



Designation: D6748 – 02a (Reapproved 2017)

Standard Test Method for Determination of Potential Instability of Middle Distillate Fuels Caused by the Presence of Phenalenes and Phenalenones (Rapid Method by Portable Spectrophotometer)^{1,2}

This standard is issued under the fixed designation D6748; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers a procedure³ for the rapid determination of phenalenes and phenalenones in middle distillate fuels, including marine, automotive, heating, and gas turbine fuel such as those specified in Specifications **D396**, **D975**, **D2069**, and **D2880**. Phenalenes and phenalenones affect the potential instability of fuels, leading to fuel degradation products during storage, which may cause performance problems.

1.2 This test method is applicable to both dyed and undyed fuels at all points in the distribution chain from refinery to end-user. It is not applicable to fuels containing residual oil. The portable apparatus allows the whole test to be conducted on site or in a laboratory and does not require the test sample to be heated.

1.3 This test method is suitable for testing samples with a relative absorbance of up to 5.00 absorbance units (AU).

NOTE 1—The precision of the test method has been established on relative absorbance up to 1.00 AU. For relative absorbance above 1.00 AU the precision may not apply.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.14 on Stability, Cleanliness and Compatibility of Liquid Fuels.

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² This test method is being jointly developed with the Institute of Petroleum where it is designated IP 463.

³ This process is covered by US Patent 5,378,632. Interested parties are invited to submit information regarding the identification of an alternative(s) to this patented item to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 ASTM Standards:⁴

D396 Specification for Fuel Oils

D975 Specification for Diesel Fuel Oils

D2069 Specification for Marine Fuels (Withdrawn 2003)⁵

D2880 Specification for Gas Turbine Fuel Oils

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

D4306 Practice for Aviation Fuel Sample Containers for Tests Affected by Trace Contamination

D4625 Test Method for Middle Distillate Fuel Storage Stability at 43 °C (110 °F)

D5854 Practice for Mixing and Handling of Liquid Samples of Petroleum and Petroleum Products

D6468 Test Method for High Temperature Stability of Middle Distillate Fuels

E131 Terminology Relating to Molecular Spectroscopy

E275 Practice for Describing and Measuring Performance of Ultraviolet and Visible Spectrophotometers

2.2 Other Standards:⁶

Def Stan 05-50 Methods for Testing Fuels and Lubricants and Associated Products, Part 40 — Storage Stability of Diesel Fuels

⁴ For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

⁵ The last approved version of this historical standard is referenced on www.astm.org.

⁶ Available from United Kingdom Defence Standardization, Room 1138, Kentigern House, 65 Brown Street, Glasgow, G2 8EX. United Kingdom.

Def Stan 91–4 Fuel, Naval, Distillate NATO Code: F76 Joint Service, Designation DIESO F76

3. Terminology

3.1 Definitions:

3.1.1 For definitions of terms relating to absorption spectroscopy see Terminology [E131](#). Terms of particular significance are the following:

3.1.2 *radiant energy, n*—energy transmitted as electromagnetic waves.

3.1.3 *radiant power P, n*—the rate at which energy is transported in a beam of radiant energy.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *absorbance, A, n*—the logarithm to the base 10 of the reciprocal of the transmittance, *T*. In symbols:

$$A = \log_{10}(1/T) = -\log_{10}T \quad (1)$$

where:

T = transmittance as defined in [3.2.5](#).

3.2.2 *absorbance units (AU), n*—units of relative absorbance reported by the portable spectrophotometer.

3.2.3 *pathlength of test portion b, n*—the distance in mm, measured in the direction of propagation of the beam of radiant energy, between the surfaces of the portion on which the radiant energy is incident and the surface of the test portion from which it emerges.

3.2.4 *relative absorbance, n*—the difference between the absorbance (near infrared-visible band results) measured at the two wavelengths, and reported in AU.

3.2.5 *transmittance, T, n*—the ratio of the radiant power transmitted by the test portion in the test cuvette to the radiant power transmitted by the Reagent 1 control in the cuvette. Expressed in the following equation.

$$T = P_T/P_{R1} \quad (2)$$

where:

P_T = radiant power transmitted by the test portion, and

P_{R1} = the radiant power transmitted by the Reagent 1 control.

4. Summary of Test Method

4.1 A 5 mL volume of middle distillate fuel is mixed with an equal volume of an immiscible reagent solution. A second reagent is then added, the new blend mixed and allowed to settle for 30 min. for two phases to separate. The top reagent layer (darker) is then placed in the portable spectrophotometer and the relative absorbance of near infrared and visible light, at fixed single wavelengths, is measured instantaneously and automatically, and reported in AU.

4.2 When mixed with the oil sample containing phenalenes, the oxidizing Reagent 2 oxidizes the phenalenes to phenalenones which are subsequently converted to colored indolylphenalene salts by the acidic Reagent 1. The spectrophotometer measures the absorbance of light caused by the colored salts while eliminating the effect of the initial color of the fuel. The absorbance of light is a measure of the concentration of the colored salts that are formed.

5. Significance and Use

5.1 Storage stability depends on complex interactions. It varies with feedstock type and source, and the processing used. The rate of degradation may not change uniformly with temperature. Chemical reactions may lead to a change in color followed by the formation of soluble gums and insoluble sediments. Insoluble sediments may overload filters, and plug nozzles and injectors.

5.2 This test method, which does not require the test sample to be heated, uses a portable apparatus and allows tests to be carried out on site or in the laboratory to give a result within 35 min.

5.3 The potential beneficial effects of stability additives in fuels may not be recognized by this test method. Therefore, the actual storage stability of middle distillate fuels with stability additives may not be correctly indicated by these test results.

5.4 The unstable reactive compounds (phenalenes and phenalenones) detected by this test method may be present in fuels containing catalytically cracked or straight run materials and can affect the potential instability of the fuel.

5.5 If this test method is used by any party for a rapid assessment of stability, it is the responsibility of parties concerned to decide whether or not this procedure yields meaningful results.

5.6 Interpretation of results and correlation with other test methods is given in [Appendix XI](#).

6. Apparatus⁷

6.1 *Spectrophotometer*; double beam, capable of measuring absorbance at one visible wavelength in the range 600 nm to 800 nm and one near infrared wavelength in the range 800 nm to 850 nm with a spectral bandwidth of 10 nm ± 2 nm and an accuracy of ±3 nm (see [Fig. 1](#)). The display shall have a resolution of 0.01 of AU, and a repeatability of measurement of ±0.02 AU for ranges up to 1.00 AU. See Practice [E275](#).

NOTE 2—The two wavelengths used are proprietary and are not adjustable.

6.1.1 *Zero Adjustment*, a facility shall be incorporated to allow the absorbance measured at the two individual wavelengths to be set to zero when using Reagent 1 as a control.

6.1.2 *Absorbance Display*, the relative absorbance, in AU, shall be the instantaneous difference between the absorbance measured at the two wavelengths. The peak wavelength represents the absorption due to the colored indolylphenalene salts which are formed, and the baseline wavelength is for normalizing the result.

6.2 *Timer*; capable of measuring 35 min to an accuracy of ±0.2 min.

⁷ The equipment, as listed in RR:D02-1522 was used to develop the precision statement. The apparatus described in Section 6 and the reagents listed in Section 7 are both supplied by Stanhope-Seta, Chertsey, Surrey KT16 8AP, United Kingdom. To date, no other equipment has demonstrated through ASTM interlaboratory testing the ability to meet the precision of this test. This is not an endorsement or certification by ASTM International.