### FOREWORD

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This report covers work performed from initiation of the contract effort on June 15, 1963, to May 15, 1964.

The work was directed for Ashland Oil & Refining Company by Mr. Arnold M. Leas.

## ABSTRACT

The thermal stability of nineteen degraded JP-6 type jet fuels was improved to a level in excess of the present MIL-J-25656B specification requirement by a filtration treatment. The improved thermal stability of these reclaimed fuels was retained for more than six months of ambient temperature storage.

The ASTM-CRC Coker was used to measure thermal stability. The coker ratings could not be correlated quantitatively with the chemical and physical analyses because of the minute quantity of the contaminants. However, with the use of filter media these contaminants were concentrated sufficiently to show some degree of correlation with the coker ratings.

Many of the additives present in these military fuels as well as those considered as possible future additives were removed in varying degrees by reclamation filtration. The generation of static electricity, filter media life, process economics, and design variables were other parameters which were investigated.

This technical documentary report has been reviewed and is approved.

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## I. INTRODUCTION AND OBJECTIVES

There exists a gap in quality level of high-temperature jet fuel between the shipping point and the use point. One possible process method to close this gap in quality level is to pump the jet fuel through an in-field reclamation filter at the use point.

Prior to the date (June 14, 1963) of this contract award, the contractor had developed, independently and wholly upon his own initiative, a process for rehabilitating thermally degraded jet fuels upon which the contractor has filed a patent application on April 30, 1963, as Serial No. 276865 for United States Letter Patent. It has been demonstrated commercially within the last five years that this process has the ability to restore degraded jet fuels at the refinery or use point to a quality level as good or better than the original refined fuel at the production point. The Air Force, by acceptance of the contractor's proposal, desired the contractor to test the process with a wide range of degraded fuels to determine the full limitations and capabilities of such process for possible Air Force use. Details of this contractual agreement are available from the proper agency within the Air Force.

Business ethics made it difficult to document all of the case histories which resulted in considerable economic loss to the military, engine builders, and fuel suppliers as a result of degradation of delivered fuels which were rejected at the destination points because of loss in thermal stability. In some cases some of the degraded fuels in large storage tanks had to be downgraded into JP-4 type fuels by blending higher vapor pressure components to meet the quality standards even on the downgraded products. In other cases the fuel suppliers had to absorb the round-trip freight cost for returning the fuels to the production point and the additional rehabilitation expense at the refinery. In the interim period of such cases the consumer was either required to discontinue his operations or to procure alternate supplies. This new parameter of instability of such fuels during transit and storage is obviously untenable for both the supplier and the consumer. A few years ago a solution to this problem was found by the use of in-field reclamation filters. The success of these commercial reclamation units at the use points during the last few years attracted the attention of the military to evaluate this process for rehabilitating many different types of degraded fuels on a pilot plant basis. Hence, this research effort was initiated and completed to determine the full capabilities and limitations of this process.

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Recently the contractor has installed five additional commercial filters at use point terminals for various processing rates ranging from 150 GPM to 1500 GPM. The purpose of these new filters is to insure the removal of all degrading contaminants that may accumulate during previous storage and transportation by barge, tanker, tank car, tank truck, and pipeline.

In addition to reclamation filtering, other methods of fuel rehabilitation were considered. The redistillation method can be used quite successfully to reclaim degraded jet fuels. Table 1 shows that most of the contaminants and additives can be concentrated in the 5% distillation residue as a reject by-product. The 95% distillation overhead is relatively free of contaminants and quite thermally stable. The test data on redistillation products listed in Table 1 show that the 5% residue concentrated such contaminants as copper, lead, indenes, naphthalenes, and peroxides. The residual additives were also concentrated in the 5% distillation residue.

However, redistillation is not a very practical method to use in the field because of its prohibitive cost, i,e. the direct operating cost of redistillation reclamation ranges between 0.5 and  $2.0\phi/\text{gallon}$ . The total operating cost of such redistillation at the use point is prohibitive. The hydrotreating process can be used quite successfully to rehabilitate degraded fuels. Hydrotreating removes oxygenated, sulfonated, halogenated, and nitrogenated organic contaminants as acidic vent gases. Again, such hydrotreating operating costs are prohibitive (1 to  $3\phi/\text{gallon}$ ) with some material loss as light catalytic cracked by-products. Another adverse economic consideration is that hydrotreating catalyst is poisoned by jet fuel contaminants and additives.

At the beginning of this research project, the contractor set aside about 30,000 gallons of degraded jet fuels in isolated, clean, carbon steel storage tanks or drums to be available as required. Tables 2 and 3 show the code identification system for all of these fuels. To differentiate these storage degraded fuels for this reclamation work, the first ten fuels received from the Aero Propulsion Laboratory were recoded Reclaim Nos. 1 through 10. These Air Force coded commercial JP-6 and thermally stable fuels were produced by several different suppliers. were all specification products at the shipping point but had become thermally unstable in the customers' tankage. These fuels were drummed, retested in the Aero Propulsion Laboratory to confirm the bad coker ratings, and shipped to the contractor for processing through reclamation filters. Reclaim Nos. 11 and 12 fuels had previously been in terminal and customer's storage for about two years. These fuels were likewise shipped as specification fuels at the shipping point, but had become thermally unstable in the customer's tankage after about one year's storage, Two transport truck loads of each of these fuels were purchased by the contractor and stored in clean carbon steel tanks. Reclaim Nos. 13 through 19 were blends of Reclaim No. 12 and thermally stable fuels, thereby providing varying degrees of thermal instability.

To date about 5,000 gallons of these fuels have been processed through three coker machines and about 9,000 gallons have been processed through twelve reclamation filter pilot plants. Fifty-five gallons of each of the nineteen fuels have been filtered and placed in ambient storage without readdition of additives. An additional drum of each of Reclaim Nos. 11 through 19 has been filtered and stored with readdition of additives. All of these filtered fuels have been tested on the coker following the three and six month storage periods at ambient temperature. These fuels were stored under the same roof where the ambient temperatures during the course of this period varied from plus 10 to plus 100 F and the relative humidity varied from 20 to 100%. The storage containers were vented to the air for normal breathing. The residual filtered fuel samples have been forwarded to the Aero Propulsion Laboratory for long time storage evaluation.

This report shows performance data obtained from existing commercial and laboratory filter units as the result of processing many storage degraded fuels. The primary objectives of this research effort were to:

- 1. investigate treating methods to rehabilitate storage degraded fuels.
- 2. test the performance of a reclamation filter for processing many different jet fuels that had degraded during storage at air bases and terminals.
- 3. document all useful test data obtained from nineteen contract fuels before and after reclamation filtering to permit defining the detrimental contaminants that caused the degradation of the fuels.
- 4. recheck the coker ratings on these reclamation filtered fuels after additional storage at ambient temperature. These coker ratings were made after three months and six months to determine whether or not degradation would occur again on these reclaimed filtered fuels.
- 5. determine the effect of additives and designated contaminants on the reclamation filtering process.
- 6. determine whether the reclamation filter unit would remove detrimental contaminants, viz. biological, organic and inorganic (soluble and insoluble), and moisture.
- 7. determine the effect of design and operating variables of the reclamation filtering process.
- 8. recommend the optimum design of a field filtration unit that would be useful and practical to the military for rehabilitating storage degraded jet fuels.

## II. METHOD FOR RECLAMATION FILTRATION

As shown in Figure 1, the reclamation filter unit consists of two zones. In the first zone the fuel flows upward through a chemical dryer which consists of a free water coalescing and settling area with bottom water drain facilities and a chemical drying area removing emulsified and soluble water. In the second zone the dried fuel flows downward through a filter which consists of a fine mesh activated media which removes particulate matter (including submicronic), soluble chemical and biological contaminants, etc;; a coarse mesh activated media which completely retains the above media; and three additional layers of coarser but completely inert graded metallurgical aggregate disengaging clean fuel and completely retaining the filter media.

Carbon steel vessels have been used successfully to minimize the cost and to provide flexibility in charging and dumping the filter media. To date the standard media continue to be most successful; however, the same hardware could readily adapt itself to possible improved filter media.

With the use of this filter the data presented later in this report demonstrate that this process can remove detrimental contaminants, viz. biological, organic and inorganic (soluble and insoluble), and moisture.

### III. DISCUSSION

### A. Thermal Stability of the Test Fuels

The Standard ASTM-CRC Coker and the Modified Coker were used to measure thermal stability of the test fuel samples. Figures 2, 8, 14, and 20 (bar graphs) show the maximum preheater coker ratings (450/550/6) for the test fuel samples: the degraded fuel as received, treated fuel immediately after reclamation filtration, treated fuel six months after reclamation filtration. Figures 3 through 7, 9 through 13, 15 through 19, and 21 through 24 (photographs of coker preheater tubes) show comparative thermal stability ratings for all fuels before and after filtration treatment. In the photographic work, it was necessary to fabricate a large tuberator housing in order to obtain authentic photographs of the maximum deposits on the cylindrical preheater tubes. By this means the photographs are comparable to visual inspection through the standard tuberator.

All of the filtered fuels were improved from failing (425/525/6) to passing on thermal stability as measured by the coker rig when operated at 450/550/6 severity. Likewise this improved thermal stability rating of the filtered fuels remained at the passing level after both the three months and six months storage periods at ambient conditions.

Appendix I shows the complete coker preheater ratings from which the graphical data were obtained. Appendix II shows the coker threshold stability data on the original fuels (Reclaim Nos. 1 through 10) as rated by the Aero Propulsion Laboratory. Nearly all of the unfiltered fuels tested continued to drop in thermal stability during the same storage time and under the same conditions. The data in Appendix III show the continued degradation of the unfiltered fuels during storage through the contract period.

The coker test data listed in Table 4 show that processing doctor treated kerosene through the reclamation filter increased the initial coker threshold temperature from 375/475/6 to 475/575/6. Table 5 shows the physical and chemical test data for the original doctor treated kerosene. Generally speaking, an aged doctor treated kerosene can be filtered to gain about 50°F coker threshold temperature whereas a desulfurized aged kerosene can be filtered to gain about 150°F coker threshold temperature.



### B. Physical and Chemical Tests on Fuels

Table 6 shows comparative physical and chemical test data for all fuel samples included in this program: the degraded fuel as received, treated fuel immediately after reclamation filtration, treated fuel six months after reclamation filtration. Appendix IV lists the sources of the chemical and physical laboratory procedures.

These comparative physical and chemical test data failed to identify clearly the offensive contaminants. However, these test results did show in most cases that in the filtered fuel samples there was some reduction in olefins, peroxides, indenes, pyrrole and basic nitrogen, surfactants including organic sulfonates, naphthalenes, naphthenic acids, sulfur, iron, lead, copper, phenols, spent additives, soluble water, gums, and particulate matter. Likewise, in the filtered fuel samples the water separating characteristics were improved as measured by the water tolerance, WSI, and WSIM tests. The regeneration of the filter media also confirmed that these same contaminants were removed from the treated fuels during the filter operation. These contaminants were concentrated in the extracting solvents during the regeneration cycle. This will be discussed in more detail later in this report under section "D".

### C. Removal of Biological Contaminants With Reclamation Filter

Previous preliminary checks of fungal and bacterial removal from other petroleum products by application of the reclamation filter have indicated that microorganisms in the magnitude of even less than 0.1 micron are completely removed from hydrocarbon fuels by use of this process.

By arrangement with the Fuels and Lubricants Branch of Wright-Patterson Air Force Base, the University of Dayton Bacteriology Department supplied three different species of Pseudomonas bacteria in pure culture, one each on Bushnell-Haas mineral salts agar slants with JP-4 overlay and one each on trypticase soy agar. These were designated as B-40, B-44, and B-54. Two different species of fungi in pure culture were supplied, each on Sabouraud's agar slants. These were designated as B-29 and B-55.

In order to substantiate the preliminary work in this area, specific samples of JP-6 jet fuel were deliberately inoculated with fungi and bacteria, both separately and together, and filtered through the reclamation filter. The resultant effluent was filtered through a 0.45 micron Millipore filter to remove or to detect any remaining microorganisms. The filters were then incubated at 37 C in media suitable for the specific microorganisms for a one-week period. The cultures were inspected visually every day for potential growth and by use of both optical- and electron-microscopy every other day.



The fifteen-day cultures showed that the fuel which had passed through the chemical dryer section of the unit still contained a few of the injected microorganisms. The fuel which had filtered through both the chemical dryer and filter sections proved to be completely free of any bacteria or fungi, thus confirming the success of this process for removal of microscopic entrainments. In Figure 25 the fungi are traced through the filtration unit.

Other photomicrographs, Figures 26 through 39, illustrate the positive identification of bacteria and fungi prior to filtration and the negative identification following filtration. Figure 40 shows the effect of biological contaminants on coker preheater tubes. The coker rating confirms that fungi and bacteria leave a deposit on the preheater tube when operated at 450 F. The uninoculated fuel gave a maximum preheater code of 1 while the inoculated fuel gave a maximum preheater code of 2.

The procedures for propagating the cultures, inoculating the fuels, microscopically examining the fuel samples, and determining the results are presented in Appendix V and Tables 7, 8, and 9.

Three complete runs were made using different filter units (each conforming to the specifications of the contractor's reclamation filter). One test run was made using the bacteria alone, one using fungi only, and the final test run was made using both bacteria and fungi. It was found that in all three test runs no bacteria, no fungi, or spores were found to have passed through the active sections of the filter units. This is illustrated in photographs shown as Figures 25, 33, 35, and 37.

The depth of penetration by the microorganisms into the filter media indicated that all of these microorganisms were retained on the entrance layers of the filter media. This indicates that the filter media life for removing biological contaminants would be much greater than for other types of contaminants. Therefore biological contaminants are not the controlling contaminant for filter media.

From the results obtained, it can be concluded that the reclamation filter successfully and completely removed the injected microbiological contaminants from the JP-6 jet fuel utilized for the project.

#### D. Removal of Other Contaminants With Reclamation Filter

The other apparent detrimental contaminants to thermal stability which are removed by the reclamation filter media consist of naphthalenes, olefins, indenes, phenols, pyrrole and basic nitrogen, sulfonates, peroxides, sulfur, naphthenic acids, surfactants including organic sulfonates, lead, iron, copper, gums, spent

additives, and particulate matter. The free and emulsified water containing soluble salts is also removed by the chemical dryer. Confirmation of the removal of these contaminants was evident upon analyzing the extract from the regeneration of the filter media.

A number of the pilot plant reclamation filtration units used for determining the life of the filter media were shut down, drained, and purged with nitrogen in preparation for regenerating the filter media. Trisolvent (benzene-acetone-isopropanol) was pumped into the reclamation filter and allowed to stand 16 hours, then the unit was flushed with additional trisolvent and drained. This extract was then tested to determine the contaminants removed from the filter media. The results from this determination follow.

- Naphthalenes represented by far the greatest organic contaminant in the extract from the spent filter as measured by the ASTM D-1840-61T spectrophotometric method. A Model DU Beckman spectrophotometer was used for these analyses. Since the jet fuel processed had an Engler distillation end point of 416 F with a 0.5% residue; some of the expected interfering compounds such as phenanthrenes, dibenzothiophenes, biphenyls, benzothiophenes, and anthracenes, were minimized by the original fractionation of the jet fuel. This same processed jet fuel contained only 0.1% naphthalene both before and after the reclamation filter; therefore it appears that the 2.0 and 2.6% naphthalene found in the spent filter media from processing Reclaim Nos. 11 and 12 could have been synthesized from spent additives and contaminants and then retained. Such synthesized degradation products when extracted from the spent filter media gave a positive test for naphthalene by ultraviolet spectrophotometry. Even with such inconsistency, we can still conclude that the real or apparent naphthalene content, when determined by ultraviolet spectrophotometry (D-1840-61T), provides a contaminant identification method for projecting thermal stability. Pilot reclamation filters or their equivalent are required to concentrate such contaminants for extracting and testing.
- 2. Indenes, phenols, and pyrrole nitrogen were also found in much smaller concentrations in the extract from the spent filter media. These data are consistent with the data from the before and after reclamation filtered samples showing partial removal of such contaminants by the filter media.
- 3. Lead, iron, and copper were also found in the spent filter media. Nearly all of the pilot plant runs showed substantial removal of these inorganic contaminants by the filter media. The commercial reclamation filters showed removal of these inorganic contaminants.



The 24-foot pilot plant reclamation filter was shut down after an extended run for a similar regeneration with the exception that each 8-foot top, middle, and bottom section was analyzed separately. The data in Table 10 show the analyses of the contaminants removed from the filter media. The most detrimental contaminants and additives were completely removed in the top and middle filter sections.

When dismantling several of the reclamation pilot plant units, it was observed that a grease-like material accumulated in the void space between the chemical dryer and the filter. Most of this grease-like material had been retained on the entrance layer of the filter media and none of it had penetrated more than one inch into the filter media. Some of the other chemical reactions are not completely understood. However, the positive removal of contaminants from jet fuel is quite evident as shown in Table 11.

Some of these contaminants were deliberately injected into the jet fuel to determine their effect on thermal stability. These were petroleum sulfonates, peroxides, indenes, and naphthenic acids.

Petroleum sulfonates were retained on the filter media yielding treated fuels free of these soluble organic contaminants as shown in Table 12. While petroleum sulfonate contaminant is very detrimental to the water separometer test, such a contaminant is not detrimental to the coker test when operated at the 475/575/6 severity level on JP-6 jet fuel.

Benzoyl peroxides were retained on the filter media yielding treated fuels free of these soluble organic contaminants as shown in Table 13.

Indenes were retained on the filter media yielding treated fuels free of these soluble organic contaminants as shown in Table 14.

Naphthenic acids were retained on the filter media yielding treated fuels free of these soluble organic contaminants as shown in Table 15.

Fuels that become off-test on the copper strip corrosion during storage due to generation of elemental sulfur by bacterial action become quite troublesome in the field. Confirming our original commercial reclamation filter data on both JP-6 and Mach 3 type fuels, our more recent laboratory data indicate that:

 elemental sulfur can be generated in aged jet fuels, following improper fractionation or bacterial action, yielding a bad copper strip corrosion test during storage, 2. by adjusting the promoter agent to the filter media, the elemental sulfur can be removed from the reclamation filtered fuel without degrading other fuel qualities such as thermal stability.

The supporting data for these conclusions are shown in Table 16.

Test fuels, Reclaim Nos. 1 through 10; contained considerable phenols on the before treats (as received) while the after treated fuel samples contained only a small amount of phenols. This indicates that reclamation filtering removed phenols which in turn may have been partially responsible for the improved thermal stability rating of these types of fuels. Reclaim Nos. 11 through 19, manufactured by a different process, contained very little phenols on both the before and after treats which indicates that phenols were not responsible for the bad thermal stability ratings on these before treats. The data in Table 17 support these conclusions.

E. Effect of Additives on Reclamation Filtering Process

General observations to date from both the pilot plant filters and commercial filters indicate that:

- some approved jet fuel additives, namely antioxidants and metal deactivators, that are normally included in jet fuel usually improve the performance of the reclamation filter media.
- 2. one approved jet fuel additive, namely icing inhibitor (ethylene glycol monomethyl ether with or without glycerol), usually reduces the effective life of the reclamation filter media.

Table 18 shows the antioxidant and metal deactivator content before and after reclamation filtering on all of the fuels. These data indicate that such additives may need to be readded to the filtrate after the initial filtration treatment. The data on the before treats indicate that thermal stability on these fuels deteriorated badly even though additives were still present in the aged fuels. Of the uncombined antioxidant and metal deactivator that were originally added to the freshly produced fuels, approximately 25% was still remaining in Reclaim No. 11 degraded fuel as received. During the course of these life tests on the filter media, nearly all of this uncombined and available antioxidant and metal deactivator was eluted in the treated fuel as effective additives. Tables 2 and 3 show that the coker break points of these aged fuels are below specification. These fuels contained nearly maximum concentrations of additives when freshly produced.

The ASTM-CRC coker ratings deteriorated with the addition of the military approved corrosion inhibitors. These data on JP-6 rehabilitated fuels confirm similar data on freshly produced commercial JP-6 type fuels which show:

- 1. that coker preheater ratings at temperatures above 425 F begin to deteriorate when the fuel is treated with minimum allowable concentrations of all current military approved corrosion inhibitors. Occasionally some of these corrosion inhibitor treated fuels have a higher threshold temperature, but additional testing shows inconsistency in the occasionally improved performance. These data indicate threshold break points of the corrosion inhibitor treated fuels below that of the virgin JP-6 jet fuel.
- 2. that reclamation filters can be used to remove some of the corrosion inhibitors from such inhibited fuels at the use point terminal thereby improving the coker rating of the filtered fuels. This approach has significant value when considering that jet fuel could be corrosion inhibited at production point, transported to destination, and reclamation filtered at the use point to minimize possible corrosion of storage and shipping containers. However, individual research would be required on a specific fuel to determine which contaminant (iron rust versus corrosion inhibitor) would be controlling in a specific fuel handling system.

Figure 41 shows that the military approved corrosion inhibitors when used in JP-6 jet fuel generally degrade a coker rating. It also shows that a portion of the corrosion inhibitor can be removed by a reclamation type filter which in turn improves the coker rating at 450/550/6. Table 19 shows the detailed coker preheater ratings supporting Figure 41.

The Aero Propulsion Laboratory used their reclamation filter to process two fuels containing lubricant additives. These two fuels were sent to Monsanto's Dayton laboratories for infrared identification of these lubricant additives in the samples before and after the reclamation filtration unit. The two runs that were made on the reclamation filtration unit showed that both lubricant additives were retained on the filter media and thereby removed from the treated fuels. These data are shown in Table 20 and indicate that such lubricant additives should be injected downstream from the reclamation filter. However, if a lubricant additive would become incompatible to the fuel, such fuel could then be pumped through the reclamation filter to remove the objectionable additive.

Figure 42 shows the infrared spectra for the base fuel, duplicate runs for the base fuel plus lubricant additive No. 1, and for the filtered fuel. Figure 43 shows similar spectra for lubricant

additive No. 2. The spectra representing the filtered fuels are identical to those of the base fuel with no additive.

A typical commercial antistatic additive (3 ppm) caused the deterioration of the thermal stability of JP-6 jet fuel with the coker operating at 450/550/6. These data are shown in Table 21.

## F. Static Electricity Generated by Reclamation Filter

Most of the static electricity studies predate this contract work since safe laboratory procedures were required to conduct the original research work to develop a practical reclamation filter. In this original work, three phases of static electricity were evaluated.

#### 1. Conductivity

Since reclamation filtering involves removing contaminants from the fuel processed, conductivity of the filtered fuel is decreased, as would be anticipated, with the use of a good jet fuel filter. Conductivity measurements varied considerably depending upon the quality of the fuel charge and its additive content, the contaminants removed, and upon the quality of the treated fuel required. If the reclamation filter accomplishes its primary objectives of removing contaminants, the conductivity parameters must become of secondary consideration.

### 2. Static charge build-up

The design of the reclamation filter has been made to reduce drastically the potential static charge build-up. These design features include relatively low fluid velocities (3 feet per minute), support media with neutralizing and/or relaxing components, grounding facilities, and media promoting agents that tend to lessen generation of static charge.

#### 3. Fire hazard measurements at effluent product receiver

Drastic laboratory environment was arranged to initiate combustion at the product receiver. Ungrounded metal receivers were used to receive the fuel by top fill splash loading in an air atmosphere. The third component for ignition of the fuel became the controlling variable -- static electrical charge. The original laboratory work included varying the fuel flow rate from 1 to 300% of the recommended design flow rate, varying the fuel type to cultivate explosive ranges in the lean and rich zones and on both sides of these air-vapor ratios at the receiving tank, varying flow temperatures from minus 20 to plus 130 F (above and below flash points of

different fuels), etc. To date, no actual fire flashes have been ignited by the static electricity generated by these reclamation filter runs.

While the number of laboratory runs prior to this effort were more numerous and more conducive to static electrical fires than the present laboratory runs, these runs also consisted of top splash filling in ambient air of about 1800 five-gallon metal cans (ungrounded) with treated product stream from the pilot plant reclamation filters. The potential hazards using any method of fuel handling should never be minimized. However, these laboratory runs as described above and actual commercial runs indicate that with well grounded reclamation filters — the static electrical discharge from reclamation filters is considerably less than for many other existing high fluid velocity filter-separators. The normal laboratory configuration for reclamation filter units should incorporate grounding of product metal containers with submerged filling spouts.

When testing for static electricity build-up on both laboratory and commercial reclamation filters, it was found that:

- 1. negligible static electricity is generated in processing fuel as it flows through this unit.
- 2. the ability of both the unfiltered and filtered fuel to carry away artificially induced electricity would vary considerably with the type of fuel being processed (with or without additives).
- proper grounding of metal filter units is required for general safety reasons regardless of whether or not a fuel is being processed.
- 4. when the product receiver in the reclamation filter was arranged in an environment which was very conducive to supporting combustion if static charge was generated, no ignition occurred indicating negligible static generated within the reclamation filter.

#### G. Life of Filter Media

Figure 44 shows the treating costs of the reclamation filter media when processing different degraded fuels. Appendix VI shows supporting data for Figure 44.

Life of the filter media is decreased with increasing soluble organic contaminants or increasing coker severity. Both pilot

units and commercial reclamation filter units have demonstrated that:

- 1. the greater the concentration of soluble organic contaminants, the lower the life of the filter media. However, this relationship is not directly proportional to the total amount of soluble organic contaminants since some organic contaminants elute sooner than others and some are more detrimental than others.
- 2. the greater the severity of the coker test conditions on the treated product, the lower the effective life of the filter media used for reclamation. This relationship approaches a logarithmic function.
- 3. the analytical complexity of reclamation filtering is such as to limit precise predictions of the life of filter media, however field experience and pilot runs give a reasonably good basis for design calculations on all grades of jet fuel processing. Obviously the degree of accidental contamination cannot always be predicted.

A specification JP-6 jet fuel containing antioxidant and metal deactivator in allowable concentrations when processed through a reclamation filter soon saturates the filter media with these additives without affecting the ability of the filter media to remove other contaminants. After filtering approximately 100 barrels of fuel per ton of filter media, these additives elute into the effluent in their original effective form. However, icing inhibitors do seriously decrease the life of the filter media. It was found that injecting the icing inhibitor following the reclamation filter provided a preferable operating procedure. While processing a very bad JP-6 jet fuel (Reclaim No. 11), the filter media life for complete reclamation for improving coker threshold temperature from 350 to 475 F exceeded 13,000 barrels of fuel per ton of filter media. The continued processing of this same fuel with a 16,000 barrels per ton rate showed a gradual increase in naphthalene, indenes, pyrrole nitrogen, copper, lead, and iron bleeding into the fuel filtrate.

The foregoing data which show the life of the filter media when processing badly degraded fuels are somewhat distorted since two commercial units have considerably less operating cost when processing larger quantities of fuels. In practice, such filters could be used continuously to process both bad and good fuels in any sequence thereby insuring that all of the filtered fuels will be thermally stable. The continuous processing of all incoming fuels through reclamation filters would insure that all the fuel delivered to the aircraft would be thermally stable and on specification with considerable saving in testing time and cost of

testing the fuels. Additional additives that may be requested for flight benefits could be injected into the effluent stream from the reclamation filter unit with an automated proportional additive pump.

### H. Design Variables of Reclamation Filter

Specifications for jet fuel filter-separators have been well documented for the removal of physical contaminants - namely solids and free water. With the continued more rigid requirement for the removal of physical contaminants, new requirements have been added to remove also chemical and biological contaminants from hightemperature jet fuels at the use point terminals. The usual design variables for filter-separators are viscosity, interfacial tension, temperature, differential density of fuel and water, and the type, quantity, and nature of contaminants to be removed. The new design criteria include the nature, type, and quantity of soluble chemical and biological contaminants and their ease of removal in the presence or absence of fuel additives. These design criteria may incorporate the supplemental flexibility of different filter media, different filter supports, different disengaging hardware along with the efficient disposal and replacement facilities for the active filter media. Optimum flow rates have been established for pilot and commercial filtration units with standard filter media for the different current types of military high-temperature jet fuels based on effective removal of typical contaminants, viz. physical, chemical, and biological.

Figure 45 shows the filter pressure drop versus the flow rate for fuels with varying viscosities. Figure 46 shows the filter pressure drop versus the flow rate for JP-6 fuel for varying filter bed depths.

Table 22 shows practical design data for maximum permissible flow rates on the reclamation filtration unit as taken from the curves in Figures 45 and 46. While other variables may be controlling, it is believed the maximum flow rates through the reclamation filtration unit listed in Table 22 provide a reasonably good design basis for insuring good performance and optimum economics. The cross sectional area of the chemical dryer can vary between 30 and 60% of the cross sectional area of the reclamation filter, depending on the anticipated drying duty. The depth of the filter bed and drier bed can vary with the permissible cost of the hardware but preferably with the anticipated effective-contaminant-load in the fuel to be processed. The effective-contaminant-load will vary with the coker severity level requirement on the treated fuel. For some operating requirements, considerably higher flow rates can be realized provided the charge pump and hardware are designed for the higher pressure drops.



In general, the performance of the commercial reclamation filter units is better than the simulated pilot units. Some of the contributing factors that favor the commercial unit are:

- 1. more consistent jet fuel charge quality
- less contact of the fuel with the metal containers -- gallons fuel/ft<sup>2</sup> metal surface
- 3. less contact of the fuel with air
- 4. fewer experimental operating variables

Since the pilot filter units are less efficient, design data based upon pilot runs provide a greater contingency allowance in commercial installations for accidental mishandling of fuel. In any event, mishandling of fuel should be avoided whenever possible.

### IV. SUMMARY

This research program included evaluating methods of reclamation for storage degraded jet fuel; viz. hydrotreating, redistillation, and reclamation filtering. The data indicate that hydrotreating and redistilling accomplish the reclamation, but these processes would not be practical in field applications where the problem exists. Reclamation filtration has been used commercially for several years in the field. Filter pilot plants were used in this research work to rehabilitate nineteen different degraded fuel samples. It can be concluded that reclamation filters are practical, economical, effective, simple, and safe. Commercial filters can be constructed and installed quickly within new or existing fuel facilities. Perhaps such a filter could be operated effectively with or without filter-separators.

The use of the filter unit could permit deleting some jet fuel additives during the production and transportation of these fuels. The filter could be used at the use point to rehabilitate the fuel and to allow also the injection of additives into the filtered fuel to meet the specific demands of the aircraft during flight. Hot residual fuel from returning aircraft could also be filtered at the air base and then returned to and commingled with fresh fuel.

Since the quality of the reclaimed filtered fuel is possibly better than the freshly refined fuel, laboratory testing could be minimized along with its corresponding time delays. The research data indicate that indenes and other contaminants are generated during ambient temperature storage. Since the greatest demand for stability of fuels is during flight, it is logical to provide for the greatest stability level just prior to fueling aircraft and then to fortify with additives for the specific flight. The use point filter could permit this optimum approach.

The filter also provides a means of concentrating and removing jet fuel constituents for supplemental measuring, identifying, and monitoring purposes. The coker test is much more sensitive than physical and chemical tests for determining thermal stability of high-temperature jet fuels. The filter removes and concentrates most of the contaminants regardless of what type or quantity causes the instability of jet fuels. The filter media are in sufficient quantity to enable several months or possibly several years operation prior to regeneration or recharging of the unit.

The rehabilitation of all of these fuels processed by the pilot filters helps to justify new installations of commercial reclamation filters for processing any storage degraded fuels at military tank farms. The processing of good fuels through filters produces even a higher quality fuel. The occasional or continuous processing and rehabilitating of bad fuels avoids downgrading of these bad fuels. Following their



reclamation filtration they can be commingled with the good fuels in any or all proportions since they have equivalent thermal stability ratings and compatibility characteristics. While most fuel suppliers expect the delivered fuel quality to be satisfactory, too many failures have already been documented at the threshold of the supersonic jet age. The installation of reclamation filters at the use point terminals could close the gap in quality levels and insure the delivery to the aircraft of as clean, dry, and thermally stable jet fuels as is possible with the present state of the art.

### V. RECOMMENDATIONS

- l. Install commercial reclamation filters at several fuel facilities to gain additional experience under full scale application and to provide field developmental data to determine feasibility of more extensive applications.
- 2. Conduct additional research work to develop an in-field thermal stability monitoring instrument to record automatically contaminant levels of jet fuels in storage tanks.
- 3. Conduct additional research work on correlating contaminants (extracted from filter media) with coker ratings on the before and after treat samples from reclamation filters.
- 4. Conduct additional research work on improving regenerative procedures for reclamation filters.

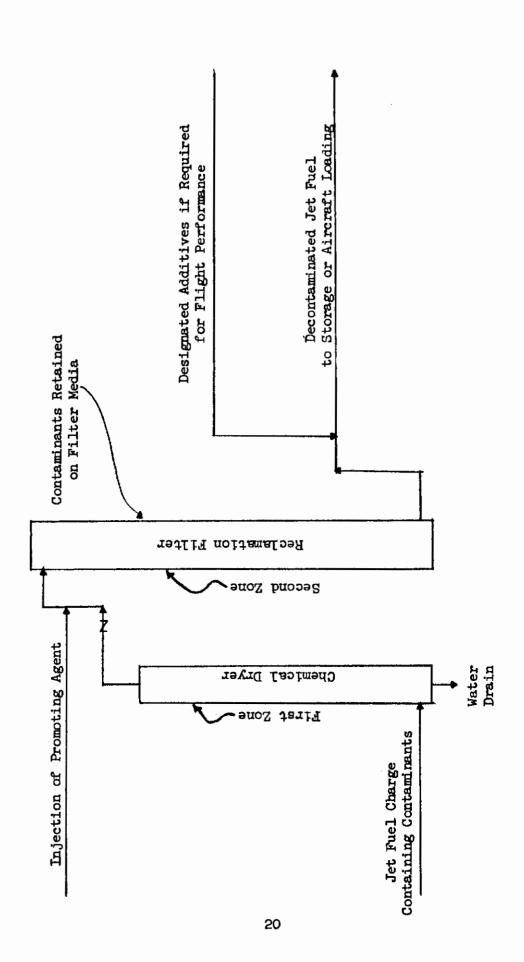
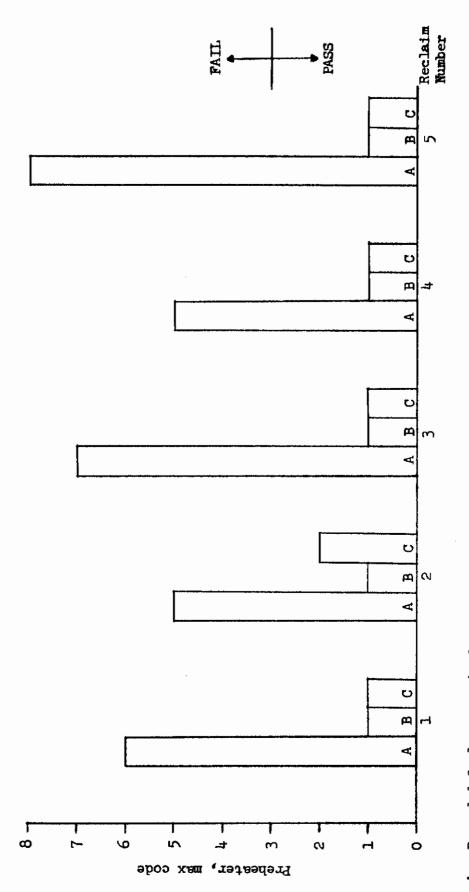


Figure 1. Process Flow of a Typical Reclamation Filter Unit



A - Degraded fuel as received
B - Treated fuel immediately after reclamation filtration
C - Treated fuel six months after reclamation filtration

Figure 2. Thermal Stability Data - Reclaim Nos. 1 Through 5

ASTM-CRC Comparative Coker Ratings (450/550/6)

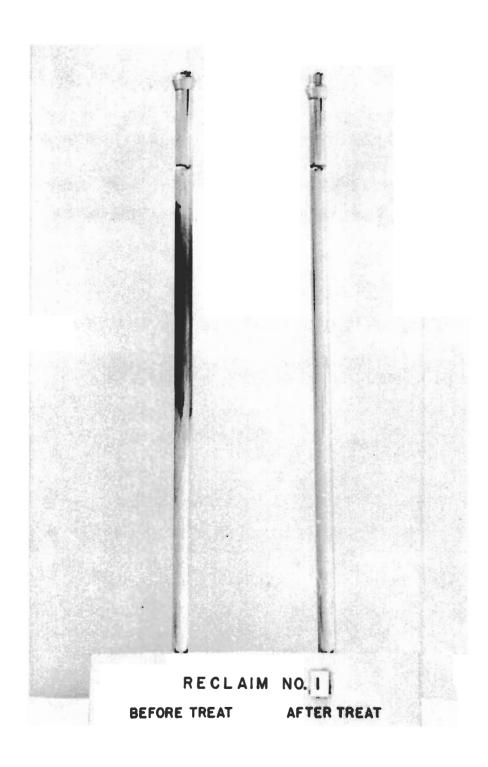


Figure 3. Comparative Coker Preheater Tubes for Reclaim No. 1

"Before Treat" (Code No. 6) represents charge to filter and "After Treat" (Code No. 1) represents effluent product from reclamation filter.

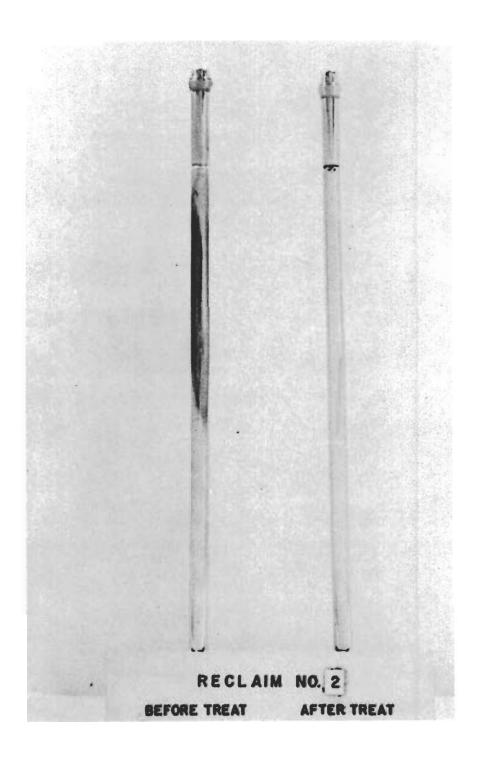


Figure 4. Comparative Coker Preheater Tubes for Reclaim No. 2

"Before Treat" (Code No. 5) represents charge to filter and "After Treat" (Code No. 1) represents effluent product from reclamation filter.

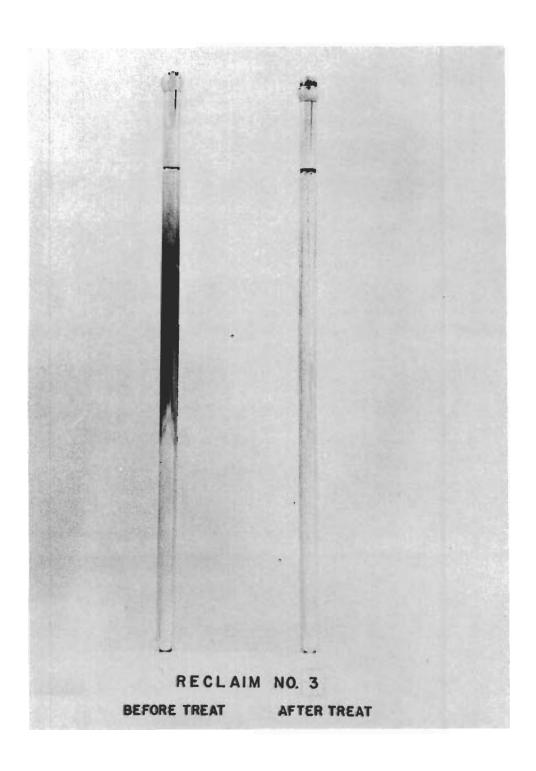


Figure 5. Comparative Coker Preheater Tubes for Reclaim No. 3

"Before Treat" (Code No. 7) represents charge to filter and "After Treat" (Code No. 1) represents effluent product from reclamation filter.

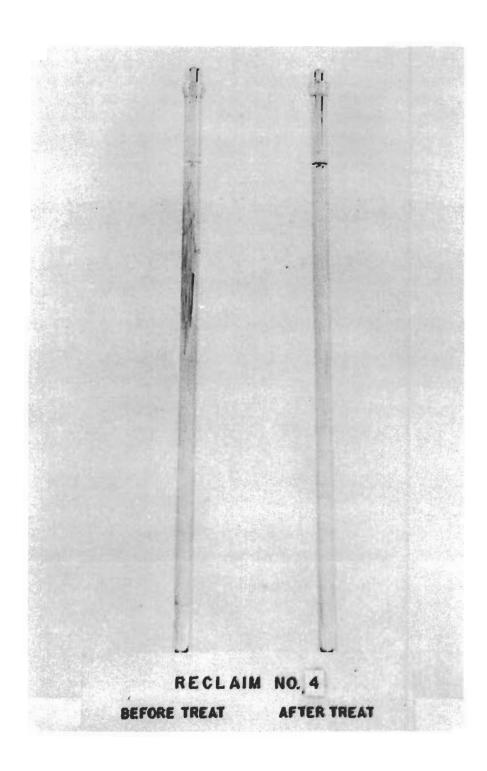


Figure 6. Comparative Coker Preheater Tubes for Reclaim No. 4

"Before Treat" (Code No. 5) represents charge to filter and "After Treat" (Code No. 1) represents effluent product from reclamation filter.

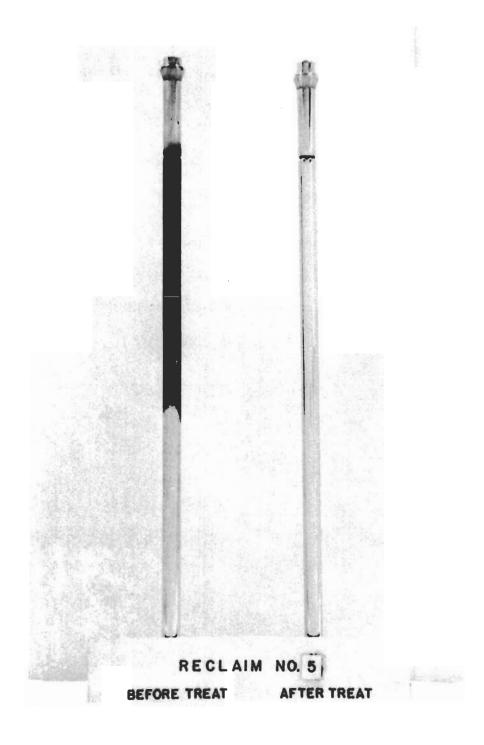
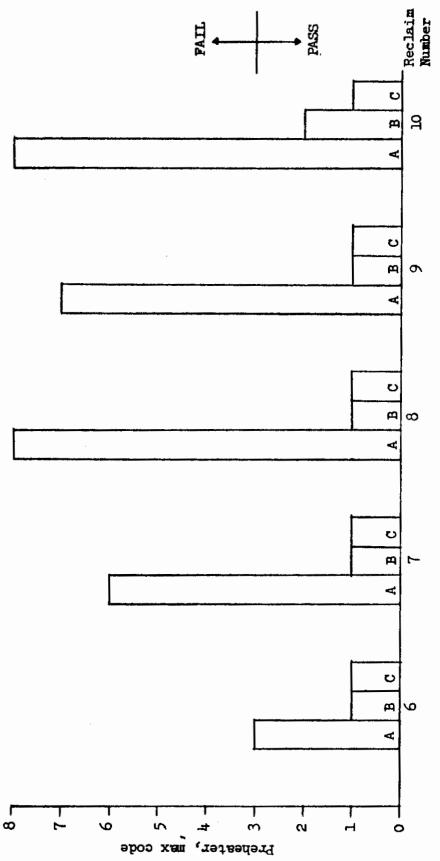


Figure 7. Comparative Coker Preheater Tubes for Reclaim No. 5

"Before Treat" (Code No. 8) represents charge to filter and "After Treat" (Code No. 1) represents effluent product from reclamation filter.



- Degraded fuel as received

A - Degraded fuel as received
 B - Treated fuel immediately after reclamation filtration
 C - Treated fuel six months after reclamation filtration

Treated fuel six months after reclamation filtration

Thermal Stability Data - Reclaim Nos. 6 Through 10 Figure 8.

ASTM-CRC Comparative Coker Ratings (450/550/6)

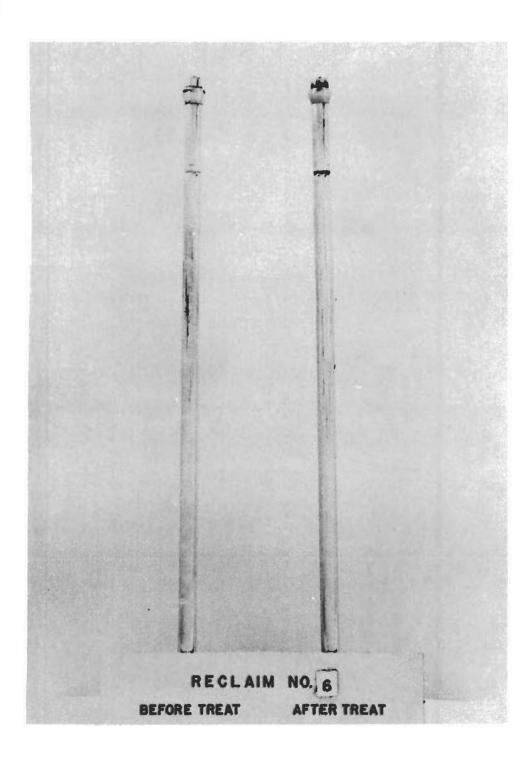


Figure 9. Comparative Coker Preheater Tubes for Reclaim No. 6

"Before Treat" (Code No. 3) represents charge to filter and "After Treat" (Code No. 1) represents effluent product from reclamation filter.

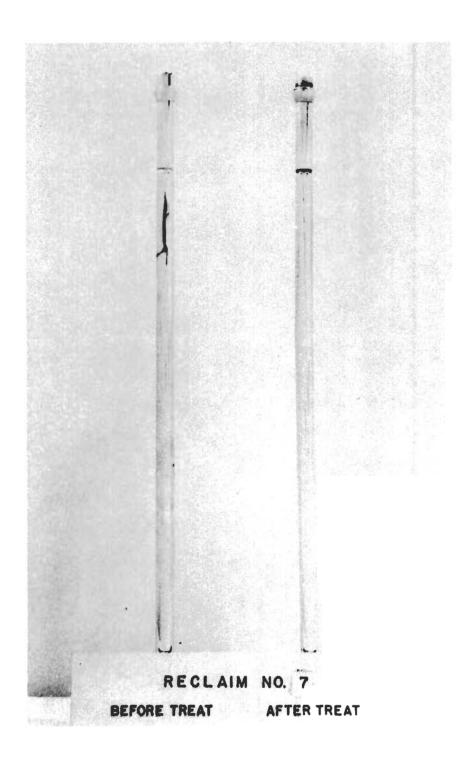


Figure 10. Comparative Coker Preheater Tubes for Reclaim No. 7

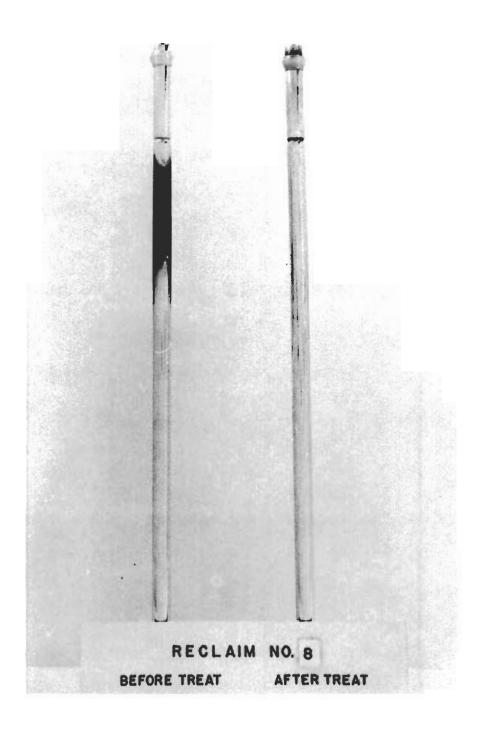


Figure 11. Comparative Coker Preheater Tubes for Reclaim No. 8

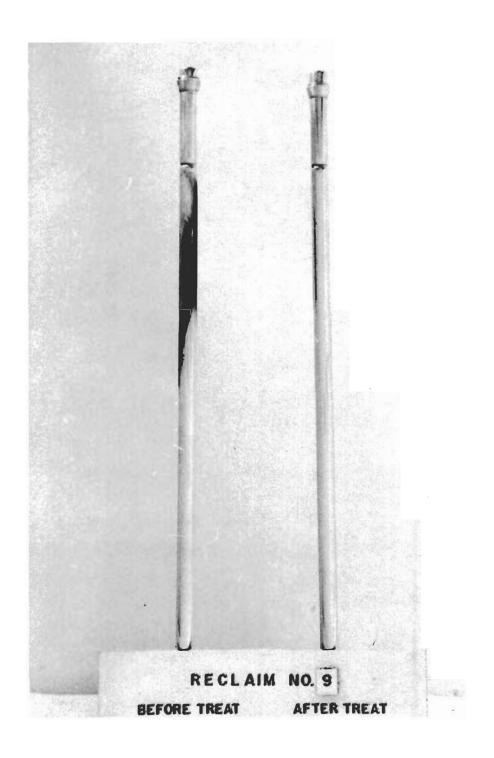


Figure 12. Comparative Coker Preheater Tubes for Reclaim No. 9

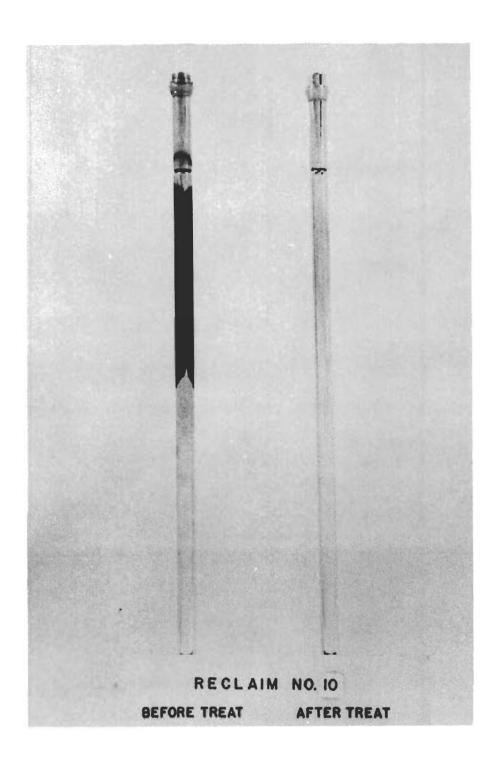
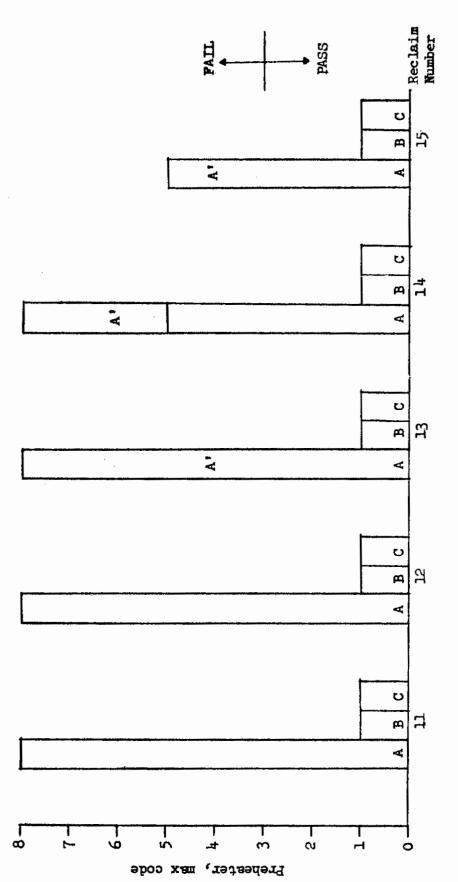


Figure 13. Comparative Coker Preheater Tubes for Reclaim No. 10



A' - second run at close of contract. A - Degraded fuel as received. A' - second run at close of Treated fuel immediately after reclamation filtration C - Treated fuel six months after reclamation filtration - Treated fuel six months after reclamation filtration

Figure 14. Thermal Stability Data - Reclaim Nos. 11 Through 15

ASTM-CRC Comparative Coker Ratings (450/550/6)

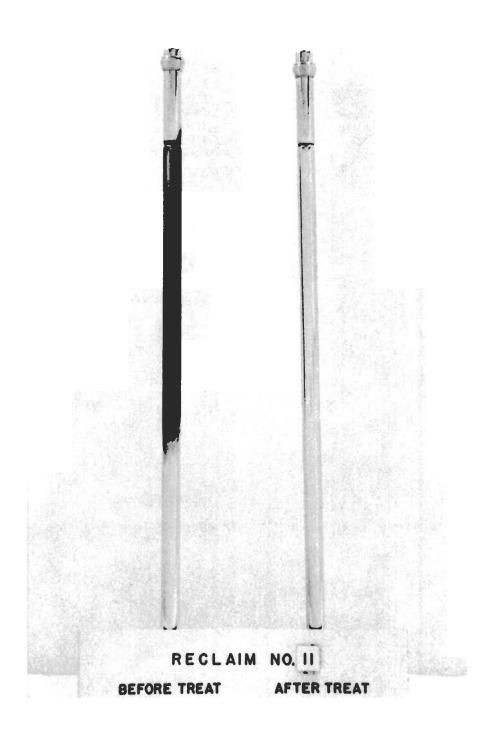


Figure 15. Comparative Coker Preheater Tubes for Reclaim No. 11

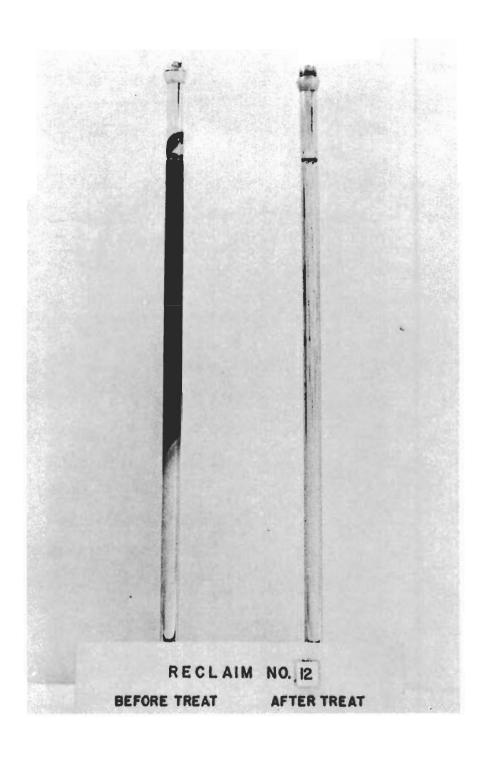


Figure 16. Comparative Coker Preheater Tubes for Reclaim No. 12

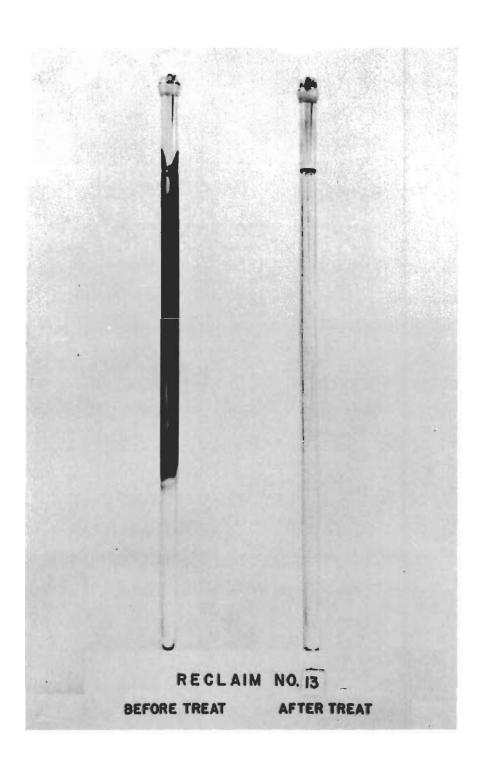
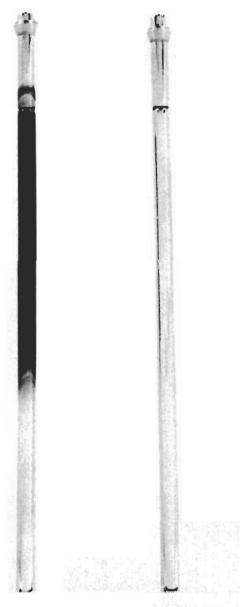


Figure 17. Comparative Coker Preheater Tubes for Reclaim No. 13



RECLAIM NO. 14

BEFORE TREAT

AFTER TREAT

Figure 18. Comparative Coker Preheater Tubes for Reclaim No. 14

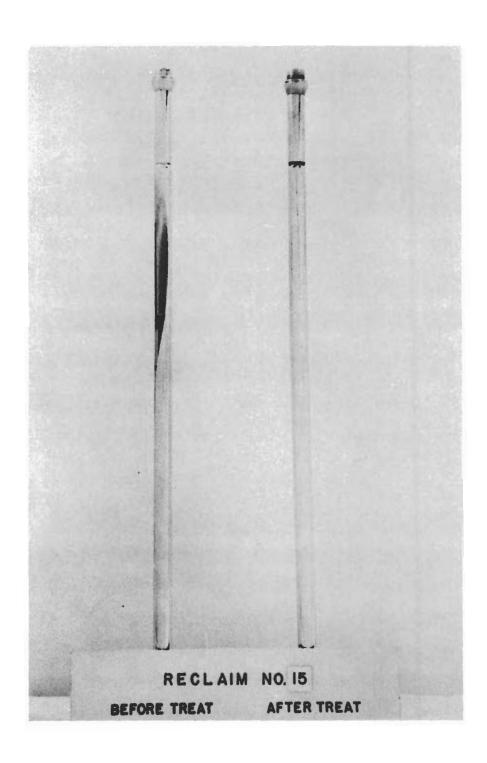
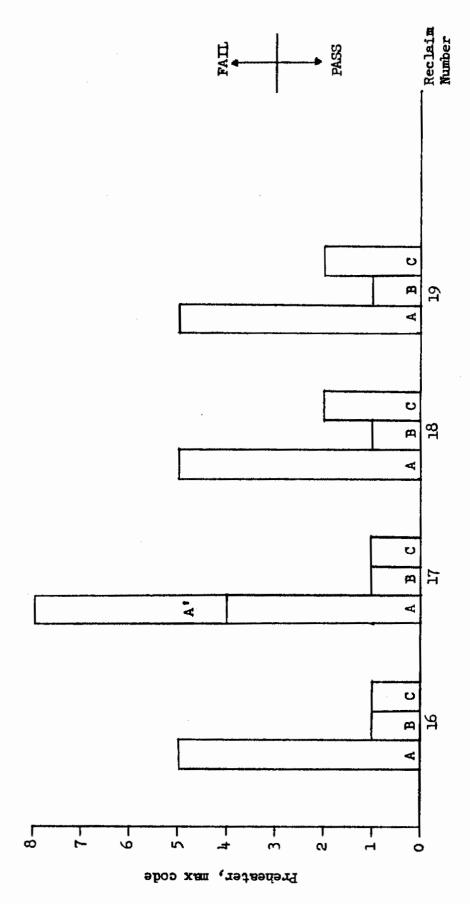


Figure 19. Comparative Coker Preheater Tubes for Reclaim No. 15



A' - second run at close of contract. A - Degraded fuel as received. A' - second run at close of a lineated fuel immediately after reclamation filtration C - Treated fuel six months after reclamation filtration

Figure 20. Thermal Stability Data - Reclaim Nos. 16 Through 19

ASTM-CRC Comparative Coker Ratings (450/550/6)

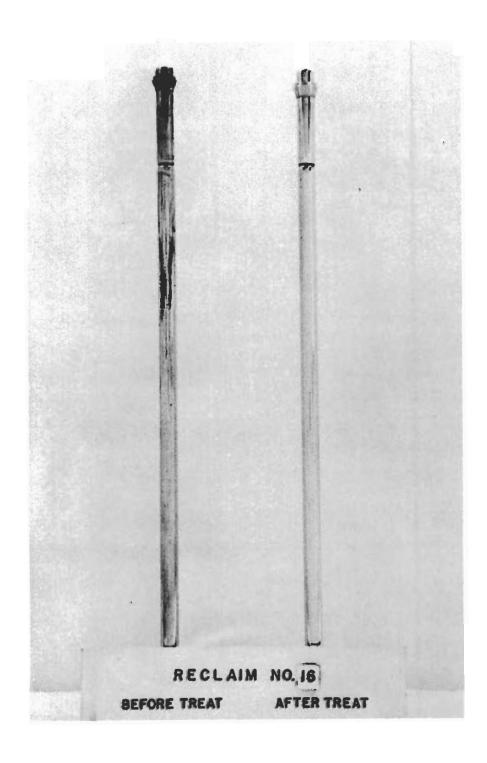


Figure 21. Comparative Coker Preheater Tubes for Reclaim No. 16

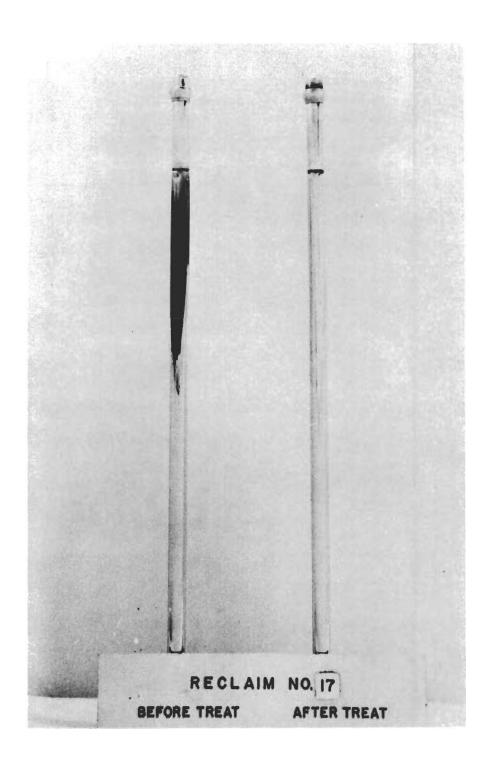


Figure 22. Comparative Coker Preheater Tubes for Reclaim No. 17

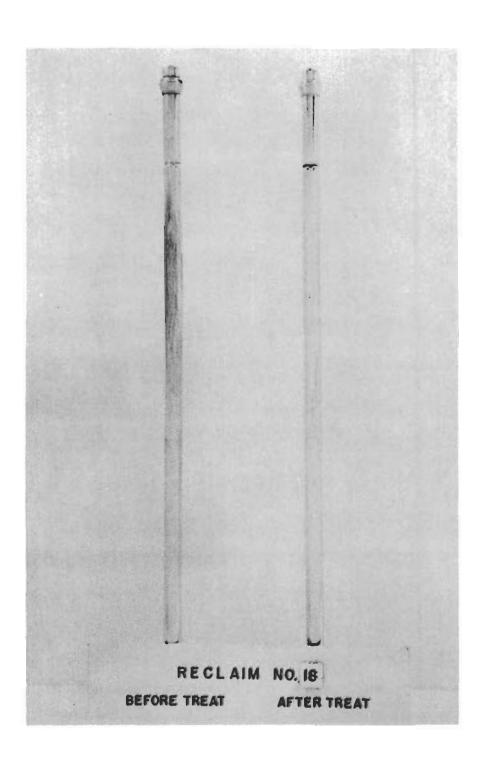


Figure 23. Comparative Coker Preheater Tubes for Reclaim No. 18

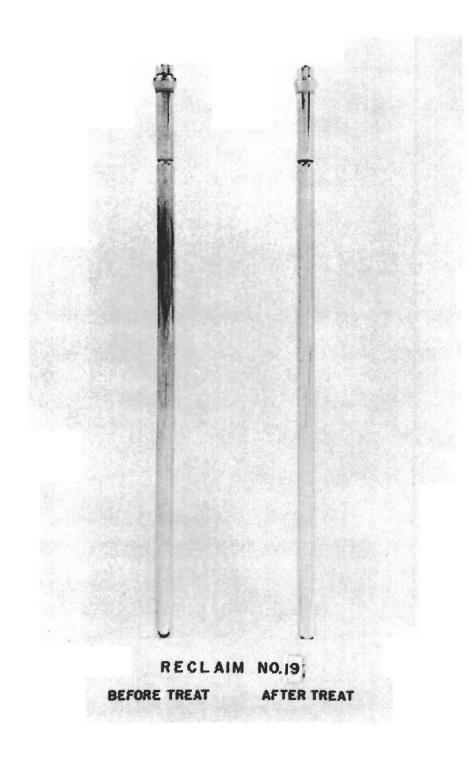


Figure 24. Comparative Coker Preheater Tubes for Reclaim No. 19

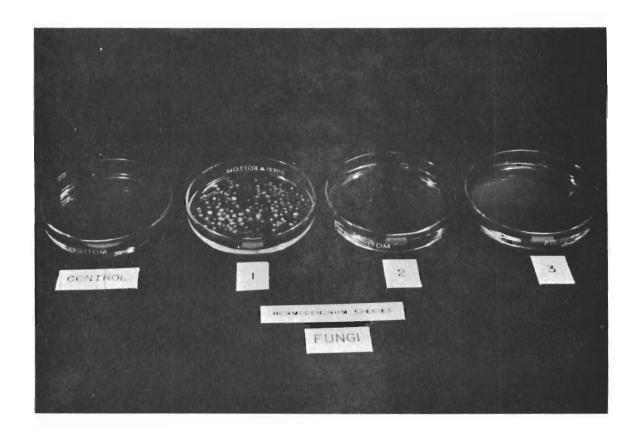


Figure 25. Tracing Fungi Through Filtering Process

Hormodendrum Species (Following 72-hours incubation)

KEY: Control: Petri dish containing sterile Sabouraud's medium

Petri 1: Inoculated drum, JP-6 fuel (~250 colonies)

Petri 2: JP-6 fuel following chemical dryer section ( $\sim$  12 colonies)

Petri 3: JP-6 fuel following the reclamation filter section

### OPTICAL MICROSCOPY SECTION

Photomicrographs, Figures 26 Through 29

Microscope: Leitz Triocular Ortholux Microscope

Oil immersion achromatic objective

with focal length of 1.9 mm

Camera; Lei

Leica M-l Body Camera with microattachment

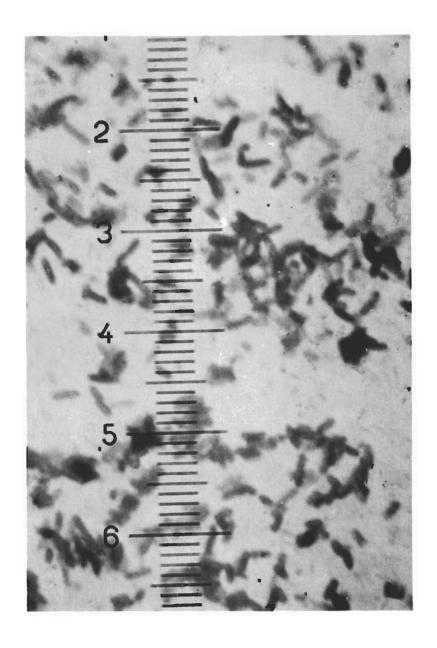


Figure 26.

Typical Bacteria Pseudomonas Species

Total magnification: 2450 X

511

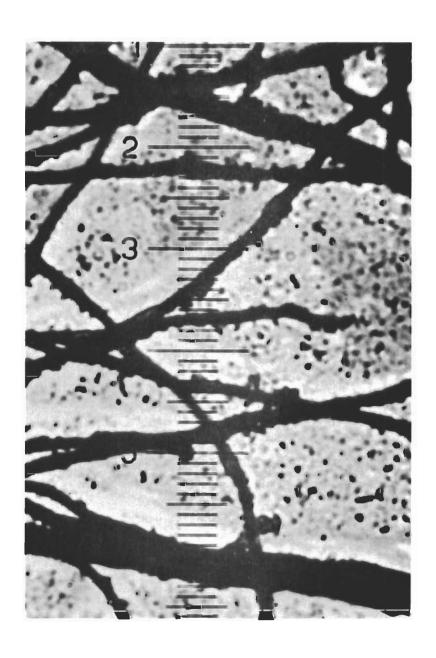


Figure 27.

Typical Fungi Hormodendrum Species

Total magnification: 1300 X

, 10,44,

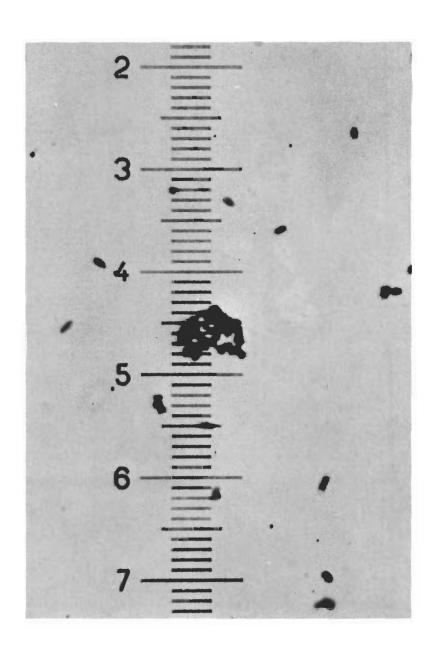


Figure 28. Bacteria-Inoculated JP-6 Fuel in Charge Drum

Total magnification: 2450 X

54

Note intimate dispersion of bacteria throughout fuel sample.

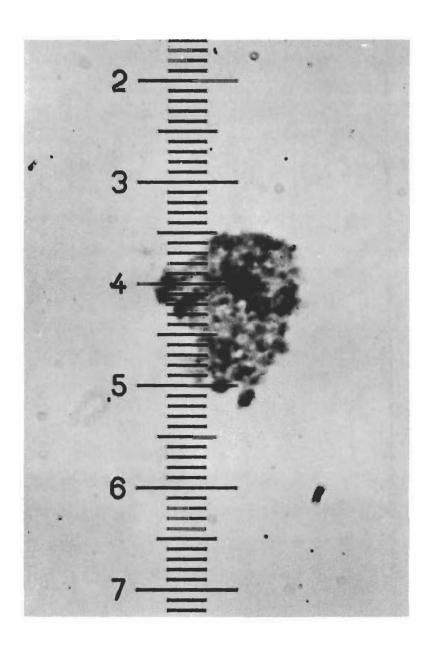


Figure 29.

Chemical Dryer Effluent

Total magnification: 2450 X

<u>54</u>,

Note bacterial clump in center of photograph.

#### ELECTRON MICROSCOPY SECTION

Electronphotomicrographs, Figures 30 Through 33

Microscope: Phillips Model EM-75

Continually variable magnification of 1500 - 15,000 diameters. Resolving

power, 75 angstroms.

Camera: Phillips, 35-mm

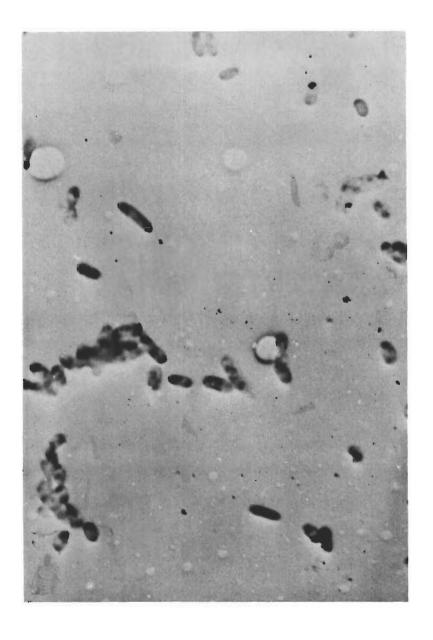


Figure 30.

Typical Bacteria Pseudomonas Species

Total magnification: 7500 diameters

щ,



Figure 31.

Typical Fungi Hormodendrum Species

Total magnification: 7500 diameters

Щ,

52

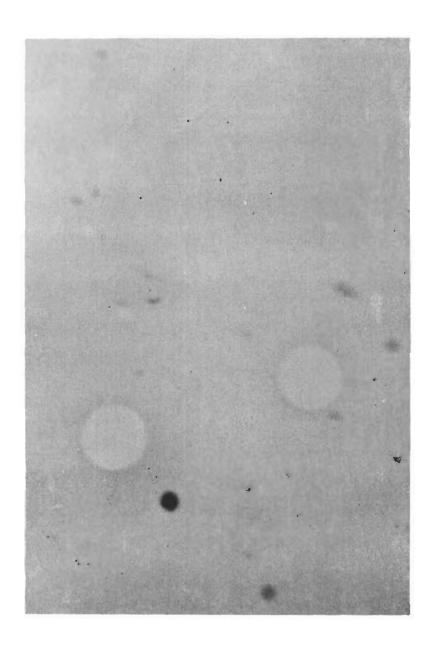


Figure 32. Uninoculated JP-6 Fuel in Charge Drum

Total magnification: 7500 diameters

144

Note: 1. Large spheres present due to collodion film.

2. Darkened areas due to inorganic and not biological particles.

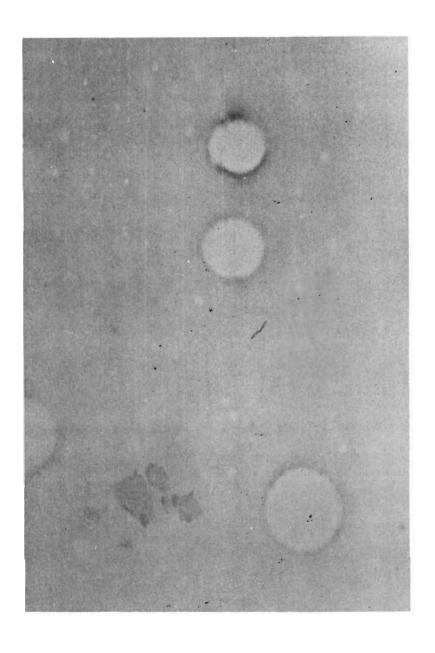


Figure 33.

Reclamation Filter Effluent

Total magnification: 7500 diameters

LUL

Note: 1. Dark particle present due to aerial contamination of grid.

2. Cleanliness of fuel at this point.

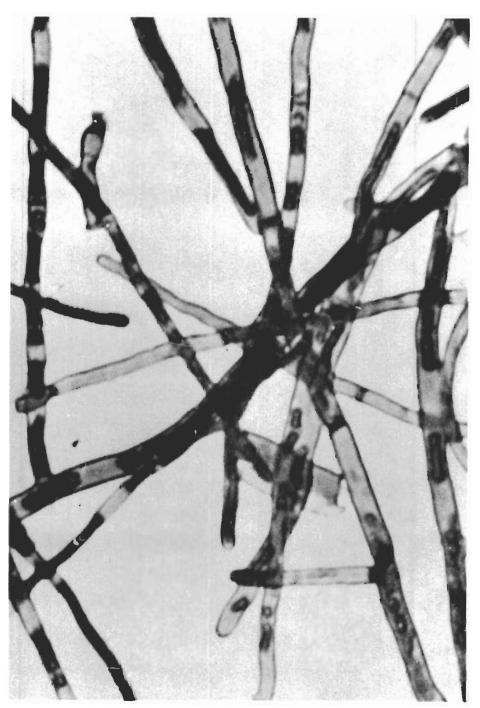


Figure 34. Fungi from Top Entrance Layer in Filter Media

(Optical Micrograph) Total magnification: 2450 X

Note fungi present at top of filter media.

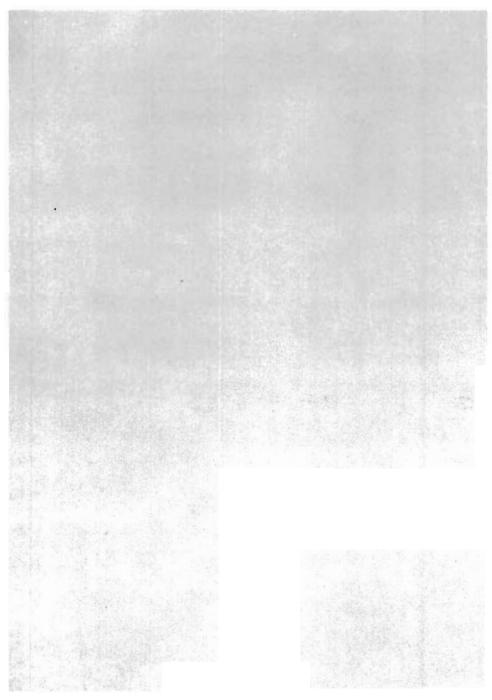


Figure 35. Bottom Exit Layer in Filter Media - Fungi Specimen

(Optical Micrograph) Total magnification: 1250 X

Note absence of fungi at bottom of filter media.



Figure 36. Bacteria from Top Entrance Layer in Filter Media

(Optical Micrograph) Total magnification: 1250 X

Note presence of bacteria at top of filter media.

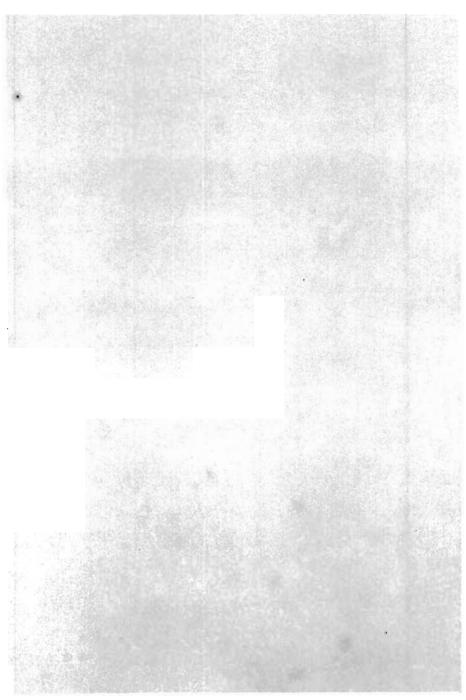


Figure 37. Bottom Exit Layer in Filter Media - Bacteria Specimen

(Optical Micrograph) Total magnification: 1250 X

Note absence of bacteria in bottom of filter media.

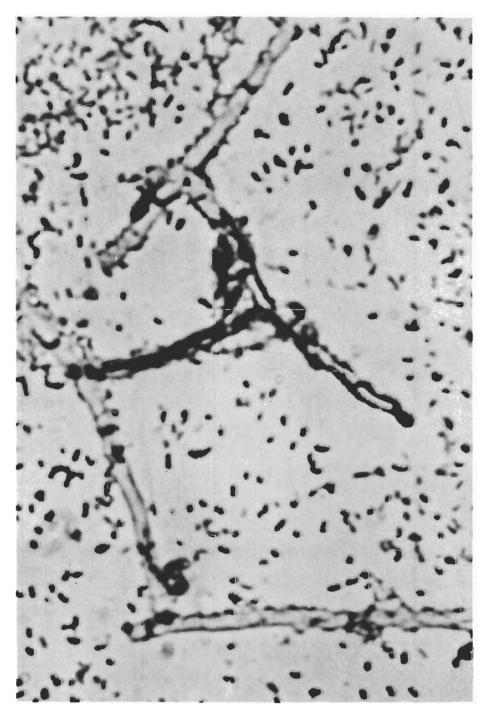


Figure 38. Inoculated Fuel Charge to Coker - Bacteria and Fungi

(Optical Micrograph) Total magnification: 1250 X

Note presence of bacteria and fungi in fuel charge to coker.

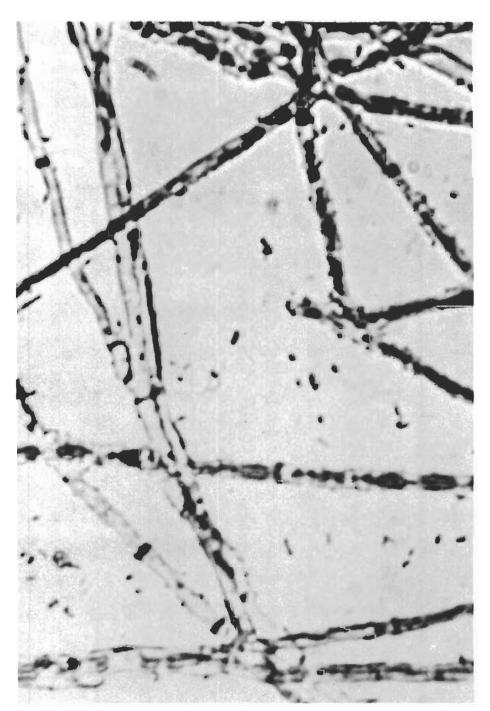


Figure 39. Coker Effluent from Inoculated Fuel - Bacteria and Fungi

(Optical Micrograph) Total magnification: 1250 X

Note presence of bacteria and fungi in fuel effluent from coker.

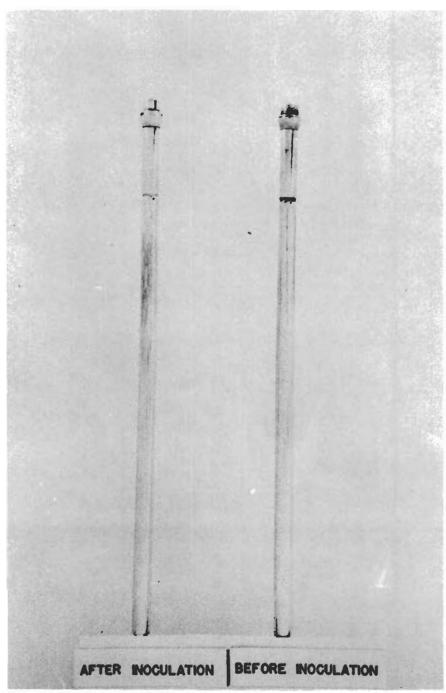
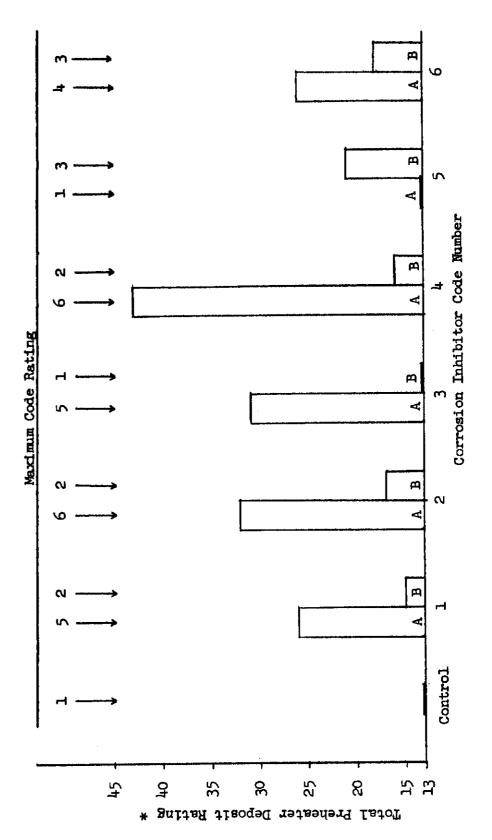


Figure 40. Effect of Biological Contaminant on Coker Preheater Tube

Note slight deposit at top of tube after inoculation.

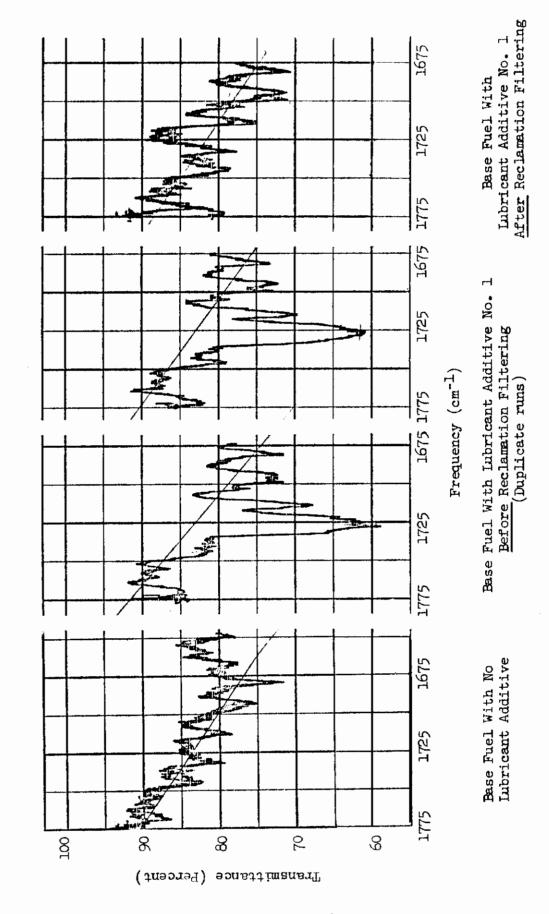
After inoculation, preheater code No. 2 Before inoculation, preheater code No. 1



corrosion inhibitor, followed by reclamation filtration - Control fuel with addition of corrosion inhibitor.

Effect of Corrosion Inhibitors on Coker Ratings at 450/550/6 Figure 41.

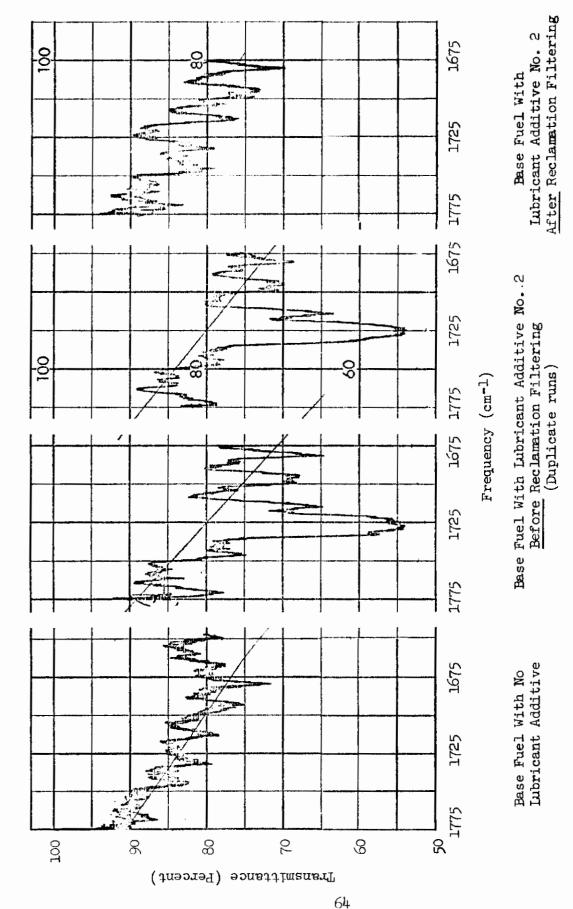
The "Total Preheater Deposit Rating" is defined as the sum of the code rating numbers on the thirteen individual sections of the coker preheater tube as rated by the tuberator.



Removal of Lubricant Additive No. 1 from Fuel by Reclamation Filtering Figure 42.

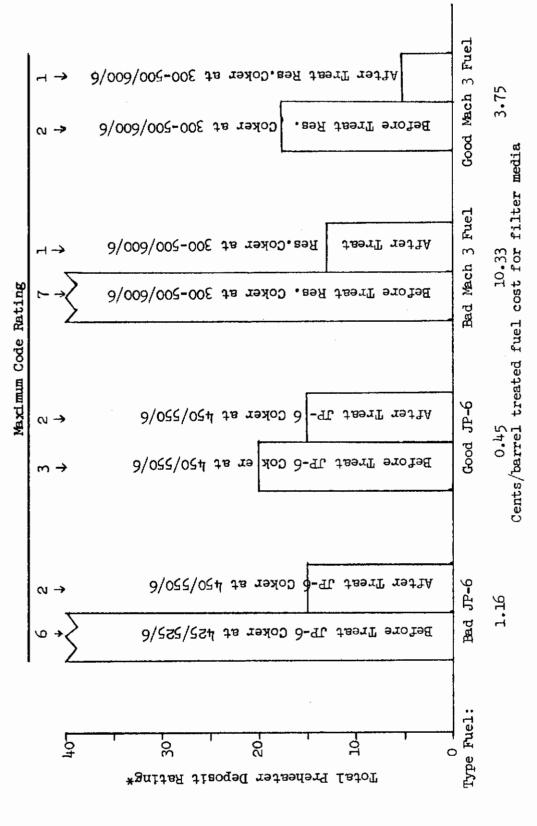
Infrared Spectra by Monsanto's Dayton Iaboratories





Removal of Lubricant Additive No. 2 from Fuel by Reclamation Filtering Figure 43.

Infrared Spectra by Monsanto's Dayton Laboratories



\* The "Total Preheater Deposit Rating" is defined as the sum of the code rating numbers on the thirteen individual squares of the coker preheater tube as rated by the tuberator.

Figure 44. Filter Media Life Expressed as  $\phi/{
m bbl}$  Treating Cost

65



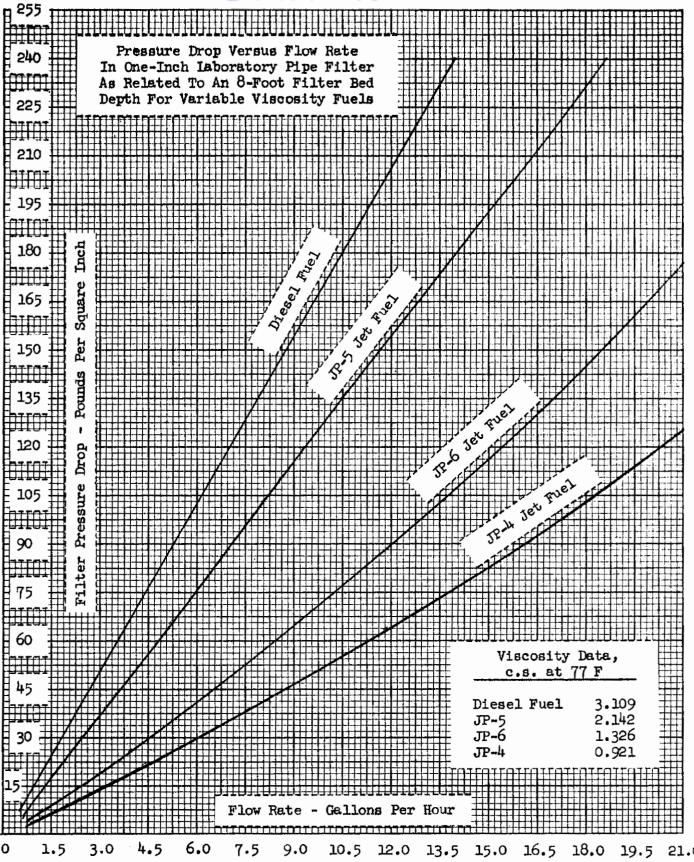


Figure 45. Filter Design Data With Varying Viscosities



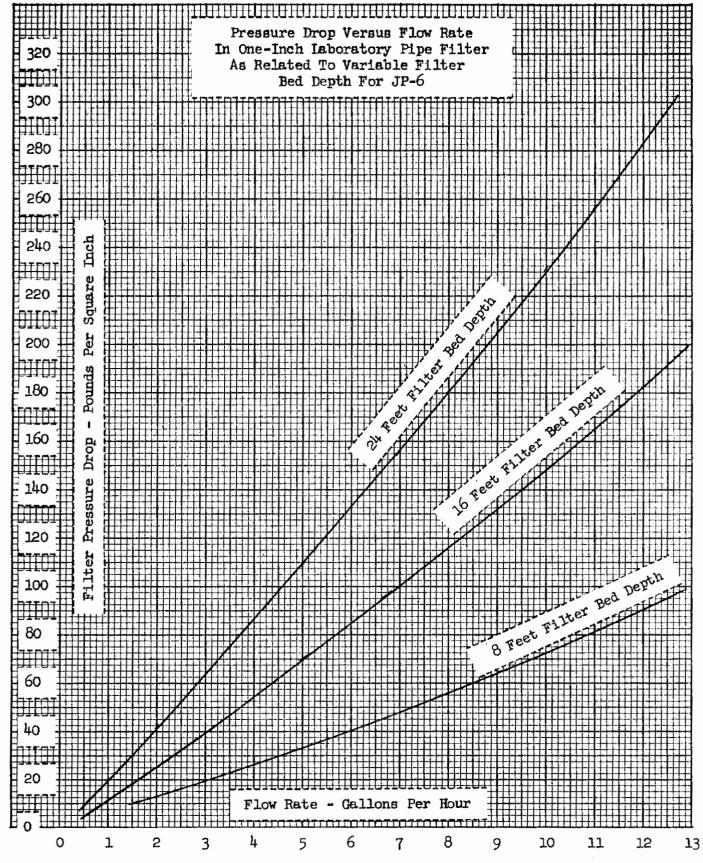


Figure 46. Filter Design Data With Varying Filter Bed Depths



Table 1
Test Data on Redistillation Fractions

	Reclaim		Reclaim	No. 12
	95%	5%	95%	5%
	Overhead	Residue	Overhead	Residue
Coker (450/550/6)				
Preheater, max code	3	8	2	8
Diff. Pressure, in. Hg	0.3	>25	0.3	>25
Coker (450/550/6) Plus Reclam	ation Filte	er		
Preheater, max code	1		1	
Diff. Pressure, in. Hg	0,3		0.0	
Metal Deactivator,				
lbs/1000 bbls	0.95	16.7	0.83	14.9
Paraphenylenediamine*,				
lbs/1000 bbls	Nil	1.05	$\mathtt{Nil}$	0.70
Corrosion, copper strip				
at 212 F	3 <b>A</b>	1B	3A	1B
Copper, ppb	Nil	40	Nil	80
Indene, ppm	46	900	7474	865
Lead, ppb	40	5110	51.	4333
Naphthalene, weight %	0.07	0.61	0.07	0.56
Nitrogen: Basic, ppm	1.9	11.5	2.1	6.5
Pyrrole, ppm	0.2	0.4	0.3	0.3
Peroxide, ppm	1,68	16. <i>6</i> 4	1.44	11.60
Sodium, ppm	2,00	5 <b>•5</b> 5	1.75	14.73
Sulfonate, ppm	0.08	1.13	0.12	1.20

<sup>\*</sup> Analyzed before readdition of antioxidant



Table 2

Code Identification for Air Force Furnished Fuels

Contractor	Air Force		ASTM-CRC Coker Break Point Temperature		
Fuel Code No.	Code	Type	Preheater, F	Filter, °F	
Reclaim No. 1	sf6-6201	JP-6	425	525	
Reclaim No. 2	SF6-6202	JP <b>-</b> 6	425	500	
Reclaim No. 3	SF6-6203	JP <b>-</b> 6	425	500	
Reclaim No. 4	TSF-6206	*TSJF	425	500	
Reclaim No. 5	sf6-6207	JP-6	425 <del>+</del>	525	
Reclaim No. 6	sf6-6208	JP-6	425+	500	
Reclaim No. 7	sf6 <b>-</b> 6209	JP <b>-</b> 6	400	500	
Reclaim No. 8	sf6 <b>-</b> 6213	JP <b>-</b> 6	425+	525	
Reclaim No. 9	SF6-6214	JP <b>-</b> 6	425+	525+	
Reclaim No. 10	TSF-6312	*ISJF	400	475	

<sup>\*</sup> Thermally Stable Jet Fuel, MIL-F-25524A (USAF)



Table 3

Code Identification for Contractor Blended Fuels

Contractor Fuel Code No.	Source	Type	ASTM-CRC Break Point T Preheater, F	
Reclaim No. 11	Ashland Tk 76	JP <b>-</b> 6	375	525
Reclaim No. 12	Ashland Tk 78	JP <b>-</b> 6	375	550
Reclaim No. 13	50% Ash. Tk 78 50% Fresh JP-6 No additives	JP <b>-</b> 6	425	575
Reclaim No. 14	50% Ash. Tk 78 50% Fresh JP-6 With additives	JP-6	450	575
Reclaim No, 15	50% Ash. Tk 78 50% Fresh TSJF With additives	*TSJF	425	550
Reclaim No. 16	50% Ash. Tk 78 50% Fresh JP-6 With anti-icing	JP <b>-</b> 6	375	500
Reclaim No. 17	10% Ash. Tk 78 90% Fresh JP-6 No additives	JP <b>-</b> 6	400	525
Reclaim No. 18	10% Ash. Tk 78 90% Fresh JP-6 With additives	JP <b>-</b> 6	450	575
Reclaim No. 19	10% Ash. Tk 78 90% Fresh JP-6 With additives	JP <b>-</b> 6	450	575

<sup>\*</sup> Thermally Stable Jet Fuel, MIL-F-25524A (USAF)

Table 4
Thermal Stability Data for Doctor Treated Kerosene

# As Received (No Treatment)

Coker Conditions	Preheater	<u> </u>
325/425/6	11111111111111	3.0-300
350/450/6	1111111111111	25,0-175
375/475/6	1111111112321	25 <b>.</b> 0 <b>-2</b> 63

### Following Processing by Reclamation Filter

Coker Conditions	Preheater	$\triangle$ P-Minutes
400/500/6	1111111111111	0.3-300
450/550/6	1111111111111	0.0-300
475/575/6	1111111233331	0.2-300

Note: The analysis of the original doctor treated kerosene (Table 5) is representative of all the fuels used for the research work shown above.



# Table 5

## Analysis of Doctor Treated Kerosene

Gravity, °API	43.7
Distillation: IBP, °F	342
10% Evap., °F	364
50% Evap., °F	406
90% Evap., °F	460
EP, °F	482
Residue, volume %	1
Loss, volume %	0
Saybolt Color	25
Freezing Point, °F	-56
Viscosity, cs at -30 F	8.26
Flash Point, F	132
Gum: Existent, mg/100 ml	2.0
Potential, mg/100 ml	3.6
Water Tolerance: Interface	1
Vol. change	0
Water Separometer: WSIM	89
Moisture, ppm	58
Net Heat of Combustion, Btu/lb	18602
Aniline-Gravity Product	6297
Corrosion	lA
Sulfur: Mercaptan, weight %	0,001
Total, weight %	0.063
Doctor Test	Sweet
Aromatics, volume %	12.3
Olefins, volume %	3.0
Smoke Point, mm	26
Naphthalene, weight %	2.02
Copper, ppb	8.8
Indene, ppm	38
Iron, ppm	0,05
Lead, ppb	34.7
Nitrogen: Basic, ppm	1.8
Pyrrole, ppm	0.93
Peroxide, ppm	0.89
Phenol, ppm	70
Saponification Number	0.20
Sodium, ppm	4.64
Sulfonate, ppm	Nil

Table 6

Physical and Chemical Tests on Fuels

Reclaim No. 1

	Before Treat	After Treat	Aged After Treat
Gravity, "API Distillation: IBP, "F  10% Evap., "F  50% Evap., "F  90% Evap., "F  Residue, volume %  Loss, volume %  Loss, volume %  Saybolt Color  Freezing Point, "F  Viscosity, cs at -40 F  Particulate Matter, mg/gal, 0.45 Filter  Gum: Existent, mg/100 ml  Potential, mg/100 ml  Water Reaction Index  Water Separometer: WSI  WSIM  Moisture, ppm  Specific Heat at 300 F  Net Heat of Combustion, Btu/lb  Aniline-Gravity Product  Corrosion, copper strip at 212 F  Sulfur: Mercaptan, weight %  Total, weight %  Doctor Test  Aromatics, volume %  Olefins, volume %  Smoke Point, mm	1.0	50.0 290 305 326 360 404 1.0 1.0 Yellow -70- 3.70 1.1 0.4 2.1 1 82.3 56.0 48.0 0.612 18,725 7475 1A 0.0006 0.044 Sweet 9.5 0.9 25.0	0.8
Naphthalene, weight % Copper, ppb Indene, ppm Iron, ppm	0.25 35.0 61 28	0.10 50.0 9 0.68	0.06 4.0 34
Lead, ppb Nitrogen: Basic, ppm Pyrrole, ppm Oxygen, Dissolved, ppm Peroxide, ppm Sodium, ppm Sulfonate, ppm	2.1	22.8 1.9 0.1 35.0 1.50 2.39 0.055	27.2 0.6 Nil 0.44 4.85 Nil

Table 6 (Continued)

# Physical and Chemical Tests on Fuels

	Before Treat	After Treat	Aged After Treat
Gravity, °API		49.8	
Distillation: IBP, °F 10% Evap., °F		290 300	
50% Evap., °F	,	316	
90% Evap., °F		344	
EP, °F		384	
Residue, volume %		0.5	
Ioss, volume %		0.5	
Saybolt Color:		+30	
Freezing Point, °F		-70-	
Viscosity, cs at -40 F Particulate Matter, mg/gal, 0.45 M Filter		3.52 1.1	
Gum: Existent, mg/100 ml		0.2	0.2
Potential, mg/100 ml		2.1	2.8
Water Reaction Index		1	
Water Separometer: WSI		85.6	
WSIM		57.0	0
Moisture, ppm		45.0	51.9
Specific Heat at 300 F	18,	0.612	
Net Heat of Combustion, Btu/lb Aniline-Gravity Product		18,625 6524	
Corrosion, copper strip at 212 F		1A	
Sulfur: Mercaptan, weight %		0.0006	
Total, weight %		0.052	
Doctor Test		Sweet	
Aromatics, volume %		8.2	
Olefins, volume %	3.1	3.1	
Smoke Point, mm		23.0	
Naphthalene, weight %	0.07	0.02	0.10
Copper, ppb	33•0	38.0	15.0
Indene, ppm	6	0 0.42	3
Iron, ppm	17	14.2	25.3
Lead, ppb Nitrogen: Basic, ppm	0.7	<1.0	7•3
Pyrrole, ppm	0.2	0.1	Nil
Oxygen, Dissolved, ppm	· · · · · ·	32.5	2
Peroxide, ppm		0.50	0.44
Sodium, ppm	1.3	0.00	3.00
Sulfonate, ppm		0.029	Nil

Table 6 (Continued)

# Physical and Chemical Tests on Fuels

	Before Treat	After Treat	Aged After Treat
Gravity, °API Distillation: IBP, °F 10% Evap., °F 50% Evap., °F 90% Evap., °F EP, °F Residue, volume % Loss, volume % Saybolt Color		49.8 290 304 318 344 379 1.0 0.5 +30	
Freezing Point, °F		-70-	
Viscosity, cs at -40 F		3.28	
Particulate Matter, mg/gal, 0.45 µ Filter Gum: Existent, mg/100 ml		1.3 0.2	0.4
Potential, mg/100 ml		0.6	0.6
Water Reaction Index		1	
Water Separometer: WSI		89.7	
WSIM Moisture, ppm		58.0	1 6
Specific Heat at 300 F		34.3 0.612	45.6
Net Heat of Combustion, Btu/lb		18,623	
Aniline-Gravity Product		6499	
Corrosion, copper strip at 212 F		lA	
Sulfur: Mercaptan, weight %		0.0006	
Total, weight %		0.053	
Doctor Test		Sweet	
Aromatics, volume %		10.5	
Olefins, volume %	2.8	1.2	
Smoke Point, mm	1	28.0	1
Naphthalene, weight %	0.14	0.04	0.04
Copper, ppb	26.0	11.0	10.0
Indene, ppm Iron, ppm	6 18.0	1	17
Léad, ppb	10.0	0,62 16.0	24.7
Nitrogen: Basic, ppm	1.3	2.0	7.3
Pyrrole, ppm	0.2	0.2	Nil
Oxygen, Dissolved, ppm	- <del></del>	31.7	
Peroxide, ppm		0.80	0.20
Sodium, ppm		1.00	5.01
Sulfonate, ppm		0.110	Nil

Table 6 (Continued)

## Physical and Chemical Tests on Fuels

	Before Treat	After Treat	Aged After <u>Treat</u>
Gravity, °API Distillation: IBP, °F 10% Evap., °F		47•9 326 340	
50% Evap., °F 90% Evap., °F EP, °F Residue, volume % Loss, volume %		358 400 440 1.0	
Saybolt Color		+23	
Freezing Point, °F		-80=	
Viscosity, cs at -40 F		5.13	
Particulate Matter, mg/gal, 0.45µ Filter		1.9	0.0
Gum: Existent, mg/100 ml		0.2 1.2	0.2
Potential, mg/100 ml Water Reaction Index		1	1.7
Water Separometer: WSI		98.7	
WSIM		36.0	
Moisture, ppm		38.2	
Specific Heat at 300 F		0.608	
Net Heat of Combustion, Btu/lb		18,639	
Aniline-Gravity Product		6658	
Corrosion, copper strip at 212 F		JA _	
Sulfur: Mercaptan, weight %		0.0006	
Total, weight %		0.032	
Doctor Test		Sweet	
Aromatics, volume %		11.7	
Olefins, volume %		0.6	
Smoke Point, mm		27.0 0.29	0.04
Naphthalene, weight % Copper, ppb		24.0	12.5
Indene, ppm		5	33
Iron, ppm		ó.61	<b>)</b>
Lead, ppb		4.0	
Nitrogen: Basic, ppm		3.8	4.4
Pyrrole, ppm	0.5	0.1	Nil
Oxygen, Dissolved, ppm		31.4	
Peroxide, ppm		1.70	1.50
Sodium, ppm		1.90	
Sulfonate, ppm		0.110	Nil

Table 6 (Continued)

# Physical and Chemical Tests on Fuels

	Before Treat	After Treat	Aged After Treat
Gravity, "API Distillation: TBP, "F  10% Evap., "F  50% Evap., "F  90% Evap., "F  EP, "F  Residue, volume %  Loss, volume %  Saybolt Color Freezing Point, "F  Viscosity, cs at -40 F  Particulate Matter, mg/gal, 0.45 µ Filter  Gum: Existent, mg/100 ml  Potential, mg/100 ml  Water Reaction Index  Water Separometer: WSI  WSIM  Moisture, ppm  Specific Heat at 300 F  Net Heat of Combustion, Btu/lb  Aniline-Gravity Product  Corrosion, copper strip at 212 F  Sulfur: Mercaptan, weight %		50.2 290 305 320 366 414 0.6 +16 -70- 3.59 1.7 0.0 2.0 1 83.9 47.0 40.2 0.612 18,646 6727 1A 0.0006	0.4 1.6
Total, weight % Doctor Test Aromatics, volume % Olefins, volume %	0.7	0.021 Sweet 9.4 0.8	
Smoke Point, mm Naphthalene, weight % Copper, ppb Indene, ppm Iron, ppm	0.19 137.0 96	30.0 0.11 18.0 14 0.28	0.10 5.0 37
Lead, ppb Nitrogen: Basic, ppm Pyrrole, ppm Oxygen, Dissolved, ppm	1.6	10.8 4.0 0.1 41.4	4.4 Nil 0.44
Peroxide, ppm Sodium, ppm Sulfonate, ppm		0.70 2.85 0.005	Nil

Table 6 (Continued)

# Physical and Chemical Tests on Fuels

	Before Treat	After Treat	Aged After Treat
Gravity, °API		49.7	
Distillation: IBP, °F		290	
10% Evap., °F		302	
50% Evap., °F		312	
90% Evap., °F		337	
EP, °F		390	
Residue, volume %		0.7	
Ioss, volume %		1.3	
Saybolt Color		+30	
Freezing Point, °F		-70 <b>=</b>	
Viscosity, cs at -40 F		3.31	
Particulate Matter, mg/gal, 0.45µ Filter		1.5	0.1
Gum: Existent, mg/100 ml		0.0	0.4
Potential, mg/100 ml Water Reaction Index		2.6 1	5.2
-			
Water Separometer: WSI WSIM		93.0 41.0	
Moisture, ppm		53.1	
Specific Heat at 300 F		0.611	
Net Heat of Combustion, Btu/lb		18,606	•
Aniline-Gravity Product		6337	
Corrosion, copper strip at 212 F		1A	
Sulfur: Mercaptan, weight %		0.0006	
Total, weight %		0.023	
Doctor Test		Sweet	
Aromatics, volume %		12.1	
Olefins, volume %	1,2	0.5	
Smoke Point, mm		28.0	
Naphthalene, weight %	0.12	0.02	0.21
Copper, ppb	15.0	25,0	9.0
Indene, ppm	8 .	0	
Iron, ppm		0.38	
Lead, ppb		9.4	
Nitrogen: Basic, ppm	1.1	2.0	4.4
Pyrrole, ppm	0.2	0.1	Nil
Oxygen, Dissolved, ppm		38.6	0.00
Peroxide, ppm		0.60	0.36
Sodium, ppm		0.76	707.2 7
Sulfonate, ppm		0.014	Nil

Table 6 (Continued)

# Physical and Chemical Tests on Fuels

	Before Treat	After Treat	Aged After Treat
Gravity, "API Distillation: IBP, "F  10% Evap., "F  50% Evap., "F  90% Evap., "F  Residue, volume %  Loss, volume %  Saybolt Color  Freezing Point, "F  Viscosity, cs at -40 F  Particulate Matter, mg/gal, 0.45µ Filter  Gum: Existent, mg/100 ml  Potential, mg/100 ml  Water Reaction Index  Water Separometer: WSI  WSIM  Moisture, ppm  Specific Heat at 300 F  Net Heat of Combustion, Btu/lb  Aniline-Gravity Product  Corrosion, copper strip at 212 F  Sulfur: Mercaptan, weight %  Total, weight %  Doctor Test  Aromatics, volume %  Olefins, volume %  Smoke Point, mm	0.7	49.0 300 311 334 372 412 0.5 0.5 +16 -70- 4.27 2.2 1.0 1.0 1 99.7 58.0 39.2 0.610 18,632 6591 1A 0.0006 0.018 Sweet 11.6 0.6 30.0	0.8
Naphthalene, weight % Copper, ppb Indene, ppm Iron, ppm	0.39 146.0 145 128	0.17 <10.0 0 0.72	0.17 7.0
Lead, ppb Nitrogen: Basic, ppm Pyrrole, ppm Oxygen, Dissolved, ppm	1.7	19.9 <b>&lt;</b> 1.0 0.1 31.6	4.4 Nil
Peroxide, ppm		0.90	0.53
Sodium, ppm Sulfonate, ppm		0.160	Nil

# Table 6 (Continued)

# Physical and Chemical Tests on Fuels

	Before Treat	After Treat	Aged After Treat
Gravity, "API Distillation: IBP, "F  10% Evap., "F  50% Evap., "F  90% Evap., "F  EP, "F  Residue, volume %  Loss, volume %  Saybolt Color  Freezing Point, "F  Viscosity, cs at -40 F  Particulate Matter, mg/gal, 0.45 Filter  Gum: Existent, mg/100 ml  Potential, mg/100 ml  Water Reaction Index  Water Separometer: WSI  WSIM  Moisture, ppm  Specific Heat at 300 F  Net Heat of Combustion, Btu/lb  Aniline-Gravity Product  Corrosion, copper strip at 212 F  Sulfur: Mercaptan, weight %  Total, weight %  Doctor Test  Aromatics, volume %  Olefins, volume %  Smoke Point mm	0.8	48.8 304 320 340 377 414 0.2 0.8 +16 -70- 4.37 1.0 1.8 3.0 1.8 3.0 1.8 3.0 1.8 3.0 1.8 6612 1A 0.0006 0.022 Sweet 11.1 0.7	0.2 3.5
Smoke Point, mm Naphthalene, weight % Copper, ppb Indene, ppm	0.42 49.0 94	29,5 0.21 <b>(</b> 10.0 14	0.15 20.0
Iron, ppm Lead, ppb Nitrogen: Basic, ppm Pyrrole, ppm	262 1.9 0.3	0.63 19.1 1.0 0.1	27.8 4.4
Oxygen, Dissolved, ppm Peroxide, ppm		38.2 0.60	0,44
Sodium, ppm Sulfonate, ppm		0.00 0.180	0.002

Table 6 (Continued)

# Physical and Chemical Tests on Fuels

	Before Treat	After Treat	Aged After Treat
Gravity, "API Distillation: IBP, "F  10% Evap., "F  50% Evap., "F  90% Evap., "F  Residue, volume %  Loss, volume %  Saybolt Color Freezing Point, "F  Viscosity, cs at -40 F  Particulate Matter, mg/gal, 0.45µ Filter  Gum: Existent, mg/100 ml  Potential, mg/100 ml  Water Reaction Index  Water Separometer: WSI  WSIM  Moisture, ppm  Specific Heat at 300 F  Net Heat of Combustion, Btu/lb  Aniline-Gravity Product  Corrosion, copper strip at 212 F  Sulfur: Mercaptan, weight %  Total, weight %  Doctor Test  Aromatics, volume %  Olefins, volume %  Smoke Point, mm	1.1	49.4 300 314 332 370 400 0.4 0.6 +16 -70- 5.09 3.2 1.0 186.3 37.0 38.2 0.611 18,638 6644 1A 0.0006 0.019 sweet 11.4 0.9 30.0	0.2
Naphthalene, weight % Copper, ppb Indene, ppm	0.29 48.0 95 90	0.18 <b>(</b> 10.0 8	0.09
Iron, ppm Lead, ppb Nitrogen: Basic, ppm Pyrrole, ppm	2.1 0.3	0.72 16.3 1.0 0.1	25•3 3•5
Oxygen, Dissolved, ppm Peroxide, ppm Sodium, ppm Sulfonate, ppm		40.3 1,42 2.05 0.000	0.36 Nil
			į

# Table 6 (Continued)

# Physical and Chemical Tests on Fuels

	Before Treat	After Treat	Aged After Treat
Cravity, "API Distillation: IBP, "F  10% Evap., "F  50% Evap., "F  50% Evap., "F  EP, "F  Residue, volume %  Loss, volume %  Saybolt Color  Freezing Point, "F  Viscosity, cs at -40 F  Particulate Matter, mg/gal, 0.45 Filter  Gum: Existent, mg/100 ml  Potential, mg/100 ml  Water Reaction Index  Water Separometer: WSI  WSIM  Moisture, ppm  Specific Heat at 300 F  Net Heat of Combustion, Btu/lb  Aniline-Gravity Product  Corrosion, copper strip at 212 F  Sulfur: Mercaptan, weight %  Total, weight %  Doctor Test  Aromatics, volume %  Olefins, volume %  Smoke Point, mm	0.7	47.6 316 332 358 390 420 0.3 0.7 +28 -80 - 5.53 2.1 0.8 1.4 1 74.3 28.0 53.9 0.607 18,628 6545 1A 0.0030 0.025 sour 12.8 0.8 28.0	0.4
Naphthalene, weight % Copper, ppb Indene, ppm	0.81 13.0 38 59	0.13 13.0 16 1.11	0.27 10.0
Iron, ppm Lead, ppb Nitrogen: Basic, ppm Pyrrole, ppm	1.7	22.0 1.0 0.1	25•3 5•3
Oxygen, Dissolved, ppm Peroxide, ppm		37•7 1•36 0•65	0.44
Sodium, ppm Sulfonate, ppm		0.054	0.062

Table 6 (Continued)

## Physical and Chemical Tests on Fuels

	Before Treat	After Treat	Aged After Treat
Gravity, API	46,5	46.5	
Distillation: TBP, °F	330	325	
10% Evap., °F	340	334	
50% Evap., °F 90% Evap., °F	355	356	
90% Evap., °F EP, °F	376 416	378 412	
Residue, volume %	0.5	1.2	
Loss, volume %	1.0	1.3	
Saybolt Color	+15	+30	
Freezing Point, °F	-70-	-80-	
Viscosity, cs at -40 F	5.52	4.99	
Particulate Matter, mg/gal, 0.8 M Filter	2.3		
0.45M Filter	7.6	1.5	
Gum: Existent, mg/100 ml	0.4	0.6	0.2
Potential, mg/100 ml	4.3	2.8	1,2
Water Reaction Index	4	1	
Water Separometer: WSI	8.3	89.3	
WSIM	27.0	55 <b>,</b> 0	16.2
Moisture, ppm Specific Heat at 300 F	36.0 0.607	28.8 0.605	16.3
Net Heat of Combustion, Btu/lb	18,615	18,604	
Aniline-Gravity Product	6426	6314	
Corrosion, copper strip at 212 F	3B	1B	
Sulfur: Mercaptan, weight %	0.0006	0.0006	
Total, weight %	0.016	0.020	
Doctor Test	Sweet	Sweet	
Aromatics, volume %	11.7	12.6	
Olefins, volume %	3.0	1.5	
Smoke Point, mm	23.0	24.0	0.70
Naphthalene, weight %	0.11	0.11	0.19
Copper, ppb	30.0	49.0	12.5 62
Indene, ppm	89 0.32	51 0.002	02
Iron, ppm Lead, ppb	55.6	2.5	51.4*
Nitrogen: Basic, ppm	3.0	5.0	5.9
Pyrrole, ppm	0.30	ó. <b>0</b> 0	0.01
Oxygen, Dissolved, ppm	32.6	46.0	
Peroxide, ppm	3.40	3 <b>.6</b> 0	0,98
Sodium, ppm	1.86	2.08	1.49
Sulfonate, ppm	0.507	0.100	0.000

<sup>\*</sup> Lead content is abnormally high because of using tin coated storage cans. 83

Table 6 (Continued).

# Physical and Chemical Tests on Fuels

	Before Treat	After Treat	Aged After Treat
Gravity, "API Distillation: IBP, "F 10% Evap., "F 50% Evap., "F 90% Evap., "F EP, "F Residue, volume % Ioss, volume % Saybolt Color Freezing Point, "F Viscosity, cs at -40 F Particulate Matter, mg/gal, 0.8  Filter	46,5 322 340 354 376 412 0.7 1.3 +15 -70- 5.30 13.2	46.5 324 340 356 378 401 1.2 0.8 +30 -80- 4.84	
Gum: Existent, mg/100 ml Potential, mg/100 ml Water Reaction Index Water Separometer: WSI WSIM	15.1 0.2 1.2 4 15.6	1.9 0.6 2.9 1 70.3 50.0	0.2 1.6
Moisture, ppm Specific Heat at 300 F Net Heat of Combustion, Btu/lb Aniline-Gravity Product Corrosion, copper strip at 212 F Sulfur: Mercaptan, weight % Total, weight % Doctor Test Aromatics, volume % Olefins, volume % Smoke Point, mm	62.6 0.607 18,615 6426 3B 0.0006 0.018 Sweet 11.5 2.8 23.0	40.8 0.605 18,604 6314 1A 0.0006 0.023 Sweet 13.1 1.6 24.0	25.5
Naphthalene, weight % Copper, ppb Indene, ppm Iron, ppm	0.11 25.0 85 0.40	0.10 49.0 56 0.01	0.17 7.0 56
Lead, ppb Nitrogen: Basic, ppm Pyrrole, ppm Oxygen, Dissolved, ppm	468 3.0 0.50 48.5	7.5 2.0 0.20 38.9	50.6* 5.9 0.00
Peroxide, ppm Sodium, ppm Sulfonate, ppm	3.40 1.69 0.356	2.90 1.88 0.110	0.71 1.51 0.030

<sup>\*</sup> Lead content is abnormally high because of using tin coated storage cans.

Table 6 (Continued)

# Physical and Chemical Tests on Fuels

	Before Treat	After Treat	Aged After Treat
Gravity, °API	46.9	46.9	
Distillation: IBP, °F	312	334	
10% Evap., °F	336	340	
50% Evap., °F	351	352	
90% Evap., °F	374	374	
EP, °F	396	<b>4</b> 16	
Residue, volume %	1.0	1.0	
Loss, volume %	1.0	1.0	
Saybolt Color	+22	+30	
Freezing Point, °F	-70 <b>-</b>	-80-	
Viscosity, cs at -40 F	5.19	5.37	
Particulate Matter, mg/gal, 0.45 Filter	6.8	3.0	
Gum: Existent, mg/100 ml	0.4	0.2	0.2
Potential, mg/100 ml	3•9	1.9	1.0
Water Reaction Index	1	1	
Water Separometer: WSI	62.6	97.6	
WSIM	23.0	43,0	
Moisture, ppm	38,7	28.8	27.5
Specific Heat at 300 F	0.606	0.606	
Net Heat of Combustion, Btu/1b	18,613	18,604	
Aniline-Gravity Product	6402	6322	
Corrosion, copper strip at 212 F	la	1B	
Sulfur: Mercaptan, weight %	0.0006	0.0006	
Total, weight %	0.039	0.014	
Doctor Test	Sweet	Sweet	
Aromatics, volume %	10.7	13.0	
Olefins, volume %	2.5	1.8	
Smoke Point, mm	26.0	26.0	
Naphthalene, weight %	0.08	0.08	0.12
Copper, ppb	26.0	7.0	10.0
Indene, ppm	44	25 .	30
Iron, ppm	0.28	0.04	
Lead, ppb	109	5.0	55.1 <b>*</b>
Nitrogen: Basic, ppm	<1.0	<1.0	
Pyrrole, ppm	0.50	0.30	0.02
Oxygen, Dissolved, ppm	34.5	50.3	- (-
Peroxide, ppm	1.22	0.89	0.62
Sodium, ppm	3.59	1.12	5.31
Sulfonate, ppm	0.132	0.000	0.005

<sup>\*</sup> Lead content is abnormally high because of using tin coated storage cans.

Table 6 (Continued)

## Physical and Chemical Tests on Fuels

	Before Treat	After Treat	Aged After Treat
Gravity, °API	46.9	47.1	
Distillation: IBP, °F 10% Evap °F	318	323	
—— (* ··································	336	336	
	351	350	
90% Evap., °F EP, °F	374 401	373	
Residue, volume %	0.5	39 <sup>)</sup> +	
Loss, volume %	1.5	1.0 1.0	
Saybolt Color	+22	+25	
Freezing Point, °F	-70-	-80-	
Viscosity, cs at -40 F	5.13	5.39	
Particulate Matter, mg/gal, 0.45 Filter	1.5	3.0	
Gum:Existent, mg/100 ml	0.6	0.2	0.4
Potential, mg/100 ml	2.7	1.0	1.0
Water Reaction Index	1	1	
Water Separometer: WSI	55•7	97•3	
WIIM	28.0	64.0	
Moisture, ppm	44.7	12.0	23.5
Specific Heat at 300 F	0.606	0.606	
Net Heat of Combustion, Btu/1b	18,613	18,607	
Aniline-Gravity Product	6402	6349	
Corrosion, copper strip at 212 F	lA	1B	
Sulfur: Mercaptan, weight %	0.0006	0.0006	
Total, weight %	0.035	0.009	
Doctor Test	Sweet	Sweet	
Aromatics, volume %	13.1	12.0	
Olefins, volume % Smoke Point, mm	1.6	1.9	
Naphthalene, weight %	27.0 0.08	25.0 0.08	0.09
Copper, ppb	27.0	3.0	5.0
Indene, ppm	48	20	22
Iron, ppm	0.41	0.40	
Lead, ppb	40.3	20.0	51,9 <del>*</del>
Nitrogen: Basic, ppm	<b>&lt;1.</b> 0	1.0	<i>y</i> , <i>y</i>
Pyrrole, ppm	0.40	0.10	0.00
Oxygen, Dissolved, ppm	34.5	48.6	
Peroxide, ppm	1.22	0.71	0.62
Sodium, ppm	0.72	0.66	4,36
Sulfonate, ppm	0.129	0.000	0.000

<sup>\*</sup> Lead content is abnormally high because of using tin coated storage cans.

Table 6 (Continued)

## Physical and Chemical Tests on Fuels

	Before Treat	After Treat	Aged After Treat
Gravity, °API	46.9	47.0	
Distillation: IBP, °F	322	329	
10% Evap., °F	336	336	
50% Evap,, °F	352	350	
90% Evap., °F	376	37 <b>4</b>	
EP, °F	404	408	
Residue, volume %	1.0 1.0	1.0 1.0	
Loss, volume %	+22	+30	
Saybolt Color Freezing Point, °F	-80 <i>-</i>	-80-	
Viscosity, cs at -40 F	4.87	5.30	
Particulate Matter, mg/gal, 0.45 Filter	4.9	2.0	
Gum: Existent, mg/100 ml	2.4	0.4	0.4
Potential, mg/100 ml	3.7	1.3	1.3
Water Reaction Index	1	1	
Water Separometer: WSI	50.9	93•9	
WSIM	22.0	48.0	
Moisture, ppm	42.1	<b>18.</b> 6	18.4
Specific Heat at 300 F	0.606	0.606	
Net Heat of Combustion, Btu/lb	18,615	18,607	
Aniline-Gravity Product	6425	634 <b>5</b>	
Corrosion, copper strip at 212 F	lA .	1B	
Sulfur: Mercaptan, weight %	0.0006	0.0006	
Total, weight %	0.034	0.020	
Doctor Test	Sweet	Sweet	
Aromatics, volume %	10.8	13.6	
Olefins, volume %	2.6	1.6	
Smoke Point, mm	27.0	27.0	0.08
Naphthalene, weight %	0.08	0,08	0.08
Copper, ppb	33.0	<10.0 16	15.0
Indene, ppm	33		33
Iron, ppm	0.92 37.8	0 <b>.7</b> 7 8.5	22.2*
Lead, ppb Nitrogen: Basic, ppm	2.0	3.0	3.0
Pyrrole, ppm	0.10	0.10	0.00
Oxygen, Dissolved, ppm	27.6	40.1	
Peroxide, ppm	1.06	0.71	0.58
Sodium, ppm	2.05	2.64	7.59
Sulfonate, ppm	0.177	0.000	0.000
,			

<sup>\*</sup>Lead content is abnormally high because of using tin coated storage cans.

Table 6 (Continued)

## Physical and Chemical Tests on Fuels

	Before Treat	After Treat	Aged After Treat
Gravity, "API Distillation: IBP, "F 10% Evap., "F 50% Evap., "F 90% Evap., "F EP, "F Residue, volume % Loss, volume % Saybolt Color Freezing Point, "F	46.9 322 338 352 370 408 0.5 0.5 +27	47.1 332 338 352 374 394 1.0 1.0 +30 -80=	
Viscosity, cs at 40 F Particulate Matter, mg/gal, 0,45 µ Filter	5•23 2•3	5.32 1.2	0.6
Gum: Existent, mg/100 ml Potential, mg/100 ml	0.6 1.8	0.8 3.7	0.6 1.4
Water Reaction Index Water Separometer: WSI WSIM	1 55.6 26.0	1 92,9 46.0	
Moisture, ppm Specific Heat at 300 F Net Heat of Combustion, Btu/lb Aniline-Gravity Product Corrosion, copper strip at 212 F Sulfur: Mercaptan, weight % Total, weight % Doctor Test Aromatics, volume % Olefins, volume %	34.4 0.606 18,610 6378 1A 0,0006 0,024 Sweet 11.7	36.9 0.606 18,612 6391 2A 0.0006 0.020 Sweet 12.7 1.4	23.5
Smoke Point, mm Naphthalene, weight % Copper, ppb Indene, ppm Iron, ppm Lead, ppb Nitrogen: Basic, ppm Pyrrole, ppm Oxygen, Dissolved, ppm Peroxide, ppm Sodium, ppm	28.0 0.07 30.0 44 0.40 92.0 <1.0 0.00 25.8 1.22 3.08	25.0 0.08 <10.0 23 0.83 5.1 6.5 0.00 39.4 0.89 1.67	0.12 30.0 28 24.7* 0.00 0.67 6.99
Sulfonate, ppm	0.183	0.000	0.009

<sup>\*</sup>Lead content is abnormally high because of using tin coated storage cans.

Table 6 (Continued)

# Physical and Chemical Tests on Fuels

	Before Treat	After Treat	Aged After <u>Treat</u>
Gravity, API	48.1	48.0	
Distillation: IBP, F	324	326	
10% Evap., °F	332	332	
50% Evap., °F	342	343	
90% Evap., °F EP, °F	366 406	366 388	
Residue, volume %	1.0	388 1.0	
Loss, volume %	0.0	1.0	
Saybolt Color	+30	+30	
Freezing Point, °F	-80-	-80-	
Viscosity, cs at -40 F	5.21	4.96	
Particulate Matter, mg/gal, 0.454 Filter	3.4	2.7	
Gum: Existent, mg/100 ml	0.4	0.4	0.4
Potential, mg/100 ml	1.6	0.6	1.1
Water Reaction Index	1	1	
Water Separometer: WSI	71.9	997	
WSIM	35.0	50.0	07.1
Moisture, ppm Specific Heat at 300 F	36,4 0. <i>6</i> 08	38 <b>.</b> 2 0.608	21.4
Net Heat of Combustion, Btu/lb	18,633	18,626	
Aniline-Gravity Product	6595	6528	
Corrosion, copper strip at 212 F	1A	1B	
Sulfur: Mercaptan, weight %	0.0006	0.0006	
Total, weight %	0.023	0.015	
Doctor Test	Sweet	Sweet	
Aromatics, volume %	9.5	10.7	
Olefins, volume %	1.7	1.5	
Smoke Point, mm	25.0	27.0	
Naphthalene, weight %	0.15	0.06	0.10
Copper, ppb	33.0	<10.0	22,5
Indene, ppm Iron, ppm	16 0.06	7 0.22	1.3
Lead, ppb	49.7	27.9	22,2
Nitrogen: Basic, ppm	<1.0	2.8	3.0
Pyrrole, ppm	0.30	0.30	0.00
Oxygen, Dissolved, ppm	39.6	47.1	
Peroxide, ppm	2.84	0.71	0.62
Sodium, ppm	1.79	0.97	5.06
Sulfonate, ppm	0.039	0.000	0.000

# Table 6 (Continued)

## Physical and Chemical Tests on Fuels

	Before Treat	After Treat	Aged After Treat
Gravity, °API	47.4	47.2	
Distillation: IBP, °F	328	330	
10% Evap., °F	336	336	
50% Evap., °F	346	348	
90% Evap., °F	368	370	
EP, F	404	410	
Residue, volume %	1.0	1.0	
Loss, volume %	0.0	1.0	
Saybolt Color	+29	+25	
Freezing Point, °F	-80-	-80-	
Viscosity, cs at -40 F	5.23	5.11	
Particulate Matter, mg/gal, 0.45 Filter	4.9	1.9	_
Gum: Existent, mg/100 ml	0.4	0.4	0.2
Potential, mg/100 ml	2.0	1.0	1.1
Water Reaction Index	1	1	
Water Separometer: WSI	95.0	98.3	
WSIM	27.0	30.0	00 1
Moisture, ppm	42.0	43.7	22.4
Specific Heat at 300 F	0.606	0.606	
Net Heat of Combustion, Btu/lb	18,621	18,615	
Aniline-Gravity Product	6484	6419	
Corrosion, copper strip at 212 F	1A	1A	
Sulfur: Mercaptan, weight %	0.0006	0.0006	
Total, weight %	0.021	0.018	
Doctor Test	Sweet	Sweet 11.6	
Aromatics, volume %	11.9 1.5	1.5	
Olefins, volume % Smoke Point, mm	25.0	26.0	
Naphthalene, weight %	0.10	0.06	0.06
Copper, ppb	19.0	<b>&lt;10.</b> 0	20.0
Indene, ppm	23	12	12
Iron, ppm	0.34	0.22	
Lead, ppb	52.7	4.6	21.5*
Nitrogen: Basic, ppm	2.5	3.0	3.0
Pyrrole, ppm	0.30	0.30	0.00
Oxygen, Dissolved, ppm	47.9	36.3	
Peroxide, ppm	0.89	0.71	0.62
Sodium, ppm	0.68	0.72	5.06
Sulfonate, ppm	0.035	0,005	0.000

<sup>\*</sup> Lead content is abnormally high because of using tin coated storage cans.

Table 6 (Continued)

## Physical and Chemical Tests on Fuels

	Before Treat	After Treat	Aged After Treat
Gravity, °API	47.3	47,2	
Distillation: IBP, F	326	328	
10% Evap., °F	33 <sup>1</sup> 4	336	
5 <b>0% Evap., °F</b> 90 <b>% Evap., °</b> F	344 368	3 <sup>4</sup> 7	
EP, °F	403	370 414	
Residue, volume %	1.0	1,0	
Loss, volume %	1.0	1.0	
Saybolt Color	<b>+</b> 27	+25	
Freezing Point, F	-80-	-80-	
Viscosity, cs at -40 F	5.31	5,12	
Particulate Matter, mg/gal, 0,45, Filter	4.5	2.9	. 1
Gum: Existent, mg/100 ml	0.2	0.2	0.4
Potential, mg/100 ml Water Reaction Index	1.7 1	3,8 1	1,0
Water Separometer: WSI	79.1	99.9	
WSIM	22.0	50.0	
Moisture, ppm	36.4	29.1	53.0
Specific Heat at 300 F	0,606	0.606	, ,
Net Heat of Combustion, Btu/1b	18,620	18,616	
Aniline-Gravity Product	6471	6429	
Corrosion, copper strip at 212 F	lA	lA.	
Sulfur: Mercaptan, weight %	0.0006	0.0006	
Total, weight %	0.017	0.027	
Doctor Test	Sweet	Sweet 11.9	
Aromatics, volume % Olefins, volume %	11.0 1.7	2,2	
Smoke Point, mm	25.0	26.0	
Naphthalene, weight %	0.11	0.07	0.04
Copper, ppb	43.0	<10,0	5.0
Indene, ppm	18	4	14
Iron, ppm	0.62	0.02	
Lead, ppb	48.3	5,2	24.7*
Nitrogen: Basic, ppm	1.5	2.5	1.5
Pyrrole, ppm	0.30	0.30	Nil
Oxygen, Dissolved, ppm	51.1 0.71	33,4	0.21
Peroxide, ppm Sodium, ppm	0.71 0.92	0.89 0.88	0.31 3.00
Sulfonate, ppm	0.000	0.010	0.005
	3,000	3 , U MV	/

<sup>\*</sup> Lead content is abnormally high because of using tin coated storage cans.



Table 7

Growth Characteristics of Bacteria

Pseudomonas Species

Bacterial Designation, _University of Dayton	Culture Media, University of Dayton	Culture Media, Contractor	Growth Rate
B-40	B. Haas slant; JP-4	B. Haas liquid; JP-6	Prolific
B-40	TSA slant	B. Haas liquid; JP-6	Moderate
B-40	TSA slant	Nutrient broth	Slight
B-44	B. Haas slant; JP-4	B. Haas liquid; JP-6	Prolific
B-44	TSA slant	B. Heas liquid; JP-6	Slight
B-44	TSA slant	Nutrient broth	Slight
B-54	B. Haas slant; JP-4	B. Haas liquid; JP-6	Prolific
B-54	TSA slant	B. Haas liquid; JP-6	Prolific
B-54	TSA slant	Nutrient broth	Prolific



Table 8
Growth Characteristics of Fungi

Fungal Designation, University of Dayton	Culture Media, University of Dayton	Culture Media, Contractor	Growth Rate
B-29	Sab. agar slant	B. Haas; JP-6	Slight
B-29	Sab. agar slant	Sab. liquid	Prolific
B~55	Sab. agar slant	B. Haas; JP-6	Moderate
B-55	Sab. agar slant	Sab. liquid	Moderate

Notes: B-29 is Cladosporium resinae f. avellaneum.

B-55 is Hormodendrum hordei.



Table 9
Sample Schedule for Biological Research
Standard Reclamation Filter

Control	Inoculated Drum	Following Chemical Dryer	Following Reclamation Filter
х	х	l gallon through	l gallon through
		5 gallons through	5 gallons through
		15 gallons through	15 gallons through

Notes: Column size of chemical dryer: 0.5 inch x 60 inches

Column size of reclamation filter: 1.0 inch x 96 inches



Table 10

Analysis of Extract Removed from Filter Media

		Weight, %	
Constituents Removed	Top	Middle	Bottom
Metal Deactivator	1,38	0.30	0.25
Naphthalene	0.53	0.47	0.26
Sodium	0.099	0.014	0,011
Lead	0.086	0.084	0.056
Indene	0.051	0.025	0.017
Paraphenylenediamine	0.042	0.0008	Nil
Sulfonate	0.0047	0.0011	Nil
Basic Nitrogen	0.0018	0.0002	Nil
Iron	0.0015	0.0015	0.0019
Pyrrole Nitrogen	0.0002	Nil.	Nil
Copper	0.00012	Nil	Nil



Table 11
Analysis of Grease Synthesized and Retained

Constituents	Weight %	Dom
Apparent Naphthalene	8.00	
Sulfur	2.87	
Indene	1.34	13400
Sulfonate	0.078	777
Paraphenylenediamine	0,063	629
Metal Deactivator	0.027	272
Peroxide	0.027	266
Iron	0.020	200
Lead	0.017	172
Copper	0.0017	17.5
Saponification Number	0.0 (Neutral)	
Ash Content	23.68	
Total Analyzed Contaminants	12.44	

#### Table 12

#### Effect of Petroleum Sulfonates on Thermal Stability

Reclaim No. 12 (Plus Filtration)

Coker Conditions	Preheater	Diff. Pressure, in. Hg
425/525/6	1111111111111	0.0
450/550/6	111111111111111111111111111111111111111	0.0
475/575/6	1111111111221	0.0

Modified Water Separometer (WSIM): 98.0

Reclaim No. 12 (Plus 2 ppm Petroleum Sulfonate then Plus Filtration)

 Coker Conditions
 Preheater
 Diff. Pressure, in. Hg

 475/575/6
 1111111111221
 0.0

Modified Water Separometer (WSIM): 99.0

Reclaim No. 12 (Plus Filtration then Plus 2 ppm Petroleum Sulfonate)

 Coker Conditions
 Preheater
 Diff. Pressure, in. Hg

 475/575/6
 1111111111121
 0.0

Modified Water Separometer (WSIM): 23.0

Reclaim No. 12 (Plus 0.5 ppm Petroleum Sulfonate then Plus Filtration)

Modified Water Separometer (WSIM): 98.0

Reclaim No. 12 (Plus Filtration then Plus 0.5 ppm Petroleum Sulfonate)

Modified Water Separometer (WSIM): 53.0



# Table 13 Effect of Peroxides on Thermal Stability

<u>Fuel</u>		Preheater, max code 450/550/6
1	JP-6 with no treatment	ı
2	JP-6 plus 10 ppm benzoyl peroxides	3
3 🔩	Fuel 2 inoculated and filtered	ı

# Table 14 Effect of Indenes on Thermal Stability

Fuel		Preheater, max code 450/550/6
1	JP-6 with no treatment	1
2	JP-6 plus 25 ppm indenes	4
3	Fuel 2 inoculated and filtered	1

Table 15

Effect of Naphthenic Acid on Thermal Stability

Treatment	Operating Conditions	Preheater	Diff. Pressure, in. Hg
Control, JP-6 with no treatment	450/550/6	11111111111111	0.0
Control, JP-6 with no treatment	475/575/6	1111111123331	0.1
JP-6 plus 1 ppm naphthenic acid	450/550/6	1111111112221	0.3
JP-6 plus 3 ppm naphthenic acid	450/550/6	1111111433331	0.0
JP-6 plus 5 ppm naphthenic acid	450/550/6	1111223366443	0.0
JP-6 plus 5 ppm naphthenic acid			
then reclamation filtration	450/550/6	11111111111111	0.0
Second run for confirmation	450/550/6	11111111111111	0.0

Notes: The last run was a duplicate of the preceding run to confirm that filtration removes naphthenic acid and therefore rehabilitates the fuel.

All treatments were made with the same JP-6 jet fuel.

Table 16

Removal of Elemental Sulfur by Reclamation Filtration

Preheater	111111134554	1111111178876	111111133543	ננוננננננננ	111111234554	
ASTM-CRC Coker Conditions	9/005/004	9/005/004	9/005/004	9/005/004	575/650/2 <b>.</b> 5 575/650/2 <b>.</b> 5	
Corrosion, copper strip at 212 F	3B	3.B	3B	A1	3B 1A	
Type of Reclamation	Control, no treatment	Filtration through 0.01 $\mu$ Millipore filter	Filtration through 30/60 clay	Standard reclamation filtration	Control, no treatment Standard reclamation filtration	
Fuel	A	А	A	Ą	д д	

The control samples with no treatment gave positive identification of the presence of elemental sulfur by the mercury test. Notes:

The standard reclamation filtered samples gave negative results for elemental sulfur by the mercury test.

Table 17
Phenol Content (ppm) Before and After Filtration

Reclaim No.	Before Treat	After Treat
1	250	10.0
2	40	0.0
3	35	0.0
4		0.0
5	290	0.0
6	63	0.0
7	320	0.0
8	315	12.5
9	285	10.0
10	250	15.5
11	5.0	10.0
12	5.0	8.0
13	4.0	4.0
14	6.0	2.0
15	6.0	0.5
16	2.0	0.0
17	0.0	0.0
18	0.0	0.0
19	0.0	8.0

Note: The <u>before treat</u> results represent phenol content on fuel samples prior to filtration. The <u>after</u> treat results represent phenol content on the fuel samples after filtration.



Table 18

Additive Content Before and After Reclamation Filter

	Paraphenylenediamine Antioxidant, lbs/1000 bbl		mine Metal Deacti 00 bbl lbs/1000 b	
Reclaim No.	Before	After	Before	After
1	Nil	0,00	2.69	0.00
	0.35	0.00	Nil	0,00
3	6.98	0.00	0.88	0.00
Ĭ	7,36	0.35	1.40	0.00
5	0.87	0.00	1.54	0,00
2 3 4 5	2.44	0.00	Ni.1	0.00
	Nil	0.00	2.27	0.00
7 8	0.17	0.00	2.36	0.00
9	1.05	0.00	1.82	0.00
ıó	Nil	0.00	1.01	0.00
11	2.27	0.00	0.33	0.00
12	7.33	0.00	1.42	0.07
13	1.57	0.00	0.93	0.00
14	7,86	0.00	1.83	0.07
15	4.19	0.00	1.46	0.16
<u>1</u> 6	1.80	0.35	0.94	0.30
17	1.40	0.00	0.40	0.02
18	2.79	0.00	2.50	0.00
19	2.27	0.00	1.10	0.00

Table 19

Detailed Thermal Stability Data for Corrosion Inhibitor Treated Fuels

Diff. Pressure, in. Hg	0.0000.0000.000000000000000000000000000	0.0000000000000000000000000000000000000
Preheater	11111111111 11111123454 11111111111111111111111111111111111	111111111221 1111122221 1111111111111 111111
Corrosion Inhibitor	Control 1 2 3 4 5 5	ው <i>ጣ</i> ተማ ከ ሥ
Treating Sequence	Filtered only Filtered, 2. Corrosion inhibited	1. Corrosion inhibited, 2. Filtered 1. Corrosion inhibited, 2. Filtered

Notes: Fuel used throughout: Reclaim No. 11

ASTM-CRC coker conditions used throughout: 450/550/6

Military approved corrosion inhibitors: minimum allowable concentration



Table 20
Lube Oil Additive Analysis by Infrared

Run	% Lube A Before Treat	Additive After Treat
1	0.024	0.0
2	0.022	0.0

Table 21

Effect of Antistatic Additive on Thermal Stability

<u>Fuel</u>		Preheater, max code	Diff. Pressure,
1	JP-6 with no additives	1	0
2	Fuel No. 1 with antistatic additive	3	1.2
3	Fuel No. 2 after reclamation filter	1	0

Table 22

Recommended Fuel Flow Rates for Filtration Unit

Fuel Type	Viscosity at 77 F, cs	Max Flow Rate, gals/hour/ft <sup>2</sup>		re Drop, ter Bed D 16 ft.	
JP-4	0.921	400	8	19	28
JP-6	1.326	375	12	28	42
JP-5	2.142	350	21	49	7 <b>4</b>
Diesel	3.109	315	28	65	97



Appendix I

Detailed Thermal Stability Data for Reclaimed Fuels

ASTM-CRC Comparative Coker Ratings (450/550/6)

		Preheater	
Reclaim No.	<u> </u>	B	С
1	1111235666664	11111111111111	111111111111111
2	1111111113335	11111111111111	11111111112221
3	1112223677765	1111111111111	11111111111111
4	1111114555543	111111111111111	111111111111111
5	1111111188888	11111111111111	11111111111111
6	1111111133332	13111111111111	11111111111111
7	1111111111665	11111111111111	11111111111111
8	1111111688886	111111111111111111111111111111111111111	111111111111111
9	1111111137754	111111111111111	11111111111111
10	11123688888888	1111111111222	11111111111111
11	1111111156666	11111111111111	11111111111111
12	1111235778887	1311111111111111	11111111111111
13	1111688888888	111111111111111	11111111111111
14	1111168888888	13111111111111	11111111111111
15	1111111245332	11111111111111	111111111111111111111111111111111111111
16	11111111112454	11111111111111	111111111111111
17	1111111666885	11111111111111	111111111111111111111111111111111111111
18	1111111155532	11331111111111	11111111112221
19	1111111111555	11111111111111	1111111112221

A - Degraded fuel as received

B - Treated fuel immediately after reclamation filtration

C - Treated fuel six months after reclamation filtration

Appendix II

# Threshold Thermal Stability Data

ASTM-CRC Coker Ratings by the Aero Propulsion Laboratory on Reclaim Nos. 1 Through 10

Reclaim No.	Date	Test Conditions, °F	△P-Minutes	Preheater
1 1	7/1 /63 7/25/63 7/1 /63	400/500/6 400/500/6 425/525/6	0.2-300 0.7-300 17.5-300	11111111111111 11111111111111 11111111
2	7/22/63	375/475/6	8.8-300	1111111111111
2	7/2 /63	400/500/6	25.0-280	22222222233
2	7/2 /63	425/525/6	25.0-188	11111111112433
3	7/23/63	375/475/6	3.4-300	1111112211111
3	7/3 /63	400/500/6	25.0-249	3333333311333
3	7/3 /63	425/525/6	25.0-234	1123331111343
<u>ነ</u> ተ <u>ን</u> ተ	7/24/63 7/15/63 7/15/63	375/475/6 400/500/6 425/525/6	25.0-300 25.0-128 25.0-191	111111111111 0000001134441 11111111114443
5	7/23/63	375/475/6	1.7-300	1111111122222
5	7/8 /63	400/500/6	1.7-300	1111111111322
5	7/8 /63	425/525/6	25.0-300	11111111111
6	7/23/63	375/475/6	9.1-300	1111111222221
6	7/9 /63	400/500/6	14.8-300	2222222222332
6	7/9 /63	425/525/6	25.0-178	1111133211111
7	7/24/63	375/475/6	6.2-300	1111111111112
7	7/10/63	400/500/6	25.0-198	11111111224332
7	7/10/63	425/525/6	25.0- 68	11111111111
8	7/11/63	400/500/6	6.7-300	111111111111111111111111111111111111111
8	7/24/63	400/500/6	1.1-300	
8	7/11/63	425/525/6	14.2-300	
9	7/12/63	400/500/6	0.5-300	111111111111111111111111111111111111111
9	7/12/63	425/525/6	0.4-300	
10 10 10 10	7/18/63 7/18/63 7/22/63 7/25/63	400/500/6 400/500/6 375/475/6 375/475/6	25.0-260 25.0-235 23.6-300 11.6-300	1111111112441 1111111124442 11111111111



Appendix III
Storage Stability Data of Unfiltered Fuels

Reclaim	Date	ASTM-CRC Coker Operating Conditions at Threshold Temp.	Preheater
No.	<u>Tested</u>	at the estate temp.	1101100000
11 11 11 11	8/14/63 8/15/63 2/11/64 2/12/64	375/475/6 350/450/6 325/425/6 300/400/6	1111111116655 1111111111111 1111111136541 1111111111111
12 12 12	8/14/63 8/15/63 4/3 /64 4/3 /64	375/475/6 350/450/6 350/450/6 325/425/6	1111111115555 1111111111111 1111111113453 1111111111
14 14 14 14 14	8/27/63 8/24/63 4/7 /64 4/7 /64 4/7 /64	450/550/6 425/525/6 450/550/6 425/525/6 400/500/6	1111111124532 11111111111121 1111111478542 1111111111243 1111111111111
17 17 17 17 17 17	8/29/63 8/29/63 8/30/63 4/10/64 4/11/64 4/13/64	450/550/6 400/500/6 375/475/6 450/550/6 375/475/6 350/450/6	1111111244432 11111111114432 1111111112221 1111111135432 1111111122211

# Appendix IV

## Laboratory Procedures Used for Physical and Chemical Testing

Test	Federal Std. No. 791	ASTM Standard	Others
Gravity, API		D 287	
Distillation		D 86	
Saybolt Color		D 156	
Freezing Point		D 1477	
Viscosity		D 445	
Particulate Matter		Proposed	
Existent Gum		D 381	
Potential Gum		D 873	
Water Reaction Index	3251		
Water Separometer Index	3255		
Water Separometer Index Modified	3256		
Moisture		D 1364-62 Vol. II	
Specific Heat			Calculated
Net Heat of Combustion			National Bureau of Standards
Aniline-Gravity Product		D 287 and D 611	
Corrosion		D 130	
Sulfur, Mercaptan		D 1323	
Sulfur, Total		D 1266	
Doctor Test			UOP 41-59

Test	Federal Std. No. 791	ASTM Standard	Others
Aromatics		D 1319	
Olefins		D 1319	
Smoke Point		D 1322	
Naphthalene		D 1840	
Copper			AORCO (Photometric)
Indene			Monsanto 2521-2
Iron			AORCO (Photometric)
Lead			AORCO (Photometric)
Nitrogen, Basic			Sinclair
Nitrogen, Pyrrole			UOP 276-59
Oxygen, Dissolved			Phillips GC
Peroxide			Louisville Neoprene Lab #S-17.08
Sodium			AORCO (Emission)
Sulfonate			California Research Corporation
Phenol			Shell
Saponification Number		D 939	
Thermal Stability		р 1660	

#### Appendix V

### Bacteriological Procedures

## A. Culture Propagation

In order to propagate the quantity of both bacteria and fungi necessary to inoculate adequately 15-gallon drum samples, subcultures were prepared using for the bacteria Bushnell-Haas liquid medium with JP-6 (sterile) overlay and nutrient broth (see Table 7). For the fungi, both Bushnell-Haas mineral salts liquid medium with JP-6 overlay and Sabouraud's liquid medium were used (see Table 8). These subcultures were allowed to incubate at 37 C for several days prior to their inoculation into the fuel.

As the growth rates shown in the tables indicate, the bacteria proliferate more readily in a mineral salts-jet fuel environment than the two fungal species.

## B. Fuel Inoculation, Sampling, and Culturing Procedures

1. Isolation of microorganisms

Each subculture was isolated from the liquid culture medium by means of decantation and filtration. The suction of the filtration assembly was released when approximately 30 milliliters of organism-containing medium remained. To preclude aerosol contamination, the apparatus was covered with foil and moved to the reclamation filter pilot plant units.

2, Actual drum inoculation procedure

Prior to inoculation of the JP-6 with the microorganisms, a stream of cylinder-nitrogen was delivered through a fritted agitator into the fuel with such velocity that the fuel was mixing violently at the time of inoculation. At this point, the microorganisms were emptied into the fuel and allowed to circulate for five minutes prior to sampling of the inoculated 15-gallon drum of JP-6. Figure 28 shows the intimate dispersion of the bacteria in the fuel.

Millipore pyrex filter assembly; 0.45 micron pore diameter, type HA filter.

3. Sampling procedure for inoculated drum

A one-quart sample was siphoned from the inoculated drum through sterilized tygon tubing into a sterilized quart bottle. This was returned to the bacteriological laboratory for culturing.

4. Isolating and culturing procedure for samples

Aseptic conditions were maintained in the laboratory at all times and elsewhere during the course of this project as required. The quart fuel samples taken at various points (see Sample Schedule, Table 9) were filtered through a 0.45 micron Millipore filter. Each empty sample bottle was rinsed with four 25-milliliter increments of sterile water. These incremental washes were added to the respective Millipore filtration assembly and suction applied to aid in drawing the wash-material through the cellulose filter. Finally, the Millipore cellulose filter was carefully placed in previously sterilized petri culture dishes containing the appropriate medium to optimize growth of the organisms sought. (Sabouraud's liquid medium in the case of the fungi and Bushnell-Haas mineral salts in the case of the bacteria.) These were incubated at 37 C for fourteen days.

#### C. Culture Examinations and Determinations of Results

#### 1. Visual examination

Visual examinations were made daily and a log kept of the presence or absence of biological growth. These inspections revealed that fungal growth could be detected within 24 hours. Following a 72-hour incubation period of a Hormodendrum-species (fungi)-inoculated-drum sample, approximately two hundred and fifty colonies were counted. Figure 25 shows the culture of these fungi through the test sequence. The control sample and the sample following the reclamation filter (designated as number 3), show a complete absence of fungal growth; whereas number 1, which is the inoculated drum sample, and number 2, the fuel following the chemical dryer section of the unit, show 250- and 12-colonies, respectively. (The fungi grew at the fuel-culture medium interface while the bacteria dispersed throughout the culture medium phase only.) Visual inspection for bacteria was less successful since approximately fortyeight hours were necessary to produce turbidity within the Bushnell-Haas medium, great enough to assure positive or negative identification. It was therefore decided that microscopic checks were more reliable in this area.

2. Optical microscopic checking procedure

One-inch by three-inch slides were prepared using various stains to accentuate contrast between the cell walls of the microorganisms and their background which in turn produced better photomicrographs. Loeffler's staining procedure proved to be adequate for this application.

Optical microscopy was found to be adequate for determination of the presence of both bacteria and fungi. Electron microscopy was also utilized both because it is more definitive and because it provides a means of double checking.

3. Electron microscopic<sup>3</sup> checking procedure

Nickel grids were covered with the culture in question by means of a looped inoculating needle. These grids were then scanned in order to detect micronic and submicronic particles in addition to microbiological contaminants. This served a very useful purpose of not only revealing the fuel to be completely free of bacteria and fungi following the reclamation filtration, but also to be virtually free of extraneous matter of all types. The electron photomicrograph 7500 X magnification enhances the cell wall differentiation and enables the viewer to obtain a better concept as to its cellular structure.

<sup>2</sup> Leitz Ortholux microscope, maximum magnification (oil) 1225 X.

<sup>3</sup> Phillips Electron Microscope, Model 75.

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Life of Standard Filter Media	Diff. Pressure, in. Eg	A 25.000.0000.000000000000000000000000000	0000000000
	Preheater	11111267888 1111111111111111111111111111111111	111235778887 11111111111111111111111111111111
	Filter Media Life Barrels of Fuel Per Ton Filter Media	381 6096 12192 14859 16774 19288	381 8763 16383 19812 20574 20955 22860
	Coker Run No.	13 13 13 13 13 13 13 13 13 13 13 13 13 1	3 4 3 3 8 8 4 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5
	Reclaim No. 11	Control, no treatment Filtered through pilot unit No. 1	Control, no treatment Filtered through pilot unit No. 2