PANEL DISCUSSION

Introduction

The following includes prepared comments by participants of the panel discussion held at the Symposium on Distillate Fuel Contamination, Cincinnatti, OH, on 24–25 June 1987. The topics discussed by each author originated from questions asked during the panel discussion concerning trends on additives, testing research, user needs, and microbiological outlooks.

Future Distillate Fuel Properties and Problems

By Robert N. Hazlett[1]

I was asked by the Chairman to discuss the future. That is a rather broad but nebulous task so I have decided to limit my comments to future properties and problems of distillate fuels.

I will consider two time periods, 5 years and 5 squared, or 25 years. For the longer time frame, I predict we will have two major worldwide upsets in crude oil supply. I expect to see substantial use of alternate fuels both from nonpetroleum fossil energy sources and biological sources. The fuels around the year 2010 will have higher viscosities and densities. Consequently, we will have problems removing water and other contaminants. The recent progress in control of microbiological growth with additives should reach an advanced state in 25 years. The stability of fuels will degrade as poorer quality petroleum crudes and alternate sources are used to produce distillate product. However, our improved knowledge on the chemistry of instability and on the mechanisms of additive action should enable us to resolve most problems. If anyone encounters me 25 years from now you can tell me how bad these predictions were.

Predictions for the near future (four to six years) may be of more interest, and I will spend more time on the short range. I expect the fuel situation will be fairly settled. Alternate fuels will contribute little to our overall energy picture. Crude quality will exhibit a modest decrease. The property trends observed for No. 2D diesel fuel over the past 20 years will continue. To review, some changes were as follows:

- Cetane number: 50 to 45.
- Kinematic viscosity: 2.6 to 2.9.
- Distillation end Point: 610 to 642°F (321 to 339°C).
- Distillation 90% Point: 565 to 600°F (296 to 316°C).
- Distillation 10% Point: no change at 430°F (221°C).

Other probable changes in properties and composition will be increases in density, aromatics, and polar compounds. The storage stability of distillate fuel will continue to decline.

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These changes will arise from two major factors: deterioration of crude quality and the incorporation of greater amounts of light cycle oil (LCO) into middle distillates. Requirements for cetane number and stability may limit the amount of LCO that can be blended into diesel fuels. However, if these properties can be relaxed or managed by other means, such as additives, cleanup systems will be severely tested. Water and sediment will settle slower in blends high in LCO. Increases in density, viscosity, and polar compounds will make centrifuges, coalescers, and filters less effective and mandate more frequent servicing.

Microbiological contamination problems could become more severe if water removal is impaired. We do have hope of controlling this problem, however, if we make more widespread use of well-known housekeeping techniques and use biocides wisely.

In summary, I would suggest three implications for this symposium:

1. Explore new ideas for fuel cleanup.
2. Define the capability of additives, including interactions, for controlling key properties, such as stability, cetane number, and microbiological growth.
3. Determine the degree of hydrotreatment needed to reduce key polar compounds and improve stability.

Fuel Stability and Cleanliness Research: Where Do We Go From Here?

By Dennis W. Brinkman[2]

I believe the time has come to focus our research efforts related to fuel stability and cleanliness. Active research has been going on in this area for well over 30 years. We have been working on it at my facility for over 40 years.

However, I believe the work described in this symposium and in the current literature implies that the quality and sophistication of our analytical techniques are now sufficient that we can start to provide some hard answers to hard questions. We are beyond the point where we either take pure compounds and measure their reactivity or take actual fuel samples and determine their bulk properties.

One of the first things we can do is to sort out the problems from the complaints. Our new experience is that about half the fuel complaints really are handling or testing problems, rather than fuel problems. Once we have identified fuel problems, then we need to apply the advanced analytical capabilities we have heard described here to quickly and efficiently pinpoint the sources of the problems. I believe we are now at the point where we can become as specific as the situation calls for, right down to identifying compound types initiating the problem. That is something we currently do on a regular basis for various clients.

This does not mean further research is not required. It is a continuing struggle to convince some people that the quality of current commercial fuels is a concern, even as we document specific incidents. A recent report acknowledged that the only problem it could see as serious was that a certain fuel could not regularly pass the oxidative stability test. Thus, their solution was to eliminate that test for the specification! Are we really that naive?

I believe there are three objectives that we should have as we move on from this meeting to further the research in fuel stability and cleanliness:

1. Develop quick protocols for identifying problem fuels, separating them from complaints that may have nothing to do with the fuels.
2. Enhance the analytical tools necessary to pinpoint the cause of the problems, so that we can work on technically sound solutions.

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3. Formulate processing, additive, or other solutions that are based on the understanding provided by the detailed analyses now available.

If we build on what we learn, and take advantage of the advances, the next time we get together we will be able to talk about important advances toward maintaining a high-quality reliable fuel supply.

Trends in Middle Distillate Stability

By Cyrus P. Henry[3]

There are a number of factors that tend to affect the stability properties of middle distillate fuels; some of these are important across the breadth of the industry; others significantly affect properties for product produced at individual refineries (Table 1).

There is a strong trend, worldwide, toward cracking of residual components to make transportation fuels. These cracking processes in general give a product with poor stability but can be readily improved by hydrogenation. When satisfactorily hydrogenated, these materials are quite stable, and their use results in no loss in pool stability. Stability properties are very poor with no hydrogen treating; hydrogen treating to reduce sulfur content may give product with marginal stability properties.

Relatively speaking, crude oil is in abundant supply at this time, providing refiners an opportunity to choose crude oils that match their process capabilities. This has resulted in some instances in improved stability relative to several years ago. Long term, however, that trend will reverse again and likely give some loss in pool stability properties or a spot basis for individual refiners. Good crude availability has also improved overall cetane quality.

The refining industry profit margin has been small in recent years; this has in general resulted in greater awareness of quality giveaway. On the other hand, there has been a resurgence in interest in stability as evidenced by recent symposiums both nationally and internationally; refiners are more aware and capable of maintaining satisfactory quality without excessive stabilization.

Additive suppliers continue to improve the quality of their products. Most of these improvements, however, have been incremental, and there have been no quantum leaps in benefits achievable with stabilizing additives. It is likely that we can look forward to further improvements in the future.

Importation of finished products from abroad continues to increase; the implications for stability properties are not clear.

The significance of all the above, however, may be small compared to the affect from mandated sulfur restrictions in the future. These may come about to meet vehicular emission requirements, to provide "smog-free air," or to reduce acid rain. There are already sulfur restrictions in place in most of the United States, and these have resulted in hydrogen treating to reduce sulfur levels in many products with a significant improvement in stability. New restrictions now being discussed may lead to further hydrogen treatment and further improvement in stability.

In general, refiners continue to produce a good-quality product with satisfactory stability and other properties for normal use. Normal competitive pressures will continue that trend. Those who store fuel for prolonged periods (greater than one year) should do so with special care in procurement, monitoring, and possibly use supplemental additive treatment of fuels. Finally, there are considerable socioeconomic pressures at work that require political so-

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TABLE 1—*Trends in middle distillate stability.*

Factor	Effect on Stability	
	Recent Trend	Long Term
Resid cracking	− or 0	− or 0
Crude availability	+	−
Reduced quality giveaway	−	− or 0
Information and communications	+	+
Improved additives	+	+
Increased product imports	?	?
Sulfur restrictions	+	+ +
Prognosis		
continued good performance in "normal" use		
special care for long-term storage		
significant socioeconomic factors		

lutions. A near-term shortage of crude oil is more likely to be politically induced than by the naural laws of supply and demand. Stability improvement or reduction in sulfur content by hydrogen treating requires the use of some of the energy in the crude oil. The balance between these factors is decided by society at large rather than by those of us here.

Diesel Fuel System Contamination: A User's Look to the Future

L. L. Stavinoha[4]

As the user becomes more aware of the effects of fuel stability and cleanliness properties related to equipment operation and maintenance costs, a greater demand for test methods to monitor these fuel properties may evolve [*1*].

This presentation emphasizes the need for the user to control the origin and handling of diesel fuel for problem-free application in end-use vehicles. Diesel fuel control (Table 2) at the refinery is based upon fuel specifications for physical and chemical properties. In the distribution system, the control is the responsibility of the custodian who places certain surveillance requirements on the product. In user's storage, the user must provide his own surveillance for quality control. In the user's vehicle, experience becomes very important in knowing the impact of operation and fuel quality/contamination on system reliability and performance.

Some possible general diesel fuel property trends affecting future quality are suggested in Table 3. Higher density of diesel fuel is being impacted by the use of higher density cracked stocks. Lower sulfur in the future may come about by the Environmental Protection Agency (EPA) mandate requiring very low (0.05 maximum) percent by weight sulfur contents [*2*]. Lower cloud point could result from refinery processing to reduce sulfur through more use of hydrodesulfurization. Higher olefin, phenol, and thiophenol concentrations in diesel fuel result from the use of untreated light-cycle oil from fluid cat cracking [*3*]. Lower biocidal resistance could be the result of refinery processing to reduce sulfur resulting in reduced naturally occurring biocides.

Table 4 is a summary of properties and specification limits for No. 2 diesel fuels from two specifications, that of ASTM Practice for Preparing Test Specimens from Basic Refractory Ramming Products by Pressing (D 975), Grade No. 2D and VV-F-800D, Grade

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TABLE 2—*Diesel fuel control.*

Location	Control
Refinery	Fuel specification
↓	
Distribution system	Custodian surveillance
↓	
User storage	User surveillance
↓	
User vehicle	User experience

DF-2[*4–5*]. Specification limits for CONUS (Continental United States) and OCONUS (outside Continental United States) procurement are provided for in VV-F-800D. Important properties to the user include cloud point, which is a local requirement as is pour point that may not be required; kinematic viscosity at 40°C, which impacts low-temperature operability/startability; and the distillation, which is controlled at the 90% point and the end point in the case of VV-F-800. Carbon residue on 10% bottoms is controlled by both specifications. Neutralization number, or total acid number, is controlled by VV-F-800 for OCONUS but not in the other specifications. Particulate contamination, crucial to good filter operation, is not controlled in ASTM D 975. A more stringent requirement for water and sediment is placed on VV-F-800 through the use of clear and bright as opposed to a 0.05 value for water and sediment in D 975; this limit can be a very high number in terms of filter plugging if the 0.05% existed as sediment and microparticulates.

Table 5 summarizes fuel stability and cleanliness properties and their effects. The results of instability (thermal/oxidative) are sediment that can plug filters. Gum can potentially foul injectors. The method used for determining gum in diesel fuels is ASTM Test for Existent Gum in Fuels by Jet Evaporation (D 381) (steam jet method). Both gum and unfiltered particulates are thought to contribute to engine combustion chamber deposits. Acid is related to corrosion of containers, pumps, and possibly injector nozzles. Additionally, it is thought that acids are related to accelerated fuel deterioration in much the same manner as certain metals (for example, copper) act as catalysts [*6*]. While color of a fuel can indicate chemical deterioration of a fuel, it is not directly harmful to the handling or use of diesel fuel. Sulfur fuel components may form corrosive elemental sulfur or acids during storage [*7*]. If a fuel forms water haze rather than shedding water, injectors or injector pump wear corrosion, or both, may result. Microbiological contamination and growth at fuel-water interfaces can rapidly plug filters. Microbiological growth produces acids through metabolism of fuel, and thus, both decreases fuel stability and increases the corrosivity of water bottoms.

TABLE 3—*General diesel fuel property trends.*[a]

Property Trend	Reason for Change
Higher density	Higher density cracked stocks
Lower sulfur	EPA mandated
Lower cloud point	Refinery processing to reduce sulfur
Higher olefin, phenol, and thiophenol concentration	Use of untreated light cycle oil from fluid catalytic cracking
Lower biocidal resistance	Refinery processing to reduce sulfur

[a] Crude oil and refinery dependent.

TABLE 4—*Summary of properties and specification limits for No. 2 diesel fuels.*

Properties	ASTM Method Number	Specification Values: ASTM D 975 Grade No. 2D	Specification Values: VV-F-800D Grade DF-2: Conus	Specification Values: VV-F-800D Grade DF-2: Oconus
Density, kg/L at 15°C	D 1298	NR	Report	0.800 to 0.860
Flash point, °C min	D 93	52	52	56
Cloud point, °C max	D 2500	Local	Local	Local
Pour point, °C max	D 97	NR	Report	Local
Kinematic viscosity at 40°C (20°C), cSt	D 445	1.9 to 4.1	1.9 to 4.1	1.8 to 9.5
Distillation, °C	D 86			
50% evaporated		NR	Report	Report
90% evaporated, max		338	338	357
end point, max		NR	370	370
residue, vol%, max		NR	3	3
Carbon residue on 10% bottoms, mass%, max	D 524	0.35	0.35	0.20
Sulfur, mass%, max	D 1552 or D 2622	0.50	0.50	0.30
Copper strip corrosion, 3 hr at 50°C, max rating	D 130	3	3	1
Ash, mass%, max	D 482	0.01	0.01	0.02
Accelerated stability total insolubles mg/100 mL, max	D 2274	NR	1.5	1.5
Neutralization number, tan, max	D 974	NR	NR	0.10
Particulate contamination, mg/L, max	D 2276	NR	10	10
Cetane number, min	D 613	40	40	45
Water and sediment, vol%	D 1796	0.05	NR#	NR#
	D 4176	NR	C&B	C&B

NOTE: NR = not required. NR# = shall be free from undissolved water, sediment, and suspended matter and shall be clean and bright at the ambient temperature or 70°F (21°C), whichever is higher.

TABLE 5—*Fuel stability and cleanliness properties and their effects.*

Fuel Property	Fuel Effects
Stability (thermal/oxidative)	
sediment	Plugs filters
gum	Injector fouling and combustion deposits
acid	Corrosion
color	None
Cleanliness (particulates)	Plugs filters
Sulfur stability	Corrosion
Water haze	Injector/injector pump wear
Microbiological contamination	Filter plugging, corrosion, fuel instability

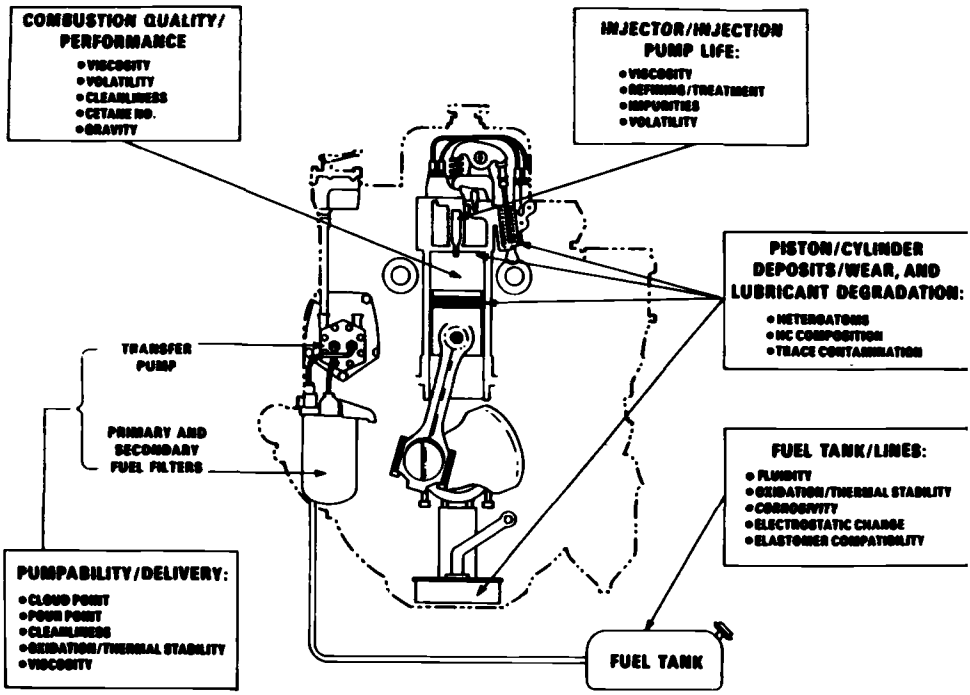

FIG. 1—*Diesel fuel property effects on vehicle engine and fuel tank.*

Figure 1 summarizes the various properties of the fuel and its impact on various areas of the engine, fuel tank, primary and secondary fuel filters, transfer pump, and injectors. Combustion quality and performance can be affected by viscosity, volatility, cleanliness, cetane number, and gravity. Injector/injection pump life is dependent upon viscosity, refinery treatment, impurities, and volatility. Piston/cylinder deposits/wear, and lubricant degradation are dependent upon heteroatoms in the fuel, hydrocarbon composition, and trace contamination. Pumpability/delivery are controlled by cloud point, pour point, which impact flow properties, cleanliness, oxidation/thermal stability, and viscosity of the fuel. Fuel tank/lines operations are dependent upon fluidity of the fuel, oxidation/thermal stability, corrosivity, electrostatic charge, and elastomer compatibility. Generally, while an engine is in operation, the fuel serves as a coolant for the injector pump system by returning heated unused fuel to the fuel tank via a return line not shown in Fig. 1. Vehicle diesel fuel systems can be particularly harsh on the fuel in that not only is it used as a coolant, but it is also exposed to air containing moisture, oxygen, and carbon dioxide caused by tank breathing. Also, the diesel fuel subjected to daily temperature extremes (including fuel tank direct sun exposure) is exposed to various metal surfaces, and generally, the fuel system receives no preventive maintenance such as fuel tank draining and cleaning. Since fuel tanks generally are not completely drained under fill/use operation, some fuel remains in the tank for extended periods and contributes to enhanced fuel degradation of new fuel/old fuel mixtures. Combined effects of poor fuel stability, continued intermittent vehicle operation, contamination, and lack of preventive maintenance can lead to severe premature fuel filter plugging [*8*].

Copper generally acts as a catalyst for fuel deterioration and is present in many vehicle systems either as pure copper or an alloy. The user installation of fuel preheaters made of

copper piping on diesel equipment to handle high cloud point fuels may relieve temporary filter plugging caused by wax separation in cold weather but can cause premature filter plugging by increasing insoluble deterioration products.

Suggested fuel specification limits for fuel stability and cleanliness properties are shown in Table 6. Also, included in Table 6 are test methods suggested for determining the fuel properties. Since some of the test methods have not yet been standardized, the status of the test methods is also given in Table 6.

Oxidative/Thermal Stability

Technical Division E (on burner, diesel, and turbine fuel oils) under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricants established Section E-V on Fuel Stability and Cleanliness in 1979. The scope of this section's activities is to prompt, oversee, and participate in the development of methods for predicting and monitoring the stability, cleanliness, and microbiological contamination of fuels under the jurisdiction of Technical Division E.

A symposium organized by Section E-V under the sponsorship of ASTM D-2 was held in June 1980 to assess the state-of-the-art and to define current and future needs for tests to predict and monitor the stability and cleanliness of distillate fuels (excluding aviation turbine fuels). Topics included appraisals of present-day distillate fuel stability and cleanliness, requirements for various applications, anticipated trends, future fuels, fuel distribution problems, microbiological contamination, test methods for stability and cleanliness,

TABLE 6—*Possible user specification limits for diesel fuel stability and cleanliness properties.*

Fuel Property	Test Method	Test Method Status	Possible Fuel Specification Limit
Stability	(1) 43°C test	ASTM standard	4 weeks; 2 mg/100 mL
storage	(2) 150°C test	ASTM cooperative development	90 min; 2 mg/100 mL or #3 pad rating
	(3) ASTM D 2274	ASTM standard	1.5 mg/100 mL
Thermal	D 3241	ASTM standard	Not defined
Acid number	(1) ASTM D 974	ASTM standard (using 40-G fuel sample)	0.15 mg KOH/g of fuel (using 40 g of fuel)
	(2) ASTM D 3242	ASTM standard	0.15 KOH/g of fuel
Cleanliness			
(particulates)	(1) D 2276	ASTM standard	10 mg/L
	(2) Filterability ratio	Not under formal development	1.3
	(3) Light transmittance	Not under formal development	10 mg/L
Soluble gum	ASTM D 381	ASTM standard	10 mg/100 mL
Sulfur stability	None defined	Not defined	1-ppm free sulfur
Water haze	(1) D 4176	ASTM standard	"Clear and bright"
	(2) Microseparator? D 3948 (modified)	Capability defined by ASTM round robin	50 mini
Microbiological contamination		ASTM task group to	
presence	None defined	write standard test	1 mg/L max
growth	None defined		Zero
fuel's ability to support growth	Innoculation	Not under formal development	Zero

and refinery processes that improve stability. Papers presented at this symposium were later published in a special technical publication (STP 751), *Distillate Fuel Stability and Cleanliness*, by ASTM [*9*].

Diesel fuel handling and storage as addressed by the user depends on guidance and experience. An appendix entitled "Long-Term Storage of Distillate Fuel" first appeared in the 1981 version of the ASTM D 975 specification for diesel fuel [*4*]. This appendix attempts to provide guidance for consumers of distillate fuels who may wish to store quantities of fuels for extended periods. Fuel selection, storage conditions, and monitoring of properties before and during storage are addressed.

A considerable amount of work has been performed in developing tests that predict the storage stability of distillate fuels. A large variety of predictive tests, referred to as accelerated aging tests, has been used or is in use for evaluating fuel stability. In a 1958 presentation by MacDonald and Jones, 26 different test methods were tabulated [*10*]. While time and temperature are accepted variables in accelerated tests, the range in containers, conditions, and especially in the method of sample evaluation, causes tremendous variation in results.

The Navy-Coordinating Research Council Barge Storage Program [*11*], conducted in the 1950s to determine the scale-down factor from barge to bottle or drum storage, provided data indicating the usefulness of storage at 43°C as a predictive method for long-term storage. Bottle storage at 43°C for 13 weeks is reported to be approximately equivalent to either drum or bottle storage at ambient (18 to 24°C) temperatures for one year [*12–15*].

In a Department of Energy (DOE) sponsored program, a literature search was conducted to provide a list of stability test techniques and their interpretations that could be used in a correlative middle distillate fuel stability test program [*16*]. A large number of test techniques from numerous sources (approximately 116) were reviewed. The 43°C storage test method used in this program represented a generalized method incorporating a cross-sectional selection of details found in six different 43°C tests. This method formed the basis for more extensive cooperative testing by ASTM to develop a 43°C standard test, ASTM Test Method for Distillate Fuel Storage Stability at 43°C (100°F) (D 4625).

While most industry experts agree that the ambient storage stability of distillate fuels can by adequately assessed using 43°C storage test methods, these methods are not satisfactory for quality control or surveillance, since 18 weeks may be required to complete the test. A great variety of "quality control" tests (that is, tests that can be completed in less than one or two days) are in use, including ASTM Test Method for Oxidation Stability of Distillate Fuel Oil (Accelerated Method) (D 2274). None of these tests are fully accepted within the petroleum industry because the fuel stability is not predicted with complete reliability. This lack of reliability is due to inherent limitations in accelerating fuel degradation, complicated by variation in the nature of the degradation process. Those who monitor fuel stability properties choose or develop a test that serves their purposes and gradually accumulate interpretative skills and correlations with use experience; their test serves them well under normal conditions. When problems arise, most experts will utilize a variety of tests with varying severity and ultimately correlate results with field experience or 43°C storage tests. There appears to be merit in standardizing some of the alternative quality-control tests for this and other purposes. The duPont version of the 150°C test [*17*] has been used to form the basis for developing a rapid ASTM standard method.

In Table 6, storage stability can be tested using either 43 or 150°C stability tests. The 43°C test is an ASTM standard. Suggested specification limit at four weeks is 2 mg of total insolubles per 100 mL. The 150°C test is under ASTM cooperative development. Suggested possible specification limit at 90 min would be a filterable insoluble limit of 2 mg/100 mL or a No. 3 (visual) pad rating. A third test, ASTM D 2274, is an ASTM standard. Most military and federal specifications limit this value in the specification to 1.5 mg per 100 mL.

Thermal stability could be controlled through ASTM Test Method for Thermal Oxidation Stability of Aviation Turbine Fuels (JFTOT Procedure D 3241) normally used for jet fuels. However, the possible fuel specification limit has yet to be defined for diesel fuels.

Acid Number

Acid number can be tested through ASTM D 974, which is an ASTM standard using 40 g of fuel for sample. A 0.15 mg of potassium hydroxide (KOH) per gram of fuel limit is recommended for consideration. Another method, ASTM Test Method for Total Acidity in Aviation Turbine Fuel (D 3242,) is an ASTM standard, and the same limit of 0.15 mg KOH per gram of fuel should be considered.

Cleanliness (Particulates)

Essentially eight types of materials can be found as particulates in diesel fuel. These types are shown in Table 7 with some typical examples.

ASTM Test Methods for Particulate Contaminant in Aviation Turbine Fuels (D 2276) can be used for diesel fuels. This method uses a 0.8-μm porous membrane filter. Fuel filters have a nominal pore size of 5 to 10 μm. In order to evaluate actual fuel filter loading ability, the Society of Automotive Engineers (SAE) J 905 test method [*18*] should be employed and then related to D 2276 values to establish tolerable limits. The limit of 10 mg/L suggested in Table 6 is based on the limit in the diesel fuel Federal Specification VV-F-800D.

Another test used for determining fuel cleanliness is the "Filterability Ratio Test" [*8*]. In this test, the time required to filter a given amount of contaminated fuel is compared with the filtering time for an equal portion of the same fuel that has been prefiltered. A value of 1.0 for the test indicates the fuel is clean. A value of 1.5 has been suggested as a "go/no-go" limit. Because of the unique characteristics of different types of particulates in diesel fuel and their ability to plug filters, the "Filterability Ratio Test" may be a more realistic test than the D 2276 test, which measures the weight of particulate material rather than its ability to plug a filter. The suggested filterability ratio limit of 1.3 in Table 6 was based on a value of 1.0 for clean, and a "no-go" value of 1.5 for a dirty fuel and was not correlated to actual fuel filters. Part of an actual fuel filter element could be used as the filter in this test to make it even more realistic.

The light transmittance test for particulates is actually a modified D 2276 test in that light transmittance through the test filter is used as a measure of particulate contamination rather than using a gravimetric weighing procedure. The contaminated filter measuring device for

TABLE 7—*Particulates (or Debris) in fuels.*

Type or Category	Examples
1. Inorganic compounds	Dirt, dust, sand
2. Fuel filter media	Paper, cotton
3. Metals	Fuel pump wear particles; vehicle and bulk fuel tank corrosion debris; corrosion debris from filler pipe screens, walls, and caps
4. Fuel container surfaces	Resins, fiberglass, high-density polypropylene, polymers/elastomers, and polyurethane foams
5. Fungus	*Cladosporium resinae*
6. Yeast	*Candida humicola*
7. Bacteria	*Pseudomonae*
8. Organic compounds	Fuel-derived sediment and/or gum

this method is described in a recent publication [*19*]. The major advantage of this test method is speed and simplicity, which makes it useful as a field test monitor. This approach could be used for product acceptance and monitoring of fuel in bulk storage tanks as well as vehicle fuel tanks.

Sulfur Compound Stability

If elemental sulfur and sulfur-containing acids are formed after a fuel is transported from the refinery or is delivered to the user, mild steel and copper corrosion inhibition may not be present. Limited evidence suggests that some fuels that show adequate corrosion inhibition at the time of refining, subsequently form small quantities (15 to 30 ppm) of elemental sulfur. Probable source of elemental sulfur is oxidation of hydrogen sulfide H_2S formed by sulfur-reducing bacteria in distribution systems, especially piping. Corrosion inhibition for this sulfur is not provided for at the refinery. Such may have been the case with Paraho-II shale fuels refined for a recent military fuel program. Model studies showed that the combined presence of free (elemental) sulfur and mercaptans, each at about the 10-ppm level, can cause failure of the copper corrosion test [*20*]. Investigations are needed to determine if diesel fuel sulfur component stability is a potential problem related to fuel corrosion changes during distribution or storage. If an accelerated test is developed for the formation of elemental sulfur, a fuel specification limit of 1-ppm elemental sulfur is suggested in Table 6.

Water Haze

If a fuel tends to form water haze rather than shed water, its lubricity may be reduced, causing wear of moving fuel-handling parts. A better test method to define "Clear and Bright" fuel is needed. An ASTM task group is actively developing an approach to better define water haze. The suggested fuel specification limit in Table 6 for water haze is "Clear and Bright" by Test Method for Free Water and Particulate Contamination in Distillate Fuels (Clear and Bright/Fuel Proceedings) (D 4176) or "50" by a modified ASTM Method for Determining Water Separation Characteristics of Aviation Turbine Fuels by Portable Separometer Field (D 3948) (microseparometer method).

Microbiological Contamination

What seems to be the most elusive and intriguing of all fuel contaminants fall into the general category of hydrocarbon-utilizing microorganisms (sometimes jokingly referred to as "HUM-bugs," for short) [*21*]. Because of the sporadic occurrence of filter plugging caused by organisms and lack of good methods to identify the problem, a great deal of confusion still exists regarding this subject. A task group has been established by ASTM to develop a standard test procedure for identifying microbial problems associated with petroleum fuels. Generally, the organisms involved are aerobic and are found at fuel-water interfaces. A recent article on "What's the Problem With Diesel Fuel" pinpointed sulfur and water as being the most troublesome to fuel users [*22*]. This water that seems to be abundant in diesel fuel systems is necessary for microbiological growth to occur in fuel systems. A fuel filter (usually easily wetted by water) provides an ideal surface for microbial activity. Nutrients, including oxygen, water, minerals, and organic matter, are carried into the filter by circulating fuel. Waste products that would normally accumulate and eventually inhibit growth are carried away as fuel passes through the filters. Thus, an ideal dynamic culture system is set up for the production of larger masses of microbial organisms and eventual

filter plugging. A more common occurrence is the rapid plugging of fuel filters when a portion of fibrous mat is introduced from the bulk storage tank or from the fuel-water interface in a vehicle fuel tank. Thick microbial growth typical of *Cladosporium resinae* is readily identified visually because of its feel and appearance as a fibrous, resinous mat [*23*]. Analytically, the microbiological contamination can be distinguished from fuel degradation sediment. However, standardized methods to identify the presence of microbial debris are needed. A second type of method is needed to identify the potential for microbial growth in a fuel. Both of these methods are being addressed by the ASTM task group on microbiological contamination. With respect to suggested fuel specification limits in Table 6, a value of <1 mg of microbial debris per litre of fuel and a zero growth potential are suggested.

Conclusions and Recommendations

Distillate fuel stability and cleanliness received major attention in the 1950s, which waned somewhat in the 1960s, saw renewed attention in the 1970s and appears to have potential for greatly maturing in its understanding and control in the 1980s.

The suitability of diesel fuels for various end-uses depends on the crude oil (or synthetic crude) source, refinery processing of the components in the finished fuel blend, additives used, method of fuel distribution and storage, and the sensitivity of the end-use to various contaminants and other fuel properties. All of these factors may affect ultimate performance in a particular user's application. In addition to those factors that determine the properties of the fuel, requirements will vary depending on the level of contamination that can be tolerated by specific end-use systems. Specifications for diesel fuel should contain guidance information for the user to determine how to maintain adequate stability and cleanliness in storage and use. Evidence has been presented to support the potential need for diesel fuel specifications that provide limits on the following properties:

- Stability (oxidative/thermal)
- Acid number
- Cleanliness (particulates)
- Soluble gum
- Sulfur compound stability
- Water haze
- Microbiological contamination

The status of test methods to measure these properties has been summarized, and user-based specification limits have been suggested.

Microbiological Outlooks

By E. C. Hill[5]

For those who know nothing about the Institute of Petroleum, it is a United Kingdom organization which is (1) a trade organization and represents its members in conflict with government or in cooperation with governments, (2) it is a technical institution with a large number of technical committees of which the microbiology committee is one, and (3) it sees itself in some sort of an educational role. Although it exists primarily for the benefit of its

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members, it also invites other European participants. The microbiology committee is an active committee, and the members are from the main oil companies, from academic institutions, from the major users, and so forth. It organizes normally two symposia a year. These are either published by John Wiley or they are published internally by the Institute of Petroleum. For example, last year there was a very successful meeting in Aberdeen called Microbiological Problems in the Offshore Oil Industry. This was published by John Wiley. It so happens that many of the fuel problems we have been experiencing have been on the offshore rigs and supply ships, so naturally these proceedings actually do contain some papers of relevance fuels. The second symposium we had last year was actually called the Microbiology Fuels and was published internally by the Institute of Petroleum. I think everybody who was involved in this was extremely pleased with the outcome because in Europe over the last five years or so we have all been wondering whether we are looking at a genuine upsurge in microbial problems or whether it was an apparent upsurge because recognition was better. We decided to hold a symposium in which the first four papers were presented by non-microbiologists, but these were people who were involved with refining, blending, additives, and distribution. The outcome was that the microbiologists who were present were able to identify real factors for the first time, which have contributed in Europe to a very considerable upsurge in microbiological problems. I will quote you the last sentence or so from a paper by Mr. Berryman of Shell International Petroleum: "Middle distillate fuel demand forecasts for the next 20 years would indicate the fuel quality may become even more favorable for microbial activity. Education of users is required to heighten the awareness, improve housekeeping facilities, and make available a range of treatment products which are effective and involvement acceptable." At the end of this meeting we all believed we were facing a genuine change in Europe. Microbiologists have seen it in other ways because we have recognized that the organisms that we were isolating and identifying were different, and that the fuels affected were different. *Cladosporium* was not the only cause of our problems, but we were seeing other micro-organisms. In particular we were seeing *Candida* problems, a variety of fungal problems, and pseudomonads. So from the microbiology point of view we could anticipate that the fuels themselves had changed chemically in some way so that they stimulated a different spectrum for organisms. As an outcome of this meeting, there was so much interest that the Institute formed a task force. The objectives of this task force are to look at the symptoms of fuel malfunction caused by microbial activity, to look at sampling methods and their significance, to look at microbiological test methods, to look at interpretation of data and at remedial techniques, the outcome of remedial techniques, and the factors that contribute to microbiological problems. We are looking for international representation on this in the first case because a lot of our problems are with ships, and these ships move internationally. We do not want to be in the position where people were uplifting samples in different countries, getting different tests run and getting different interpretations. We were anxious that we should have this task force organized on a European basis, and the main reason why I am here is that we are also anxious that as these ships move across the ocean to North America they should also have consistent advice. So I am very anxious that from an Institute of Petroleum point of view we work very closely with you on standard test methods, both laboratory and on-site, where we take samples and finally how we interpret the results that we get. This task force has in fact met twice, and it is making fairly plodding progress as you might imagine. When a number of people come for the third International Symposium on Fuel Stability in London next year, I intend to organize a meeting of this task force either immediately before or immediately after. Hopefully you will come along to this meeting, and we can have round-table discussions of the list of problems that we are addressing.

If anyone is interested in the minutes of the first two meetings, they can write to me in London.[5] I have been interested in all that I have been listening to over these two days but some things strike me as being different in Europe and America. In some ways we are a little more advanced and in some ways we are a little more backward.

In Europe but not mentioned here we are now seeing microbial problems with black oils. This is a bacterial problem. On the European scene the sources of the crudes have changed dramatically. They now come from the North Sea and even from Russia. In processing different crudes, we are obviously making different kinds of distillates. There are also many blending changes and changes in additives that result in the fuel chemistry changing. One of the factors that was identified was that there are far more soluble carbon compounds now in the water phase. If we are putting more soluble nutrients into the water phase we are stimulating microorganisms there. I would like to draw your attention to the much greater use of additives. Little "bugs" require carbon, nitrogen and phosphorus, and many additives contain these. This is a stimulation factor. There is work on the effect of these on the microbiology of fuel. As you might expect, some of them actually stimulate microbial growth because we are providing a few vital nutrients, and some of them conversely actually inhibit microbial growth.

On the subject of anti-microbial strategies that are used, these are all more or less the same as conducted here. In some ways, I think we are less advanced than you are. There is a need to improve housekeeping, and everyone here is aware of this and hopefully they will do it in the future. Typical physical methods that have been investigated are radiation, pasteurization centrifugation, and filtration. On the technical chemical side, you have two strategies: either to put a preservative in or to actually disinfect the system when trouble arises. There is a great deal of resistance in Europe to the use of fuel preservatives. Obviously, the glycol ethers have been around for a long time; they are used mainly by the military because they need the antifreeze properties. The civilian user tends not to like spending money on fuel preservatives. He would rather wait until he has trouble and then use a fuel disinfectant. Disinfection has been used from time to time, sometimes with the approval of engine builders and sometimes without. In most cases fuel users are very much going for disinfection rather than use preservatives. On this basis, there is a much greater call for on-site microbiological testing. I do not think I have time to say much about this, but if you do on-site microbiological testing, you also must interpret the results that you get.

There is a problem with detection, as you know, that most people can cope with water phase detection and that is fine provided you have a water bottom sample. You can then do more or less conventional microbiological tests on it. It is when you have oil phase samples that you run into trouble; it has been mentioned already that a lot of microorganisms die out very quickly once they get into the oil phase, and by the time the sample is a few days old you may test and obtain a completely wrong answer. You may believe you have no microorganisms in the system, but in fact the system is very heavily contaminated. There is a great need for a quick microbiological test on the oil phase so that you do not have to ship it back to the laboratory. We are thoroughly interested in this development, but at the moment, the test that is most widely used is a laboratory based membrane filtration test, and the Institute of Petroleum has a committee working on standardizing this.

It has now been submitted not as a standard test procedure (because it is not that rigid), but it is a recommended test procedure. An alternative to using membrane filtration is to take the fuel and emulsify it with nontoxic emulsifier and then apply conventional microbiology. I think you have to be very cautious to interpret the test when you only have a fuel phase sample to examine.

One tool that we do have and you do not is a little monitor that will monitor levels of biocide. So often, as you know, you work on some biocide accurately in the laboratory and

you then give a guy a bucket, and say "put in 24 ppm." You have no idea whether he puts it in at the correct concentration, whether it gets into the water phase and how long it remains in the water phase. We have a little test kit, which is a strip of plastic with a pad on the end, which contains an organism that is very sensitive to most biocides. It is immersed in fluid, withdrawn, and incubated. If it stays white, the organism is inhibited; this tells you that the biocide is above a specific concentration. So what? Well, we can arrange dilutions of that water bottom, test them all, and then we are able to say which of these dilutions contains inhibitory concentrations of biocide. Hence we can deduce the original concentration. Thus, you have a simple technique for actually measuring how much biocide you really have in your tank water bottoms.

To conclude, the two meetings next year at which we would really like to see ASTM members are the Third International Meeting on Fuel Stability and the Fuels Task Force.

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