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LEVEL II

**A FILTERABILITY STUDY OF  
CORROSION INHIBITED JP-4**

*FUELS AND LUBRICATION DIVISION  
FUELS BRANCH*

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TECHNICAL REPORT AFAPL-TR-78-44  
Final Report August 1974 - June 1976

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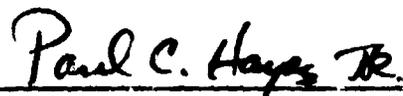
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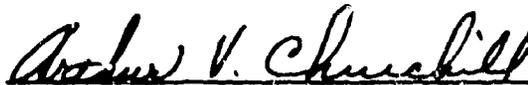
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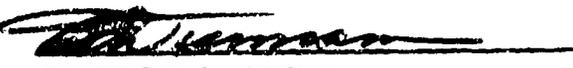


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REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM	
1. REPORT NUMBER AFAPL-TR-78-44 ✓	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER	
4. TITLE (and Subtitle) A Filterability Study of Corrosion Inhibited JP-4.	5. AUTHOR(s) C. Paul Hayes, Jr	6. PERFORMING ORGANIZATION NAME AND ADDRESS AF Aero Propulsion Lab AFAPL/SFF, WPAFB OH 45433	7. CONTROLLING OFFICE NAME AND ADDRESS Fuels and Lubrication Division (SF) Air Force Aero Propulsion Laboratory Wright-Patterson Air Force Base, OH 45433
8. PERFORMING ORGANIZATION NUMBER	9. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS 6203F 3048 304805, 30480591	10. NUMBER OF PAGES 62	11. SECURITY CLASS. (of this report) UNCLASSIFIED
12. DISTRIBUTION STATEMENT (of this Report) Approved for public release; distribution unlimited.		13. DECLASSIFICATION/DOWNGRADING SCHEDULE	
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)			
15. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)			
16. SUPPLEMENTARY NOTES			
17. KEY WORDS (Continue on reverse side if necessary and identify by block number) JP-4 Jet Fuel                      Fuel System Icing Inhibitor Filtration Time                      Water Bottoms Filterability Corrosion Inhibitors			
18. ABSTRACT (Continue on reverse side if necessary and identify by block number) The filterability of JP-4 fuel indicates the extent of contamination with solid particulate matter, refinery carry-overs, and/or reaction products generated from the mixing of inhibited fuel and water bottoms. This report examines the creation of filter-plugging precipitants via the interaction of a fuel corrosion inhibitor with entrained water. Further work revealed methods to prevent the formation of the reaction product and steps to reclaim such a synthetically created high filtration time JP-4.			

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SUMMARY Sept. AUG 74 - JUNE 78

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FOREWORD

This report was prepared by the Fuels Branch, Fuels and Lubrication Division, Air Force Aero Propulsion Laboratory (AFAPL/SFF) under Project 3048, Task 30480591, "Aero-Propulsion Fuels." The work was performed in-house in AFAPL facilities. The Air Force project leader for this work was Paul C. Hayes, Jr. Valuable technical assistance in the laboratory work was provided by Mr. Ralph Wagner. The time period covered by the work was August 1974 through June 1976. This report was submitted by the Author in May 1978.

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SECTION I  
INTRODUCTION

1. Background

For years the premature failure of filter-separator elements has caused serious disruptions in jet fuel bulk storage and distribution systems throughout the Air Force. Most of the problems were traced to inadequate refinery procedures, alleged improper handling and inspection of tanker loadings, and poor housekeeping and handling techniques at support terminals and air bases. Apparently the blame does not rest with the type of lining in tankers and barges since coated vessels exhibited just as poor a fuel quality record as uncoated ones. A filtration time test was devised by the Directorate of Aero-Space Fuels, San Antonio Air Logistics Center, to monitor the tendency of JP-4 fuel to clog filter separators. These test results correlated with filter-separator failures in the field. The filtration time test has subsequently been refined and in its present form requires one gallon of JP-4 to pass through a 0.8 micron filter membrane in less than 15 minutes under a prescribed set of laboratory conditions (Reference 1; Appendix A).

JP-4 fuel contains a corrosion inhibitor to control corrosion in pipelines and to improve the fuel's lubricating qualities. If the JP-4 comes into intimate contact with water, especially sea water, a gelatinous precipitant may be formed that can plug filters and cause a high filtration time fuel. This has been a recurrent problem with JP-4 shipped by barge and tanker.

Work by the Naval Research Laboratory describes gelatinous precipitants that plugged filter membranes (Reference 2). This contaminant formed in fuel containing a corrosion inhibitor that came in contact with synthetic sea water and bare metal. Although not isolated and rigorously identified, it was theorized that metal oxides

and/or heavy metal ions can react with fatty acid-type corrosion inhibitors to form membrane-clogging metal "soaps."

Previous work by the AFAPL Laboratory had resulted in a method to artificially create the filter-plugging precipitant (Reference 3). JP-4 fuel stock, clay-treated to remove polar contaminants and additives, was doped with a test corrosion inhibitor, a synthetic sea water bottom [as per American Society for Testing and Materials (ASTM) D665-135, Appendix B] and soft steel shim stock. This mixture was tumbled end-over-end periodically in a five gallon epoxy-lined can, and after a settling time of approx. 40 hours, the filtration time was determined. The short-comings of this procedure included very poor sample to sample filtration time repeatability and a week long reaction time.

Subsequently, ultrasonic agitation was selected as a possible substitute for the occasional but severe mixing that the fuel and water phases might experience in the field.

## 2. Project Objectives

The dual purpose of this work is to establish the components and conditions necessary to artificially create a high filtration time JP-4 and to investigate possible schemes to prevent or remove the resultant membrane-plugging metal soap. The first objective, therefore, was to identify the various combinations of ingredients and conditions crucial in producing the precipitant. The second objective was directed toward preventing the reaction or lowering the filtration time of a "soap"-contaminated JP-4 to below the 15 minute specification limit.

SECTION II  
TEST EQUIPMENT AND PROCEDURE

1. Filtration Time Test Method

The filtration time test method used for the tests discussed herein is detailed in Appendix A. The only procedural deviation was in not weighing the filter membranes before and after each filtration time run.

2. Generation of High Filtration Time Fuel

A non-additive JP-4 stock was clay-treated using Atapulgu clay to remove polar impurities as per ASTM D2550-A4. The reaction vessels employed were five gallon glass bottles which were washed several times with tap water, then "delimer", then tap water again, and finally rinsed with distilled water. Described below is the step-by-step procedure and the equipment used for generating a high filtration time JP-4:

- a. A five gallon glass bottle was filled with 15 liters of clay-treated JP-4 stock.
- b. The test corrosion inhibitor was added at the desired concentration and well mixed.
- c. Approximately 1.5 to 2 volume percent of ASTM synthetic sea water (D665-135; Appendix B) or hard WPAFB tap water was then added.
- d. Using the apparatus described in Figure 1, the fuel-water mixture was circulated at a rate of 125 ml/min via a peristaltic pump to and from an ultrasonic agitation chamber.
- e. After the "Tygon" and glass tubing sections had filled with alternating columns of phases, the ultrasonic probe (set at maximum power) was turned on.
- f. The circulation and agitation was continued for 30 minutes.

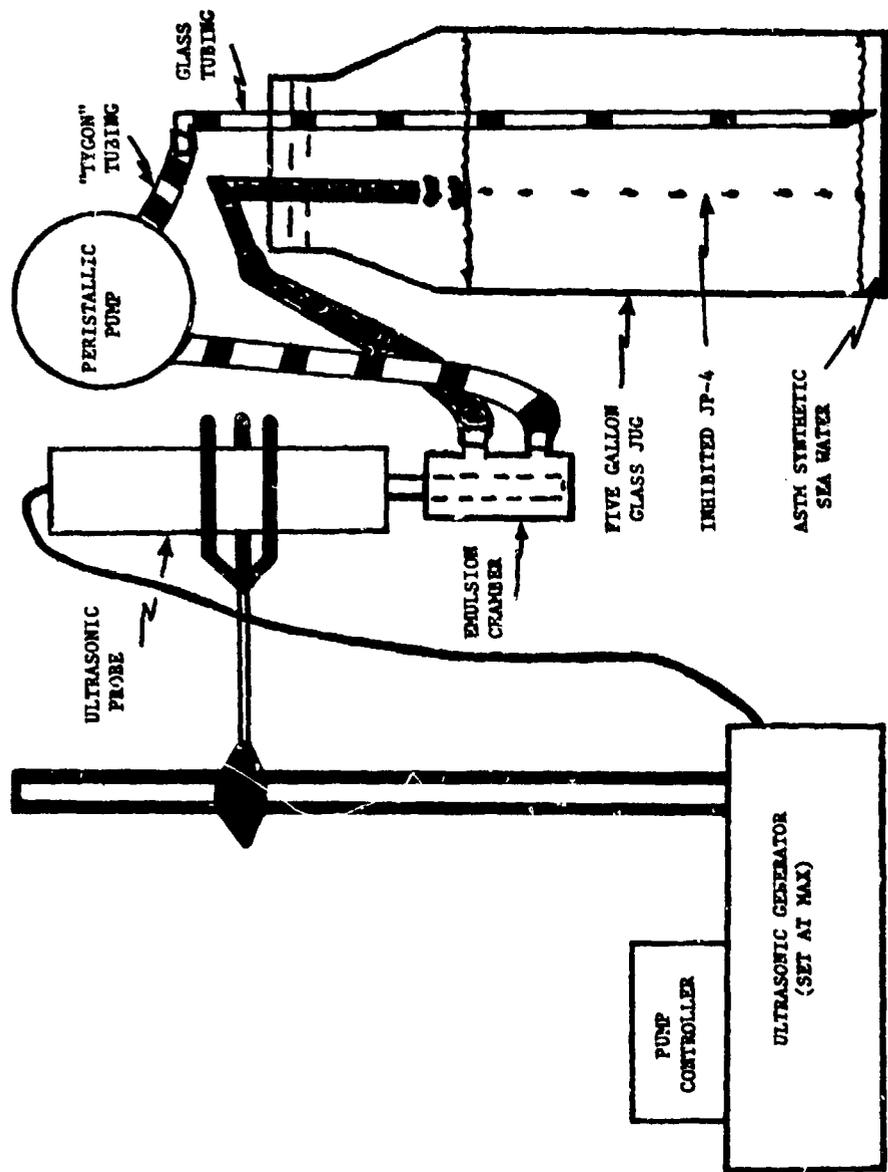


Figure 1. Ultrasonic Agitation and Circulation Apparatus

g. By the end of the agitation period, the fuel phase was a milky-white, translucent suspension of water droplets.

h. The glass jug was then transferred to a hood and kept there until sampled or re-agitated.

The following test equipment was utilized:

a. Circulation pump: "Master-Flex" pump and controller, Model 7017, 3200 rpm maximum; distributed by Cole-Parmer, 7425 North Oak Park Ave., Chicago, IL 60648.

b. Ultrasonic agitator: "Sonic Dismembrator," Model 150, Artek Systems Corp., 275 Adams Blvd., Farmingdale, NY 11735.

SECTION III  
DISCUSSION OF RESULTS

1. Control Runs

Control samples without a corrosion inhibitor or other additives but exposed to soft steel shim and ASTM sea water were prepared. The object was to determine what effect high levels of rust and entrained water in the JP-4 phase had on the filtration time of the fuel. Table I summarizes these data. The results are listed according to sample "bottle age," i.e., the time elapsed since the initial contact of the two phases. The "settling time" was the interval from the end of the bottle's last ultrasonic agitation until its sampling for a filtration test run. The filtration time tests were terminated at about 60 minutes, the fuel volume remaining was measured and listed in parenthesis next to the time in the "FILTRATION TIME (+XXX ml)" column. Three one-gallon samplings and filtrations were possible per bottle. Some bottles, as noted in the table, were not ultrasonically agitated at the time the fuel and water phases were initially mixed.

The data indicated that immediately following ultrasonic agitation of the fuel-water mixture, the entrained water in the clay-treated JP-4 would increase the filtration time by 4 to 5 minutes above the nominal 4 minute filtration time for JP-4 without a water bottom. The rate of rust formation was greatly accelerated when ultrasonic agitation was frequently performed. Eventually, rust particles would accumulate and plug the filter membrane of a one gallon filtration run, increasing the sample's filtration time well beyond the acceptable limit. However, a settling period of about 1 1/2 days was sufficient to return a sample's filtration to below 15 minutes.

2. HITEC E-515 (With Metal) Runs

Since HITEC E-515 has been a widely utilized corrosion inhibitor, its ability to create a high filtration time JP-4 was investigated first. Mixtures were

TABLE I. Filterability of Control Samples

Bottle I.D.	Bottle Age	Settling Time	Bottle Sampling	Filtration Time (+XXX ml)	Bottle Agitation History And Test Comments
I	1/2 hr	none	first	6.0 min	ultrasonically agitated on initial mixing of phases
I	1 1/2 hrs	1 hr	second	4.5 min	not ultrasonically agitated since last sampling
I	6 days	none	third	5.9 min	ultrasonically agitated immediately prior to this sampling
II	1/2 hr	none	first	7.1 min	ultrasonically agitated on initial mixing of phases
IV	7 days	none	first	60 min (+400 ml)	ultrasonically agitated on initial mixing of phases and periodically, noted thick rust deposit on filter membrane
V	7 days	none	first	8.3 min	never ultrasonically agitated until immediately prior to this sampling
V	16 days	none	second	19.1 min	ultrasonically agitated immediately prior to this sampling
VI	7 days	none	first	8.8 min	never ultrasonically agitated until immediately prior to this sampling
VI	9 days	36 hrs	second	4.0 min	ultrasonically agitated only once in its history (36 hrs ago)

Table I Test Constituents: a. Sanded soft steel shim stock (6" x 3")  
 surface area/fuel volume ratio =  $0.03 \text{ in}^2/\text{in}^3$   
 b. 250 ml ASTM D-665 synthetic sea water with pH 8.0 (about 2 volume percent fuel)  
 c. 15 liters of non-additive, clay-treated JP-4

prepared containing HITEC E-515 at its minimum effective concentration in JP-4 with soft steel shim and ASTM synthetic sea water present. Varying intervals of ultrasonic agitation and settling preceded each filtration run as outlined in Table II.

As in the control runs, each bottle's agitation history greatly influenced its resultant filtration time. As quickly as 2-3 hours after the initial ultrasonic agitation of inhibited fuel and sea water, a 1-hour filtration time JP-4 would occur. Mere pouring of the water through the fuel phase in the container, without efficient mixing, failed to generate a high filtration time fuel. Pre-rusted steel shim stock did not accelerate the formation of a problem fuel. In fact, subsequent HITEC E-515 experiments without steel shim revealed elemental metal not to be a crucial variable to "soap" formation. As the data in Table II indicates, the initial contribution of a metal surface and/or rust toward causing a high filtration time fuel was minor compared to the interaction of corrosion inhibitor and sea water.

### 3. FSII Runs

To circumvent ice formation as well as bacteriological growth, fuel system icing inhibitor\* (FSII) is added to JP-4 to a concentration of 0.10 to 0.15% of the fuel volume. Unavoidably, however, tank water bottoms leach out the alcohol-like FSII. Levels of this additive in water bottoms may accumulate to as high as 20-40% of the water volume. It was the intent of this phase of the JP-4 filterability investigation to evaluate the influence of FSII on the fuel-sea bottom interactions, but in the absence of rusting metal. This work is summarized in Table III.

Upon mixing the FSII into a sea water bottom, the 15% and 30% bottoms warmed and immediately turned milky. In the case of a 40% FSII water bottom, a white, flocculant precipitant formed. Repeated ultrasonic agitations of uninhibited fuel-water mixtures, followed by immediate filtrations indicated no filterability

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\* ethylene glycol, monomethyl ether

TABLE II. Filterability of HITEC E-515 Samples (W/Metal)

Bottle I.D.	Bottle Age	Settling Time	Bottle Sampling	Filtration Time (+XXX ml)	Bottle Agitation History And Test Comments
VII	1/2 hr	none	first	7.2 min	ultrasonically agitated on initial mixing of phases
VII	3 1/2 hrs	none	second	60 min (+800 ml)	ultrasonically agitated immediately prior to this sampling
VIII	1/2 hr	none	first	6.1 min	ultrasonically agitated on initial mixing of phases
VIII	4 days	none	second	60 min (+1000 ml)	ultrasonically agitated immediately prior to this sampling
VIII	18 days	none	third	12.9 min	ultrasonically agitated immediately prior to this sampling; noted thick rust deposit on filter membrane
IX	1/2 hr	none	first	5.6 min	ultrasonically agitated on initial mixing of phases
IX	2 1/2 hrs	none	second	60 min (+920 ml)	ultrasonically agitated immediately prior to this sampling
X	1 1/2 hrs	none	first	25.6 min	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling
XI	1 day	none	first	60 min (+1425 ml)	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling
XII	1 day	1 day	first	4.3 min	never ultrasonically agitated
XII	6 days	none	second	40.4 min	ultrasonically agitated immediately prior to this sampling and 3 days ago

Table II Con't

Bottle I.D.	Bottle Age	Settling Time	Bottle Sampling	Filtration Time (+XX.X min)	Bottle Agitation History And Test Comments
XII	8 days	2 days	third	18.7 min	not ultrasonically agitated since last sampling 2 days ago
XIII	3 days	none	first	23.4 min	not ultrasonically agitated on initial mixing of phases; ultrasonically agitated immediately prior to this sampling
XIII	8 days	1 day	second	22.8 min	ultrasonically agitated 1 day ago prior to this sampling
XIII	9 days	2 days	third	5.5 min	ultrasonically agitated 2 days prior to this sampling
XIV	3 days	3 days	first	4.2 min	never ultrasonically agitated
XIV	7 days	1 hr	second	60 min (+540 ml)	ultrasonically agitated 1 hour ago prior to this sampling
XV	6 days	6 days	first	4.0 min	never ultrasonically agitated
XV	7 days	7 hrs	second	57.5 min	ultrasonically agitated 7 hours ago prior to this sampling
XV	8 days	1 3/4 days	third	10.0 min	ultrasonically agitated 1 3/4 days ago prior to this sampling
XVI	7 days	none	first	7.1 min	never ultrasonically agitated, merely circulated periodically via peristaltic pump
XVI	16 days	none	second	3.6 min	never ultrasonically agitated, merely circulated periodically via peristaltic pump
XVII	7 days	none	first	43.1 min	never ultrasonically agitated until immediately prior to this sampling
XVII	17 days	none	second	30.2 min	ultrasonically agitated immediately prior to this sampling; noted thick rust deposit on filter membrane

Table II Test Constituents: a. sanded soft steel shim stock (5" x 3")  
surface area/fuel volume ratio = 0.03 in<sup>2</sup>/in<sup>3</sup>

b. 250 ml ASTM D-665 synthetic sea water with pH 8.0 (2 volume percent fuel)

c. HITEC E-515 at minimum effective concentration in 15 liters of clay-treated JP-4

TABLE III. Filterability of HITEC E-515 With FSII

Bottle I.D.	Bottle Contents	Bottle Age	Settling Time	Bottle Sampling	Filtration Time (+XXX ml)	Bottle Agitation History And Test Comments
XXB	no HITEC E-515 present; 0.1 Vol % FSII in fuel; 15 Vol % FSII in syn sea water bottom.	1 day	none	first	5.33 min	ultrasonically agitated on initial mixing of phases; water bottom warmed and turned cloudy.
XXBBB	no HITEC E-515 present; no FSII in fuel; no FSII in syn sea water bottom.	1 day	none	first	5.20 min	ultrasonically agitated on initial mixing of phases.
XXIB	no HITEC E-515 present; 0.1 Vol % FSII in fuel; 30 Vol % FSII in syn sea water bottom.	1 day	none	first	5.47 min	ultrasonically agitated on initial mixing of phases; water bottom warmed and turned cloudy.
		2 days	none	second	5.44 min	ultrasonically agitated immediately prior to this sampling.
XXIIBB	no HITEC E-515 present; no FSII in fuel; 40 Vol % FSII in syn sea water bottom.	2 1/2 hrs	none	first	8.78 min	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling; water bottom warmed and white, flocculent precipitant formed during initial mixing step.
XXII	HITEC E-515 present; no FSII in fuel; 40 Vol % FSII in distilled water bottom.	2 1/2 hrs	none	first	6.42 min	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling precipitant appeared in water bottom during initial mixing.
		1 day	none	second	9.41 min	ultrasonically agitated immediately prior to this sampling.

TABLE III Con't

Bottle I.D.	Bottle Contents	Bottle Age	Settling Time	Bottle Sampling	Filtration Time (+XXX ml)	Bottle Agitation History And Test Comments
XXIII	HITEC E-515 present; no FSII in fuel; 40 Vol % FSII in distilled water bottom.	2 1/2 hrs	none	first	5.83 min	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling; no warming or precipitant appeared in water bottom buffered to pH = 6.0 during initial mixing.
XXIV	HITEC E-515 present; no FSII in fuel; no FSII in syn sea water bottom.	2 1/2 hrs	none	first	39.99 min	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling.
XXV	HITEC E-515 present; no FSII in fuel; 20 Vol % FSII in syn sea water bottom.	1 day	none	second	50 min (+800 ml)	ultrasonically agitated immediately prior to this sampling.
XXVI	HITEC E-515 present; no FSII in fuel; 40 Vol % in syn sea water bottom.	2 1/2 hrs	none	first	55 min (+515 ml)	ultrasonically agitated on initial mixing of phases.
		2 1/2 hrs	none	first	17.13 min	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling; white, flocculent precipitant formed during initial mixing step.

Table III Test Constituents: a. HITEC E-515 (when present) at minimum effective concentration in 15 liters of clay-treated JP-4.

b. All distilled water bottoms, unless marked otherwise, buffered to pH = 8.0 with Na<sub>2</sub>CO<sub>3</sub>

problem. The precipitant was not fuel soluble and, consequently, did not influence the filtration time.

In other experiments, a distilled water bottom was selected instead of synthetic sea water so that no heavy metal ions could react with the FSII or with the corrosion inhibitor (when present). Also, the water bottom was adjusted to a basic pH to ensure dissociation of the active agent of the HITEC E-515, namely an aliphatic (fatty) di-acid. No fuel suspendable, filter-plugging product ever formed as was evidenced by the low filtration times.

It is believed that heavy metal ions probably reacted with the alcohol portion of the FSII. However, this fuel-insoluble precipitant demonstrated no JP-4 filtration effects. It was concluded that FSII in the fuel, in the water, or in both phases had no influence on JP-4 filterability, even under the severe conditions tested.

#### 4. Water Type

The next variable investigated was the effect of water bottom type on JP-4 filterability. Previous studies (Reference 3) indicated water bottom hardness affected filter plugging tendencies. Distilled water, Wright-Patterson AF Base tap water\*, and ASTM synthetic sea water\*, were used with HITEC E-515 as the JP-4 corrosion inhibitor.

Table IV summarizes the water bottom effects. It is theorized that heavy metal ions, i.e.,  $Mg^{+2}$  and  $Ca^{+2}$  in particular, react with fatty acid-type corrosion inhibitors to form metal "soaps" that are fuel-suspendable and plug filter separators. The absence of these ions should preclude any such reaction or filtration time problem. This idea was substantiated when distilled water bottoms, void of heavy metal ions, could not generate a high filtration time JP-4. Both the ASTM synthetic sea water and WPAFB tap water caused a high filtration time fuel in only a couple of hours under identical agitation conditions.

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\* analyzed by SFQLA, WPAFB, OH (See Appendix C)

TABLE IV. Filterability of HITEC E-515 With Different Water Types

Bottle I.D.	Bottle Contents	Bottle Age	Settling Time	Bottle Sampling	Filtration Time (+XXX ml)	Bottle Agitation History And Test Comments
XXXIV	HITEC E-515 present; 250 ml syn sea water bottom; pH = 8.0	1 day	none	first	50 min (+1280 ml)	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling.
XXXVI	HITEC E-515 present; 250 ml syn sea water bottom; pH = 8.0	1 day	none	first	50 min (+1400 ml)	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling.
XL I	HITEC E-515 present; 300 ml syn sea water bottom; pH = 8.0	1 day	none	first	60 min (+1000ml)	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling.
XLII	HITEC E-515 present; 300 ml WPAFB tap water bottom; pH = 7.4	3 1/2 hrs	none	first	64.16 min	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling.
XL	HITEC E-515 present; 245 ml distilled water bottom; pH = 6.0	1/2 hr	---	---	---	added 10 ml syn sea water then ultrasonically agitated phases.
		1 day	none	first	5.23 min	ultrasonically agitated immediately prior to this sampling.
		2 days	---	---	---	added 25 ml more of syn sea water and ultrasonically agitated phases.
		3 days	none	second	5.06 min	ultrasonically agitated immediately prior to this sampling; then added 25 ml more of syn sea water.
		4 days	none	third	11.68 min	ultrasonically agitated immediately prior to this sampling.

TABLE IV. Con't

Bottle I.D.	Bottle Contents	Bottle Age	Settling Time	Bottle Sampling	Filtration Time (+XXX ml)	Bottle Agitation History And Test Comments
XXXB	HITEC E-515 present; 240 ml distilled water bottom; pH made basic	1/2 hr	---	---	---	added 10 ml syn sea water, then ultrasonically agitated phases.
		1 day	none	first	4.09 min	ultrasonically agitated immediately prior to this sampling; then added 90 ml more of syn sea water.
		2 days	none	second	50 min (+275 ml)	ultrasonically agitated immediately prior to this sampling.
XXXBS	HITEC E-515 present; 215 ml distilled water bottom; pH made basic	1/2 hr	---	---	---	added 35 ml syn sea water, then ultrasonically agitated phases.
		1 day	none	first	6.05 min	ultrasonically agitated immediately prior to this sampling; then added 25 ml more of syn sea water.
		2 days	none	second	21.09 min	ultrasonically agitated immediately prior to this sampling.

TABLE IV Test Constituents: a. HITEC E-515 at minimum effective concentration in 15 liters of clay-treated JP-4.

Other experiments were conducted on inhibited fuels whose distilled water bottoms were doped every 24 hours with additional volumes of synthetic sea water and ultrasonically agitated after test samplings were taken. The comparatively low concentration of HITEC E-515 was constant for all of these mixtures (30 ppm). In this way, the inhibitors were made the limiting reagent in the soap-forming reaction. Increasing the amount of synthetic sea water in the distilled water bottoms revealed a threshold concentration of sea water that could cause filterability problems. From Table V, a re-grouping of data from Table IV, a distilled water bottom altered with approximately 20-30 volume percent synthetic sea water could generate a high filtration time JP-4 after ultrasonic agitation. The synthetic sea water used in the above series was analyzed <sup>and found</sup> by ~~CFDA~~ to contain 730 ppm calcium and 720 ppm magnesium. A 25 volume percent synthetic sea water in a distilled water bottom therefore had a combined divalent metal ion concentration of about 360 ppm. The WPAFB tap water was also analyzed to have 235 ppm calcium and 61 ppm magnesium with a combined divalent ion concentration of nearly 300 ppm. Further experiments using distilled water bottoms doped only with magnesium or calcium oxide and adjusted to a pH of 8.0 with NaOH revealed even lower heavy metal ion concentrations could generate a high filtration time JP-4. Filterability problems resulted when there was less than 100 ppm of either calcium or magnesium ions. Therefore, given efficient intermixing of inhibited fuel and water phases, a relatively low concentration of heavy metal ions, particularly magnesium and/or calcium, could produce a high filtration time JP-4 even in distilled water bottoms.

Only a limited effort was directed in finding the minimum percentage of water bottom volume of any water type necessary to create a high filtration time fuel. As was stated, 1.5 - 2.0 water volume percent was normally used, but synthetic

TABLE V. Extraction of TABLE IV Data for Sea Water Volume Effect

<u>Bottle I.D.</u>	<u>Varying Bottoms Composition</u>			<u>Actual Vol %</u>	<u>Filtration Time</u>
	<u>Total Syn Sea Volume</u>	<u>Total Bottoms Volume</u>	<u>Syn Sea Water</u>	<u>(+XXX ml)</u>	
XXXI	10 ml	in 255 ml	4%	5.23 min	
	35 ml	in 280 ml	13%	5.06 min	
	60 ml	in 305 ml	20%	11.68 min	
XXXB	10 ml	in 250 ml	4%	4.09 min	
	100 ml	in 340 ml	30%	50 min (+275 ml)	
XXXBS	35 ml	in 250 ml	14%	6.05 min	
	60 ml	in 275 ml	22%	21.09 min	
XXXIV	250 ml	in 250 ml	100%	50 min (+1280 ml)	

TABLE V Test Constituents: a. Water bottom variations of synthetic sea water in distilled water  
 b. HITEC E-515 at minimum effective concentration in 15 liters of clay-treated JP-4

sea bottoms as low as 1/4 volume percent (or 35 mls bottom per 15 liters JP-4) were successful in generating a fuel whose filtration time exceeded the 15 minute limit.

#### 5. Water Bottom pH

A crucial factor influencing soap formation is the pH of the water bottom. It was theorized that since many corrosion inhibitors are fatty acids, an acidic pH would retard their dissociation and, consequently, block a reaction with heavy metal ions. Table VI summarizes the experimental data generated to test this hypothesis.

As with other experiments, the synthetic sea water bottoms were 1.5 - 2.0 volume percent of the HITEC E-515 inhibited JP-4. The pH of the water bottoms was adjusted with  $\text{Na}_2\text{CO}_3$  for more basicity or  $\text{HCl}$  for more acidity. The ASTM synthetic sea water bottoms of all previous filterability tests had had a pH of 8.0 prior to agitation.

Neutral or acidic water bottoms prevented the formation of the filter-plugging soap. On the other hand, a slightly basic water bottom readily generated a high filtration time JP-4.

Unfortunately, attempts to quickly disintegrate the reaction product by acidifying the water bottom, agitating ultrasonically, and immediately running a filtration time test proved fruitless. For example, Bottle LI had an initial water bottom pH adjusted to 10.0 prior to its first agitation and circulation. As a bottle age of one day, its pH was retested and found to be 8.95. The water bottom was acidified by  $\text{HCl}$  addition to  $\text{pH} = 5.50$ . The fuel/water phases were then agitated ultrasonically and circulated for 1/2 hour. At a bottle age of two days, the water bottom's pH had to be readjusted from 7.90 back to 5.40. The bottle was then agitated and circulated for 1/2 hour and a one gallon sample filtered immediately afterwards. No significant decrease in the filtration time was observed.

TABLE VI. Filterability of HITEC E-515 with Different Water Bottom pH's

Bottle I.D.	Bottle Contents	Bottle Age	Settling Time	Bottle Sampling	Filtration Time (+XXX ml)	Bottle Agitation History And Test Comments
LV	HITEC E-515 present; 250 ml syn sea water bottom.	2 1/2 hrs	none	first	7.20 min	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling; initial bottom pH = 4.0
LIV	HITEC E-515 present; 250 ml syn sea water bottom.	2 1/2 hrs	none	first	7.04 min	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling; initial bottom pH = 6.0
XLII	HITEC E-515 present; 300 ml syn sea water bottom.	3 1/2 hrs	none	first	6.03 min	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling; initial bottom pH = 6.0
LIII	HITEC E-515 present; 250 ml syn sea water bottom.	2 1/2 hrs	none	first	7.55 min	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling; initial bottom pH = 7.0
XLIII	HITEC E-515 present; 300 ml MPAFB tap water bottom.	3 1/2 hrs	none	first	64.16 min	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling; initial bottom pH = 7.4
LII	HITEC E-515 present; 250 ml syn sea water bottom.	2 1/2 hrs	none	first	55 min (+1950 ml)	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling; initial bottom pH = 8.0
		1 day	none	second	52.86 min	found pH = 7.0, then adjusted to 5.20 and ultrasonically agitated immediately prior to this sampling.

TABLE VI CON't

<u>Bottle I.D.</u>	<u>Bottle Contents</u>	<u>Bottle Age</u>	<u>Settling Time</u>	<u>Bottle Sampling</u>	<u>Filtration Time (+XXX ml)</u>	<u>Bottle Agitation History And Test Comments</u>
LI	HITEC E-515 present; 250 ml syn sea water bottom.	2 1/2 hrs	none	first	55 min (+2000 ml)	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling; initial bottom pH = 10.0.
		1 day	---	---	---	found pH = 8.95, then adjusted to 5.50 and ultrasonically agitated phases.
		2 days	none	second	55 min (+1350 ml)	found pH = 7.90, then adjusted to 5.40 and ultrasonically agitated immediately prior to this sampling.

TABLE VI Test Constituents: a. HITEC E-515 at minimum effective concentration in 15 liters of clay-treated JP-4.

This pH shift back to basicity even after repeated HCl acidifications was observed with several other runs. The next section will further investigate the pH shift phenomenon.

#### 6. Buffered Water Bottoms

As seen above, HCl acidification of the reacted water bottoms had altered the pH only temporarily. Within 24 hours the pH had traversed from acidic (about 5.5) back to basic (about 8.0). Obviously something more than a simple one-step neutralization was occurring. ASTM D665 synthetic sea water contains a sulfate, a bicarbonate, and is initially adjusted to a pH of between 7.8 and 8.2 with a carbonate (see Appendix B). Each of these salts acts as a buffering agent to maintain a basic pH. Probably the free hydroxyl ions were first neutralized as well as the heavy metal hydroxides. Apparently though, not enough hydronium ions ( $H_3O^+$ ) were available after reaction with the buffering agents to significantly alter the fatty acid product concentration and, therefore, the sample's filtration time. A "reservoir of Hydronium ions," on the other hand, could overwhelm the basic buffering agents and possibly degrade the fatty acid soap back into its free acid and heavy metal ion form.

Such a source of acidifying potential is an acid buffer solution. Two candidates for acidic buffering salts were sodium phosphate and ammonium acetate with the following specifications:

- a. Sodium Phosphate (Monobasic):  $NaH_2PO_4 \cdot H_2O$  F.W. = 137.99  
pH of a 5% solution at 25°C = 4.1 - 4.5
- b. Ammonium Acetate:  $NH_4C_2H_3O_2$  (or  $NH_4OAc$ ) F.W. = 77.08  
pH of a 5% solution at 25°C = 6.7 - 7.3

Table VII shows the experimental results of these buffering agents on the JP-4 filterability.

A neutrally buffered water bottom ( $NH_4OAc$ ) was only partially successful in reducing the filtration time of what was initially a high filtration time JP-4.

TABLE VII. Filterability of HITEC E-515 with Buffered Water Bottoms

Bottle I.D.	Bottle Contents	Bottle Age	Settling Time	Bottle Sampling	Filtration Time (+XXX ml)	Bottle Agitation History And Test Comments
XXXII	HITEC E-515 present; 250 ml syn sea water bottom.	1/2 hr	---	---	---	added $\text{NaH}_2\text{PO}_4$ to 5 weight % level, then ultrasonically agitated phases.
		1 day	none	first	4.66 min	ultrasonically agitated immediately prior to this sampling.
XXXIII	HITEC E-515 present; 250 ml syn sea water bottom.	1/2 hr	---	---	---	ultrasonically agitated on initial mixing of phases.
		1 day	none	first	5.01 min	added $\text{NaH}_2\text{PO}_4$ to 5 weight % level, then ultrasonically agitated immediately prior to this sampling.
		2 days	none	second	4.39 min	ultrasonically agitated immediately prior to this sampling.
2 XXXIV	HITEC E-515 present; 250 ml syn sea water bottom.	1/2 hr	---	---	---	ultrasonically agitated on initial mixing of phases.
		1 day	none	first	50 min (+1280 ml)	ultrasonically agitated immediately prior to this sampling; after sampling, added $\text{NaH}_2\text{PO}_4$ to 5 weight % level and re-agitated ultrasonically.
		2 days	none	second	4.82 min	ultrasonically agitated immediately prior to this sampling.

TABLE VII Con't

Bottle I.D.	Bottle Contents	Bottle Age	Settling Time	Bottle Sampling	Filtration Time (+XXX ml)	Bottle Agitation History And Test Comments
XXXV	HITEC E-515 present; 250 ml syn sea water bottom.	1/2 hr	---	---	---	ultrasonically agitated on initial mixing of phases.
		1 day	none	first	29.08 min	added NH <sub>4</sub> OAC to 5 weight % level, then ultrasonically agitated immediately prior to this sampling.
		2 days	none	second	7.33 min	ultrasonically agitated immediately prior to this sampling.
XXXVI	HITEC E-515 present; 250 ml syn sea water bottom.	1/2 hr	---	---	---	ultrasonically agitated on initial mixing of phases.
		1 day	none	first	50 min (+1400 ml)	ultrasonically agitated immediately prior to this sampling; after sampling, added NH <sub>4</sub> OAC to 5 weight % level and re-agitated ultrasonically.
		2 days	none	second	40.43 min	ultrasonically agitated immediately prior to this sampling.
		3 days	none	third	10.18 min	ultrasonically agitated immediately prior to this sampling.

TABLE VII Test Constituents: a. HITEC E-515 at minimum effective concentration in 15 liters of clay-treated JP-4.

However, settling for a one day interval did bring the fuel batch into the acceptable filtration time range.

Reclamation results with  $\text{NaH}_2\text{PO}_4$  were far more encouraging. Nearly instantaneous improvement in the filtration time of a high filtration time JP-4 was observed.

#### 7. Reclamation Efforts

The buffered water bottoms test data led to two conclusions:

(1) The neutral or slightly acidic buffering of a fuel's water bottom blocked the formation of a membrane plugging "soap".

(2) In the case of HITEC E-515, at least, the acidic buffering action can destroy the "soap" much more quickly and thoroughly than a neutral buffer.

These results prompted work involving two theoretical approaches to the problem of fuel reclamation. Given a fuel tank with a water bottom and a high filtration time JP-4, one could acidically buffer the water bottoms, pump them out and "shower" them back down through the fuel phase. Alternately, one could pump the contaminated fuel up through a "tower" containing an aqueous buffer solution, and back into the tank.

##### a. "Shower Method":

The proposed "shower" method is similar to the procedure thus far employed in generating the high filtration time JP-4's of this report (see Figure 1). However, the Laboratory's ultrasonic agitation chamber would not be necessary in the field. For field adaptation, a coarse screen fitted into the outlet of the hose returning the buffering bottom to the tank could divide the flow into enough streams to flush the soap from the fuel phase. Using the ultrasonic agitation chamber, in the Laboratory version of the "shower" method, HITEC E-515 as well as several other corrosion inhibitors were examined to determine the effectiveness of the "shower" reclamation method as outlined in Table VIII.

TABLE VIII. Reclamation of Synthetically Crystallized High Filtration Time JP-4's Via "Shower" Method

Bottle I.D.	Bottle Contents	Bottle Age	Settling Time	Bottle Sampling	Filtration Time (±XX ml)	Bottle Agitation History And Test Comments
XXXIV	HITEC E-515 present; 250 ml syn sea water bottom.	1/2 hr	---	---	---	ultrasonically agitated on initial mixing of phases.
		1 day	none	first	50 min (+1280 ml)	ultrasonically agitated immediately prior to this sampling; after sampling, added NaH <sub>2</sub> PO <sub>4</sub> to 5 weight % level and re-agitated ultrasonically.
		2 days	none	second	4.82 min	ultrasonically agitated immediately prior to this sampling.
LXI	DCI-4A present; 250 ml syn sea water bottom.	1/2 hr	---	---	---	ultrasonically agitated on initial mixing of phases.
		1 day	none	first	19.52 min	ultrasonically agitated immediately prior to this sampling.
		2 days	none	second	34.34 min	ultrasonically agitated immediately prior to this sampling; after sampling, added NaH <sub>2</sub> PO <sub>4</sub> to 5 weight % level and re-agitated ultrasonically.
		3 days	none	third	6.78 min	ultrasonically agitated immediately prior to this sampling.
LXIII	PR1-19 present; 250 ml syn sea water bottom.	1/2 hr	---	---	---	ultrasonically agitated on initial mixing of phases.
		1 day	none	first	44.04 min	ultrasonically agitated immediately prior to this sampling.
		2 days	none	second	50.74 min	ultrasonically agitated immediately prior to this sampling; after sampling, added NaH <sub>2</sub> PO <sub>4</sub> to 5 weight % level and re-agitated ultrasonically.

Table VIII Con't

Bottle I.D.	Bottle Contents	Bottle Age	Settling Time	Bottle Sampling	Filtration Time (+XXX ml)	Bottle Agitation History And Test Comments
LXII	LUBRIZOL 541 present; 250 ml syn sea water bottom.	3 days	none	third	8.04 min	ultrasonically agitated immediately prior to this sampling.
		1/2 hr	---	---	---	ultrasonically agitated on initial mixing of phases.
		1 day	none	first	50 min (+2370 ml)	ultrasonically agitated immediately prior to this sampling; after sampling, added $\text{NH}_4\text{OAC}$ to 5 weight % level and re-agitated ultrasonically.
		2 days	1 day	second	50 min (+2100 ml)	ultrasonically agitated 1 day ago prior to this sampling; after sampling, added $\text{NaH}_2\text{PO}_4$ to 5 weight % level and re-agitated ultrasonically.
		3 days	none	third	50 min (+2475 ml)	ultrasonically agitated immediately prior to this sampling.
LXXI	LUBRIZOL 541 present; 250 ml syn sea water bottom.	1/2 hr	---	---	---	ultrasonically agitated on initial mixing of phases.
		1 day	none	first	50 min (+2090 ml)	ultrasonically agitated immediately prior to this sampling; after sampling, added $\text{NaH}_2\text{PO}_4$ to 15 weight % level and re-agitated ultrasonically.
		2 days	none	second	18.38 min	ultrasonically agitated immediately prior to this sampling.
		3 days	1 day	third	4.84 min	ultrasonically agitated 1 day ago prior to this sampling.

TABLE VIII Test Constituents: a. All inhibitors at minimum effective concentration in 15 liters of clay-treated JP-4.

Three high filtration fuels, each generated using a different corrosion inhibitor, were reclaimed in one day after agitation of their bottoms following the addition of 5 weight percent  $\text{NaH}_2\text{PO}_4$ . Lubrizol 541, with the highest acid number of all the corrosion inhibitors currently qualified to MIL-I-25017, resulted in the highest filtration time of all problem JP-4 mixtures yet generated. Ammonium acetate had no corrective effect. Further, the acetate-buffered bottom with a 5 weight per cent  $\text{NaH}_2\text{PO}_4$  addition proved futile. However, a 15% weight percent  $\text{NaH}_2\text{PO}_4$  buffered water bottom was ultimately successful. For such difficult reclamation cases in the field, a second buffer shower of considerably greater concentration would be recommended.

A valid test of the capability of the "shower" method to rejuvenate an otherwise unacceptable high filtration time JP-4 would be an actual field problem fuel. Such a "naturally" occurring high filtration time fuel was shipped to AFAPL/WPAFB by DFSC from a tank located in Pasadena, Texas. According to DFSC, even settling did not significantly improve this high filtration time JP-4. Table IX presents data on the "shower" method of reclamation for the DFSC JP-4.

An aqueous buffered bottom of 5 weight percent  $\text{NaH}_2\text{PO}_4$  in distilled water was added to the DFSC JP-4. The phases were ultrasonically agitated and continuously recirculated down through the fuel phase. The resultant fuel filtration time was drastically reduced. Ammonium acetate had the same effect. The volume of buffered bottom necessary to achieve the same level of reclamation was decreased and the distilled water replaced with hard WPAFB tap water in subsequent experiments. A 5 weight percent buffered tap water bottom of 1/3 volume percent of the total fuel volume rectified the filtration time of DFSC fuel in only one day. The "shower" method, if employed in the field on such a

TABLE IX. Reclamation of DFSC High Filtration Time JP-4 Via "Shower" Method

Bottle I.D.	Bottle Contents	Bottle Age	Settling Time	Bottle Sampling	Filtration Time (+XXY ml)	Bottle Agitation History And Test Comments
DFSC -SH-1	DFSC JP-4 only; initially w/o water bottom.	--	--	--	50 min (+785 ml)	baseline filtration time determined.
		1/2 hr	none	first	50 min (+275 ml)	added a 250 ml distilled water bottom buffered with $\text{NaH}_2\text{PO}_4$ to 5 weight % level and ultrasonically agitated phases immediately prior to this sampling.
DFSC -SH-2	DFSC JP-4 only; initially w/o water bottom.	1 day	none	second	10.75 min	ultrasonically agitated immediately prior to this sampling.
		5 days	4 days	third	5.37 min	ultrasonically agitated 4 days ago prior to this sampling.
		1/2 hr	---	---	---	added a 250 ml distilled water bottom buffered with $\text{NaH}_2\text{PO}_4$ to 5 weight % level and ultrasonically agitated phases.
DFSC -SH-3	DFSC JP-4 only; initially w/o water bottom.	1 day	1 day	first	5.74 min	ultrasonically agitated 1 day ago prior to this sampling.
		1/2 hr	---	---	---	added a 250 ml distilled water bottom buffered with $\text{NH}_4\text{OAC}$ to 5 weight % level and ultrasonically agitated phases.
		1 day	1 day	first	3.42 min	ultrasonically agitated 1 day ago prior to this sampling.
		1 day + 1/2 hr	none	second	7.36 min	ultrasonically agitated immediately prior to this sampling.

TABLE IX Con't

Bottle I.D.	Bottle Contents	Bottle Age	Settling Time	Bottle Sampling	Filtration Time (+XXX ml)	Bottle Agitation History And Test Comments
LXIV	DFSC JP-4 only; initially w/o water bottom.	1/2 hr	---	---	---	added a 50 ml WPAFB tap water bottom buffered with NH <sub>4</sub> OAC to 5 weight % level and ultrasonically agitated phases.
		1 day	1 day	first	3.99 mlr	ultrasonically agitated 1 day ago prior to this sampling.

TABLE IX Test Constituents: a. Added buffered water bottoms to 15 liters of the DFSC High Filtration Time base fuel.

problem fuel would produce a cloudy fuel/tap water mixture in the tank, but a one-day settling interval should yield a clear JP-4.

b. "Tower" Method:

The buffered "tower" approach to JP-4 reclamation was originally proposed by Mr. James McCoy (AFAPL/SFF) and entailed a reservoir tower filled with a concentrated buffer solution through which the contaminated fuel would be circulated. The lab version of the buffer "tower" was a modified Millipore filtration assembly. Pipe fittings were threaded into a stainless steel pressure cylinder (Millipore Part No. XX4004700) having a fluid capacity of 100 milliliters. The internal cross sectional area was 11.35 cm<sup>2</sup>. Several coarse mesh screens were inserted into the tower's filter holder base. The buffer solution was poured into the tower. A plug of glass wool was inserted in the pipe leading from the tower's cap, to retain the aqueous phase, and the top screwed onto the barrel. The test fuel was pumped into the tower's base and divided into droplet streams via the screens. The fuel then rose through the buffer solution to the top of the tower. The fuel coagulated there and then drained back into the 5 gallon glass jug. A peristaltic pump recirculated the test fuel at a rate of 325 ml/min, or at 5 gallons/hour. At this flow rate, a circulation period of four hours meant about five passes of all the fuel through the buffer "tower." Experiments with the "tower" method are summarized in Table X.

Preliminary reclamation runs utilized a low strength buffer solution and gave little initial improvement in filtration time. However, as the tower's buffer strength was increased, the filtration time of the test JP-4 decreased in nearly a linear fashion for a given circulation period. Figure 2 is a plot of the filtration time of the DFSC JP-4 after circulation for 2 or 4 hours through different tower buffer strengths (data extracted from Table X). The most resounding success in the fewest number of fuel passes was with the 50 weight percent NaH<sub>2</sub>PO<sub>4</sub> solution.

TABLE X. Reclamation of High Filtration Time JP-4 Via "Tower" Method

Bottle I.D.	Buffer Tower Strength	Bottle Age	Settling Time	Bottle Sampling	Filtration Time (+XXX ml)	Total Time of Circulation	Bottle Agitation History (Through Tower) and Test Comments
DFSC -TW-1	Mah <sub>2</sub> PO <sub>4</sub> in WPAFB tap water; 12.5 g/35 ml (26 weight %)	4 hrs 3 days	none 2 days	first second	50.25 min 3.04 min	4 hrs 4 hrs	circulated for 4 hours immediately prior to this sampling. not circulated for 2 days prior to this sampling.
DFSC -TW-3	Mah <sub>2</sub> PO <sub>4</sub> in WPAFB tap water; 62.5 g/100 ml (38 weight %)	2 hrs 1 day	none 3/4 day	first second	35.02 min 3.81 min	2 hrs 6 hrs	circulated for 2 hrs (but at 300 ml/min instead of usual 325 ml/min) immediately prior to this sampling; then circulated for 4 more hours and let settle. not circulated for 3/4 day prior to this sampling.
DFSC -TW-2	Mah <sub>2</sub> PO <sub>4</sub> in WPAFB tap water; 37.5 g/50 ml (43 weight %)	2 hrs 4 hrs 1 day	none none 3/4 day	first second third	26.60 min 14.63 min 11.24 min	2 hrs 4 hrs 4 hrs	circulated for 2 hours immediately prior to this sampling. circulated for 2 more hours immediately prior to this sampling. not circulated 3/4 day prior to this sampling.

TABLE X Con't

Bottle I.D.	Buffer Tower Strength	Bottle Age	Settling Time	Bottle Sampling	Filtration Time (+XXX ml)	Total Time of Circulation	Bottle Agitation History (Through Tower) and test comments
DFSC -TW-4	NaH <sub>2</sub> PO <sub>4</sub> in WPAPB tap water; 50.0 g/50 ml (50 weight %)	2 hrs	none	first	16.80 min	2 hrs	circulated for 2 hours immediately prior to this sampling.
*LUB -TW-1	NaH <sub>2</sub> PO <sub>4</sub> in WPAPB tap water; 50.0 g/50 ml (50 weight %)	5 hrs	none	second	5.24 min	4 hrs	circulated for 2 more hours immediately prior to this sampling.
		1 day	none	first	50 min (+1000 ml)	4 hrs	circulated for 4 hours immediately prior to this sampling.
		2 days	none	second	5.12 min	8 hrs	circulated for 4 more hours immediately prior to this sampling.

TABLE X Test Constituents: a. 15 liters of the DFSC High Filtration Time Base fuel.

\*b. SOLE EXCEPTION: LUB-TW-1 which was a synthetically generated high filtration time JP-4 using LUBRIZOL 541 at the minimum effective concentration in 15 liters of clay-treated JP-4 with a 250 ml synthetic sea water bottom. In this example, only the fuel phase was circulated through the buffer tower.

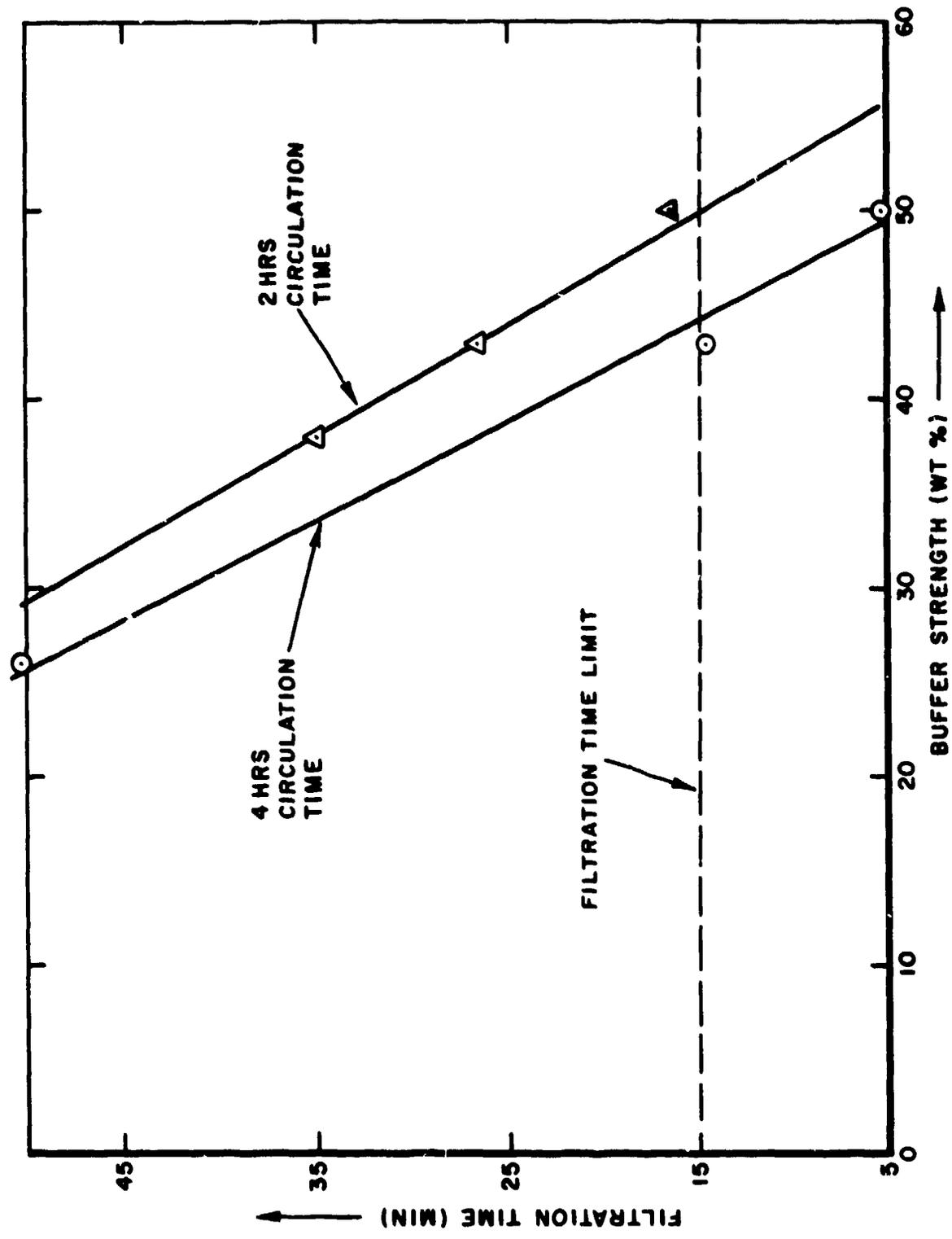


Figure 2. Reclamation Efficiency of "Tower" Method Vs Circulation Time

The "tower" method of reclamation was applied to the high filtration time fuel artificially generated from the ultrasonic agitation of Lubrizol 541 inhibited JP-4 with a synthetic sea water bottom. Four hours of circulation of the fuel phase through the 50% strength buffer tower did not reduce the filtration time. Only after four additional hours of circulation on the next day did the fuel phase pass the filtration time test limit. Obviously, if the "tower" method of fuel reclamation were employed in the field, several passes of the tank's fuel should be circulated through as concentrated a buffer solution of as large a volume as is practicable.

Each reclamation process has its own advantages and disadvantages. The "shower" method requires the addition of a buffered bottom to the fuel tank of about 1 volume percent of the fuel. However, only simple equipment (fuel pump, hose, shower head screens, etc.) is needed. Unfortunately, the water-washed fuel could not be used until about 24 hours later, after the suspended water droplets had settled back down to the tank's bottoms. The tank bottoms would then have to be drained for fear of corrosion by the acidically buffered solution. The "tower" method requires a fuel circulation pump and a buffer "tower" of approximately 100-200 gallon capacity to treat @100,000 gallons of contaminated fuel. The processing of a "soap"-type contaminated fuel supply via this method would definitely take longer than the initial "shower" treatment but the fuel would be available immediately since no settling time of water droplets is necessary.

#### 8. Filterability Effect of Other Corrosion Inhibitors

Table XI is the current list of Air Force approved corrosion inhibitors as of January 1975 along with their minimum effective concentrations and their equivalent amounts per 15 liters of JP-4 for the filterability studies. Each inhibitor was dissolved in clay-treated JP-4. Ultrasonic agitation and circulation followed,

TABLE XI. Approved AF Corrosion Inhibitors  
(QPL-25017-11, 24 June 1975)

<u>Corrosion Inhibitor</u>	<u>Min. Eff. Conc. (#/KBBL)</u>	<u>Equivalent Amounts Added Per 15 Liters Clay-Treated JP-4 (grams)</u>
AFA-1	4.5	0.1926
CONOCO T-60	6.0	0.2567
DCI-4A	3.0	0.1283
EMERY 9885	4.5	0.1926
HITEC E-515	7.5	0.3210
HITEC E-580	3.0	0.1283
LUBRIZOL 541	3.0	0.1283
NALCO 5402	3.0	0.1283
NALCO 5403	3.0	0.1283
PRI-19	3.0	0.1283
TOLAD 245	7.5	0.3210
TOLAD 246	3.0	0.1283
UNICOR J	3.0	0.1283
<hr/>		
NALCO 5400-A (Deleted)	3.0	0.1283

using an ASTM synthetic sea water bottom under the same conditions as was used with HITEC E-515 in its generation of a high filtration time JP-4. Table XII is a compilation of the filterability behavior of the different inhibitors.

Under the prescribed reaction conditions, most but not all the corrosion inhibitors surpassed the >50 minute filtration time mark after two days of a half-hour per day ultrasonic agitation. Of the exceptions, only one corrosion inhibitor consistently remained well below the 15 minute filtration time specification limit even with the high levels of entrained water normally introduced. This was the Nalco 5400-A, which is no longer on the Qualified Products List for corrosion inhibitors.

The referenced compilation reveals a wide variance in filtration time repeatability on runs performed with the same corrosion inhibitor. If, however, one selects only those filtration time results that meet the criteria: "one-day" in bottle age and a settling time of "none", then distinctive filterability classifications emerge. The three filtration time ranges listed in Table XIII were sufficient to circumscribe all of the one day filtration results for each corrosion inhibitor.

Most corrosion inhibitors required longer than 50 minutes for a one gallon filtration run on one-day old samples. Besides Nalco 5400-A, only AFA-1 displayed filtration time runs that were in the "less than 20 minute" category.

Again, on a qualitative level, there was a noticeable difference in the rate at which the high filtration time mixtures settled or re-approached the 15 minute filtration time limit when left undisturbed. Of those inhibitors in Table XIII that had all of their runs fall in the "greater than 50 minute" filtration time classification, only HITEC E-515 repeatedly gave a 10-20 minute filtration time runs after one day of undisturbed settling. Most required more than one week to display a similar filtration time.

TABLE XII. Filterability of Different Corrosion Inhibitors with Synthetic Sea Water Bottoms

CORROSION INHIBITOR	BOTTLE I.D.	BOTTLE AGE	SETTLING TIME	BOTTLE SAMPLING	FILTRATION TIME (+XXX ml)	BOTTLE AGITATION HISTORY AND TEST COMMENTS
AFA-1	I-AFA-H	1 day	none	first	9.64 min	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling
		2 days	none	second	10.53 min	ultrasonically agitated immediately prior to this sampling
		3 days	none	third	16.33 min	ultrasonically agitated immediately prior to this sampling
	II-AFA-D	1 day	none	first	16.69 min	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling
		2 days	none	second	50 min (+370 ml)	ultrasonically agitated immediately prior to this sampling
		3 days	1 day	third	28.50 min	ultrasonically agitated 1 day ago prior to this sampling
	III-AFA-C	1 day	none	first	8.66 min	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling
		2 days	none	second	10.78 min	ultrasonically agitated immediately prior to this sampling
		3 days	none	third	9.30 min	ultrasonically agitated immediately prior to this sampling
	IV-AFA-K	1 day	none	first	20.23 min	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling
		2 days	none	second	50 min (+610 ml)	ultrasonically agitated immediately prior to this sampling

TABLE XII. continued

CORROSION INHIBITOR	BOTTLE I.D.	BOTTLE AGE	SETTLING TIME	BOTTLE SAMPLING	FILTRATION TIME (+XXX ml)	BOTTLE AGITATION HISTORY AND TEST COMMENTS
AFA-1	IV-AFA-K	3 days	1 day	third	22.21 min	ultrasonically agitated 1 day ago prior to this sampling
	V-AFA-L	1 day	none	first	3.76 min	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling
		2 days	none	second	23.24 min	ultrasonically agitated immediately prior to this sampling
		3 days	1 day	third	62.67 min	ultrasonically agitated 1 day ago prior to this sampling
COMOCO T-60	I-COM-H	1 day	none	first	27.99 min	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling
		2 days	none	second	50 min (+1550 ml)	ultrasonically agitated immediately prior to this sampling
		3 days	1 day	third	50 min (+875 ml)	ultrasonically agitated 1 day ago prior to this sampling
	II-COM-D	1 day	none	first	33.53 min	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling
		2 days	none	second	50 min (+800 ml)	ultrasonically agitated immediately prior to this sampling
		4 days	2 days	third	27.13 min	ultrasonically agitated 2 days ago prior to this sampling
	III-COM-I	1 day	none	first	50 min (+435 ml)	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling
		2 days	1 day	second	20.84 min	ultrasonically agitated 1 day ago prior to this sampling

TABLE XII. continued

<u>CORROSION INHIBITOR</u>	<u>BOTTLE I.D.</u>	<u>BOTTLE AGE</u>	<u>SETTLING TIME</u>	<u>BOTTLE SAMPLING</u>	<u>FILTRATION TIME (+XXX ml)</u>	<u>BOTTLE AGITATION HISTORY AND TEST COMMENTS</u>
COMOCO T-60	(III-COM-I)	3 days	2 days	third	50 min (+1000 ml)	ultrasonically agitated 2 days ago prior to this sampling
	IV-COM-K	1 day	none	first	50 min (+1700 ml)	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling
		2 days	1 day	second	50 min (+900 ml)	ultrasonically agitated 1 day ago prior to this sampling
		3 days	2 days	third	50 min (+250 ml)	ultrasonically agitated 2 days ago prior to this sampling
	V-COM-L	1 day	none	first	50 min (+1865 ml)	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling
		2 days	1 day	second	37.34 min	ultrasonically agitated 1 day ago prior to this sampling
		3 days	2 days	third	22.24 min	ultrasonically agitated 2 days ago prior to this sampling
DCI-4A	I-DCI-H	1 day	none	first	31.55 min	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling
		2 days	none	second	26.37 min	ultrasonically agitated immediately prior to this sampling
		3 days	1 day	third	16.82 min	ultrasonically agitated 1 day ago prior to this sampling
	II-DCI-C	1 day	none	first	50 min (+1610 ml)	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling
		2 days	1 day	second	50 min (+245 ml)	ultrasonically agitated 1 day ago prior to this sampling

TABLE XII. continued

COMBOSION INHIBITOR	BOTTLE I.D.	BOTTLE AGE	SETTLING TIME	BOTTLE SAMPLING	FILTRATION TIME (+XX ml)	BOTTLE AGITATION HISTORY AND TEST COMMENTS
DCI-4A	(II-DCI-C)	5 days	2 days	third	27.92 min	ultrasonically agitated 2 days ago prior to this sampling
	III-DCI-K	1 day	none	first	50 min (+1310 ml)	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling
		2 days	1 day	second	15.63 min	ultrasonically agitated 1 day ago prior to this sampling
		3 days	2 days	third	10.45 min	ultrasonically agitated 2 days ago prior to this sampling
	IV-DCI-L	1 day	none	first	50 min (+1780 ml)	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling
		2 days	1 day	second	25.37 min	ultrasonically agitated 1 day ago prior to this sampling
		3 days	2 days	third	13.22 min	ultrasonically agitated 2 days ago prior to this sampling
	I-224-E	1 day	none	first	50 min (+1020 ml)	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling
	II-224-H	1 day	none	first	50 min (+360 ml)	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling
		2 days	1 day	second	47.26 min	ultrasonically agitated 1 day ago prior to this sampling
		3 days	2 days	third	50 min (+580 ml)	ultrasonically agitated 2 days ago prior to this sampling

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TABLE XII, Continued

COMPOSITION IDENTIFICATION	BOTTLE I.D.	BOTTLE AGE	SETTLING TIME	BOTTLE SAMPLING	FILTRATION TIME (+XXX ml)	BOTTLE AGITATION HISTORY AND TEST COMMENTS
EMERY 9955	III-204-1	1 day	none	first	50 min (+1580 ml)	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling.
		2 days	1 day	second	50 min (+1380 ml)	ultrasonically agitated 1 day ago prior to this sampling
		3 days	2 days	third	50 min (+1500 ml)	ultrasonically agitated 2 days ago prior to this sampling
RTTC E-515	XXIV	1 day	none	first	50 min (+800 ml)	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling
	XXIV	1 day	none	first	50 min (+1280 ml)	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling
	XXVI	1 day	none	first	50 min (+1400 ml)	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling
	XI(1)	1 day	none	first	60 min (+1425 ml)	ultrasonically agitated on initial mixing of phases but agitated 1 hour prior to this sampling
	XIV(1)	7 days	1 hr	second	60 min (+540 ml)	not ultrasonically agitated on initial mixing of phases but agitated 7 hours prior to this sampling
	XV (1)	7 days	7 hrs	second	57.5 min	ultrasonically agitated 1 1/2 days ago prior to this sampling
	XIII(1)	8 days	1 1/2 days	third	10.0 min	not ultrasonically agitated on initial mixing of phases, but agitated 1 day ago prior to this sampling.
		8 days	1 day	second	22.8 min	

TABLE XII. continued

CORROSION INHIBITOR	BOTTLE I.D.	BOTTLE AGE	SETTLING TIME	BOTTLE SAMPLING	FILTRATION TIME (+XXX ml)	BOTTLE AGITATION HISTORY AND TEST COMMENTS
HITEC E-515	(XIII)	9 days	2 days	third	5.5 min	ultrasonically agitated 2 days ago prior to this sampling
HITEC E-580	I-H58-E	1 day	none	first	50 min (+415 ml)	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling
	II-H58-H	1 day	none	first	28.36 min	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling
		2 days	none	second	10.10 min	ultrasonically agitated immediately prior to this sampling
	III-H58-I	1 day	none	first	50 min (+1090 ml)	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling
		2 days	1 day	second	50 min (+200 ml)	ultrasonically agitated 1 day ago prior to this sampling
		3 days	2 days	third	26.12 min	ultrasonically agitated 2 days ago prior to this sampling
	IV-H58-K	1 day	none	first	50 min (+725 ml)	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling
		2 days	1 day	second	13.43 min	ultrasonically agitated 1 day ago prior to this sampling
		3 days	2 days	third	8.62 min	ultrasonically agitated 2 days ago prior to this sampling
LUBRIZOL 541	LXXI	1 day	none	first	50 min (+2090 ml)	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling

TABLE XII. continued

COMPOSITION INHIBITOR	BOTTLE I.D.	BOTTLE AGE	SETTLING TIME	BOTTLE SAMPLING	FILTRATION TIME (+XXX ml)	BOTTLE AGITATION HISTORY AND TEST COMMENTS
LUBRIZOL S41	LXII	1 day	none	first	50 min (+2370 ml)	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling
		2 days	1 day	second	50 min (+2100 ml)	ultrasonically agitated 1 day ago prior to this sampling
		1 day	none	first	50 min (+1100 ml)	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling
MALCO 5402	I-N02-J	2 days	1 day	second	31.38 min	ultrasonically agitated 1 day ago prior to this sampling
		3 days	2 days	third	6.43 min	ultrasonically agitated 2 days ago prior to this sampling
		1 day	none	first	19.24 min	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling
	II-N02-D	2 days	none	second	27.45 min	ultrasonically agitated immediately prior to this sampling
		3 days	none	third	16.41 min	ultrasonically agitated immediately prior to this sampling
		1 day	none	first	50 min (+500 ml)	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling
	III-N02-I	2 days	1 day	second	12.73 min	ultrasonically agitated 1 day ago prior to this sampling
		3 days	2 days	third	14.74 min	ultrasonically agitated 2 days ago prior to this sampling

TABLE XII. continued

CORROSION INHIBITOR	BOTTLE I.D.	BOTTLE AGE	SETTLING TIME	BOTTLE SAMPLING	FILTRATION TIME (+XXX ml)	BOTTLE AGITATION HISTORY AND TEST COMMENTS
MALCO 5402	IV-N02-K	1 day	none	first	50 min (+1245 ml)	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling
		2 days	1 day	second	8.02 min	ultrasonically agitated 1 day ago prior to this sampling
		3 days	2 days	third	5.19 min	ultrasonically agitated 2 days ago prior to this sampling
MALCO 5403	I-N03-E	1 day	none	first	33.87 min	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling
		2 days	none	second	50 min (+150 ml)	ultrasonically agitated immediately prior to this sampling
	II-N03-I	1 day	none	first	50 min (+410 ml)	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling
		2 days	1 day	second	48.16 min	ultrasonically agitated 1 day ago prior to this sampling
		3 days	2 days	third	41.45 min	ultrasonically agitated 2 days ago prior to this sampling
	III-N03-L	1 day	none	first	50 min (+1100 ml)	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling
		2 days	1 day	second	49.52 min	ultrasonically agitated 1 day ago prior to this sampling
		3 days	2 days	third	11.21 min	ultrasonically agitated 2 days ago prior to this sampling

TABLE XII. continued

CORROSION INHIBITOR	BOTTLE I.D.	BOTTLE AGE	SETTLING TIME	BOTTLE SAMPLING	FILTRATION TIME (+XXX ml)	BOTTLE AGITATION HISTORY AND TEST COMMENTS
PRI-19	I-PRI-J	1 day	none	first	50 min (+400 ml)	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling
		2 days	1 day	second	27.68 min	ultrasonically agitated 1 day ago prior to this sampling
		3 days	2 days	third	27.19 min	ultrasonically agitated 2 days ago prior to this sampling
II-PRI-L	I-PRI-L	1 day	none	first	49.05 min	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling
		2 days	none	second	50 min (+1880 ml)	ultrasonically agitated immediately prior to this sampling
		3 days	1 day	third	50 min (+515 ml)	ultrasonically agitated 1 day ago prior to this sampling
TOLAD 245	I-T45-D	1 day	none	first	50 min (+2270 ml)	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling
		2 days	1 day	second	50 min (+1615 ml)	ultrasonically agitated 1 day ago prior to this sampling
		3 days	2 days	third	50 min (+2300 ml)	ultrasonically agitated 2 days ago prior to this sampling
II-T45-K	I-T45-K	1 day	none	first	50 min (+3150 ml)	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling
		2 days	1 day	second	50 min (+1040 ml)	ultrasonically agitated 1 day ago prior to this sampling
		7 days	6 days	third	50 min (+740 ml)	ultrasonically agitated 6 days ago prior to this sampling

TABLE XII. continued

CORROSION INHIBITOR	BOTTLE I.D.	BOTTLE AGE	SETTLING TIME	BOTTLE SAMPLING	FILTRATION TIME (+XXX ml)	BOTTLE ACITATION HISTORY AND TEST COMMENTS
TOLAD 246	I-T46-E	1 day	none	first	50 min (+820 ml)	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling
	II-T46-I	1 day	none	first	50 min (+440 ml)	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling
		2 days	1 day	second	50 min (+1150 ml)	ultrasonically agitated 1 day ago prior to this sampling
		3 days	2 days	third	50 min (+1080 ml)	ultrasonically agitated 2 days ago prior to this sampling
UNICOR J	I-UNI-J	1 day	none	first	50 min (+2140 ml)	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling
		2 days	1 day	second	50 min (+1220 ml)	ultrasonically agitated 1 day ago prior to this sampling
		3 days	2 days	third	50 min (+525 ml)	ultrasonically agitated 2 days ago prior to this sampling
	II-UNI-D	1 day	none	first	50 min (+410 ml)	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling
		4 days	3 days	second	20.47 min	ultrasonically agitated 3 days ago prior to this sampling
MALCO 5400A (DELETED FROM 1975 QPL)	I-NOA-D	1 day	none	first	5.87 min	ultrasonically agitated on initial mixing of phases and immediately prior to this sampling
		2 days	none	second	7.97 min	ultrasonically agitated immediately prior to this sampling
		3 days	none	third	11.23 min	ultrasonically agitated immediately prior to this sampling

TABLE XII Continued

<u>CORROSION INHIBITOR</u>	<u>BOTTLE I.D.</u>	<u>BOTTLE AGE</u>	<u>SETTLING TIME</u>	<u>BOTTLE SAMPLING</u>	<u>FILTRATION TIME (+XXX ml)</u>	<u>BOTTLE AGITATION HISTORY AND TEST COMMENTS</u>
MALCO 5400A	II-NOA-E	3 days	none	first	9.10 min	ultrasonically agitated on initial mixing of phases, once each day, as well as immediately prior to this sampling

TABLE XII Test Constituents: a. All inhibitors at their minimum effective concentration in 15 liters of clay-treated JP-4.

b. 250 ml synthetic sea water bottoms.

c. (1) MEANS THESE RUNS WERE IN THE PRESENCE OF SOFT STEEL SHIM STOCK.

TABLE XIII. Extraction of TABLE XII Data for Inhibitors' Repeatability

<u>All Filtration Runs Less Than 20 Min</u>	<u>All Filtration Runs From 20 to 50 Min</u>	<u>All Filtration Runs Greater Than 50 Min</u>
AFA-1	CONOCO T-60	EMERY 9885
NALCO 5400-A*	DCI-4A	HITEC E-515
	NALCO 5403	HITEC E-580
	NALCO 5402	LUBRIZOL 541
		TOLAD 245
		TOLAD 246
		UNICOR J
		PRI-19

\* Deleted from 1975 QPL for Corrosion Inhibitors.

## 9. Miscellaneous Work

### a. Filterability with Ester vs. Silver Membranes

At the request of the Defense Fuel Supply Center (DFSC), filtration time tests were conducted on a series of JP-4 mixtures utilizing two different kinds of filter membranes. The standard filtration time test uses Millipore filters composed of mixed cellulose esters with the following specifications: 0.8 micron pore size, 47 mm diameter (Part No. AAWPO 4700). The ester filters disintegrate upon exposure to methanol, ethanol, some ether alcohols (like FSII), ketones, esters (including phosphate), dioxane, and most acids and bases. The comparison silver membranes are products of Flotronics, Inc. (Part No. 83485-03) with similar physical dimensions but composed of silver and, consequently, resistant to all fluids except a few concentrated acids.

Some field personnel have claimed that the filtration time of certain high filtration time JP-4's could artificially be reduced by replacing the standard ester membrane with a silver one. However, mixtures of high and low filtration time JP-4's failed to bear out this filtration behavior. The results of tests with ester and silver type filter membranes on the same fuel sample are summarized in Table XIV.

Five gallon batches of each sample composition listed in Table XIV were prepared and continuously stirred magnetically while sampled via a stainless steel siphon. Generally, the filtration time repeatability for a given five gallon fuel batch was better for the ester membranes. Furthermore, the silver membranes usually had longer filtration times.

In some experiments, water bottoms were present and each filtration immediately followed a one-half hour ultrasonic agitation period. Consequently, a high level of synthetic sea water would be entrained in these samples. The silver membrane required a significantly longer time to filter a one gallon aliquot than its ester membrane counterpart.

TABLE XIV. Filterability of Different JP-4 Mixtures Using Ester vs. Silver Membranes

Test Fuel Composition and Agitation History	Filtration Times (+XXX ml)	
	Ester Membrane	Silver Membrane
Clay-treated JP-4	3.31 min (a)	3.24 min (b)
Fuel phase mixture of synthetically generated high filtration time JP-4's using several corrosion inhibitors with synthetic sea water bottom.	5.9 min (+765 ml) (a)	50 min (+690 ml) (b)
1/4 by volume above high filtration time fuel mixture + 3/4 by volume clay-treated JP-4.	12.63 min (a)	21.25 min (b)
BOTTLE XXB: No corrosion inhibitor, 0.1 vol % FSII in fuel phase, 15 vol % FSII in syn sea water bottom; ultrasonically agitated and fuel phase immediately sampled for filtrations.	5.33 min (a)	6.30 min (b)
BOTTLE XXIB: No corrosion inhibitor, 0.1 vol % FSII in fuel phase, 30 vol % FSII in syn sea water bottom; ultrasonically agitated and fuel phase immediately sampled for filtrations.	5.47 min (a)	20.41 min (b) 6.27 min (c)
BOTTLE XXBBB: No corrosion inhibitor, no FSII in fuel phase, no FSII in syn sea water bottom; ultrasonically agitated and fuel phase immediately sampled for filtrations.	5.20 min (a)	32.70 min (b)
1/2 by volume DFSC Field Problem JP-4 + 1/2 by volume clay-treated JP-4.	11.00 min (a)	14.29 min (b)
	10.87 min (c)	10.87 min (d)

TABLE XIV Continued

<u>Test Fuel Composition and Agitation History</u>	<u>Filtration Times (+XXX ml)</u>	
	<u>Ester membrane</u>	<u>Silver Membrane</u>
2/3 by volume DFSC Field Problem JP-4 + 1/3 by volume clay-treated JP-4.	23.45 min (a)	25.80 min (b)
	22.58 min (c)	25.52 min (d)
3/4 by volume DFSC Field Problem JP-4 + 1/4 by volume clay-treated JP-4.	32.53 min (a)	29.44 min (b)
	30.03 min (c)	19.69 min (d)

TABLE XIV Test Constituents: a. (a,b,c,d.) Denotes filtration sequence for a given fuel composition.

For all of these filtrations, the ester filter membranes had been screened by weight (75 to 85 milligram range) in accordance with the filtration time test procedure (see Reference 1). No such screening was performed on the silver filters. However, each set of duplicated runs on a given five gallon fuel batch employed filter membranes from the same box.

b. Concrete Effect on Filterability

Concrete storage chambers of World War II vintage are still in use and were suspected of causing filtration time problems in Japan. Lime (CaO), one of the chief constituents of concrete, could have been the source of heavy metal ions which are known to react with corrosion inhibited JP-4 to create a high filtration time fuel. In a laboratory reenactment of the situation, several large chunks of weathered pavement concrete (5" x 3" x 1-1/2") were placed in five gallon glass jars with their smooth face down. After addition of inhibited JP-4, enough distilled water (pH = 6.5) was poured into the reaction jar to cover the concrete's bottom face and half-way up the sides. This resulted in a 3 volume percent of water in the fuel. Control jars without the corrosion inhibitor but with a 3 volume percent of synthetic sea bottom or with a distilled water bottom along with concrete were also prepared. The fuel/water phases were then circulated through an ultrasonic agitation chamber via a peristaltic pump in order to simulate severe mixing. The filterability effect of the concrete is displayed in Table XV.

Probably, the lime leached out of the concrete and into the water bottom yielding two of the necessary conditions for generating a fatty acid "soap": conversion of the slightly acidic distilled water bottom to basicity and the release of a substantial quantity of  $Ca^{+2}$  ions. With HITEC E-515 present, the result was a high filtration time JP-4. The control runs without inhibitor, produced no such filterability problems.

TABLE XV. Filterability of Concrete-Exposed JP-4 with Water. HITEC E-515

Bottle I.D.	Bottle Contents	Bottle Age	Settling Time	Bottle Sampling	Filtration Time (+XXX ml)	Bottle Agitation History And Test Comments
LMC-B (CONTROL)	No concrete present; no HITEC E-515 present; 500 ml distilled water bottom of initial pH 6.5	3 days	none	first	4.73 min	Ultrasonically agitated on initial mixing of phases, once each day, as well as immediately prior to this sampling.
LMC-A (CONTROL)	Concrete chunks present; no HITEC E-515 present; 500 ml 300 sea water bottom of initial pH 2.0	3 days	none	first	7.30 min	ultrasonically agitated phases once each day as well as immediately prior to this sampling.
LMC	Concrete chunks present; HITEC E-515 present; 500 ml distilled water bottom of initial pH 6.5	3 days	none	first	50 min (+1845 ml)	ultrasonically agitated on initial mixing of phases, once each day, as well as immediately prior to this sampling.
		4 days	1 day	second	36.04 min	ultrasonically agitated 1 day age prior to this sampling.
		5 days	none	second	5.89 min	ultrasonically agitated phases once each day, as well as immediately prior to this sampling.
		6 days	none	second	7.60 min	ultrasonically agitated phases once each day as well as immediately prior to this sampling.

TABLE XV Test Constituents: a. HITEC E-515 at minimum effective concentration in 19 liters of clay-treated JP-4.

b. Weathered concrete chunks from same locale.

### c. Filter Porosity Effect on Filterability

DFSC expressed interest in filtration time as a function of filter membrane pore size. Two moderately high filtration time JP-4 batches were generated via the usual method, i.e., ASTM synthetic sea water bottom, inhibited fuel, ultrasonic agitation and circulation, etc. Repeated agitation the following day preceded a 16 hour settling period. All but the water bottom was siphoned into a clean five gallon jug. Enough clay-treated JP-4 was added to dilute each mixture to permit five one-gallon filtrations (see Table XVI). Each such five gallon batch was magnetically stirred prior to and between samplings. All filter membranes (Millipore brand) were heated in a 90°C oven for one-half hour and cooled for one-half hour in a dessicator. A change in the membrane pore size from 0.8 microns to 1.2 microns was enough to easily permit an acceptable filtration time (<15 minutes) of an otherwise high filtration time fuel.

TABLE XVI. Filter Porosity Effect on Filtration Time

<u>Test Fuel Composition and Agitation History</u>	<u>Settling Time</u>	<u>Bottle Sampling</u>	<u>Membrane Porosity</u>	<u>Filtration Time (+XXX ml)</u>
Synthetically generated high filtration time JP-4 using DCI-4A and syn sea water; then siphoned off fuel phase only, diluted to 19 liters with clay-treated JP-4, and sampled for filtrations.	16 hrs	first	0.80 $\mu$	26.99 min
	16 hrs	second	0.80 $\mu$	26.84 min
	16 hrs	third	1.20 $\mu$	5.29 min
	16 hrs	fourth	3.00 $\mu$	7.23 min
	16 hrs	fifth	5.00 $\mu$	1.39 min
Synthetically generated high filtration time JP-4 using TOLAÜ 245 and syn sea water; then siphoned off fuel phase only, diluted to 19 liters with clay-treated JP-4, and sampled for filtrations.	16 hrs	first	0.80 $\mu$	41.86 min
	16 hrs	second	0.80 $\mu$	43.99 min
	16 hrs	third	1.20 $\mu$	9.71 min
	16 hrs	fourth	3.00 $\mu$	9.01 min
	16 hrs	fifth	5.00 $\mu$	1.59 min

TABLE XVI. Test Constituents: a. Inhibitors initially at minimum effective concentration in 15 liters of clay-treated JP-4 prior to siphoning.

b. 250 ml synthetic sea water bottom initially present.

SECTION IV  
CONCLUSIONS

The following conclusions were derived:

1. High levels of entrained free water in non-additive JP-4 did not result in high filtration time fuels. The filtration time of the stock JP-4 increased from the usual 4 minutes to around 8-9 minutes.

2. The severity and frequency of the mixing of the fuel/water phases is absolutely crucial to the creation of a high filtration time JP-4. Mere pouring of the inhibited fuel and basic, hard water phases into the same container or even with simple circulation via a peristaltic pump to and from the glass reaction jugs failed to generate high filtration time fuels.

3. No filterability problem ever arose when one of the following was missing:

- a. Corrosion inhibitor in the fuel phase
- b. A water bottom with a basic pH
- c. High levels of calcium and/or magnesium ions (total > 100 ppm) in a water bottom (i.e., tap water, sea water, etc).

4. Freshly polished or pre-rusted soft steel shim stock was not necessary to create a high filtration time JP-4. However, rusting steel did eventually introduce sufficient rust to plug the filter membrane even in the absence of corrosion inhibitor.

5. Control runs without corrosion inhibitor but with 20 to 40 volume percent of the fuel system icing inhibitor (FSII) in a basic hard water bottom gave no fuel-suspendable precipitant and, consequently, no effect on the fuel's filtration time.

6. A hard water bottom initially buffered to a neutral or an acidic pH did not generate a high filtration time JP-4.

7. The reclamation of high filtration time JP-4 samples was achieved via two different processes:

a. The first procedure involved a "shower" of the acidically buffered water bottom falling through the fuel phase.

b. The other successful method entailed pumping the high filtration time JP-4 up through a water buffer "tower" containing a concentrated solution of a buffering agent. The fuel phase was first broken up into small droplets to increase the contact area.

8. The corrosion inhibitors tested revealed major differences in the settling period required to return the fuel's filtration time to less than 15 minutes. Under the laboratory conditions of ultrasonic agitation of a basic, hard water bottom and corrosion inhibited fuel, all currently accepted inhibitors produced a very high filtration time fuel in two days. Only AFA-1 did not generate a problem fuel in the first day of agitation. However, NALCO 5400-A, deleted from the 1975 list of qualifying inhibitors, failed to cause a problem JP-4 under any test conditions.

SECTION V  
RECOMMENDATIONS

Based on experimental results in this Lab, the following measures should be enacted to ensure that the lowest possible filtration-time JP-4 be readily available:

- a. Maintain a minimum of water bottoms in all fuel storage, transfer, and transport systems.
- b. Gently buffer all easily accessible water bottoms to a slightly acidic pH.
- c. Study further the "shower" and "tower" approaches to fuel reclamation, or any procedure involving an acidic water-washing of problem fuel.

The most promising answer to the "soap" formation problem does not lie in the judicious selection of the "right" corrosion inhibitor, but rather in controlling the water bottoms volume and chemistry.

SECTION VI  
REFERENCES

1. Hayes, Paul C., Jr., Development of an Improved Fuel Filtration-Time Test, AFAPL-TR-74-33, September 1974.
2. McLaren, G. W., Krynitsky, and Hazlett, R. N., Effect of Corrosion Inhibitors on Jet Fuel Filtration, NRL Memorandum Report 1660, November 1965.
3. Martel, Charles R., et al., Aircraft Turbine Engine Fuel Corrosion Inhibitors and Their Effects on Fuel Properties, AFAPL-TR-74-20, July 1974.
4. ~~NRL-7-5624K~~  
*Specification MIL-T-5624K, Aviation turbine fuel, grades JP-4 and JP-5.*

APPENDIX A  
FILTRATION TIME-SOLIDS LEVEL TEST

**EQUIPMENT:**

1. Millipore Filter Membranes; 0.8 micron, 47 mm diameter, plain white No. AAWP04700
2. Millipore Stainless Steel Hydrosol Filter Holder with grounding clamp No. XX2004720
3. Four liter filter flask
4. Four liter graduated cylinder
5. Vacuum pump with gauge; 0-30 inches of mercury
6. Timer; in minutes and hundredths of minutes
7. Balance reading to at least 0.1 mg
8. Petri dishes; pronged bases with covers, approx. 50 mm diameter
9. Drying oven; dust free, maintained at 90°C
10. Particle free petroleum ether (filtered through 0.45 micron membrane)
11. Dessicator with indicating Drierite
12. Thermometer reading from 0 - 100°F
13. Tweezers, stainless steel, flat-tipped
14. Plastic wrap

**SAMPLE:**

One gallon of fuel in a vessel (previously particle-freed)

**PROCEDURE**

1. Particle free wash Hydrosol Filter Holder, graduated cylinder, and petri dishes.
2. Oven dry Millipore filter membranes in petri dishes for 30 min at 90°C.
3. Allow filters to cool for 30 min in a dessicator
4. Weigh membranes to nearest 0.1 mg, discard any outside the 75-85 mg range.

5. Place one gallon stainless steel orificed disk onto filter base, then filter on top of this, then secure cap to base and cover with plastic wrap.
6. Attach grounding clamp, install assembly into filter flask, and connect vacuum line.
7. Fill separatory funnel from vigorously shaken one gallon jug with 3785.3 ml of fuel and stopper.
8. Record fuel temperature after several minutes to the nearest 0.2°C only if within 20-30°C range.
9. Release fuel into filter cup; switch on pump and timer.
10. Regulate vacuum to 25 +/- 0.2 Hg throughout the run.
11. **CAUTION:** NEVER ALLOW FILTER PAD TO RUN DRY DURING THE TEST!
12. As last of fuel passes through filter, stop timer and record the filtration time in minutes and hundredths of minutes.
13. Wash sample container with four 25 ml flushes of filtered pet ether and empty into cup.
14. Wash down cup with pet ether, shut off vacuum, remove top of filter holder.
15. Gently wash particles on filter pad from the outside inward with pet ether.
16. Place filter in pronged petri dish, cover, oven dry for 30 min at 90°C, cool in dessicator for 30 min.
17. Weigh membrane and record to nearest 0.1 mg.
18. Calculate any weight change by difference.
19. Report filtration time and membrane weight gain as solids level.

APPENDIX B  
SYNTHETIC SEA WATER

(EXCERPT FROM D665-135)

6.2 The solution can be conveniently prepared as follows. The method avoids any precipitation in concentrated solutions with subsequent uncertainty of complete resolution. Using cp chemicals and distilled water, prepare the following stock solutions:

<i>Stock Solution No. 1:</i>	
MgCl <sub>2</sub> ·6H <sub>2</sub> O	3885 g
CaCl <sub>2</sub> (Anhydrous)	406 g
SrCl <sub>2</sub> ·6H <sub>2</sub> O	14 g
Dissolve and dilute to 7 liters	
<i>Stock Solution No. 2:</i>	
KCl	483 g
Na <sub>2</sub> CO <sub>3</sub>	140 g
KBr	70 g
H <sub>3</sub> BO <sub>3</sub>	21 g
NaF	2.1 g
Dissolve and dilute to 7 liters.	

6.2.1 To prepare the synthetic sea water, dissolve 245.4 g of NaCl and 40.94 g of Na<sub>2</sub>SO<sub>4</sub> in a few liters of distilled water, add 200 ml of Stock Solution No. 1 and 100 ml of Stock Solution No. 2 and dilute to 10 liters. Stir the 10-liter batch and add 0.1 N Na<sub>2</sub>CO<sub>3</sub> until the pH is between 7.8 and 8.2. One or two milliliters of the carbonate solution will be required.

APPENDIX C

CALCIUM AND MAGNESIUM ANALYSIS

<b>AEROSPACE FUELS LABORATORY TEST REPORT</b>		<b>ITEM TO BE TESTED</b> <b>WATER SAMPLES</b>
<b>TEST LABORATORY AND LOCATION</b> Aerospace Fuels Laboratory SFQLA WPAFB OH		<b>SPECIFICATION NUMBER</b> N/A
<b>SAMPLE NUMBER</b> 74-1369-M & 74-1370-M	<b>DATE SAMPLE RECEIVED</b> 8 October 1974	<b>DATE SAMPLE TESTED</b> 15 November 1974
<b>CONTRACTOR</b> N/A		<b>CONTRACT NUMBER</b> N/A
<b>MANUFACTURER</b> N/A		<b>PURCHASE ORDER NO AND/OR FEDERAL STOCK NO</b> N/A
<b>SUBMITTED BY</b> AFAPL/SFF Attn: Paul Hayes		<b>SAMPLE MARKED</b> Tap Water & Synthetic Sea Water
<b>TEST RESULTS</b>		
<b>PURPOSE OF INVESTIGATION:</b> To determine the total solids and the amount of calcium and magnesium present in each sample.		
<b>DESCRIPTION OF SAMPLES:</b> One quart of each material was submitted for analysis.		
<b>DESCRIPTION OF ANALYSIS:</b> The total solids analysis was performed using the standard procedure. The values for tap water and synthetic sea water were, 611 ppm and 45,308 ppm, respectively.		
The calcium and magnesium content were determined by the Atomic Absorption Method. The magnesium content was 61 ppm for tap water and 720 ppm for synthetic water. The calcium content was 235 ppm for tap water and 730 ppm for synthetic water.		
<b>CONCLUSION:</b> None, the results were reported to the initiator. The initiator believed the calcium result might be slightly low. However, he admitted the container with the synthetic sea water had a precipitate on the bottom, possibly a calcium compound, explaining the low value.		
<b>REMARKS</b>		
<b>REVIEWED BY (Signature, date and organization symbol)</b> WILLIAM J. CRAWFORD 12 Dec 1974		<b>APPROVED BY (Signature, date and organization)</b> THOMAS J. O'SHAUGHNESSY Chief, Test Laboratory Directorate of Aerospace Fuels

SAAMA FORM 216 JUN 72 REPLACES SAAMA FORM 215B WHICH WILL BE USED.