DISTILLATE FUEL STABILITY AND CLEANLINESS

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Foreword

The symposium on Distillate Fuel Stability and Cleanliness was presented at Chicago, Illinois, 24 June 1980. The symposium was sponsored by The American Society for Testing and Materials through its Committee D-2 on Petroleum Products and Lubricants. L. L. Stavinoha, Southwest Research Institute, and C. P. Henry, E. I. duPont de Nemours and Co., presided as symposium chairmen and editors of this publication.

Related ASTM Publications

The Relationship Between Engine Oil Viscosity and Engine Performance, STP 621 (1977), $15.00, 04-621000-12

The Relationship Between Engine Oil Viscosity and Engine Performance, STP 621-S1 (1977), $12.00, 04-621010-12

The Relationship Between Engine Oil Viscosity and Engine Performance, STP 621-S2 (1977), $15.00, 04-621020-12

The Relationship Between Engine Oil Viscosity and Engine Performance, STP 621-S3 (1978), $15.00, 04-621030-12

The Relationship Between Engine Oil Viscosity and Engine Performance, STP 621-S4 (1980), $18.00, 04-621080-12

Single Cylinder Engine Tests, Part I, Caterpillar IG2 Test Method, STP 509A (1979), $9.75, 04-509011-12

Single Cylinder Engine Tests, Part II, Caterpillar IH2 Test Method, STP 509A (1979), $9.75, 04-509020-12

Single Cylinder Engine Tests, Part III, Caterpillar ID2 Test Method, STP 509A (1979), $9.75, 04-509030-12

Single Cylinder Engine Tests, Part IV, Caterpillar L38A Test Method, STP 509A (1980), $7.25, 04-509040-12

Significance of Tests for Petroleum Products, STP 7C (1977), $11.75, 04-007030-12

A Note of Appreciation to Reviewers

This publication is made possible by the authors and, also, the unheralded efforts of the reviewers. This body of technical experts whose dedication, sacrifice of time and effort, and collective wisdom in reviewing the papers must be acknowledged. The quality level of ASTM publications is a direct function of their respected opinions. On behalf of ASTM we acknowledge with appreciation their contribution.

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Introduction

A symposium 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, and refinery processes which improve stability. This publication contains the proceedings.

The symposium was organized by Section E-V of Technical Division E on Burner, Diesel and Turbine Fuel Oils, which is under the jurisdiction of ASTM Committee D-2 on Petroleum and Petroleum Products. Section E-V on Fuel Stability and Cleanliness was organized in 1979 with a scope of activities defined as follows:

> The Section shall 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 (on Burner, Diesel and Turbine Fuel Oils).

A primary objective of Section E-V members—both initially and on a continuing basis—is to accurately determine what test methods are needed, can be standardized, and will be accepted by the petroleum industry and those it serves.

To the casual observer, needs with respect to distillate fuel stability and cleanliness testing may seem readily apparent—one test of limited value has been standardized to determine oxidative stability [ASTM Test for Oxidation Stability of Distillate Fuel Oil (Accelerated Method) (D 2274-74)], and ASTM methods for cleanliness have been developed for other purposes [ASTM Test for Water and Sediment in Crude Oils and Fuel Oils by Centrifuge (D 1796-68) and ASTM Tests for Particulate Contaminant in Aviation Turbine Fuels (D 2276-79)]. No methods are standardized to evaluate freedom from water haze or microbiological contamination. A similar symposium was held in 1958 (*Symposium on Stability of Distillate Fuel Oils, ASTM STP 244*); a primary purpose was to determine if standardized tests for stability were needed. Indeed, a second purpose was to determine if a section such as E-V should be formed.

Some of the concerns raised in 1958 are still unanswered, largely due to complexities with respect to needs, testing, and acceptability. Fuel stability and cleanliness needs are not simply defined; requirements for various

burners, diesel engines, and turbines vary over a wide range and are somewhat adjustable to tolerate normally available fuel. Test methods, particularly accelerated stability tests useful for quality control, are complicated by poorly understood variables which affect the precision and significance of results. These uncertainties lead to diverse opinions and viewpoints which hamper broad acceptance of new methods. Progress in developing standardized, broadly accepted ASTM methods for fuel stability and cleanliness will result from the patient development of consensus on needs and test methods—but recognizing that needs vary and tests have limitations.

The papers included in this publication have helped provide direction for further work and update the state of the art since the 1958 symposium. A careful perusal also helps the reader assess the balance between current and future fuel quality and requirements for satisfactory end-use performance.

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L. L. Stavinoha[1] *and S. R. Westbrook*[1]

Accelerated Stability Test Techniques for Middle Distillate Fuels

REFERENCE: Stavinoha, L. L. and Westbrook, S. R., **"Accelerated Stability Test Techniques for Middle Distillate Fuels,"** *Distillate Fuel Stability and Cleanliness, ASTM STP 751,* L. L. Stavinoha and C. P. Henry, Eds., American Society for Testing and Materials, 1981, pp. 3-21.

ABSTRACT: Improved test techniques are needed for evaluating the inherent stability of middle distillate fuels both in surveillance and in procurement activities. A project was initiated to define and evaluate the contributing conditions leading to the formation of deleterious products in accelerated aging tests of middle distillate fuels and to relate these results to an experimental definition of more repeatable/reliable middle distillate fuel stability test technique(s).

A literature search was conducted to provide a list of stability test techniques and their interpretations which could be used in a correlative middle distillate fuel stability test program. For this program, seven accelerated stability tests were chosen and evaluated using a set of six test fuels. The test techniques were selected to represent a wide variety of test conditions, including temperature, aging time, and oxygen availability. These six test fuels were purposely chosen to represent a wider range of stabilities than would necessarily be commonly available for procurement. The fuel properties generally measured included both adherent and suspended particulates, steam jet gum, color, and light absorbance at 540 nm. Accelerated stability test results were then related to test results obtained at a storage temperature of 43.3°C, which has generally been regarded as showing good correlation with long-term ambient storage.

KEY WORDS: deterioration, storage stability, stability tests, fuel stability, diesel, distillate fuels, fuel test techniques, accelerated stability

In April 1977 the U.S. Army Research Office, in cooperation with the U.S. Army Mobility Equipment Research and Development Command (MERADCOM), sponsored a seminar at Southwest Research Institute to promote basic research in the area of "Diesel Fuel Stability" in support of test technique developmental activities. As an overview, a presentation of

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"A Review of Diesel Fuel Deterioration and Related Problems" was made by Army representatives and later published through the Defense Documentation Center [*1*].[2] While no basic research has been prompted thus far, Army-sponsored diesel fuel stability research and development studies have continued.

From a review standpoint, extensive work has been performed to develop tests which predict the storage stability of distillate fuels. A large number of tests have been used or are in use for evaluating fuel stability. A presentation by MacDonald and Jones [*2*] tabulated 26 different methods. The Navy-Coordinating Research Council Barge Storage Program [*3*], conducted in the 1950's to determine the scale-down factor from barge to bottle or drum storage, provided data indicating the usefulness of storage at 43.3°C as a predictive method for long-term storage. With the accumulation of additional data, most researchers have accepted the results of aging fuels at 43.3°C as consistent with those obtained under actual storage conditions. Bottle storage at 43.3°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 [*4–7*]. Although the American Society for Testing and Materials (ASTM) has accepted several accelerated tests for evaluating petroleum products, only the ASTM Test for Stability of Distillate Fuel (D 2274-74) is directly applicable to distillates [*8*].

The ASTM Method D 2274-74 accelerated stability technique is currently specified in the Federal diesel fuel specification, VV-F-800b, as a prediction of storage stability for bulk fuel deliveries at the time of procurement for use outside the continental United States or for storage in the continental United States. The present VV-F-800b specification has a D 2274 limit of 1.5 mg of total insolubles in 100 ml of sample. However, the stated repeatability of ASTM D 2274 in the range of 0 to 1 mg/100 ml is ± 0.3 mg/100 ml with a reproducibility of ± 1.0 mg/100 ml. Two additional test methods are used in VV-F-800b specifically as a measure of contaminants: (1) ASTM Test for Particulate Contaminant in Aviation Turbine Fuels (D 2276-79) modified to use 1.2-μm filters to determine particulates, and (2) ASTM Test for Existent Gum in Fuels by Jet Evaporation (D 381-70) (steam jet method).

These same types of methods have proved useful in determining the stability of diesel in storage in a 2-year, 100-barrel, above-ground steel tank storage program using four diesel fuels meeting Federal Specification VV-F-800a [*9*]. Three government specification documents describing diesel fuel limit the total insolubles to a 1-mg/100-ml maximum; namely, the U.S. Air Force Purchase Description No. AFPID 9140/1, a Canadian Specification 3-GP-30, and a British Specification 91-9/Issue 1 [*9*]. In addition to this, a recent Society of Automotive Engineers paper [*10*]

[2]The italic numbers in brackets refer to the list of references appended to this paper.

recommended that a maximum limit of 0.2 mg/100 ml for the accelerated stability requirements be applied to commercial diesel fuels to ensure adequate stability. However, it is felt that this low value would not be practical from the standpoint of supply and logistics.

One approach in terms of new predictive methodologies being considered by the Army for diesel fuels has involved use of a dynamic oxidation autoclave wherein maximum aeration of a liquid occurs under pressure [*11*]. This technique has been successfully applied to gasolines.

An extensive program to evaluate the storage stability of gasoline was summarized in a 1972 publication [*12*] in which the Bureau of Mines reported the development of a rapid and precise method for predicting storage stability of gasolines, a method which may be applicable to diesel fuels.

Recent actions impacting fuel stability and creating greater awareness and concern for fuel stability have been fuel curtailments (leading to increased fuel storage), increased replacement cost of stored fuel, greater variability in crude oil sources/refinery techniques, and the potential for introduction of potentially less-stable synthetically derived fuels. In February 1979 a program was initiated to review and selectively evaluate laboratory test techniques which may be applicable to distillate fuel stability.

This report summarizes the progress of this program during the first year's effort to evaluate accelerated stability test techniques for diesel (or distillate) fuels. The program has been covered in a more detailed report [*13*].

Discussion

A literature search was conducted in order to develop a catalog of test techniques from which to choose the test methods that would be evaluated in this program. A large number of test techniques from numerous sources were reviewed and cataloged based upon an annotated bibliography consisting of 116 entries, a detailed listing of which is contained in Ref *13*. Using this approach, two sets of possible test techniques were tabulated from which eight test methods were studied.

The 43.3°C storage test method (in Table 1) represented a generalized method incorporating a cross-sectional selection of details found in six different 43.3°C tests. The method was used in both a vented and nonvented configuration for comparing the effects of oxygen availability and retention of volatile reactive products. Since storage at 43.3°C (for one week) is widely accepted as representative of storage under ambient temperatures (for approximately four weeks), this test method provided inherent fuel stability base-line comparison data for evaluating the data from the other accelerated stability test methods. Inherent stability is here defined as the relative stability exhibited under "sterile" laboratory conditions as opposed to actual stability under normal storage conditions in the presence of metallic containers and other environmental considerations such as dirty

TABLE 1—*Summary of selected test methods.*

Test No.	Test Method Title	Test Duration; Temperature	Sample Size, ml	Test Conditions
1	Storage test at 43.3°C (110°F)	4, 8, 16, 24, & 32 weeks; 43.3°C	650	vented and nonvented borosilicate glass bottles in light proof box
2	Storage test at 150°C (300°F)	1.5, 3.0, 4.5 h; 150°C	50	vented borosilicate glass test tubes
3	Storage test at 80°C (175°F)	3, 7, 14 days; 80°C	100	vented borosilicate glass bottles
4	ASTM Method D 2274 (modified)	16 h; 95°C	350	oxygen is bubbled through the fuel while it is aging
5	ASTM Method D 873 (modified)	16 h; 100°C	100	the fuel is heated in a bomb that is pressurized to 689 kPa (100 psi) with oxygen
6	ASTM Method D 3241 (modified)	2.5 h; 204, 229, & 254°C	600	the fuel is passed over a metal tube that is heated to the desired temperature and then flowed through an in-line filter
7	Storage test at 93.3°C (200°F)	16 h; 93.3°C	250	nonvented soft glass bottles
8[a]	Chromic acid oxidation test	N/A; 20°C	2	fuel is chemically oxidized and the reaction is followed spectrophotometrically

[a]This technique was evaluated only to the extent that it was found that an homogeneous solution could not be prepared, due to a prerequisite to spectrophotometric evaluation.

water bottoms in storage tanks. The second set of possible test techniques consisted of 55 accelerated stability tests from which seven test methods were then formulated. The eight test methods thus derived are summarized in Table 1.

The test methods in Table 1 were selected to represent a wide variety of test conditions, including temperature, aging time, and oxygen availability. Some of the methods were modified so that the test data for the aged fuel would be similar for each test technique. The "work-up" tests were kept similar in order to simplify any comparisons; and, since the work-up tests to be performed on the aged fuel samples were to be fairly consistent for all test techniques, this factor was not considered crucial in the selection process.

Fuels aged using the methods in Table 1 were evaluated for (1) insoluble particulates (adherent and suspended), (2) steam jet gum, (3) color, (4) light absorbance, and (5) acid number. Additionally, test results unique to a particular test method were also obtained (for example, heater tube deposit rating and pressure drop across the filter for Test No. 6 in Table 1). Glass fiber filters having a nominal pore size of 1.2 μm were used to measure suspended particulates/insolubles.

After the aged fuel had been filtered through the glass fiber filter, it was filtered through two membrane filters of 0.45-μm pore size. This second filtration was performed in an attempt to determine what amount, if any, of particulates were passing through the glass fiber filter having a nominal pore size of 1.2 μm.

Adherent insolubles are also known as wall adherent gum or insoluble gum. This gum is the insoluble material that is found to adhere to the walls of the aging container. Since the ASTM Test for Thermal Oxidation Stability of Aviation Turbine Fuels (D 3241-77) (JFTOT Procedure) apparatus has no specific aging container, no adherent insolubles were reported for this test technique.

The total insolubles reported for each test is the sum of the particulates from the glass fiber filtration and the adherent insolubles. For the methods that have duplicate results for either particulates or adherent insolubles, the average of the two results was used for the total insolubles value. When an average value was used, the total insolubles were reported as average total insolubles. Due to variability in test results for these type of analyses, duplicate analyses are always recommended. In the case of storage tests at 80 and 150°C, the aged fuel samples were mixed to provide an averaging effect.

Steam jet gum, also known as soluble gum, was determined on the filtered fuel by evaporating the fuel, using a jet of superheated steam, and leaving the soluble gum behind. The sum of the steam jet gum value and total insolubles was reported as total gum. Average values were used where applicable and were reported as such.

The aged fuel tests selected were those thought to be the most likely to change during storage of diesel-type fuels. Particulates tend to plug filters, while steam jet gum is thought to be related to injector fouling and may be a precursor to particulate formation. Both gum and unfiltered particulates are thought to contribute to engine combustion chamber deposits. Acid number is related to corrosion of containers, pumps, and possibly injector nozzles. Additionally, it is thought that acid number is related to accelerated deterioration of a fuel in much the same manner as certain metals (for example, copper) act as catalysts. While color of a fuel can indicate chemical deterioration (oxidation), it is not directly harmful to the handling or use of diesel fuel. Light absorbance at 540 nm is thought to be related to color body formation and soluble gum formation in a fuel. However, this belief has not been proven nor has light absorbance been demonstrated to relate directly to harmful effects on fuel handling or engine operation.

In this program, 27 candidate fuels were received from various locations in the United States and were used directly or as blends to form a set of six test fuels.

Table 2 lists the six selected test fuels (identified as Fuels A-F) in order of their stability as predicted by ASTM Method D 2274 with the most stable fuel at the top and the least stable fuel at the bottom.

It is noted at this point that some of the selected test fuels are less stable than might commonly be commercially available, but were selected to provide a wide range of stabilities. Table 3 lists physical-chemical characterization data for each of the test fuels.

Table 4 summarizes the D 2274 data for fuel samples A through F. Included in Table 4 are the total insolubles, filterable and adherent insolubles, steam jet gum, total gum, particulates on a 0.45-μm filter, color, light absorbance, and total acid number. Similar extensive data for the

TABLE 2—*Selected test fuels.*

Fuel	Description	ASTM Method D 2274 Total Insolubles (mg/100 ml)
A	straight run diesel	0.7
B	diesel fuel potentially unstable from California crude	0.9
C	same as Fuel E below but treated with antioxidant-dispersant at 4.5 kg/1000 bbl (10 lb/1000 bbl)	2.9
D	Cat 1-H specification fuel from San Antonio, Tex. refinery	4.2
E	blend of low aromatic naphtha, light cycle oil, and light catalytically cracked stock	4.8
F	blend of No. 2 fuel oil and Fuel D from above	10.0

TABLE 3—*Physical-chemical characterization data for selected test fuels.*

	Sample Fuel					
	A	B	C	D	E	F
FIA, D 1319[a]						
% aromatics	27.2	31.4	35.9	29.3	37.5	36.1
% olefins	3.5	3.3	3.5	2.3	3.7	3.2
% saturates	69.3	65.3	60.6	68.4	58.8	60.7
Total acid number, D 664[b]						
mg KOH/g sample	0.03	0.34	0.25	0.03	0.23	0.04
Steam jet gum, D 381,[c]						
mg/100 ml	5.6	23.2	4.8	4.8	10.4	4.6
Sulfur by XRF, weight %	0.18	0.27	0.50	0.41	0.50	0.60
API gravity, D 287[d] at 15.5°C						
(60°F)	39.6	34.6	30.8	35.0	30.8	34.7
Density at 15°C	0.8270	0.8515	0.8714	0.8494	0.8714	0.8509
Light transmittance at						
650 nm	0.004	0.061	0.194	0.010	0.206	0.015
575 nm	0.017	0.181	0.275	0.026	0.328	0.033
540 nm	0.033	0.327	0.406	0.041	0.509	0.049
500 nm	0.071	0.614	0.652	0.071	0.825	0.083
Distillation by D 86,[e] °F(°C)						
IBP	361(183)	408(209)	470(243)	424(218)	466(241)	428(220)
10% off	406(208)	448(231)	519(271)	480(249)	518(270)	484(251)
50% off	472(244)	512(267)	566(297)	526(270)	567(297)	530(277)
90% off	598(314)	638(337)	610(321)	600(316)	610(321)	587(308)
EP	670(354)	706(374)	647(342)	680(360)	645(341)	653(335)
Calculated cetane index, D 976[f]	51.9	48.3	48.8	51.0	48.8	51.0

[a] ASTM Test for Hydrocarbon Types in Liquid Petroleum Products by Fluorescent Indicator Adsorption (D 1319-77).
[b] ASTM Test for Neutralization Number by Potentiometric Titration (D 664-58).
[c] ASTM Method D 381-70 (see text).
[d] ASTM Test for API Gravity of Crude Petroleum and Petroleum Products (Hydrometer Test) (D 287-67).
[e] ASTM Test for Distillation of Petroleum Products (D 86-78).
[f] ASTM Test for Calculated Cetane Index of Distillate Fuels (D 976-66).

TABLE 4—*Data for fuel aged by ASTM Method D 2274 (modified).*

	A	B	C	D	E	F
Total insolubles, mg/100 ml	0.7	0.9	2.9	4.2	4.8	10.00
Filterable insolubles, mg/100 ml	0.4	0.6	2.4	1.6	3.8	3.1
Adherent insolubles, mg/100 ml	0.3	0.3	0.5	2.7	1.0	6.9
Filtrate Tests						
Steam jet gum, D 381,[a] mg/100 ml	7.9	23.5	12.6	11.5	18.2	21.0
Total gum, mg/100 ml	8.6	24.4	15.5	15.7	23.0	31.0
Particulates D 2276[b] (modified) 0.45-μm second filtration, mg/100 ml	0.21	0.50	2.02	0.24	3.50	0.02
Color, D 1500[c] (before/after aging)	1.0/2.5	3.5/6.0	2.0/3.5	1.0/2.5	1.5/4.5	1.0/3.0
Light absorbance at						
650 nm	0.010	0.116	0.089	0.030	0.133	0.044
575 nm	0.038	0.456	0.161	0.085	0.273	0.121
540 nm	0.065	0.796	0.297	0.142	0.440	0.202
500 nm	0.131	0.339	0.630	0.271	0.816	0.386
Total acid no., D 664,[d] 40-g sample, mg KOH/g	0.03	0.34	0.28	0.06	0.11	0.07

[a] See Table 3.
[b] See text.
[c] ASTM Test for Color of Petroleum Products (ASTM Color Scale) (D 1500-64).
[d] See Table 3.

other test methods are summarized elsewhere [*13*] and are used here for general discussion and conclusions.

Some of the results of the filtration through a 0.45-μm pore size filter are found in Table 4. It can be seen from the data that in some cases the amount of particulate that was trapped by the membrane filter was nearly the same as that trapped by the glass fiber filters. However, in the majority of the remaining data (not presented here), the amount of particulates trapped by the membrane filters is small relative to that trapped by the glass fiber filter. The large amounts of particulates trapped by the membrane filters in the D 2274 test may possibly be due to a lack of time for the smaller particles to agglomerate into particles sufficiently large enough to be trapped by the glass fiber filter.

Figure 1 (based on data in Table 4) provides a graphical presentation of D 2274 derived soluble gum, adherent insolubles, and filterable insolubles, the total sum having been defined as total gum.

The analytical test method data were selectively placed in a data base and mathematically compared to produce correlation coefficients using a computer-assisted Pearson Product-Moment Correlation [*14*]. In the majority of cases, a correlation coefficient in the range of 0.4 to 0.5 and above indicates a significant linear relationship between the data. For this program, values of 0.75 or greater were considered to be the most reliable for comparison purposes.

The most useful coefficients for comparison purposes are those which show the correlation between similar physical parameters. The parameters that were given the major emphasis for this program were total insolubles, steam jet gum, and total gum.

Examination of the coefficients for comparison of analogous parameters showed good correlation between vented and nonvented 43.3°C storage data. Only the coefficients for the four-week data showed a value of less than 0.8800. While nonvented storage could be more severe in that volatile reactive materials would be kept in the vessel and thereby contribute to deleterious product formation, there was no evidence to support this effect.

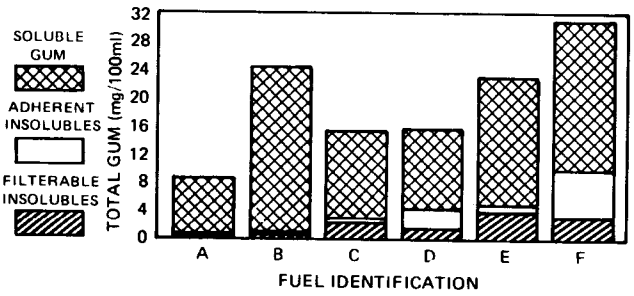

FIG. 1—*Data for fuel aged by ASTM Method D 2274 (modified).*