

ACCELERATED FUEL OIL STABILITY

UOP Method 835-82

SCOPE

This method is for determining the relative stability of distillate fuels such as home heating oils or diesel fuels under short-term, high temperature, aging conditions involving air exposure. It may also be used to evaluate the effectiveness of additives in inhibiting sediment formation and color degradation of distillate fuels under conditions of the test. The test does not correlate well with field storage or 3 month 43 C (110 F) dark storage.

The method, and modifications of it, have been used since 1942 (C & NW RR), and it has been known by a variety of titles including “EMD-Diesel Fuel Stability Test”, “Union Pacific Diesel Blotter Test (U P-Blotter Test)”, “Santa Fe Blotter Test”, “Nalco 300 F Test” and the DuPont F21-61 Test. Of the tests listed, the DuPont publication is probably the most commonly used.

OUTLINE OF METHOD

A sample of distillate fuel is aged (heated) at 150 C (300 F) for 90 minutes. After cooling, any insoluble sediment is collected by vacuum filtration on a filter paper which is generally referred to as a “blotter”. The reflectance of the sediment is measured, or it may be visually compared to a set of reference blotters, to obtain a numerical rating. The fuel oil percent transmittance or color is measured on the filtered sample before and after aging to determine the color stability of the sample.

APPARATUS

Cell, spectrophotometer 40-mm path length, UV Silica, Beckman Instruments Inc., Cat. No. 580016, or equivalent

Dish, Petri, borosilicate glass, 60-mm diameter

Filter holder, stainless steel, “Milipore Hydrosol” filter holder, Millipore Corp., No. XX 20 047 20, or equivalent

Flask, filtering, with side arm, 500-mL

Oil bath, Blue M Electric Co., Model MW 1145 A-1, or equivalent

IT IS THE USER'S RESPONSIBILITY TO ESTABLISH APPROPRIATE PRECAUTIONARY PRACTICES AND TO DETERMINE THE APPLICABILITY OF REGULATORY LIMITATIONS PRIOR TO USE. EFFECTIVE HEALTH AND SAFETY PRACTICES ARE TO BE FOLLOWED WHEN UTILIZING THIS PROCEDURE. FAILURE TO UTILIZE THIS PROCEDURE IN THE MANNER PRESCRIBED HEREIN CAN BE HAZARDOUS. MATERIAL SAFETY DATA SHEETS (MSDS) OR EXPERIMENTAL MATERIAL SAFETY DATA SHEETS (EMSDS) FOR ALL OF THE MATERIALS USED IN THIS PROCEDURE SHOULD BE REVIEWED FOR SELECTION OF THE APPROPRIATE PERSONAL PROTECTION EQUIPMENT (PPE).

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Pipet, volumetric transfer, 50-mL capacity, with wide tip opening for rapid delivery, Sargent-Welch Scientific, Cat. No. S-69514-K, or equivalent

Pipet, Mohr, 1.0-mL capacity, 0.01-mL subdivisions

Pump, vacuum, heavy duty, Sargent-Welch Scientific, Cat. No. S-71380, or equivalent

Reference blotters, available from E. I. DuPont De Nemours Inc. (see Ref. 1)

Reflectometer, paper brightness meter, Photovolt Corp., Model 670, Cat. No. 00-570-32, with Search Unit W, complete with standards, or equivalent

Spectrophotometer, Bausch & Lomb Model 710, or equivalent. Modify cell holder, if required, to accept a 40-mm cell.

Stirring apparatus, test tube, variable speed, "Vortex-Genie", Sargent-Welch Scientific, Cat. No. S-76509-20, or equivalent

Test tubes, chemical, with lip, Pyrex, Corning No. 9800, 200-mm length, 25-mm OD, 70-mL capacity, available from Sargent-Welch Scientific, Cat. No. S-79515-M or equivalent

Test tube rack, to fit inside the oil bath. A custom made version capable of holding up to 28 tubes may be constructed of two horizontally aligned stainless steel plates each measuring 2 mm thick by 175 by 265 mm and held vertically 175 mm apart using four screw rods of 225 mm length as corner legs. A grid of 7 by 4 holes of 28 mm diameter in the upper plate and 20 mm diameter in the lower plate is made. To facilitate good oil circulation, the lower stainless steel plate should also have 3 by 6 or 18 additional holes of about 12 mm diameter centered between the test tube holes. Finally, a stainless steel hook is attached to the four corner legs above the upper plate, so that the entire tray may be easily removed from the oil bath.

REAGENTS AND MATERIALS

All reagents shall conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society, when such specifications are available, unless otherwise specified. References to water mean deionized or distilled water.

Filter paper, medium, Whatman No. 1, 4.25-cm diameter, Sargent-Welch Scientific, Cat. No. S-33215A, or equivalent

n-Heptane, 99% minimum purity, available from Phillips Petroleum Co., or equivalent

Mineral oil, heavy 300-320 SUS at 38 C viscosity, Sargent-Welch Scientific, Cat. No. SC 13635, or equivalent

Toluene, 99% minimum purity, available from Phillips Petroleum Co., or equivalent

PROCEDURE

Locate the oil bath in a fume hood shielded from light to avoid exposure of the samples during aging. Alternatively, a special ventilation hood may be fitted over the bath to permit free air circulation without exposure of the samples to light. Fill the bath with mineral oil to a sufficient depth (about 3/4 full) so that the oil will be above the level of the sample when the test tubes are inserted. Adjust the bath temperature controller so that a temperature of 150 ± 2 C (300 ± 5 F) is maintained.

Using the Millipore filtration apparatus, vacuum filter through a Whatman No. 1 filter paper sufficient fuel sample, *previously brought to ambient temperature*, to provide a 50-mL fuel sample (see Note 1). If samples are to be run in duplicate or if the sample is to be tested with varying additions of an inhibitor, filter enough sample so that 50 mL will be available for each test. Determine the % transmittance of the filtered sample at a wavelength of 650 nm using a 40-mm cell with water as a reference (see Note 2).

Transfer 50-mL of the filtered sample into a Pyrex test tube positioned in the rack using a volumetric pipet. Place the sample rack containing the samples into the oil bath and allow the fuel samples to age for 90 minutes at 150 ± 2 C in *total darkness* with *good ventilation and free access of air*. Then, remove the samples from the bath and allow them to cool for 90 minutes in the absence of light. Allow the samples to cool gradually because rapid cooling may cause super cooling, thereby leading to erroneous results. When cool, wipe the outside of the test tube free of mineral oil with a paper towel.

Quantitatively vacuum filter the sample through a Whatman No. 1 filter paper in the Millipore filter holder (see Note 3). Remove the filtered sample from the filtering flask and, without delay, place the sample in the cell and determine % transmittance with the spectrophotometer (see Note 2). Replace the filtering flask and rinse the sample test tube with three, 10-mL portions of *n*-heptane, transferring each wash to the filter holder. Thoroughly wash the filter paper and holder with *n*-heptane, using vacuum, to remove all traces of fuel oil. Any residual fuel oil remaining on the filter will influence sediment measurement. Discard the *n*-heptane filtrate.

Allow the filter paper to air-dry in an appropriately labeled Petri dish. Measure the reflectance of the filter with the reflectometer using the ceramic standard supplied with the instrument as an absolute reference (see Note 4).

Additive Testing

When the effectiveness of additives is to be evaluated, they are usually added at very low concentration levels. Accurately prepare solutions of the additive to be tested in toluene. Adjust the concentration of the additive so that when 0.5 mL of the solution is added to 50 mL of sample, the desired additive concentration is obtained.

Transfer 50 mL of the filtered sample into a Pyrex test tube. To the sample, accurately add exactly 0.50 mL of the additive solution with a 1.0-mL pipet. Place the sample test tube in a test tube stirring apparatus such as the "Vortex-Genie" and thoroughly mix by obtaining a vortex 10 times. Place the sample in the bath and process it from this point on as described above.

REPORT

Report the percent transmittance of the sample before and after aging. Also report the percent reflectance which may be converted to a blotter rating if desired.

INTERPRETATION OF RESULTS

A fuel producing a filter which has a percent reflectance matching reference blotter No. 7 or lower is generally considered as "passing" the test; all others are considered to "fail" the test. The numerical rating offers a means of comparing various fuels or additives.

The percent transmittance or ASTM color of the "before" and "after" aging samples provides a means of determining the color stability of the test fuel.

NOTES AND PRECAUTIONS

1. Prefiltering of fuel before aging removes any insoluble matter which may be present prior to aging. Filtering must be accomplished within 24 hours of starting the test.
2. Alternatively, the color may be determined according to ASTM D 1500 using a colorimeter meeting the specifications of that method.
3. Filter the aged sample and determine the transmittance soon after reaching ambient temperature to avoid changes in composition.
4. Alternatively, the filter paper can be visually compared to a set of DuPont Reference Standards (see Ref. 1) and depending on the degree of shading, assigned a numerical value, referred to as a blotter rating. Values obtained using the reflectometer can be correlated to visual blotter ratings through the use of the attached table.
5. Filter paper may be sprayed with a clear lacquer after measurements are made to preserve the appearance for future reference.
6. The filter holder must be grounded during the filtering operation to prevent an electrostatic charge from accumulating (see Ref. 2).

PRECISION

Using a reflectometer for measurement, the estimated standard deviation (esd) based on the indicated pairs is shown below. Duplicate results by the same operator should not be considered suspect unless they differ by more than the amounts shown in the “allowable difference” column (95% probability).

<u>Type of Sample</u>	<u>Number of Pairs</u>	<u>Level, % Reflectance</u>	<u>esd, % Reflectance</u>	<u>Allowable Difference, % Reflectance</u>
Fuel Oil Blends	8	40-81	2.14	7.1

The % Reflectance can be converted to a Blotter Rating by referring to the attached table.

TIME FOR ANALYSIS

The elapsed time for one analysis is 4.0 hours. The labor requirement is 1.0 hour. In groups of ten determinations, the elapsed time is 6.5 hours and the labor requirement is 0.25 hours per sample.

REFERENCES

1. “300 F Accelerated Fuel Oil Stability Test,” DuPont Method F21-61, Dec., 1962; Petroleum Chemicals Division, E. I. DuPont De Nemours Inc.
2. “Static Electricity Can Be A Fire Hazard,” F. F. Gorup, National Safety Council—Research Newsletter, Aug., 1979, page 4, Operational and Environmental Safety Division, U.S. Dept. of Energy.

SUGGESTED SUPPLIERS

Bausch & Lomb, 340 Melvin Dr., Northbrook, IL 60002
 Beckman Instruments Inc., Scientific Instruments Div., 2500 Harbor Blvd., Fullerton, CA 92634
 Blue M Electric Co., Blue Island, IL 60406
 E. I. DuPont De Nemours & Co., Inc., Petroleum Chemicals Div., Wilmington, DE 19800
 Millipore Corp., Bedford, MA 01730
 Photovolt Corp., 1115 Broadway, New York, NY 10010
 Phillips Petroleum Co., Special Products Div., Bartlesville, OK 74004
 Sargent-Welch Scientific Co., 7300 N. Linder Ave., Skokie, IL 60077

Conversion of % Reflectance to Corresponding Blotter Rating

This correlation is based on calibration of the reflectometer with the standard white ceramic plate of 74.8% reflectance as supplied by Photovolt Corp.

% Reflectance Range*	DuPont F21-61 Blotter Rating
77.0 - 81.0	1
75.7 - 76.9	2
71.7 - 75.6	3
68.9 - 71.6	4
66.2 - 68.8	5
63.7 - 66.1	6
60.9 - 63.6	7
58.2 - 60.8	8
55.6 - 58.1	9
53.0 - 55.5	10
50.1 - 52.9	11
45.5 - 50.0	12
44.9 - 45.4	13
42.9 - 44.8	14
40.0 - 42.8	15
<40.0	>15

*The % Reflectance Range was obtained from the grand average of duplicate reflectance readings on each of the new brown and black reference blotters contained in DuPont Method F21-61.