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Standard Test Method for Oxidation Stability of Aviation Fuels (Potential Residue Method)[1]

This standard is issued under the fixed designation D 873; 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 test method has been approved by the sponsoring committees and accepted by the Cooperating Societies in accordance with established procedures.

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

1. Scope

1.1 This test method[2] 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 D 525.

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

1.3 *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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 381 Test Method for Existent Gum in Fuels by Jet Evaporation[3]

D 525 Test Method for Oxidation Stability of Gasoline (Induction Period Method)[3]

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products[4]

D 5452 Test Method for Particulate Contamination in Aviation Fuels by Laboratory Filtration[5]

E 1 Specification for ASTM Thermometers[6]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard*

3.1.1 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.

3.1.1.1 *insoluble gum*—deposit adhering to the glass sample container after removal of the aged fuel, precipitate, and soluble gum. Insoluble gum is obtained by measuring the increase in mass of the glass sample container.

3.1.1.2 *potential gum*—sum of the soluble and insoluble gum.

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

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

3.1.1.5 *total potential residue*—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

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

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

[3] *Annual Book of ASTM Standards*, Vol 05.01.

[4] *Annual Book of ASTM Standards*, Vol 05.02.

[5] *Annual Book of ASTM Standards*, Vol 05.03.

[6] *Annual Book of ASTM Standards*, Vol 14.03.

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 D 525. (**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 D 525/1980 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.

6.2 *Thermometer*, having a range as shown below and conforming to the requirements as prescribed in Specification E 1, or specifications for IP thermometers:

Thermometer Range	Thermometer Number	
	ASTM	IP
95 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 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 D 525. The liquid shall be water or a mixture of ethylene glycol and water, as required. The temperature can be controlled thermostatically at 100 ± 0.2°C, or by maintaining it at its boiling point, which must be between 99.5 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 ± 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 D 4057.

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 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 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 ± 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 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 to 25°C. Place the weighed glass sample container in the pressure vessel and add 100 ± 1 mL of test specimen. Alternatively, transfer 100 ± 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 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 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, 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 the oxidized fuel and rinses thoroughly, and preserve the mixture for the determination of soluble gum. Proceed with the test as specified in 10.6. If a precipitate is observed, and if the amount is required by specifications, determine the initial mass of the filtering crucible (see 6.5) that is to be used and filter the oxidized fuel through the crucible and save the filtrate. A vacuum filtration set-up has been found suitable to use, although precautions should be taken to avoid the potential of static discharges, such as described in Test Method D 5452. Wash the interior of the glass container twice with 10-mL portions of gum solvent to remove any gum or precipitate. Filter the washings through the crucible, adding them to the oxidized fuel filtrate, and mix thoroughly. Preserve the mixture for the determination of soluble gum.

TABLE 1 Aging Time Correction Factors

NOTE—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

10.5 Dry the crucible in an oven maintained at 100 to 150°C for at least 1 h, cool in a cooling vessel to approximately room temperature (for at least 2 h), and weigh the crucible (that is, the crucible plus residue) to determine its final mass. Subtract the initial mass of the crucible from the final mass of the crucible. Record any increase in mass as precipitate, *A*.

10.6 Dry the glass sample container in an oven maintained at 100 to 150°C for 1 h, cool in a cooling vessel, and weigh. Two hours has been found to be a suitable time to cool the glass sample container. Record any increase in mass as insoluble gum, *B*.

10.7 Divide the mixture obtained in 10.4 into two equal portions (within 2 mL), and determine the soluble gum existing therein by the procedure and test conditions described in Test Method D 381, using in each test the entire half portion instead of the 50-mL test specimen specified in Test Method D 381. Record the sum of the increase in mass of the two beakers as soluble gum, *C*, as calculated in accordance with the following equation:

$$C = 1000 \times ((D-E) + (F-G) + 2(X-Y)) \tag{1}$$

where:

C = soluble gum, mg/100 mL,
D = mass of test specimen beaker 1 + residue, g,
E = mass of test specimen beaker 1, g,
F = mass of test specimen beaker 2 + residue, g,
G = mass of test specimen beaker 2, g,
X = mass of tare beaker (before), g, and
Y = mass of tare beaker (after), g.

11. Report

11.1 Calculate the results obtained by combining the separately determined residues as prescribed in Table 2 and report as "*X*" hour aging characteristics, Test Method D 873 as x mg/100 mL or < 1mg/100 mL.

12. Precision and Bias

12.1 The precision of the test method as determined by the statistical examination of interlaboratory test results is as follows:

12.1.1 *Repeatability*—The difference between successive results obtained by the same operator with the same apparatus under constant operating conditions on identical test material

TABLE 2 Aging Characteristics

Aging Characteristic to be Reported	Residue Fractions to be Combined	
	Aviation Reciprocating Engine Fuel	Aviation Turbine Fuel
Potential gum, mg/100 mL	sum of insoluble gum and soluble gum (*B* + *C*)	...
Precipitate, mg/100 mL	precipitate (*A*) if required	...
Total potential residue, mg/100 mL	...	sum of precipitate *A* (if required) insoluble gum *B* and soluble gum *C* (*A* + *B* + *C*)

would, in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in twenty:

	Repeatability (16 h aging)	
	Aviation Reciprocating Engine Fuel	Aviation Turbine Fuel
Potential Gum, mg/100 mL		
Up to 5	2	2
Over 5 to 10	3	3
Over 10 to 20	4	5
Precipitate, mg/100 mL		
Up to 2	1	

12.1.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would, in the long run, exceed the following values only in one case in twenty:

	Reproducibility (16 h aging)	
	Aviation Reciprocating Engine Fuel	Aviation Turbine Fuel
Potential Gum, mg/100 mL		
Up to 5	3	4
Over 5 to 10	4	5
Over 10 to 20	6	7
Precipitate, mg/100 mL		
Up to 2	1	

12.2 *Bias*—Since there is no accepted reference material available for determining the bias for the procedure in Test Method D 873 for measuring oxidation stability, bias has not been determined.

13. Keywords

13.1 aviation fuels; gum (insoluble, soluble, potential); oxidation stability; potential residue; total potential residue

SUMMARY OF CHANGES

Committee D02.14 has identified the location of selected changes to this standard since the last issue (D 873–99a) that may impact the use of this standard.

(1) Added Test Method D 5452 to Section 2, Referenced Documents.
(2) Updated 10.4 to explicitly state that an initial mass of the filtering crucible is needed when a precipitate is observed in order to determine the increase in mass of the precipitate in 10.5. Identified a suitable filtration set-up that could be used and alerted users of a potential safety concern.
(3) Updated 10.5 regarding the drying time and the precipitate calculation.