MIDDLE DISTILLATE FUEL STABILITY CHARACTERISTICS—A REVIEW

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# Middle Distillate Fuel Stability Characteristics—A Review

**Abstract**

Middle distillate fuel stability is of prime concern when long-term storage is necessary. Extensive work has been undertaken over the past half century in an attempt to establish the true chemical mechanism by which this degradation takes place.

This review was undertaken as part of a basic research program having as its goal the definition of fuel deterioration and additive-inhibition mechanisms. It is intended to report some of the more pertinent highlights so that the
20. ABSTRACT (Cont'd)

reader may obtain an overview of the current state-of-the-art in fuel stability research. An annotated bibliography has also been included so that more in-depth information may be readily obtained.
FOREWORD

The review presented herein was prepared at the U.S. Army Fuels and Lubricants Research Laboratory (USAFLRL), Southwest Research Institute, San Antonio, Texas, under Contract DAAK70-82-C-0001. The work was funded by the U.S. Army Mobility Equipment Research and Development Command (USAMERADCOM), Ft. Belvoir, VA. Contracting Officer's representative was Mr. F.W. Schaekel, and the technical monitor was Mr. M.E. LePera, Fuels and Lubricants Division, Materials, Fuels and Lubricants Laboratory (DRDME-VF).
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</table>
I. INTRODUCTION

A. Purpose

The material presented in this review is an accumulation of information from many of the more significant presentations in literature. It is designed to be a ready reference document and, as such, does not contain some of the detailed information that an exhaustive review might cover. However, the inclusion of an annotated bibliography gives a base from which more specific information may be obtained in particular areas of interest.

B. Historical Information

Petroleum-like bituminous materials were described by Herodotus, Plutarch and others (1)* 2000 years ago. However, actual use of these materials spans at least 5000 years, dating to the Sumerians, Assyrians, and Babylonians. During those times, seepages were found on the Euphrates River, in Mesopotamia, and in the eastern Mediterranean areas.(2) Crude oil was probably obtained by dipping directly into the seep or by prior digging to enlarge the area and then dipping the material out.(1) Because of these seeps, the area now known as the Dead Sea was, in ancient times, known as Lake Asphaltites, because of the tarballs found on its shores.

The word "petroleum" is attributed to first use by a German mineralogist in a treatise on its recovery and refining published in 1556.(2) Other historical points are noted by Bacon and Hamor.(3)

The tars and oils which made up the substance of petroleum have been used in a wide variety of ways, including caulking, waterproofing, and illumination. In the early 19th century United States, animal fat (especially whale fat) was still the major source of illuminating oil, even though petroleum seeps had been found. With increasing population, a more abundant source of

*Underscored numbers in parentheses refer to the list of references at the end of this report.
refined oil became necessary. Distillation of coal was popular, but product volume was not sufficient to overcome the growing needs for less costly and greater volumes of lubricants and illuminating oil. These requirements and the approach of the Industrial Revolution led to completion of the first drilled oil well in the United States at Titusville, Pennsylvania in 1859. Refining of this crude took place in the already existing coal-oil refineries.(2) By 1900, Pennsylvania lubricants had displaced animal and vegetable materials as the primary oil source.(4) By this time, oil fields had been discovered in 14 states, Europe, and the Far East.(2)

From the beginning of this century to the present time, greater emphasis has continually been placed on particular specifications for various refined products. As noted, lubricants and illuminants headed the early product lists; now, approximately 2500 materials are produced, at least in part, from petroleum, not counting the 3000 petrochemicals also produced.(4)

C. Processing

Methods for refinement of crude oils have also changed considerably over the years, due to increasing demands for specific products and better overall performance of those products. Several refinery processes are currently in use. Each is generally composed of three fundamental activities:(1)

1. Primary separation of raw materials
2. Preparation of process feed
3. Fractionation of the products

These may be accomplished by:(4)

1. Crude fractionation
2. Thermal decomposition
3. Catalytic cracking
4. Catalytic reforming
5. Hydrocracking
Crude fractionation is generally used as the first step before further processing. Light gasoline, naphtha, kerosene, gas oil, and topped crude are the products obtained from this process. Materials from the process are designated as straight run products. Further refining, as described below, is required for the heavier products.

Successful cracking processes were demonstrated for use in World War I, but it was not until World War II that the thermal cracking process was maximized. Catalytic cracking became successful toward the end of World War II, but its growth has been optimized only within the past 20 years, now exceeding thermal cracking in terms of capacity. Thermal cracking treats the feed material at temperatures of 850°-1150°F (454°-621°C) and pressures of 1 to approximately 110 atmospheres for 10 minutes or less. Catalytic cracking requires less severe conditions [750°-1000°F (399°-538°C)] at 1-7 atmospheres while using the catalytic material to accelerate and guide the cracking process to produce the desired final products. Fluidized catalytic cracking (as opposed to the fixed bed process) began in 1942. In this process, vaporized feedstock lifts the powdered catalyst into the reaction bed, thus "fluidizing" the powdered material.

Catalytic reforming is not a popular process in the United States because much higher octane gasoline can be produced by alkylation and catalytic cracking. Reforming also leads to loss of light ends (gases) and therefore is not economically feasible. It does, however, substantially increase the aromatic content of naphthenic feedstock through partial dehydrogenation of the naphthenes.

Hydrocracking produces a final product almost exclusively composed of aliphatic and aromatic materials with little or no sulphur and no nitrogen. This would be the process of choice except for the expense of the hydrogen involved. Even so, use of this process is increasing, and if some of the economic factors are overcome, it could replace catalytic cracking as the major refining process.

D. Demand

For FY 1977, the total U.S. petroleum energy demand was approximately 18.6
million barrels per day (of which DOD used 2.6 percent).\(^6\) In 1980, total oil consumption in the U.S. was approximately 16.9 million barrels per day.\(^7\) During this period (1978) demand for middle distillate fuel oil (diesel, jet fuel, etc.) was approximately 3.4 million barrels per day, or 18 percent of the total demand for that year.\(^8\)

E. Fuel Stability Background

After a fuel is produced, the time lag prior to end use could vary from weeks to years. Long-term storage stability has been a requirement imposed on military fuels designed for tactical/combat uses.\(^9\) However, storage stability (and the prediction of long-term fuel stability) for diesel and distillate fuels has long been recognized as a potential problem by both government and industry.\(^10-11\) Many research programs have been undertaken to determine how fuel can be stabilized (by use of additives) as well as what causes degradation to occur (elucidation of reaction mechanisms). Various specifications have been formulated for specific use fuels. These specifications also include geographical allowances, particularly for temperature. A summary of fuel specifications for aviation and diesel fuels is given in Tables 1 and 2.

Almost a quarter of a century ago, the chemical character of fuel degradation products was postulated.\(^12\) Well over one-half century ago, it was noted that the presence of diolefinic hydrocarbons in cracked distillates was at least partially responsible for gum formation in gasoline.\(^13\) Several papers published in the late 1920's through the 1930's verify and expand upon this fact.\(^14\) The following sections summarize current knowledge in the field of middle distillate fuel stability.

II. MECHANISMS OF GUM FORMATION

A. Oxidation/Autoxidation

This mechanism is considered to be the most prominent of the degradation
### TABLE 1. SUMMARY OF FUEL SPECIFICATION PROPERTIES OF AVIATION TURBINE FUELS

<table>
<thead>
<tr>
<th>Specification:</th>
<th>MIL-T-5624K* Amendment 1</th>
<th>MIL-T-83133A**</th>
<th>ASTM D 1655***</th>
</tr>
</thead>
<tbody>
<tr>
<td>NATO Code No.:</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Properties</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Distillation Temp., °C (D 2887 limits in parentheses)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>10% recovered, max temp</td>
<td>205(185)</td>
<td>205(186)</td>
<td>204.4</td>
</tr>
<tr>
<td>20% recovered, max temp</td>
<td>205(186)</td>
<td>206(187)</td>
<td>204.4</td>
</tr>
<tr>
<td>50% recovered, max temp</td>
<td>--</td>
<td>--</td>
<td>--</td>
</tr>
<tr>
<td>90% recovered, max temp</td>
<td>--</td>
<td>--</td>
<td>--</td>
</tr>
<tr>
<td>End point, max temp</td>
<td>--</td>
<td>--</td>
<td>--</td>
</tr>
<tr>
<td>Residue, vol%, max (D 86)</td>
<td>1.5</td>
<td>1.5</td>
<td>1.5</td>
</tr>
<tr>
<td>Loss, vol%, max (D 86)</td>
<td>1.5</td>
<td>1.5</td>
<td>1.5</td>
</tr>
<tr>
<td>Flash Point, °C(°F), min</td>
<td>--</td>
<td>--</td>
<td>--</td>
</tr>
<tr>
<td>Vapor Pressure, 37.8°C(100°F), kPa</td>
<td>14-21</td>
<td>--</td>
<td>--</td>
</tr>
<tr>
<td>Freezing Point, °C(°F), max</td>
<td>-58(-72)</td>
<td>-46(-51)</td>
<td>-50(-58)</td>
</tr>
<tr>
<td>Viscosity, @ -20°C, cSt, max</td>
<td>6.5</td>
<td>8.0</td>
<td>8.0</td>
</tr>
<tr>
<td>Water Reaction, Interface rating, max</td>
<td>1b</td>
<td>1b</td>
<td>1b</td>
</tr>
<tr>
<td>Separation rating, max</td>
<td>2</td>
<td>2</td>
<td>2</td>
</tr>
<tr>
<td>Water Separation Index, modified, min</td>
<td>70</td>
<td>85</td>
<td>7/</td>
</tr>
<tr>
<td>Total Acid No., mg KOH/g, max</td>
<td>0.015</td>
<td>0.015</td>
<td>0.015</td>
</tr>
<tr>
<td>Aromatics, vol%, max</td>
<td>25.0</td>
<td>25.0</td>
<td>25.0</td>
</tr>
<tr>
<td>Olefins, vol%, max</td>
<td>5.0</td>
<td>5.0</td>
<td>5.0</td>
</tr>
<tr>
<td>Mercaptan Sulfur, wt%, max</td>
<td>0.001</td>
<td>0.001</td>
<td>0.001</td>
</tr>
<tr>
<td>Sulfur, total wt%, max</td>
<td>0.40</td>
<td>0.40</td>
<td>0.3</td>
</tr>
<tr>
<td>Copper Strip Corrosion, 2 hr @ 100°C (212°F), max</td>
<td>1b</td>
<td>1b</td>
<td>1b</td>
</tr>
<tr>
<td>Density, kg/m³, min</td>
<td>751(57.0)</td>
<td>788(48.0)</td>
<td>775(51.0)</td>
</tr>
<tr>
<td>Density, kg/m³, max</td>
<td>802(65.0)</td>
<td>845(56.0)</td>
<td>840(37.0)</td>
</tr>
<tr>
<td>Thermal Stability (JFTOT), Change in pressure drop, mm of Hg, max</td>
<td>25</td>
<td>25</td>
<td>25</td>
</tr>
<tr>
<td>Preheater deposit code, less than</td>
<td>3</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>Existent Gum, mg/100 mL, max</td>
<td>7.0</td>
<td>7.0</td>
<td>7.0</td>
</tr>
<tr>
<td>Particulate Matter, mg/liter, max 8/</td>
<td>1.0</td>
<td>1.0</td>
<td>1.0</td>
</tr>
<tr>
<td>Filtration Time, minutes, max 8/</td>
<td>15</td>
<td>--</td>
<td>--</td>
</tr>
<tr>
<td>Heating Value, Aniline-gravity product, min, or Net heat of combustion, MJ/kg (Btu/lb), min</td>
<td>5,250</td>
<td>4,500</td>
<td>4,28(18,400)</td>
</tr>
<tr>
<td>Hydrogen content, wt%, min, or</td>
<td>13.6</td>
<td>13.5</td>
<td>13.5</td>
</tr>
<tr>
<td>Smoke point, mm, min, or</td>
<td>20.0</td>
<td>19.0</td>
<td>25</td>
</tr>
<tr>
<td>Smoke point, mm, min, and</td>
<td>--</td>
<td>--</td>
<td>19</td>
</tr>
<tr>
<td>Naphthalenes, vol%, max</td>
<td>--</td>
<td>--</td>
<td>3.0</td>
</tr>
<tr>
<td>Fuel System Icing Inhibitor, vol%</td>
<td>0.10-0.15</td>
<td>0.10-0.15</td>
<td>0.10-0.15</td>
</tr>
<tr>
<td>Fuel Electrical Conductivity, pS/m</td>
<td>--</td>
<td>--</td>
<td>200-600</td>
</tr>
</tbody>
</table>

* Dated 12 November 1976
** Dated 5 May 1976
*** Dated 1978

1/ The minimum flash point shall be 38°C as measured using ASTM D 3243 and 41°C as measured using ASTM D 93.
2/ The maximum freezing point for Jet A is -40°F and for Jet A-l it is -50°C (-58°F).
3/ The minimum water separation index, modified, rating shall be 85 for JP-8 fuel without both the corrosion inhibitor and the electrical conductivity additives present or 70 for JP-8 fuel containing all required additives except for the electrical conductivity additive.
4/ Jet A with an aromatics content over 20 volume % but not exceeding 25 volume % and Jet A-l or Jet B with an aromatics content over 20 volume % but not exceeding 22 volume % is permitted provided the supplier (seller) notifies the purchaser of the volume, distribution and aromatic content under conditions mutually agreeable to both parties. This footnote is subject to reapproval in 1979.
5/ Applies only when an electrical conductivity additive is used.

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The content includes various specifications for aviation turbine fuels, detailing properties such as distillation temperatures, flash points, and other relevant characteristics. Each specification is categorized under NATO code numbers and includes details on the fuel's performance characteristics under specific conditions. The table provides a comprehensive overview of the fuel specifications required for different grades and types of aviation turbine fuels.
**TABLE 2. SUMMARY OF FUEL SPECIFICATION PROPERTIES OF DIESEL AND DISTILLATE FUELS**

<table>
<thead>
<tr>
<th>Specification:</th>
<th>VV-F-800C*</th>
<th>NATO F-54**</th>
<th>MIL-F-16884C***</th>
<th>MIL-F-24397****</th>
</tr>
</thead>
<tbody>
<tr>
<td>Product:</td>
<td>DF-A</td>
<td>DF-1</td>
<td>DF-2 CONUS</td>
<td>DF-2 OCONUS</td>
</tr>
<tr>
<td>NATO Code No.:</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>---</td>
</tr>
<tr>
<td>50% evaporated, max</td>
<td>550 (288)</td>
<td>550 (288)</td>
<td>640 (338)</td>
<td>675 (357)</td>
</tr>
<tr>
<td>90% evaporated, max</td>
<td>572 (300)</td>
<td>626 (330)</td>
<td>700 (370)</td>
<td>698 (370)</td>
</tr>
<tr>
<td>End point, max</td>
<td>3</td>
<td>3</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>Residue, vol%, max</td>
<td>100 (38)</td>
<td>100 (38)</td>
<td>125 (52)</td>
<td>133 (56)</td>
</tr>
<tr>
<td>Flash Point, °F (°C), min</td>
<td>-60 (-51)</td>
<td>27</td>
<td>27</td>
<td>27</td>
</tr>
<tr>
<td>Cloud Point, °F (°C), max</td>
<td>--</td>
<td>--</td>
<td>--</td>
<td>--</td>
</tr>
<tr>
<td>Viscosity @ 104°F (340°C), cSt</td>
<td>1.1-2.4</td>
<td>1.3-2.1</td>
<td>1.1-4.1</td>
<td>1.8-9.5</td>
</tr>
<tr>
<td>Viscosity @ 68°F (20°C), cSt</td>
<td>1.1-2.4</td>
<td>1.3-2.1</td>
<td>1.1-4.1</td>
<td>1.8-9.5</td>
</tr>
<tr>
<td>Total Acid Number, mg KOH/g, max</td>
<td>0.05</td>
<td>---</td>
<td>---</td>
<td>0.10</td>
</tr>
<tr>
<td>Sulfur, % wt., max</td>
<td>0.25</td>
<td>0.50</td>
<td>0.50</td>
<td>0.70</td>
</tr>
<tr>
<td>Copper Strip Corrosion, 3 hrs @ 122°F (50°C), max</td>
<td>3</td>
<td>3</td>
<td>3</td>
<td>1</td>
</tr>
<tr>
<td>Ash, % wt., max</td>
<td>0.01</td>
<td>0.01</td>
<td>0.01</td>
<td>0.02</td>
</tr>
<tr>
<td>Density @ 15°C, kg/L</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>0.815-0.860</td>
</tr>
<tr>
<td>Accelerated Stability, total insolubles, mg/100 ml, max 6/</td>
<td>1.5</td>
<td>1.5</td>
<td>1.5</td>
<td>1.5</td>
</tr>
<tr>
<td>Water &amp; Sediment, % vol, max</td>
<td>0.01</td>
<td>0.01</td>
<td>0.01</td>
<td>0.01</td>
</tr>
<tr>
<td>Carbon Residue on 10% bottoms, % wt., max 5/</td>
<td>0.10</td>
<td>0.15</td>
<td>0.35</td>
<td>0.20</td>
</tr>
<tr>
<td>Particulate Contamination, mg/L, max</td>
<td>10</td>
<td>10</td>
<td>10</td>
<td>10</td>
</tr>
<tr>
<td>Cetane Number, min</td>
<td>40</td>
<td>45</td>
<td>45</td>
<td>45</td>
</tr>
<tr>
<td>Appearance, visual</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>---</td>
</tr>
<tr>
<td>Color (ASTM D 1500), max</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>---</td>
</tr>
<tr>
<td>Demulsification, min, max</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>---</td>
</tr>
</tbody>
</table>

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* Dated 15 September 1980.
** From Annex C to STANAG 2845, 21 September 1978.
*** Amendment 1 dated 22 March 1978.
**** Cancelled 23 May 1977.

1/ DF-2 intended for entry into the Central European Pipeline System shall have a minimum value of 58°F (-13°C).
2/ DF-2 destined for Europe and S. Korea shall have a maximum limit of 9°F (-13°C).
3/ DF-2 destined for Europe and S. Korea shall have a maximum limit of 0°F (-18°C).
4/ This requirement is applicable only for military bulk deliveries intended for tactical, OCONUS, or long term storage (greater than six months) applications (i.e., Army depots, etc.).
5/ See Appendix of VV-F-800C. The maximum limits do not apply for samples containing cetane improvers. In those instances, the test must be performed on the base fuel blend.
mechanisms since air \( \text{O}_2 \) is normally present at all times and temperatures. The mechanism for oxygen addition to organic compounds, particularly diolefins, appears to be well agreed upon.\(^{15-18}\) The reaction is free radical in nature with an oxygen molecule adding to the host hydrocarbon, forming a peroxy radical which can then reform to either a hydroperoxide or a dialkylperoxide. From this point, polymerization may occur, forming the gums.

B. Acid/base Reactions

1. Sulfur Compounds

Certain materials, such as the polysulfides, have been shown to have a greater effect on gum formation than either mercaptans or thiophenols.\(^{19}\) Other studies\(^{20,21}\) using various sulfur compounds in conjunction with 2,5-dimethylpyrrole (stability decreaser) showed that the synergistic properties were different than the properties of the sulfur compounds alone. For example, sulfide compounds had little effect on sediment formation, while thiols showed inhibitory effects and sulfonic acids acted to increase sediment formation.

2. Nitrogen Compounds

Organo-nitrogen compounds and their effect on fuel storage stability have been the subject of many studies.\(^{19-26}\) A large number of nitrogen compounds have been shown to have no effect or even an inhibitory effect on certain gum-forming reactions. However, 2,5 substituted pyridines and 2,5 substituted pyrroles have been shown to be quite active in sediment formation, particularly when the latter compound is placed in the fuel prior to aging the sample.

C. Metal Catalysis

Certain metals, when in solution, behave as catalysts for on-going reactions, particularly the decomposition of hydroperoxides to free radicals.
For example: \((18)\)

\[
\text{ROOH} + \text{Cu}^+ \rightarrow \text{RO}^+ + \text{Cu}^{2+} + \text{OH}^-
\]

or \((16)\)

\[
\text{ROOH} + \text{Cu}^{2+} \rightarrow \text{ROO}^+ + \text{H}^+ + \text{Cu}^{2+}
\]

Iron and nickel also act in a manner similar to that illustrated for copper above.

D. Polymerization/Condensation

There are two types of polymerization reactions: (1) addition (free radical and ionic), and (2) condensation. Addition reactions occur through coupling of complete molecules to form a larger molecule. Condensation occurs with the loss of some product, usually water.

Polymerization requires the presence of a small amount of initiator (usually a peroxide). The difference between this type of reaction and autoxidation is that the final product does not necessarily include oxygen.

Examples of a condensation reaction are that which occurs between carboxylic acids and amines to yield amides or acid/alcohol to yield esters. If both components have more than one functional group, polymerization can occur. \((27)\)

E. Biological Activity

The effect of biological growth in fuel systems can be considered as causing fuel degradation from two points of view. The first actually does not cause chemical transformation of the fuel components but as the microbes grow, they form particulate matter or a film-like colony which can cause filter and/or injector fouling. The second mode of degradation is from the acidic nature of the growth byproducts. This change in pH can affect either the container (metal solubility), thus increasing potential catalytic activity or act as an initiator for a polymerization reaction (see above). Although
many types of fungal and bacterial growth have been observed in fuel tanks and on filters, the overall most abundant is the fungus *Cladosporium resinae*. Examples of common yeasts and bacteria are *Candida* and *Pseudomonas aeruginosa*, respectively. (28–33).

III. REACTION PRODUCT COMPOSITION

One of the first postulations of a "typical" sediment molecule is shown in Figure 1. (12) Variations in this structure are also suggested by replacement of one or more of the three ring groups with other components. The ester linkage remains present in each form. Some of the products resulting from reaction of 2,5 dimethylpyrrole are given in Figure 2. (23)

![Figure 1. Typical Sediment Molecule](image1)

![Figure 2. Products from the Reaction of 2,5-Dimethylpyrrole](image2)
TABLE 3. ELEMENTAL ANALYSIS OF GUMS

<table>
<thead>
<tr>
<th>Element</th>
<th>Gums From Heating Oil(10), %</th>
<th>Gums From Gasoline(17), %</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>60-78</td>
<td>70-80</td>
</tr>
<tr>
<td>H</td>
<td>6.0-7.5</td>
<td>7-10</td>
</tr>
<tr>
<td>N</td>
<td>1.5-2.8</td>
<td>0.2</td>
</tr>
<tr>
<td>S</td>
<td>0.9-3.6</td>
<td>0.4-3</td>
</tr>
<tr>
<td>O</td>
<td>7.4-17.5</td>
<td>10-20</td>
</tr>
</tbody>
</table>

The empirical formula developed from averaging the above data is $C_{6.3}H_{7.0}NO_{1.67}$. Elemental analysis has also been performed on various gums. The results of these analyses are shown in Table 3.

The involvement of sulfur in the analysis of gum material has led to the postulation of the formula shown in Figure 3.

![Figure 3. Sediment Molecule](image)

IV. FUEL STABILIZERS/ENHANCERS

The preceding discussion has concerned itself with the degradation of neat, middle distillate fuels. The following sections will be concerned with methods by which that degradation may be controlled.

A. Antioxidants

To prevent oxidation of a fuel, it is normally considered that a "chain-breaking" molecule or free radical "scavenger" needs to be employed. Phenylene diamines or hindered phenols are most commonly used for this purpose. The reactions which occur are:(18)
B. Dispersants

Particulate material, whether derived from fuel decomposition or external sources, can agglomerate to form still larger particles, which may eventually clog filters, fuel lines, and/or injector nozzles. In order to maintain a minimum of particles of large size, surfactants are employed to prevent settling. Generally, nitrogen-based chemicals are the better materials of choice. Oleoylamide is a good example. Salts of alkylbenzenesulfonic acid may also be used. To be effective, the amount of dispersant used must be in excess of that required; otherwise, it will be depleted and settling of particulates will reoccur.

C. Metal Deactivators

Elimination of the catalytic effect of dissolved metals may be effectively accomplished through addition of chelating materials to the fuel. A common chelating agent is N,N'-disalicylidene propylenediamine.

D. Sweetening

Product quality in general may be improved by treatment of the original crudes prior to further processing. For many years, a sulfuric acid wash
followed by aqueous alkali and addition of "sweetening" agents have been used. Four sweetening processes are commonly used.

1. **Doctor process**: Requires treatment with sodium plumbite.
2. **Lead sulfide process**: Involves a reaction similar to the doctor process, using lead sulfide instead.
3. **Copper sweetening**: Involves treatment with cupric chloride.

In the above processes, the major reaction is conversion of thiols to disulfides.

4. **Merox process**: The newest of these processes, this process follows use of a chelate catalyst(4) which allows this process to be used either as a sweetening agent or solvent extraction process.

V. METHODS USED TO STUDY FUEL DECOMPOSITION MECHANISMS

A. **Accelerated Decomposition**

Many methods have been used to accelerate or "test" the ability of any given fuel to degrade. It is normally assumed that the prevailing mechanism is that of oxidation. The following procedures are most commonly employed.(29)

1. Storage at 43.3°C
2. Storage at 80°C
3. Storage at 150°C
4. ASTM D 873 (modified)
5. ASTM D 2274 (modified)
6. ASTM D 3241 (modified)

The first three procedures may be carried out for any time duration of interest; normally, the higher the temperature, the shorter the time of experiment, e.g., 43.3°C maximum 6 months to 1 year, 150°C usually <10 hours. The containers, normally borosilicate glass, may be vented to the atmosphere, or sealed under nitrogen or any other gas as per study emphasis.
ASTM D 873 (modified) requires a volume of filtered fuel to be aged at 100°C for 16 hours under 100 psi oxygen.

ASTM D 2274 (modified) requires a volume of filtered fuel to be aged at 95°C for 16 hours at ambient pressure with oxygen continuously bubbled through the sample.

ASTM D 3241 (modified), also known as the jet fuel thermal oxidation test (JFTOT), requires a volume of filtered fuel to be pumped past an electrically heated metal tube, then through a 17-μm filter across which the pressure drop is measured.

In each of the above methods, "insoluble" particulates are measured by passing an aliquot of the aged fuel through a preweighed filter. The modification noted generally indicates a change in nominal pore size of this filter; usually 0.8 μm rather than the specified 1.2 μm. Adherent gums are dissolved in toluene-acetone-methanol (TAM) after a prewash with heptane. The TAM solution is then placed in a preweighed beaker and the solvent evaporated. Soluble gums may be determined by the steam jet or air jet process.

B. Correlation Between Acceleration Techniques

1. Experimental Results

The techniques listed above, plus storage at 93.3°C in a soft glass container and a chromic acid oxidation test, were employed in an extensive matrix involving six test fuels. Each fuel was evaluated for particulates (adherent and suspended), steam jet gum, color, light absorbance and acid number.

The Pearson Product-Moment correlation coefficient was calculated with major emphasis on total insolubles, steam jet gum, and total gum. The overall conclusion reached in this study was that the 43.3°C storage data did not follow a trend predictable by any one of the other test methods. However,
the 80°C test appeared to yield the closest prediction. Reference 19 also details the results of an extensive correlation test matrix with the conclusion that significant correlations are minimal.

There are several possible reasons for this. First, different reaction mechanisms may predominate at different temperatures, particularly above 160° to 180°F (71° to 82°C). Also, as temperature increases, thermal degradation, rather than oxidation, becomes the predominant mechanism. Secondly, filtration and comparison of material retained on the filter presuppose that particle size is reproducible from one experiment to the next; however, there are no published studies yielding information on this point. Other possible reasons for the variability may be sample volume (surface to volume ratio), type of filter used, flow rate through the filter, methods of drying the filters, temperature required for sample workup (particularly steam jet gum), etc. Many of these variables could be eliminated by procedural change and subsequent verification of the resulting experiments. It is obvious that, at this time, each of the tests listed must be interpreted on an individual basis, with the results perhaps being valid within a range rather than as a specific number.

2. The Arrhenius Equation

Attempts to predict correlative temperatures may also be undertaken in a theoretical sense. The equation needed for this prediction is based on the one developed by Arrhenius; \( k = Z e \left( - \frac{E_a}{RT} \right) \) where \( E_a \) is the energy of activation, \( R \) is the gas constant, and \( T \) is the temperature. Including a pressure correction term (for accelerated aging due to increased ambient pressure such as in a bomb) and assuming that \( k \) is proportional to \( \frac{1}{t} \) and integrating, the equation becomes(19)

\[
\ln \left( \frac{t_1}{t_2} \right) = A \left( \frac{1}{T_1} - \frac{1}{T_2} \right) + B \ln \frac{P_1}{P_2}
\]

The term \( A \) equals \( E_a/R \) where \( R = 1.9869 \text{ cal}(15°)/°C \text{ mole} \), and \( t \) is the time required for a specific amount of material to be formed. By rearrangement, the equation may also be used to predict amounts of material that will be formed in a given time and assuming that \( P_1/P_2 = 1 \):(21)
\[ \ln \frac{M_2}{M_1} = A\left(\frac{1}{T_2} - \frac{1}{T_1}\right) \]

where \( M \) is the amount of material formed.

Several values have also been suggested for the energy of activation.

<table>
<thead>
<tr>
<th>Type of Fuel</th>
<th>( \frac{E_a}{\text{mole}} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>Jet fuels</td>
<td>21.0(19), 10.0(34)</td>
</tr>
<tr>
<td>#2 Diesel</td>
<td>10.7(21)</td>
</tr>
<tr>
<td>JP-8</td>
<td>14.4(21)</td>
</tr>
</tbody>
</table>

VI. REFERENCES


VII. ANNOTATED BIBLIOGRAPHY


This paper is a summary of the work on physical and related properties of three military-type fuels derived from shale (JP-5, JP-8, and DFM).


Liquid hydrocarbon fuels derived from coal and from oil shale were made available for characterization and thermal and storage stability investigations. The shale-derived fuels were refined to meet the requirements of military specification jet fuels JP-8 and JP-5 and marine diesel fuel (DFM). Virtually all the specification requirements were met, and the fuels were found to be thermally and storage stable. During the 32-week, 43°C storage tests conducted on these liquids, periodic measurements were made of precipitate fuel-insolubles, fuel-soluble gum, dissolved oxygen content and peroxide number.


Shale-derived JP-5, JP-8, aviation turbine fuels and marine diesel fuel were analyzed for compliance with military specifications and evaluated for storage stability, corrosion tendencies, additive response, compatibility with petroleum fuels, and microbiological growth susceptibility.

The purpose of this program was to evaluate these products for specification requirements and other properties not necessarily defined by specification testing, and to ascertain their performance in Army engine systems as part of the overall program to develop a capability for consuming multisource fuels within the Department of Defense.


Synfuels which are currently available have demonstrated degradation problems that were predicted from work with petroleum. This paper presents basic and applied data which should aid in the tradeoff decisions between further costly processing and product stability. Various degradation mechanisms were discussed.


The objectives of the current work are to determine the storage characteristics of representative liquid fuels derived from coal, oil shale, and tar sands, to analyze representative gasolines, diesel fuels and jet fuels for hydrocarbons and nonhydrocarbons that are known precursors of deterioration products, and to provide information that will guide refiners in selecting processes to minimize fuel deterioration through removal or inhibition of reactive components.


The purpose of this research contract is to develop additional data,
methodology, computer software, and mass spectrometer facilities needed to provide quantitative analyses of these fuels.


This investigation was initiated to ascertain the variables influencing the storage stability of jet fuels and to determine the simplest and most practical methods of controlling these variables for a resultant increase in the permissible storage life of the fuels.


The title subject was investigated using soft glass bottles. JFTOT was used for deposit formation.


The storage stability characteristics of a diesel-type fuel were determined to evaluate the effect of the polar compounds in the fuel.


Syncrudes, particularly from shale rock, contain much higher N levels than petroleum materials. This work was to determine the magnitude of the N-caused sediment problem with respect to compound types, effects of light, investigate structure, and mechanism of formation of the sediment.

Syncrudes, particularly from shale rock, contain much higher N levels than petroleum materials. This work was to determine the magnitude of the N-caused sediment problem with respect to compound types, effects of light, and to investigate structure and mechanism of formation of the sediment.


The primary objective of this program was to evaluate the potential of additives for improving the low-temperature flow properties of synthetic diesel fuels derived from oil shale.


A report on the first year of a multi-year program. JP-8, n-decane, and No. 2 diesel fuel were used at 110°F and 150°F in the dark. Summary only. c.f. Frankenfeld & Taylor, First Annual Report 1980.


This report was prepared by Exxon Research and Engineering Company, Linden, NJ. A variety of organic nitrogen compounds were evaluated for their tendencies to promote sediment in hydrocarbon fuels under accelerated storage conditions. The most deleterious species to fuel stability were found to be alkylated heterocyclic nitrogen compounds. The most reactive compounds were those with two or more alkyl groups, at least one of which was situated on a carbon adjacent to the nitrogen.


Experiments started in 1969 to ascertain the storage stability of the new MIL-F-24397 Navy distillate fuel have been completed. Long-term experiments simulating actual storage at ambient temperatures and accelerated tests for predictive purposes were run.


JP-5, the Navy jet fuel, must meet many stringent requirements if satisfactory performance in aircraft and fuel-handling and storage systems is to be attained. In considering JP-5 derived from alternate fossil fuels, several properties are affected more by the chemical characteristics of the fuel than by the physical properties, and explanation of these properties are covered in this publication.


ASTM D 1563 is studied with respect to repeatability and ability to distinguish between samples.


Determined the title study with oxygen concs. <3 ppm up to saturation of approximately 300 ppm. In <3 ppm, no effect was noted; in 3-10 ppm, rapid decrease in thermal stability moderating up to 15 ppm and little response beyond that.

Fuel thermal stability has become a fuel property of increasing importance as engine designers have turned to the problems of the SST and higher Mach number aircraft.


The following topics were discussed: U.S. energy consumption, liquid fuels' demand and sources of supply, alternative sources of liquid fuels, effects of diesel vehicles on fuels' demand and quality, and effects of electric-powered vehicles on fuels' demand.


Current chemical technology including descriptions of methods for processing synthetic fuel sources.


A slow-scanning Fourier infrared Michelson interferometer was converted into an emission microspectrophotometer by replacement of the commercial source with a reflecting microscope objective (36X) focused on an adsorbate on a metal surface maintained at a temperature somewhat higher than that of the detector and with appropriate optical interfaces.

Fuel-oriented problems occurring in the field prompted an investigation to examine the thermal-oxidation characteristics of diesel fuels.


The storage stability characteristics of diesel fuels meeting the requirements of Federal Specification VV-F-800a (Fuel Oil, Diesel) were investigated by conducting a 2-year storage program involving four different diesel fuels.


This task summarizes the effect of some projected potential changes in JP-5 composition/properties and processing that may have an important impact on fuel technical problem areas.


The objective of this work is to obtain information on the mechanism of deposit and sediment formation from jet turbine fuels by looking for development, during fuel oxidations, of precursors of decreasing solubilities, and increasing oxygen contents and molecular weights. See SRI International Notebook for Monthly Progress Reports.

The objective of this work is to obtain information on the mechanism of deposit and sediment formation from jet turbine fuels by looking for development, during fuel oxidations, of precursors of decreasing solubilities, and increasing oxygen contents and molecular weights.


The object of this work is to determine the mechanism by which gum and deposits are formed in diesel fuels, and thus how to predict and prevent their formation.


An extensive review of the subject covering virtually all aspects.


Compilation of various articles by different authors relating to jet fuel stability, composition, and properties.


Origin and early use of the word "petroleum" is explained.


This seminar was held with the objective of helping to clarify the situation relating to the supply and demand outlook for mobility fuels and related influential factors.


Some aspects of stability of a jet fuel prepared from Paraho-I shale oil are reported.


Current state of diesel fuel and what the industry is doing about it is discussed.


N. Por's publication covers (a) study on subject stabilities in general and their behavior during long-term storage in particular; (b) an investigation on the effect of various storage and environmental conditions on stabilities of liquid fuels, (c) definition of mechanisms governing breakdown processes; and (d) inclusion of products of wide ranges of compositions.

Reproducibilities and significance of the ASTM D 2274 test for oxidation stabilities of diesel fuels are studied.


Six distillate diesel fuels have been stored at Rosyth for six years, and their behaviour studied both in storage and in laboratory tests.


FCC is discussed relative to stability of gasolines. Mechanism of gum formation is discussed. Types of inhibitors are also discussed. c.f. Inhibiting Gum Formation in Modern Gasolines, 1980 UOP Process Division Technology Conference, April-September 1980.


A synthetic fuels industry could start contributing to our energy supply within a decade and could assume significant proportions by the end of the century.


This paper describes the chemical characterization of JP-5 and DFM from the Shale-II project.

General information is given with regard to fuel specifications, gum formation and structure, types of inhibitors, etc.


This report represents an "overview" as presented at a seminar sponsored by ARO in April 1977 to promote basic research in the area of "Diesel Fuel Stability". Generally covers fuel deterioration primarily with diesel or distillate fuels used in compression ignition engines which power Army tactical vehicles. Selected field problems outlined.


This paper describes the results of a program to develop laboratory volatility test methods and to correlate volatility data with engine wear performance data using lubricants which have shown volatility-related performance.


This report covers a program initiated to review and selectively evaluate laboratory test techniques in an effort to develop and/or optimize accelerated stability test techniques for diesel fuels.


The purpose of this 2-year project was to study the formation of dele-
terious products in middle distillate fuels; and apply the results toward development of more reliable test methods for fuel stability. During first year, eight accelerated stability tests were chosen and evaluated using a set of 6 test fuels. During the second year, primary emphasis was placed on testing 43°C and 150°C tests which are being cooperatively developed by ASTM. In general, test results at 43°C, 80°C, 150°C, and ASTM D 2274 provided directional agreement. In an effort to standardize the 43°C and 150°C tests, planning and cooperative testing was provided to ASTM Committee D-2, Technical Division E-V.


This report reviews fuel deterioration. Primary emphasis is placed on diesel fuels used in compression ignition engines of the Army tactical and combat vehicles. Field problems regarding fuel stability and related problems are outlined. Information places in perspective the background which has prompted current research activities to detect, predict, and prevent fuel stability-associated equipment failures. Army's needs for diesel fuel stability and cleanliness are identified.


Initial results are reported from studies designed to elucidate the variables which control the rate of deposit formation from hydrocarbon jet fuels exposed to high temperature stress.


This report covers the results of studies which were aimed at estab-
lishing more precisely the deterioration characteristics of fuels and equipment in storage and devising standby storage techniques.


Data from a modified JFTOT procedure is used to obtain carbon values which are related to fuel type, anti-oxidant, fuel end point.


A laboratory test rig has shown more highly refined fuels to have poorer lubricity. A 10 to 20 percent blend of conventional treated fuel improves lubricity to normal. Addition of highly polar compounds from conventionally treated fuels significantly improves lubricity.


The most important turbine oil quality index, which determines the service life of the oil, is the oil's resistance to effects from atmospheric oxygen at elevated temperatures, is discussed.


IR spectra of fuels and deposits were obtained for heating oil and Jet A fuels.

35

Program objective was to demonstrate that shale oil could be converted into stable, specification military fuels utilizing conventional refining technology and in sufficient volumes to support an extensive engine testing program.


This report details the development of a portable device to measure the cleanliness and stability of diesel fuels in both bulk and vehicle fuel cell storage.


The influence of substituted quinolines, pyrroles, indoles, and pyridines on deposit formation in a diesel fuel is evaluated.


The influence of substituted pyridines, pyrroles, indoles, and quinolines on storage stability of conventional Jet A turbine fuel is evaluated.

60. Zamulinskii, I.M., "Distribution of Total and Basic Nitrogen in Crude

How the contents of total and basic nitrogen in straight-run petroleum cuts are related to the average boiling point of the cut is presented.
GLOSSARY OF TERMS

1. **Additive** - A chemical agent employed to assure the presence of certain desirable characteristics in fuels or other materials.
2. **Adherent gum** - See insoluble gum.
3. **Barrel** - A unit of measure fixed for certain commodities (42 gallons for petroleum).
4. **Bituminous** - Resembling or containing various mixtures of hydrocarbons (a tar) often together with their nonmetallic derivative.
5. **Break point** - 1) The time at which the rate of formation of insoluble particulates significantly increases compared to the induction period. 2) The time at which the weight of filterable particulates exceeds a specified value by 20 mg/100 mL.
6. **Crude** - Unrefined petroleum.
7. **Diesel fuel** - A hydrocarbon fuel used in diesel engines.
8. **Distillate fuel** - A fuel obtained by distillation of crude materials into fractions according to temperature.
9. **Existent gum** - The evaporation residue of aircraft fuel or heptane-insoluble portion of the evaporation residue of motor gasoline.
10. **Free radical** - An atom or group of atoms possessing an unpaired electron normally formed by cleavage of the parent molecule.
12. **Insoluble gum** - Deposit adhering to the sample container after removal of the aged fuel.
13. **Petroleum** - A complex oily, flammable liquid mixture composed of hydrocarbons and small amounts of other substances which may vary from almost colorless to black.
14. **Potential Gum** - The sum of soluble and insoluble gum.
15. **Precipitate** - Sediment and suspended material with aged fuel.
16. **Sediment** - Foreign particulate matter which settles to the bottom of a storage container.
17. **Soluble gum** - Deterioration products existing in solution in the aged fuel and the benzene-acetone soluble portion of the deposits on the container wall. Determined by air jet gum method.
18. **Storage Stability** - Resistance of a fuel to spontaneous formation of deleterious products when stored over long periods of time.

19. **Tar** - A dark odorous, viscous liquid obtained by destructive distillation of organic material.

20. **Thermal stability** - Relating to the tendency to form deposits at high temperature.

21. **Total potential residue** - Sum of the potential gum and precipitate.

22. **Unwashed gum** - The evaporation residue of motor gasoline consisting of existent gum and non-volatile additive components.
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