure. We modified the cleaning procedure and the bottle venting method. The fuels were stressed at 65° C rather than 43° C. Fuels were stressed for periods ranging from 2 to 12 weeks, as indicated in Table 3. The adherent insolubles were dried at 85° C rather than at $99\pm1^{\circ}$ C as specified in the Air-Jet Method of ASTM D381.

Table 3. Summary of stress periods used.

Experiment	Neat Fuel	Stress Time (weeks)
8541	1774	4 and 8
8542	1773	8
8544	1880	6 and 12
8645m	1879	2, 4, 6, and 8
8647	2065	2, 4, 6, and 8

PRESTRESS PROCEDURES

Cleaning

We modified the cleaning procedure slightly for the sample bottles, volumetric glassware, Gooch crucibles, beakers, and separatory funnels used in our tests. An initial TAM rinse was followed by cleaning with a mildly alkaline detergent, a tap water rinse, a rinse with distilled/deionized water, and an overnight soak with deionized water. The cleaned labware was oven-dried. The bottles were rinsed with a small amount of the sample fuel immediately before the final filling.

Bottle Venting

The 500-mL borosilicate glass bottles were vented by loosening the caps during oven stressing, rather than by using the standard glass gooseneck specified in ASTM D4625. Based on previous 65°C bottle tests, the results obtained with this method are indistinguishable from those obtained with the gooseneck vented bottles

Time and Temperature

Tests were conducted at 65°C rather than the 43°C specified in ASTM D4625.

Stress times ranged from 2 to a maximum of 12 weeks (see Table 3).

Sample Preparation

Upon receipt of the fuel samples, we filtered the neat fuel and fuel blends through 2.1-cm-diameter glass microfiber, Whatman 934-AH filters. All initial filtrations were conducted in batches large enough to fill all of the Wheaton borosilicate glass sample bottles with the required 400 mL of a given fuel. This minimized fuel variability between replicate samples. The fuels were customarily filtered in the numeric order of the NIPER-assigned identification numbers.

Oven Loading

Fuel samples were loaded into the 65°C oven in a double blind manner to minimize the effects of variations in oven temperature. One person randomly removed a labeled sample bottle from the transportation cart and handed it to a second person who then placed it in the oven.

POSTSTRESS PROCEDURES

Filterable Insolubles Determination

At the end of the temperature stress period, the samples were removed from the oven and allowed to cool in a dark place for a minimum of 6 hr but no more than 17 hr. Filterable insolubles were determined by filtering the fuel through tared Gooch crucibles fitted with the same type of Whatman 934-AH filters used in the initial filtration. The poststress filtration was conducted by series in numerical order of the NIPER identification numbers (i.e., a complete series of fuel numbers was filtered (1770, 1771, 1774, etc), then a second series, and so forth until all replicates had been filtered).

The bottles were rinsed with iso-octane to remove any remaining fuel (three rinses each of approximately 50 mL). The solution from this rinse was filtered through the Gooch crucibles, each of which then was rinsed with an additional 25-mL portion of iso-octane. The crucibles were dried in an 85°C oven overnight, until there was no solvent odor and constant weight was achieved.

Adherent Insolubles Determination

Adherent insolubles were determined by rinsing the fuel-free bottles with three portions of TAM (approximately 15 mL each) and collecting the resulting solutions in tared beakers. The beakers were allowed to evaporate overnight by standing them on the top of the drying ovens with a dust cover over each beaker. The nearly dried beakers then were placed in the drying oven until the solvent odor was gone and a constant weight was achieved.

DISCUSSION OF RESULTS

EXPERIMENTS 8541, 8542, AND 8544

Experiments 8541, 8542, and 8544 were the core of the laboratory work planned in the contract. They used the full complement of 12 stability enhancing additives.

(A compilation of all replicates, averages, and standard deviations appears in Appendix A.)

There was often little difference between the total insolubles obtained with the different additive blends, making discrimination among the additives difficult by direct comparison. To accentuate the differences, we have defined an apparent improvement percentage (AIP), which is derived from the average total insolubles of the neat fuel (additive free) and the average total insolubles of the additive-containing blend:

AIP =
$$\frac{\text{TI}_{\text{neat}} \cdot \text{TI}_{\text{blend}}}{\text{TI}_{\text{neat}}} \times 100$$

AIP values are shown in Tables 4 through 6, which summarize the data for the longest stress period in each experiment (8, 8, and 12 weeks for Experiments 8541, 8542, and 8544, respectively). The tables are divided into low- and high-additive dosage levels; the last column in each table is the calculated AIP. Table 7 shows the average AIP's in descending order on the basis of the high dosage level values. The Army additives are not included because they are not suitable for Navy use. Table A.6 of Appendix A summarized the apparent improvement percentages and the averages for all 12 additives.

Table 4. Insolubles from Experiment 8541, Week 8.

		Additive	Insolubl	es, mg/100	mL*	
Fuel	Description	Code	Filterable	Adherent	Total	AIP**
1770	SR	None	0.4	0.7	1.1	-
1771	LCO	None	2.6	8.6	11.2	-
1774	40% LCO	None	1.6	2.6	4.3	-
		Low Dose	ige Levels			
1779	40% LCO] 1	1.0	2.6	3.6	16
1785	40% LCO	2	1.3	1.6	2.9	33
1791	40% LCO	3	2.2	0.8	3.1	28
1797	40% LCO	4	2.0	2.0	4.0	9
1803	40% LCO	5	2.0	1.7	3.7	14
1809	40% LCO	6	1.7	1.9	3.6	16
1815	40% LCO	7	1.9	1.5	3.4	21
1822	40% LCO	8	1.7	2.0	3.7	14
1828	40% LCO	9	2.3	0.7	3.0	30
1834	40% LCO	11	2.6	0.8	3.3	23
1840	40% LCO	12	1.4	1.5	2.9	33
1846	40% LCO	10	1.3	1.6	2.9	33
		High Dos	age Levels			
1780	40% LCO	1 1	1.3	2.3	3.6	16
1756	40% LCO	2	1.3	1.3	2.6	40
1792	40% LCO	3	2.2	0.8	3.0	30
1798	40% LCO	4	1.2	1.9	3.1	28
1804	40% LCO	5	2.2	2.0	4.2	2
1810	40% LCO	6	1.6	1.7	3.5	19
1816	40% LCO	7	1.8	1.5	3.3	23
1823	40% LCO	8	1.3	2.2	3.4	21
1829	40% LCO	9	0.2	0.3	0.5	88
1835	40% LCO	11	0.4	0.5	0.9	79
1841	40% LCO	12	2.2	1.0	3.2	26
1847	40% LCO	10	0.8	1.6	2.4	44

*Average of triplicate determinations.

^{**}Apparent improvement percentage rounded to nearest percent.

Table 5. Insolubles from Experiment 8542, Week 8.

· 		Additive	Insolubl		mL*		
Fuel	Description	Code	Filterable	Adherent	Total	AIP**	
	·						
1773	30% LCO	None	2.3	2.5	4.8	-	
	•		· -	•	•		
	Low Dosage Levels						
1777	30% LCO	յ 1	1.8	2.7	4.5	6	
1783	30% LCO	2	1.5	2.0	3.5	27	
1789	30% LCO	3	2.4	1.3	3.7	23	
1795	30% LCO	4	1.9	2.4	4.3	10	
1801	30% LCO	5	1.7	2.5	4.1	15	
1807	30% LCO	6	2.3	2.4	4.7	2	
1813	30% LCO	7	2.6	2.2	4.8	0	
1820	30% LCO	8	1.6	2.5	4.1	15	
1020	30% 233		}				
1826	30% LCO	9	2.5	0.8	3.3	31	
1832	30% LCO	11	2.6	1.0	3.6	25	
1838	30% LCO	12	3.3	1.8	5.1	-6	
1844	30% LCO	10	1.6	1.8	3.4	29	
	•	High Do	sage Levels				
1788	30% LCO) 1	1.6	2.7	1 4.4	8	
1784	30% LCO	2	1.0	2.2	3.2	33	
1790	30% LCO	3	2.3	1.0	3.3	31	
1796	30% LCO	4	1.8	2.3	4.1	15	
1000						10	
1802	30% LCO	5	2.0	2.2	4.2	12	
1808	30% LCO	6	2.0	1.9	3.9	19	
1814	30% LCO	7	2.5	2.0	4.5	6	
1821	30% LCO	8	1.8	2.5	4.2	12	
1827	30% LCO	9	1.4	0.5	1.8	62	
1833	30% LCO	11	3.0	1.1	4.1	15	
1839	30% LCO	12	3.0	1.6	4.6	4	
1845	30% LCO	10	1.4	2.2	3.5	27	
di A	of hud-14 and d		<u> </u>		L	<u> </u>	

*Average of triplicate determinations.

^{**}Apparent improvement percentage rounded to nearest percent.

Table 6. Insolubles from Experiment 8544, Week 12.

	<u> </u>	Additive	Insolubl	es, mg/100	mL*	
Fuel	Description	Code	Filterable		Total	AIP**
1876	SR	None	0.2	0.6	0.7	
1877	LCO	None	5.6	20.0	25.6	
1880	40% LCO	None	6.4	6.9	13.3	1 - 1
		•	•	•	•	`
		Low Dos	ige Levels			j
1885	1 40% LCO	1	5.7	4.3	10.0	25
1891	40% LCO	2	0.3	5.5	5.8	56
1897	40% LCO	3	4.8	4.0	8.8	34
1903	40% LCO	4	5.6	7.8	13.3	0
	[1
1909	40% LCO	5	6.5	2.5	9.0	32
1915	40% LCO	6	4.3	8.4	12.7	5
1921	40% LCO	7	5.1	5.6	10.7	20
1927	40% LCO	8	4.6	9.0	13.7	-3
1933	40% LCO	9	2.5	1.3	3.8	71
1939	40% LCO	10	4.4	2.6	7.0	47
1945	40% LCO	ii	5.1	7.3	12.4	7
1951	40% LCO	12	3.8	6.8	10.6	20
			· · · · · · · · · · · · · · · · · · ·	•		ĺ
		High Do	sage Levels			
1886	40% LCO	1	7.7	5.0	12.7	1 5
1892	40% LCO	2	4.0	0.5	4.6	65
1898	40% LCO	3	4.4	4.0	8.5	36
1904	40% LCO	4	3.1	7.8	10.9	18
1910	40% LCO	5	7.7	3.5	11.3	1.5
1916	40% LCO	6	4.6	8.8	13.4	15
1922	40% LCO	7	4.6	6.4	11.1	-1 17
1928	40% LCO	8	3.6	7.1	10.7	20
1320	40% 100		3.0	/.1	10.7	20
1934	40% LCO	9	3.2	1.4	4.6	65
1940	40% LCO	10	1.6	6.3	7.9	41
1946	40% LCO	11	3.5	8.8	12.2	8
1952	40% LCO	12	0.7	4.4	5.1	62
L	of triplicate de	********	<u> </u>		L	L

*Average of triplicate determinations.

^{**}Apparent improvement percentage rounded to nearest percent.

As can be seen in Tables 4 through 6, the amounts of total insolubles in Experiments 8541, 8542, and 8544 generally were very low. Therefore, the task of discrimination between the various additives by direct inspection of the data is difficult. However, the AIP values in Table 7 show that Additives 2, 3, 9, and 10 averaged greater than 30% apparent improvement in the fuels tested. Additives 1, 4, 5, 6, 7, and 8 exhibited an apparent improvement between 10% and 20%.

Table 7. Average apparent improvement percentages of additives.

	Average AIP				
Additive Code	Low Concentration	High Concentration			
_	1				
9) 23	53			
2	39	46			
10	27	44			
3	28	32			
4	6	20			
8	9	18			
7	14	15			
6	8 {	12			
1	16	10			
5	20	10			

On the basis of these observations:

- Additives 2, 3, 9, and 10 appear to improve storage stability under the conditions tested;
- Additives 1, 5, 6, 7, and 8 appear to offer minor improvements or to be innocuous.

EXPERIMENTS 8645m AND 8647

Experiments 8645m and 8647 were special study experiments run as a result of concern over obtaining only low levels of total insolubles in Experiments 8541 through 8544. Experiment 8645m was run on fuels preaged by air sparging and

preliminary ambient storage prior to the normal ASTM D4625 type of accelerated aging at 65°C. Only the high concentration of additives was used in a neat fuel containing 30% LCO.

Results of the tests on preaged fuels are shown in Table 8. From the AIP levels, commercial Additives 2, 3, and 8 appear to be beneficial in these air aparged fuels; commercial Additives 4 and 12 appeared to be detrimental to the storage stability; and commercial Additives 1, 5, 6, 7, and 9 appeared innocuous.

From comparison of results from the preaged 30% LCO fuels (Table 8) and from the unaged 40% LCO fuels (Table 6) from the same refinery and fuels stocks, we found that in both cases blends containing Additives 2 and 3 were among those with the most beneficial results, and blends containing Additive 6 were among those with the poorest results.

Table 8. Insolubles from Experiment 8645m, Week 8.

	Additive	Insolubl	es, mg/100	mL*	
Fuel	Code	Filterable	Adherent	Total	AIP**
1879	None	5.1	3.2	8.3	-
1884	2	0.7	2.7	3.4	59
1932	11	3.2	0.9	4.1	51
1926	8	3.2	1.9	5.0	40
1938	12	4.1	1.9	6.0	28
1896	3	4.7	1.5	6.1	27
1920	7	4.8	1.6	6.4	27
1908	5	5.0	1,8	6.9	17
1884	1	5.0	2.3	7.3	12
1944	9	4.8	2.8	7.5	10
1914	6	4.0	3.8	7.8	6
1950	10	5.2	3.7	8.9	-7
1902	4	6.4	2.8	9.2	-11
		ļ	}		

Note: All blends were 30% LCO; all additives were at the high dosage levels.

^{*}Average of triplicate determinations.

^{**}Apparent improvement percentage rounded to nearest percent.

Experiment 8647 was conducted to explore the possible effects of the way in which a sample was taken and stored. Whereas early samples had been kept in epoxylined cans under an inert gas (argon) blanket, the samples for Experiment 8647 were taken and stored under a variety of conditions, as shown in Table 9. Note that some of the cans were unlined, some contained a small amount of water, and some were blanketed or blown with air; these are all conditions found in normal sampling. We focused on the effectiveness of only three fuel additives and did not calculate apparent improvement percentages, because our major thrust was to investigate the sampling conditions.

The data from Experiment 8647 indicate that the collection conditions do affect the production of insolubles. The condition that most directly relates to common fuel transportation and storage conditions (air blown, unlined can with water present) produced appreciably larger amounts of insolubles then the normal fuel study collection conditions (argon blanket, lined can with no water). Total insolubles of 6.9 mg/100 mL were obtained for neat Fuel 2093 (air, unlined, with water) versus 4.7 mg/100 mL for neat Fuel 2065 (argon, lined, with no water). This increase may have resulted from the presence of exposed metal. Total insolubles of 3.3 mg/100 mL were obtained for Fuel 2073 (argon, lined, with water) versus 4.7 mg/100 mL for Fuel 2065 (argon, lined, no water). Water added, in the absence of air, appeared to have only a slight negative effect on the generation of insolubles. A combination of air, water, and exposed metal surfaces appears to have increased the amounts of insoluble products generated in accelerated storage stability testing.

Table 9. Insolubles from Experiment 8647, Week 8.

[Additive	Insolubl	es, mg/100	mL*
Fuel	Description	Code	Filterable	Adherent	Total
	Argon Bla	nket. Lined	Can. No Wate	I	
2059 լ	SR	None	0.1	0.1	0.2
2060	LCO	None	0.1	0.3	0.5
2065	30% LCO	None	1.5	3.2	4.7
2066	30% LCO	1	3.0	2.5	5.5
2067	30% LCO	2	0.2	1.7	1.9
2068	30% LCO	9	1.4	2.1	3.6
	Air Blo	wn. Lined Ca	n. No Water		
 2061	LCO	None	0.1	0.7	0.8**
2069	30% LCO	None	1.4	3.0	4.4
2070	30% LCO	1	0.9	3.2	4.2
2071	30% LCO	2	0.2	1.9	2.1
2072	30% LCO	9	2.4	3.6	6.0
	Argon Blanket, Li	ned Can. 10	mL Water (5	gallons)	
2062	LCO	None	0.2	0.6	0.8
2073	30% LCO	None	0.7	2.6	3.3
2074	30% LCO	1	1.0	2.3	3.3
2075 2076	30% LCO 30% LCO	2 9	0.3 1.0	1.1 2.2	1.4 3.2
2070	30% LCO)	1.0	2.2	3.2
	Argon Blan	<u>ket. Unlined</u>	Can. No Wat	er	
2063	LCO	None	1.5	1.2	2.7
2077	30% LCO	None	3.0	2.8	5.2
2078	30% LCO	1	1.4	5.6	7.0
2079	30% LCO	2	1.8	1.9	3.7
2080	30% LCO	9	2.8	2.8	5.6
	Air Blown, Lined	Can. 10 mL	of Water (5	gallons)	
2081	30% LCO	None	1.1	4.2	1 5.4
2082	30% LCO	1	0.3	1.4	1.7
2083	30% LCO	2	1.5	2.8	4.3
2084	30% LCO	9	0.7	3.7	4.4
<u> </u>		_]
	rage of triplicate		ons.	·	
**Base	d on duplicate ana	lysis.			

Table 9. (Continued)

[Additive	Insolubl	es, $mg/100$	mL
Fuel	Description	Code	Filterable	Adherent	Total
	Alr Blow	n. Unlined C	an. No Water		
2064	LCO) None	0.6	2.8	3.4**
2085	30% LCO	None	3.6	4.3	7.9
2086	30% LCO	1	2.4	5.5	7.9
2087	30% LCO	2	1.9	2.9	4.8
2088	30% LCO	9	2.9	3.0	5.9
j			·	'	
Ar	gon Blanket, Unli	ned Can. 10	mL of Water	(5 gallons)
2089	30% LCO	None	3.1	2.8	5.9
2090	30% LCO	1	1.6	4.2	5.8
2091	30% LCO	2	0.5	2.0	2.5
2092	30% LCO	9	2.3	2.2	4.5
		, , , , , , , , , , , , , , , , , , , ,	6 11 / 5	11>	
	Air Blown. Unline	a can. 10 ml	or water (5	gallons)	
2093	30% LCO	None	2.8	4.1	6.9
2094	30% LCO	1	6.5	3.5	10.0
2095	30% LCO	2	1.5	2.2	3.8
2096	30% LCO	9	2.7	3.6	6.3
}					
	<u>Air Blank</u>	et. Unlined	Can. No Wate	r	
2106	30% LCO	None	6.1	3.8 j	10.0
2107	30% LCO	2	2.7	2.4	5.1

The use of an air blanket during collection should provide a closer representation of standard fuel conditions, because air normally is present in the ullage during collection and shipping of fuels. However, the increase in total insolubles was relatively small, and the apparent effectiveness of the three fuel additives did not change as a function of the collection conditions.

We observed that regardless of sampling and storage conditions, Additive 2 reduced the amount of total insolubles, compared to the fuel without additives.

Under most of the sampling and storage conditions, the presence of Additive 1 in the

fuel blend yielded innocuous results; i.e., the total insolubles produced in the test were within a few tenths of a mg/100 mL of those obtained with the neat fuel blend. However, in the case where the sample had water present, had been air-blown, and had been stored in a lined can, the presence of Additive 1 resulted in more total insolubles than were produced in the neat fuel under identical sampling and storage conditions. Additive 9 produced some lessening of the total insolubles, but not as much as Additive 2.

CONCLUSIONS

- 1. Additives 2, 3, 9, and 10 appear to improve storage stability under the conditions tested.
- 2. Additives 1, 4, 5, 6, 7, and 8 appear to offer minor improvements or be innocuous in the fuels and under the conditions of the experiment.
- 3. Variations in the collection condition indicate that the condition most directly related to common fuel transportation and storage conditions (air blown, unlined can, with water present) produces larger amounts of insolubles then the normal fuel study collection conditions (argon blanket, lined can, with no water). Water added, in the absence of air, appeared to have only a slight negative effect on the amount of insolubles generated.

APPENDIX A
RAW DATA

Table A.1. Experiment 8541, 4 weeks.

						In	solub	les, i	ng/10	00 mL					
			iherei	nt			Fi:	lteral					Cotal		
		Sample			Std		Sample	2]	Std		Sample		[Std
Fuel	1	2	3	Avg	Dev	1	2	3	Avg	Dev	1	2	3	Avg	Dev
1770 1771 1774	0.3 3.25 0.95	3.25 0.85	3.23 1.13	3.2 1.0	0.01 0.11	4.67 1.38	3.73 1.4	3.63 0.93	4.0	0.47	7.92 2.33	0.55 6.97 2.25	6.85 2.05	7.3	0.48 0.12
1779 1780 1785 1786 1791	1.7 1.0 0.8 0.7 0.17	0.9 0.63 0.68 0.23	1.15 0.63 0.55 0.23	1.0 0.7 0.6 0.2	0.08 0.07 0.02	0.93 1.2 1.05 1.6	1.85 1.05 1.57	0.93 1.1 1.07 1.63	1.0 1.4 1.1 1.6	0.33 0.01 0.02	1.93 2.0 1.75 1.78		1.73 1.63 1.85	2.0 2.1 1.7 1.8	0.07 0.31 0.05 0.03
1792 1797 1798 1803		0.25 1.02 1.2 0.85	0.78 1.05	1.1 1.1	0.27	1.07 0.82	1.45 1.2 0.8 1.43	1.27 1.23	1.2	0.08 0.19	2.5 1.95	2.22	3.52 2.05 2.28 2.27	2.3 2.1	0.19 0.14
1804 1809 1810 1815	0.97	0.9 1.23	0.82	0.9	0.06	1.25 1.4	1.3 1.27	1.32 1.23	1.3	0.03	2.23 2.42	2.5	2.77 2.15 2.03 4.63	2.2	0.03 0.21
1816 1822 1823 1828	0.9	1.35	1.38	1.2 1.5	0.19	1.45 0.97		1.5 0.93	1.5	0.03	2.35 2.9	2.6 2.45	2.7 2.88 2.15 1.92	2.6 2.5	0.31
1829 1834 1835 1840	0.75 0.38		0.57 0.55	0.6	0.17 0.10 0.07 0.05	1.77 0.4	1.8	1.8	1.8 0.7	0.01 0.31	2.52 0.78	1.05 2.3 1.07 2.63	2.38 1.68	2.4	0.09
1841 1846 1847	1.02 1.32 0.93	1.07	0.88 2.08 1.3	1.5	0.08 0.42 0.16	1.05	2.02	0.8	1.3	0.53	2.38	2.83 3.1 2.45	2.88	2.8	0.30

Table A.2. Experiment 8541, 8 weeks.

·						Ins	solub	les,	mg/10	00 mL					
ł			neren	<u> </u>				terab	le			Tota	1		, <u>.</u>
1		Sample			Std		Sample			Std		ample	, 		Std
Fuel	1	2	3	Avg	Dev	1	2	3	Avg	Dev	1	2	3	Avg	Dev
						0.57					0.9	1.45	1.02		0.24
						3.23				0.60		11.7	10.6	11.2	
1774			2.65				1.65			0.06	4.13	4.5			0.16
1779	2.9	2.63	2.2/	2.6	0.26	1.25	0.75	1.1	1.0	0.21	4.15	3.38	3.38	3.6	0.37
1780										0.12		3.65	3.3		0.18
1785			1.63							0.10	3.1	2.7	2.8		0.17
1						1.07					2.45	2.73	2.6		0.11
1791	17.1	0.95	0.5	0.9	0.25	2.15	2.1/	2.42	2.3	0.12	3.25	3.13	2.92	3.1	0.13
1792 1797						2.35 2.08					2.92 3.63	3.15 4.75	2.83 3.38		0.14
		1.77					1.15				3.38	2.92	3.15		0.80
						1.98					3.63	3.5	3.82		0.18
1803	11.65	1 4 . / /	1.65	1.7	0.00	1.70	1.73	2.17	2.0	0.16	3.63	3.5	3.62	3./	0.13
1804 1809						2.13 1.75				0.12	4.05 3.55	4.42 3.7	4.03 3.42	1	0.18
1810			1.63				1.55			0.10	3.8	3.38	3.35		0.21
						2.08				0.19	3.55	3.02	3.47		0.21
1012	1.40	1.52	1.43	1.5	0.04	2.00	1.5	2.05	1.9	0.27	3.33	3.02	3.4/	3.4	0.23
						1.25 1.85		2.1		0.43	2.92		3.33 3.63		0.30
		2.02					1.43				3.47				0.23
		0.65								0.08	2.65		3.3		
1020	0.75	0.65	0.66	0.7	0.04	1.9	2.58	2.38	2.3	0.28	2.03	3.23	3.05	3.0	0.24
1829						0.07					0.42	0.75	0.35		0.17
1834						2.38					3.27	3.5	3.2		0.13
1		0.33								0.07	1.25	0.7	0.88		0.23
1840	1.7	1.6	1.2	1.5	0.22	1.25	1.48	1.55	1.4	0.13	2.95	3.08	2.75	2.9	0.13
1841	1.2	1.0	0.93	1.0	0.12	2.15	2.15	2.15	2.2	0	3.35	3.15	3.08	3.2	0.12
1846	2.05					0.78				0.42	2.82		2.72		0.11
1847	1.85	1.48				0.78					2.63	2.2	2.53		0.18
				L											

Table A.3. Experiment 8542, 8 weeks.

						Ins	olub	les,	ng/10	00 mL					
		Adl	neren	t			Filt	terab	le_			Tota	1		
		ample			Std		ample			Std		ample		ļ	Std
Fuel	1	2	3	Avg	Dev	1	2	3	Avg	Dev	1	2	3	Avg	Dev
	1	2.40	2.55 2.50	2.7 2.7		1.57 1.98	1.70 1.57	2.00 1.38	1.8 1.6	0.18 0.25	4.57 4.85 5.03 3.50	4.72 4.10 4.25 3.45	5.17 4.55 3.88 3.67	4.5 4.4	0.25 0.31 0.48 0.10
1789 1790	2.85 1.27 1.02 2.38	1.15	1.40	1.3 1.1	0.10 0.05	2.45 2.38	2.20 2.13	2.50 2.25	2.4	0.13 0.10	3.60 3.73 3.40 4.60	2.72 3.35 3.13 4.00	3.22 3.90 3.38 4.22	3.7 3.3	0.36 0.23 0.12 0.25
1801	2.90 2.75 1.90 2.75	2.00	2.63	2.5	0.33	2.15 2.05	1.20 2.00	1.63 1.80	1.7 2.0	0.39 0.11	4.60 4.90 4.05 4.53	3.85 3.20 3.83 4.93	3.82 4.25 4.38 4.60	4.1 4.2	0.36 0.70 0.18 0.15
1813 1814	2.02 1.80 2.15 2.38	2.58 1.80	2.17	2.2 2.0	0.32 0.15	2.73 2.73	2.35 2.25	2.83 2.63	2.6 2.5	0.20 0.20	4.05 4.53 4.88 4.25	3.83 4.93 4.05 3.80	3.95 5.00 4.67 4.35	4.8 4.5	0.09 0.21 0.35 0.24
1826	2.73 0.85 0.53 1.27	0.80	0.78	0.8 0.5		2.27 2.92	2.35 0.68	2.80 0.45	2.5 1.4	0.16 0.23 1.12 0.22	4.30 3.13 3.45 3.52	4.00 3.15 1.35 3.63	4.40 3.57 0.68 3.70	3.3 1.8	0.17 0.21 1.18 0.07
1838 1839 1844		1.60 1.32 1.82	1.93 2.05 1.82	1.9 1.6 1.8	0.30 0.18 0.31 0.00	3.08 3.08 1.65	3.35 3.13 1.38	3.42 2.70 1.75	3.3 3.0 1.6	0.06 0.15 0.19 0.16	4.40 5.10 4.60 3.47	3.80 4.95 4.45 3.20	3.97 5.35 4.75 3.58	5.1 4.6 3.4	0.25 0.16 0.12 0.16

Table A.4. Experiment 8544, 6 weeks.

						In	solub	les, 1	ng/10	00 mL					
			neren	t				terab	le_			Tota	a1		
		ampl			Std	-	Sampl			Std		ample		Į .	Std
Fuel	1	2	3	Avg	Dev	1	2	3	AVE	Dev	1	2	3	Avg	Dev
	0					0.05				0.07			1.63		0.70
													10.60		
										0.41		4.35			0.09
1885	1.15	1.48	1.57	1.4	0.18	2.90	2.65	2.77	2.8	0.10	4.05	4.13	4.35	4.2	0.13
										0.11	4.15	3.77	4.00		0.15
										0.24	2.17	2.25	2.55		0.16
								0.72			2.27	2.15	2.20		0.05
1897	0.57	0.65	0.68	0.6	0.04	2.27	2.30	2.40	2.3	0.05	2.85	2.95	3.08	3.0	0.09
										0.06	2.57	2.65	2.70		0.05
								2.70			4.20		11.40		3.42
								2.23			4.50	4.13	4.22		0.16
1909	1.07	1.05	5.85	2.7	2.26	2.00	2.73	2.42	2.4	0.30	3.08	3.78	8.27	5.0	2.30
								3.13			1.28	1.75	3.95		1.17
								1.25			3.70	5.78	3.85		0.94
								1.82			5.22	5.18	3.52		0.79
1921	3.13	2.77	1.90	2.6	0.52	1.15	1.//	1.20	1.4	0.28	4.28	4.55	3.10	4.0	0.63
										0.55	2.70	4.30	4.50		0.81
								1.18			5.05	6.88	5.22		0.82
								1.48			5.42	5.40	3.45		0.93
1933	0.42	0.57	0.42	0.5	0.07	1.60	1.80	1.75	1./	0.08	2.02	2.38	2.17	2.2	0.14
								2.33			3.18	3.15	2.80	1 1	0.17
										0.03	2.90	3.13	3.23		0.14
								0.63			3.50	3.90	i		0.24
1945	3.92	2,90	4.33	3.7	0.60	0.97	1.60	1.57	1.4	0.29	4.90	4.50	5.90	5.1	0.59
										0.08	4.92	4.00	5.10		0.48
								0.88			3.80	3.47	2.60		0.51
1952	2.58	2.40	2.35	2.4	0.10	0.30	0.40	0.40	0.4	0.05	2.88	2.80	2.75	2.8	0.05
L			نـــــا	Щ.		L	L	<u></u>	L	L	L	<u> </u>	<u> </u>		L

Table A.5. Experiment 8544, 12 weeks.

						Inso	luble	s, mg	/100	mL					
]			nerent					terab	le				Total	,	
]		ample		[Std		Sample			Std		Sample	 -	}	Std
Fuel	1	2	3	Avg	Dev	1	2	3	Avg	Dev	1	2	3	Avg	Dev
1876	0.35	0.45	0.90							0.03					0.21
	20.20												26.60		
1880	5.63	7.53	7.50										13.20		
1885	2.27	5.15	5.60	4.3	1.4/	1.90	8.15	7.10	5./	2.73	4.1/	13.30	12.70	10.1	4.1/
1886	5.28	4.30	5.55										13.30		
1891	5.22		5.67							0.07					0.12
1892	4.20		4.22							0.06					0.16
1897	4.40	3.83	3.80	4.0	0.28	4.6/	4.9/	4.72	4.8	0.13	9.07	8.80	8.52	8.8	0.22
1898	5.25		3.92									7.78			0.79
1903	5.78		12.50										15.50		
1904	7.60		8.22										10.80		
1909	2.17	2.67	2.65	2.5	0.23	7.13	7.40	5.10	6.5	1.03	9.30	10.10	7.75	9.0	0.97
1910	3.00	3.80	3.75										11.10		
1915	8.72		7.58										13.10		
1916	8.95 5.50		8.95 5.90							1.25			14.10		
1921	3.30	3.40	3.90	3.6	0.22	3.42	0.47	3.28	3.1	1.23	0.93	11.90	11.20	10.7	1.20
1922	6.17		5.28										11.00		
1927		10.30	8.35										13.70		
1928	8.70		5.83										9.18		
1933	1.05	1.38	1.45	1.3	10.17	3.00	2.00	2.48	2.5	0.41	4.05	3.38	3.92	3.8	0.29
1934	1.57	•	1.32							0.19			1		0.09
1939	2.25		2.80							0.18			1		0.41
1940	5.10	1	8.53							0.11			10.00		1.48
1945	7.88	7.30	6.85	7.3	0.42	4.13	5.63	5.58	5.1	0.70	12.00	12.90	12.40	12.5	0.38
1946	8.85	8.10	9.30	8.8	0.49	3.40	3.60	3.48	3.5	0.08	12.30	11.70	12.80	12.2	0.44
1951	7.65	6.33	6.58										10.20		
1952	6.60	0.93	5.53								7.38	1.85			2.36
<u></u>	<u></u>	<u> </u>	<u> </u>	<u> </u>	<u></u>		<u> </u>			<u></u>	<u> </u>	<u> </u>		<u> </u>	

Table A.6. Apparent improvement percentages obtained.

	Wee	k 8	Week 12	
Additive	Experiment 8541	Experiment 8542	Experiment 8544	Average
		Low Concentration		1
1	16	6	25	16
2	33	27	56	39
2 3	28	23	34	28
4	9	10	0	6
5	14	15	32	20
5 6	16	2	5	8
7	21	0	20	14
8 9	14	15	-3	9
9	30	31	7	23
10	33	29	20	27
11	23	25	71	40
12	33	-6	47	25
		High Concentration		!
1	j 16	8	5	10
1 2 3	40	33	65	46
3	30	31	36	32
4	28	15	18	20
5 6 7	2	12	15	10
6	19	19	-1	12
7	23	6	17	15
8	21	12	20	18
9	88	62	8	53
10	64	27	62	44
11	79	15	65	53
12	26	4	41	24
: 	<u> </u>	<u> </u>		<u> </u>

Table A.7. Experiment 8645m, 2 wee's.

						Inso	luble	s, mg	/100	mL					
		Ad	herent				Fil	terab:	le				Total		I
ł		Sample	:		Std		Sampl	е		Std		Sample			Std
Fuel	1	2	3	Avg	Dev	1	2	3	Avg	Dev	1	2	3	Avg	Dev
										}					
1879	1.48	1.57	0.80	1.3	0.34	2.48	2.38	1.93	2.3	0.24	3.95	3.95	2.73	3.5	0.58
1884	0.42	0.80	1.20	0.8	0.32	0.38	0.88	0.50	0.6	0.21	0.80	1.68	1.70	1.4	0.42
1890	0.57	1.30	0.50	0.8	0.36	1.13	1.20	1.68	1.3	0.24	1.70	2.50	2.17	2.1	0.33
1896	0.60	1.15	0.55	0.8	0.27	2.48	1.88	2.42	2.3	0.27	3.08	3.02	2.97	3.0	0.04
1902	0.72	0.68	0.47	0.6	0.11	2.80	2.63	2.33	2.6	0.20	3.52	3.30	2.80	3.2	0.30
		i	1			1	ŀ								1
1908	0.38	0.10	0.40	0.3	0.14	1.43	1.50	1.52	1.5	0.04	1.80	1.60	1.92	1.8	0.13
1914	0.72	0.55	0.82	0.7	0.11	2.30	2.90	2.88	2.7	0.28	3.02	3.45	3.70	3.4	0.28
1920	0.65	1.00	0.90	0.9	0.15	3.02	3.52	3.15	3.2	0.21	3.67	4.53	4.05	4.1	0.35
1926	0.38	0.40	0.45	0.4	0.03	0.90	1.00	1.40	1.1	0.22	1.27	1.40	1.85	1.5	0.25
1932	0.42	0.55	0.72	0.6	0.12	2.20	2.73	2.38	2.4	0.22	2.63	3.28	3.10	3.0	0.27
3)	ļ	}	ĺ		ļ	,								Ì
1938	0.50	0.90	0.68	0.7	0.16	3.38	3.88	3.42	3.6	0.22	3.88	4.78	4.10	4.3	0.38
1944	1.05	0.97	0.80	0.9	0.10	2.65	2.75	2.58	2.7	0.07	3.70	3.73	3.38	3.6	0.16
1950	1.32	1.50	2.08	1.6	0.32	4.28	4.45	4.20	4.3	0.10	5.60	5.95	6.28	5.9	0.28
L	<u></u>	<u> </u>	<u> </u>	L		<u> </u>	<u> </u>						L		

Table A.8. Experiment 8645m, 4 weeks.

<u> </u>						Inso	lubles	s, mg	/100	mL					
ļ		Ad	herent					erab.					Total		
ł		Sample			Std		Sample	<u> </u>		Std		Sample			Std
Fuel	1	2	3	Avg	Dev	1	2	3	Avg	Dev	1	2	3	Avg	Dev
	1														
1879	1.05	0.80	1.48	1.1	0.28	5.63	4.10	4.13	4.6	0.71	6.67	4.90	5.60	5.7	0.73
1884	0.40	0.60	0.88	0.6	0.19	3.25	2.88	2.65	2.9	0.25	3.65	3.48	3.52	3.6	0.07
1890	1.45	1.32	1.13	1.3	0.13	1.60	1.55	1.52	1.6	0.03	3.05	2.88	2.65	2.9	0.16
1896	0.40	0.63	0.42	0.5	0.10	3.02	3.52	3.67	3.4	0.28	3.42	4.15	4.10	3.9	0.33
1902	1.10	1.05	1.23	1.1	0.07	5.22	4.85	5.33	5.1	0.20	6.32	5.90	6.55	6.3	0.27
]	[.	!				i i				ì	i		1		}
1908	0.65	0.65	0.35	0.6	0.14	3.13	3.48	3.48	3.4	0.16	3.77	4.13	3.83	3.9	0.15
1914	1.63	2.35	2.33	2.1	0.34	4.42	3.95	4.00	4.1	0.21	6.05	6.30	6.33	6.2	0.12
1920	1.18	0.95	0.90	1.0	0.12	4.83	4.85	4.85	4.8	0.01	6.00	5.80	5.75	5.9	0.11
1926	1.10	1.20	1.02	1.1	0.07	3.02	2.52	2.38	2.6	0.28	4.13	3.72	3.40	3.8	0.30
1932	0.53	0.68	0.40	0.5	0.11	2.90	2.70	2.63	2.7	0.12	3.42	3.38	3.02	3.3	0.18
}	}					ļ]	,	}]		
1938	1.00	0.97	1.10	1.0	0.05	3.63	4.08	3.65	3.8	0.21	4.63	5.05	4.75	4.8	0.18
1944	1.68	1.13	1.75	1.5	0.28		4.05		:				5.90		0.33
1950	1.95	2.10	2.02	2.0	0.06	4.58	1	I	i e	0.14			6.85	6.8	
	•		}			1		1					}	, -	

Table A.9. Experiment 8645m, 6 weeks.

						Insol	ubles	mg/	100 r	nL					
ľ		Ad	herent				Filte	erable	<u>e</u>			1	otal		
]		Sample			Std	S	ample			Std	S	ample		ł	Std
Fuel	1	2	3	Avg	Dev	1	2	3	Avg	Dev	1	2	3	Avg	Dev
								i	ł			}	}	1) .
1879	1.88	2.35	2.05	2.1	0.20	5.03	4.47	4.55	4.7	0.24	6.90	6.82	6.60	6.8	0.13
1884	1.25	1.25	1.38	1.3	0.06	3.48	3.52	3.33	3.4	0.08	4.72	4.78	4.70	4.7	0.03
1890	1.95	2.10	1.60	1.9	0.21	0.82	1.15	0.95	1.0	0.13	2.77	3.25	2.55	2.9	0.29
1896	0.70	0.80	0.68	0.7	0.05	3.90	3.73	4.63	4.1	0.39	4.60	4.53	5.30	4.8	0.35
1902	1.40	2.35	2.15	2.0	0.41	5.38	5.30	6.33	5.7	0.47	6.78	7.65	8.47	7.6	0.69
	1														
1908	0.88	1.20	0.60	0.9	0.25	3.98	3.90	4.50	4.1	0.27	4.85	5.10	5.10	5.0	0.12
1914	2.38	2.60	3.05	2.7	0.28	4.15	3.88	4.80	4.3	0.39	6.53	6.47	7.85	7.0	0.64
1920	1.25	1.57	1.48	1.4	0.14	4.83	4.90	6.20	5.3	0.63	6.08	6.48	7.68	6.7	0.68
1926	1.20	1.25	1.15	1.2	0.04	3.08	2.85	3.33	3.1	0.19	4.28	4.10	4.47	4.3	0.15
1932	0.68	0.82	0.47	0.7	0.14	3.05	3.00	3.08	3.0	0.03	3.72	3.83	3.55	3.7	0.11
ł	}	ŀ	ł	1	! !			ļ	1			[
1938	2.17	1.70	1.88	1.9	0.20	2.95	3.70	3.38	3.3	0.31	5.13	5.40	5.25	5.3	0.11
1944	1.93	2.38	2.45	2.3	0.23	4.22	3.83	4.28	4.1	0.20	6.15	6.20	6.73	6.4	0.26
1950	2.95	2.85	3.02	2.9	0.07	4.63	4.70	4.50	4.6	0.08	7.58	7.55	7.53	7.6	0.02
			<u> </u>												

Table A.10. Experiment 8645m, 8 weeks.

	Insolubles, mg/100 mL														
ł		Ad	Filterable					Total							
		Sample	1		Std		Sample	•	Std		Sample				Std
Fuel	1	2	3	Avg	Dev	1	2	3	Avg	Dev	1	2	3	Avg	Dev
}			1												
1879	3.92	2.38	3.15	3.2	0.63	4.30	5.53	5.50	5.1	0.57	8.22	7.90	8.65	8.3	0.31
1884	2.38	2.20	2.38	2.3	0.08	5.85	4.28	4.78	5.0	0.66	8.22	6.48	7.15	7.3	0.72
1890	2.95	2.40	2.65	2.7	0.22	0.40	0.80	0.93	0.7	0.22	3.35	3.20	3.58	3.4	0.15
1896	1.52	1.50	1.35	1.5	0.08	5.53	3.98	4.53	4.7	0.64	7.05	5.47	5.88	6.1	0.67
1902	3.48	2.83	2.08	2.8	0.57	7.05	5.97	6.17	6.4	0.47	10.50	8.80	8.25	9.2	0.97
1	1)	}		[]		1							
1908	1.98	2.38	1.18	1.8	0.50	7.22	3.90	4.00	5.0	1.54	9.20	6.28	5.17	6.9	1.70
1914	3.23	4.30	3.85	3.8	0.44	4.80	3.95	3.20	4.0	0.65	8.03	8.25	7.05	7.8	0.52
1920	1.82	1.50	1.60	1.6	0.14	5.30	5.72	3.35	4.8	1.03	7.13	7.22	4.95	6.4	1.05
1926	1.23	1.73	2.67	1.9	0.60	3.35	3.13	3.02	3.2	0.14	4.58	4.85	5.70	5.0	0.48
1932	1.18	0.68	0.88	0.9	0.21	3.23	3.00	3.27	3.2	0.12	4.40	3.67	4.15	4.1	0.30
}	1		ļ		{	}		j	1	[
1938	2.08	2.10	1.57	1.9	0.24	3.15	5.88	3.17	4.1	1.28	5.22	7.97	4.75	6.0	1.42
1944	3.17	2.73	2.35	2.8	0.34	4.55	4.75	5.05	4.8	0.21	7.72	7.47	7.40	7.5	0.14
1950	4.08	3.40	3.67	3.7	0.28	5.13	5.38	5.17	5.2	0.11	9.20	8.78	8.85	8.9	0.19
			.	l		L				L					İ

Table A.11. Experiment 8647, 4 weeks.

<u> </u>	Insolubles, mg/100 mL														
]	Adherent							terab	le_		Total				
i i		ample			Std		Sampl			Std	~	ample			Std
Fuel	1	2	3	Avg	Dev	1	2	3	Avg	Dev	Ĺ	2	3	Avg	Dev
2059	0	o	0	0	0 01	0 15	0 17	0.07	0 1	0.04	0.15	0.15	0.07	0 1	0.04
2060	Lost	•			0.15					0.06	Lost	0.47	0.05		0.21
2061		0.20			0.04						0.38	0.35	0.25		0.05
2062		7.05			3.15						0.50	8.80	0.93		3.82
2063		0.40			0.15						0.43	0.80	1.75		0.56
2064	0.28	4.30	0.25	1.6	1.90	0.20	2.63	0.25	1.0	1.13	0.48	6.92	0.50	2.6	3.03
2065		0.85	1.20	1.0	0.14	0.40	0.65	0.47	0.5	0.10	1.48	1.50	1.67	1.6	0.09
2066	1.52	1.23			0.15						3.57	3.63	3.90	3.7	0.14
2067	l .	0.80			0.14						1.20	0.93	1.13		0.12
2068	1.57	1.35	1.50	1.5	0.09	0.65	0.78	0.80	0.7	0.07	2.23	2.13	2.30	2.2	0.07
2069		1.23			0.08						1.70	2.08	1.63		0.20
2070		0.88			0.29						2.13	1.50	1.45		0.31
2071		0.70			0.19						1.38	0.82	0.85		0.25
2072 2073		1.90 2.38			0.47						4.00	3.77 2.73	4.15 1.85		0.15 0.48
20/3	1.32	2.36	1.43	1.7	0.47	0.28	0.33	0.40	0.3	0.03	1.60	2.73	1.65	2.1	0.48
2074		1.55			0.20						1.55	2.13	1.70		0.24
2075		0.72			0.12						0.80	0.85	0.60		0.11
2076		1.13			0.19						2.10	1.50	1.63		0.26
2077		1.15			0.07						3.22	2.92	3.17		0.13
2078	1.60	2.27	2.27	2.1	0.32	2.15	1.98	1.70	1.9	0.19	3.75	4.25	3.97	4.0	0.20
2079		1.15			0.05						2.73	2.65	2.42		0.13
2080		2.10			0.15						4.13	4.50	3.95		0.23
2081		2.23			0.28						2.63	3.13	3.02		0.22
2082		0.95			0.67						2.33	1.05	0.95		0.63
2083	0.45	0.40	0.33	0.4	0.05	1.18	1.48	1.32	1.3	0.12	1.63	1.88	1.65	1.7	0.11
2084		2.10			0.87						2.67	2.73	4.53	3.3	0.86
2085	2.02	2.05			0.15						5.13	3.95	5.15	1 1	0.56
2086		3.10								0.51		5.45	4.60		0.41
2087		1.52								0.23	2.60	2.72	2.60		0.06
2088	1.18	4.63	3.13	3.0	1.41	2.17	2.38	2.23	2.3	0.08	3.35	7.00	5.35	5.2	1.49
2089		1.45			0.16						3.38	3.53	3.10	3.3	0.18
2090		2.02			0.26						3.20	3.67	3.42		0.19
2091		1.93			0.23						1.75	2.33	1.85		0.25
2092		1.00			0.08						2.55	2.90	2.67		0.14
2093	1.70	2.00	1.95	1.9	0.13	1.57	2.75	3.08	2.5	0.64	3.27	4.75	5.03	4.4	0.77
2094	1.82		1.80								7.30	7.70	7.10	1	0.25
2095	0.88				0.17						2.63	2.80	2.65		0.08
2096	1.48				0.37						3.77	6.80	4.00		1.38
2106		1.90 1.35			0.22						6.60	4.45	6.78		1.06
2107	2.40	1.35	1.02	T . 0	0.59	<u> </u>	2.13	2.1/	14.9	11.05	6.78	3.48	3.20	4.5	1.62

Table A.12. Experiment 8647, 8 weeks.

[Insolubles, mg/100 mL														
Ì	Adherent					Filterable				Total					
		ample			Std		Sample			Std		Sample			Std
Fuel	11	2	3	Avg	Dev	1	2	3	Avg	Dev	1	2	3	Avg	Dev
2059	0.10	0.07	0.05	0.1	0.02	0.20	0.13	0.10	0.1	0.04	0.30	0.20	0.15	0.2	0.06
	0.82			0.3						0.01	1.07	0.55	0.40		0.08
1	1.07		· ·	0.7						0.07	1.27	0.38	18.90		0.45
i .	1.20			0.6						0.04	1.40	0.63	0.43		0.42
2063	1.27	1.10	3.48	1.2	0.09	1.45	1.60	2.40	1.5	0.08	2.72	2.70	5.88	2.7	0.01
	1.00			2.8				0.95			1.32	0.80	7.95		3.25
				3.2						0.47		5.38	4.30		0.48
2066				2.5						0.22		5.38	5.30		0.20
	1.95			1.7						0.11	2.30	1.60	1.68		0.31
2068	2.70	1.93	1.77	2.1	0.41	1.45	1.5/	1.2/	1.4	0.12	4.15	3.50	3.05	3.6	0.45
2069	3.52	3.00	2.52	3.0	0.41	2.23	0.88	1.07	1.4	0.59		3.88	3.60		0.96
	3.13			3.2						0.31	3.88		3.80		0.44
	1.70			1.9						0.13		1.83	2.48		0.27
	4.63			3.6						0.25		5.10	5.70		0.87
20/3	2.98	2.02	2.73	2.6	0.40	0.53	1.20	0.50	0.7	0.32	3.50	3.22	3.23	3.3	0.13
	2.17			2.3						0.53	3.90		3.05		0.43
	0.90			1.1						0.04		1.57	1.50		0.14
	2.63			2.2						0.34		i e	2.52		0.51
	2.92			2.2						0.05		1			1.16
2078	6.20	5.85	4.80	5.6	0.59	1.40	1.07	1./3	1.4	0.27	7.60	6.92	6.53	7.0	0.44
	2.10			1.9						0.06			3.45		0.23
	3.45			2.8						0.24			i e		0.33
	3.48			4.2						0.09	1		1		0.69
	1.50 3.10			1.4 2.8						0.07		1.65 5.08	1.70		0.08
2005	3.10	3.13	2.20	2.0	0.44	1.05	1.93	1.02	1.3	0.37	4.72	3.00	3.23	4.3	0.60
			3.30	3.7						0.20				4.4	0.57
	4.65			4.3		3.75	3.58	3.42	3.6	0.13			1		0.34
			6.40	5.5						0.73	(1	i		0.18
			2.80							0.45		1	1		0.57
2088	3.27	2.95	2.65	3.0	U.26	2.98	3.23	2.63	2.9	0.25	6.25	6.18	5.28	15.9	0.44
			3.08		0.18					0.18		5.97	5.90	5.9	0.06
			4.42		0.48					0.25			t .	•	0.23
	2.33			1 1	0.34					0.09			1	1	0.25
	2.05				0.37					0.24			1		0.61
2093	3.85	4.20	4.25	4.1	0.18	2.65	2.90	2.85	2.8	0.11	6.50	7.10	7.10	6.9	0.28
1	3.48				0.52							10.40	1	10.0	0.69
	2.20				0.08					0.07	1				0.11
	2.83				0.59					0.03				6.3	0.61
	2.65				0.85					0.81		10.60		10.0	L
210/	2.23	2.58	2.50	2.4	0.15	2.75	2.70	2.58	2.7	0.07	4.97	5.28	5.08	5.1	0.12

REFERENCES

- Klinkhammer, M.D., and R.E. Morris, "FY 1985 Fuel Stability Additive Evaluation Program: Collateral Studies on Fuel Characteristics," DTNSRDC Report SME-86/15 (Oct 1986).
- 2. White, E.W., DTRC, Written Communication.
- 3. Hardy, D.R., E.J. Beal, and R.N. Hazlett, "Evaluation of Commercial Stability Additives for Naval Distillate Fuels," <u>Proceedings of the 3rd International Conference on Stability and Handling of Liquid Fuels.</u> Institute of Petroleum, London, pp. 399-411 (13-16 Sep 1988).

INITIAL DISTRIBUTION

Copies		Copies	
1	OCNR (Code 123) (Dr. A. Roberts)	1	National Research Council of Canada (P.L. Strigner) Div of Mechanical Engineering
3	NAVSEA 1 SEA 05M32		Ottawa, Ontario, Canada KIA OR6
2	2 Library NAVPETOFF (L. Long)	1	ARTECH Corporation 14554 Lee Road Chantilly, VA 22021
1	NAVSSES PHILA (Code 053)	1	NIPER (K.W. Sterling)
3	NAPC (Code PE 33) 1 C.J. Nowack	25	IEP/ABCA/3/UK
	1 G. Speck 1 L.M. Turner	15	IEP/ABCA/3/Canada
5	NRL (Code 6180) 1 Dr. D. Hardy 1 Dr. R. Morris 1 E.J. Beal 1 Dr. R.N. Hazlett 1 Dr. B.H. Black	10	IEP/ABCA/3/Australia
2	DFSC 1 Code T (C. Martin) 1 Code Q (W. Carley)		
1	AFWAL Code AFWAL/POSF (S. Anderson)		
12	DTIC		
1	CNA		
3	Southwest Research Institute 1 L. Stavinoha 1 S. Westbrook 1 Dr. G. Fodor		
2	USA BRADC 1 Code STRBE-VF (M. LePera) 1 Code STRBE-VFF (J. Mengenhauser)	,	
1	E.I. duPont Company (Dr. C.P. Henry Petroleum Laboratory Chambers Works Deepwater, NJ 08023	")	

INITIAL DISTRIBUTION (Continued)

CENTER DISTRIBUTION

Copies	Code	Name
1	2759	W. Stoffel
3	2759	R. Strucko
1	28	G. Wacker
1	2801	J. Crisci
1	2801	D. Ventriglio
1	2802	T. Morton
1	2803	J. Cavallaro
1	2809	A. Malec
1	281	J. Gudas
3	283	H. Singerman
3	2832	T. Daugherty
10	2832	Dr. E.W. White
1	2832	Dr. M. Klinkhammer
1	2832	D. Smith
1	284	E. Fischer
1	342	Unclass Lib (A)
2	3431	Office Services