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Standard Test Method for Oxidation Stability of Aviation Fuels (Potential Residue Method)^{1,2}

This standard is issued under the fixed designation D873; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope Scope*

1.1 This test method³ covers the determination of the tendency of aviation reciprocating, turbine, and jet engine fuels to form gum and deposits under accelerated aging conditions. (**Warning**—This test method is not intended for determining the stability of fuel components, particularly those with a high percentage of low boiling unsaturated compounds, as these may cause explosive conditions within the apparatus.)

NOTE 1—For the measurement of the oxidation stability (induction period) of motor gasoline, refer to Test Method **D525**.

1.2 The accepted SI unit of pressure is the kilo pascal (kPa); the accepted SI unit of temperature is °C.

1.3 **WARNING**—Mercury has been designated by many regulatory agencies as a hazardous ~~material~~ substance that can cause ~~central nervous system, kidney and liver damage, serious medical issues~~. Mercury, or its vapor, ~~may have been demonstrated to be~~ hazardous to health and corrosive to materials. ~~Caution should be taken~~ Use caution when handling mercury and mercury containing mercury-containing products. See the applicable product Material Safety Data Sheet (MSDS) for details and EPA's website ~~http://www.epa.gov/mercury/faq.htm~~ for additional information. Users should be aware (SDS) for additional information. The potential exists that selling mercury and/or mercury containing products into your state or country may be prohibited by law or mercury-containing products, or both, is prohibited by local or national law. Users must determine legality of sales in their location.

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.*

1.5 *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.*

¹ This test method is under the jurisdiction of ASTM International Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of ASTM Subcommittee D02.14 on Stability, Cleanliness and Compatibility of Liquid Fuels. The technically equivalent standard as referenced is under the jurisdiction of the Energy Institute Subcommittee SC-B-8.

~~This test method has been approved by the sponsoring committees and accepted by the Cooperating Societies in accordance with established procedures.~~

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² This test method has been developed through the cooperative effort between ASTM and the Energy Institute, London. ASTM and IP standards were approved by ASTM and EI technical committees as being technically equivalent but that does not imply both standards are identical.

³ Further information can be found in the June 1978, January 1979, and June 1986 editions of the *Institute of Petroleum Review*.

*A Summary of Changes section appears at the end of this standard

2. Referenced Documents

2.1 ASTM Standards:⁴

- D381 Test Method for Gum Content in Fuels by Jet Evaporation
- D525 Test Method for Oxidation Stability of Gasoline (Induction Period Method)
- D4057 Practice for Manual Sampling of Petroleum and Petroleum Products
- D5452 Test Method for Particulate Contamination in Aviation Fuels by Laboratory Filtration
- E1 Specification for ASTM Liquid-in-Glass Thermometers

3. Terminology

3.1 The following definitions of terms are all expressed in terms of milligrams per 100 mL of sample, after “X” hours aging, “X” being the accelerated aging (oxidation) period at ~~100°C~~ 100 °C.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *insoluble gum, n*—deposit adhering to the glass sample container after removal of the aged fuel, precipitate, and soluble gum.

3.2.1.1 Discussion—

Insoluble gum is obtained by measuring the increase in mass of the glass sample container.

3.2.2 *potential gum, n*—sum of the soluble and insoluble gum.

3.2.3 *precipitate, n*—sediment and suspended material in the aged fuel, obtained by filtering the aged fuel and washings from the glass sample container.

3.2.4 *soluble gum, n*—deterioration products present at the end of a specific aging period. These deterioration products exist in solution in the aged fuel and as the toluene-acetone soluble portion of the deposit on the glass sample container.

3.2.4.1 Discussion—

The soluble gum is obtained as a nonvolatile residue by evaporating the aged fuel and the toluene-acetone washings from the glass sample container.

3.2.5 *total potential residue, n*—sum of the potential gum and the precipitate.

4. Summary of Test Method

4.1 The fuel is oxidized under prescribed conditions in a pressure vessel filled with oxygen. The amounts of soluble gum, insoluble gum, and precipitate formed are weighed. (**Warning**—In addition to other precautions, to provide protection against the possibility of explosive rupture of the pressure vessel, the pressure vessel should be operated behind an appropriate safety shield.)

5. Significance and Use

5.1 The results (of these tests) can be used to indicate storage stability of these fuels. The tendency of fuels to form gum and deposits in these tests has not been correlated with field performance (and can vary markedly) with the formation of gum and deposits under different storage conditions.

6. Apparatus

6.1 *Oxidation Pressure Vessel, Burst Disc Assembly, Glass Sample Container and Cover, Accessories and Pressure Gage*, as described in the Annex to Test Method D525. (**Warning**—Provision shall be made to safely vent any expelled gases or flames away from the operator, other personnel, or flammable materials as a safety precaution if the burst-disc ruptures.)

NOTE 2—Pressure vessels conforming to Test Method D525–80 are also suitable, but the specified burst-disc shall be attached. The burst disc assembly shall be mechanically designed to ensure that it cannot be incorrectly fitted.

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.

6.2 *Thermometer*, having a range as shown below and conforming to the requirements as prescribed in Specification E1, or specifications for IP thermometers:

Thermometer Range	ASTM	Thermometer Number	IP
95 °C to 103 °C	22C		24C

NOTE 3—Other temperature sensing devices that cover the temperature range of interest, such as thermocouples or platinum resistance thermometers, that can provide equivalent or better accuracy and precision, may be used in place of the thermometers specified in 6.2.

6.3 *Drying Oven*, air oven maintained at 100 °C to 150 °C.

6.4 *Forceps*, corrosion-resistant, steel.

6.5 *Filtering Crucible*, sintered-glass, fine porosity.

6.6 *Oxidation Bath*, as described in the Annex to Test Method D525. The liquid shall be water or a mixture of ethylene glycol and water, as required. The temperature can be controlled thermostatically at 100 °C ± 0.2 °C, or by maintaining it at its boiling point, which must be between 99.5 °C to 100.5 °C. If a liquid medium other than water is used, an appropriate mechanical stirrer/mixer shall be used to maintain uniformity of the liquid bath at 100 °C ± 0.2 °C. A non self-resettable device shall be fitted on all new baths to ensure that the heater is switched off if the liquid bath falls below a safe level. Users of older baths without this device are strongly urged to have the equipment retrofitted to ensure safe operation.

NOTE 4—Electric heating blocks are known to be used. These blocks can have heating capacities, heating rates, and heat transfer characteristics that differ from those of a liquid bath. An electric heating block may be used in place of the liquid bath as long as the sample heating rate and sample temperature are demonstrated to be equivalent to that of the liquid bath.

6.7 *Cooling Vessel*—A desiccator or other type of tightly covered vessel for cooling the beakers before weighing. The use of a drying agent is not recommended.

7. Reagents and Materials

7.1 *Gum Solvent*—A mixture of equal volumes of toluene and acetone.

7.2 *Oxygen*, commercially available extra dry oxygen of not less than 99.6 % purity.

8. Sampling

8.1 Sample in accordance with the procedure for oxidation stability, as described in Practice D4057.

9. Preparation of Apparatus

9.1 Thoroughly clean a glass sample container to remove traces of any adhering material. Immerse the container and its cover in a mildly alkaline or neutral pH laboratory detergent cleaning solution. The type of detergent and conditions for its use need to be established in each laboratory. The criterion for satisfactory cleaning shall be a matching of the quality of that obtained with chromic acid cleaning solutions (or some other equivalently strong oxidizing non-chromium containing acid cleaning solutions) on used sample containers and covers (fresh chromic acid, 6 h soaking period, rinsing with distilled water and drying). For this comparison, visual appearance and mass loss on heating the glassware under test conditions may be used. Detergent cleaning avoids the potential hazards and inconveniences related to the handling of highly corrosive and strongly oxidizing acid solutions; this procedure remains the reference cleaning practice and, as such, may function as an alternate to the preferred procedure, cleaning with detergent solutions. Remove from the cleaning solution by means of corrosion-resistant steel forceps and handle only with forceps thereafter. Wash thoroughly first with tap water and then with deionized or distilled water, and dry in an oven at 100 °C to 150 °C for 1 h. Cool the sample containers and covers for at least 2 h in the cooling vessel in the vicinity of the balance. Weigh to the nearest 0.1 mg, and record mass.

9.1.1 Experience indicates that the amount of insoluble gum is negligible in aviation reciprocating engine fuels. Therefore, the

TABLE 1 Aging Time Correction Factors

NOTE 1—To obtain the correct aging time at the operating temperature, multiply the time specified for 100 °C by the correction factor.

Temperature, °C	Correction Factor
99.5	1.06
99.6	1.04
99.7	1.03
99.8	1.02
99.9	1.01
100.0	1.00
100.1	0.99
100.2	0.98
100.3	0.97
100.4	0.96
100.5	0.95

glass sample container need not be weighed when testing such fuels unless visible evidence of insoluble matter remains in the container after treatment with gum solvent. In such cases, the test must be repeated and the mass of the container recorded.

9.2 Drain any fuel from the pressure vessel and wipe the inside of the pressure vessel and pressure vessel closure, first with a clean cloth moistened with gum solvent and then with a clean, dry cloth. Remove the filler rod from the stem, and carefully clean any gum or fuel from the stem, rod, and needle valve with gum solvent. The pressure vessel, the valve, and all connecting lines shall be thoroughly dry before each test is started. (**Warning**—Volatile peroxides, which may have formed during a previous test, may accumulate in the equipment, producing a potentially explosive environment. Special care in cleaning after each test is needed to ensure that the filler rod, stem, and needle valve are free of these peroxides.)

9.3 If a thermostatically controlled constant temperature oxidation bath is used, adjust the temperature to 100 °C ± 0.1 °C and maintain it within this temperature range for the duration of the test.

9.4 If a boiling water oxidation bath is used, adjust the temperature within the range from 99.5 °C to 100.5 °C by the addition of water or a higher boiling liquid such as ethylene glycol. Factors are given in **Table 1** to adjust the “X” hour aging time if the bath temperature at the start of the test deviates from 100 °C.

10. Procedure

10.1 Bring the pressure vessel and the fuel to be tested to a temperature from 15 °C to 25 °C. Place the weighed glass sample container in the pressure vessel and add 100 mL ± 1 mL of test specimen. Alternatively, transfer 100 mL ± 1 mL of sample into the weighed glass sample container first, before placing the glass sample container into the pressure vessel. Cover the same container, close the pressure vessel, and using a quick release coupling, introduce oxygen until a pressure from 690 kPa to 705 kPa is attained. Allow the gas in the pressure vessel to escape slowly through the needle valve at a rate not to exceed 345 kPa/min. Repeat the charging and exhausting of the oxygen once more in order to flush out the air originally present. Introduce oxygen again until a pressure of from 690 kPa to 705 kPa is attained and observe for leaks, ignoring an initial rapid drop in pressure (generally not over 40 kPa), which can be observed because of the solution of oxygen in the sample. Assume the absence of leaks, and proceed with the test if the rate of pressure drop does not exceed 15 kPa in 10 min.

10.2 Place the charged pressure vessel in one of the described oxidation baths, being careful to avoid shaking, and record the time of immersion as the starting time. Leave the pressure vessel in the oxidation bath for the specified “X” hour aging time. If the temperature at the start of a test varies from ~~100°C~~, 100 °C, adjust the “X” hour aging time by the correction factors given in **Table 1**.

10.3 At the completion of the period of oxidation, remove the pressure vessel from the bath. To minimize further oxidation of the test specimen and to provide for safe venting of the pressure vessel, cool the pressure vessel to approximately room temperature within 30 min after removal from the bath, using water ≤35 °C. Release the pressure slowly through the needle valve at a rate not to exceed 345 kPa/min. Take the pressure vessel apart, and remove the sample container.

10.4 Transfer the oxidized fuel from the glass sample container to a graduated flask, such as a graduated, stoppered cylinder, that will allow mixing of approximately 120 mL, if no visible precipitate is observed or if the amount is not specifically required by specifications. Wash the interior of the glass sample container twice with 10 mL portions of gum solvent to remove any gum. Mix