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An American National Standard

Designation 323/99

Standard Test Method for Thermal Oxidation Stability of Aviation Turbine Fuels[1]

This standard is issued under the fixed designation D3241; 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 Department of Defense.

1. Scope*

1.1 This test method covers the procedure for rating the tendencies of gas turbine fuels to deposit decomposition products within the fuel system.

1.2 The differential pressure values in mm Hg are defined only in terms of this test method.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

~~1.4~~

1.4 **WARNING**—Mercury has been designated by many regulatory agencies as a hazardous material that can cause central nervous system, kidney and liver damage. Mercury, or its vapor, may be hazardous to health and corrosive to materials. Caution should be taken when handling mercury and 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 that selling mercury and/or mercury containing products into your state or country may be prohibited by law.

1.5 *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.* For specific warning statements, see 6.1.1, 7.2, 7.2.1, 7.3, 11.1.1, and Annex A3.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1655 Specification for Aviation Turbine Fuels

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

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

2.2 *ISO Standards:*[3]

ISO 3274 Geometrical Product Specifications (GPS)—Surface Texture: Profile Method—Nominal Characteristics Of Contact (Stylus) Instruments

ISO 4288 Geometrical Product Specifications (GPS)—Surface Texture: Profile Method—Rules And Procedures For The Assessment Of Surface Texture

2.3 *ASTM Adjuncts:*[4]

Color Standard for Tube Deposit Rating

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *deposits*—oxidative products laid down on the test area of the heater tube or caught in the test filter, or both.

[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.J0.03 on Combustion and Thermal Properties.

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[2] 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.

[3] Available from International Organization for Standardization (ISO), 1, ch. de la Voie-Creuse, CP 56, CH-1211 Geneva 20, Switzerland, http://www.iso.org.

[4] Available from ASTM International Headquarters. Order Adjunct No. ADJD3241. Original adjunct produced in 1986.

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

3.1.1.1 *Discussion*—Fuel deposits will tend to predominate at the hottest portion of the heater tube, which is between the 30-mm and 50-mm position.

3.1.2 *heater tube*—an aluminum coupon controlled at elevated temperature, over which the test fuel is pumped.

3.1.2.1 *Discussion*—The tube is resistively heated and controlled in temperature by a thermocouple positioned inside. The critical test area is the thinner portion, 60 mm in length, between the shoulders of the tube. Fuel inlet to the tube is at the 0-mm position, and fuel exit is at 60 mm.

3.2 *Abbreviations:*

3.2.1 ΔP—differential pressure.

4. Summary of Test Method

4.1 This test method for measuring the high temperature stability of gas turbine fuels uses an instrument that subjects the test fuel to conditions that can be related to those occurring in gas turbine engine fuel systems. The fuel is pumped at a fixed volumetric flow rate through a heater, after which it enters a precision stainless steel filter where fuel degradation products may become trapped.

4.1.1 The apparatus uses 450 mL of test fuel ideally during a 2.5-h test. The essential data derived are the amount of deposits on an aluminum heater tube, and the rate of plugging of a 17 μm nominal porosity precision filter located just downstream of the heater tube.

5. Significance and Use

5.1 The test results are indicative of fuel performance during gas turbine operation and can be used to assess the level of deposits that form when liquid fuel contacts a heated surface that is at a specified temperature.

6. Apparatus

6.1 *Aviation Fuel Thermal Oxidation Stability Tester*[5]—~~Five~~—Six models of suitable equipment may be used as indicated in Table 1.

6.1.1 ~~Portions of this test may be automated. Refer to the appropriate user manual for the instrument model to be used for a description of detailed procedure. A manual is provided with each test rig, and the latest version of each manual is on file at ASTM as a Research Report.~~[5] ~~(~~ **Portions of this test may be automated. Refer to the appropriate user manual for the instrument model to be used for a description of detailed procedure. A manual is provided with each test rig. (Warning—No attempt should be made to operate the instrument without first becoming acquainted with all components and the function of each.)**

6.1.2 Certain operational parameters used with the instrument are critically important to achieve consistent and correct results. These are listed in Table 2.

6.2 *Heater Tube Deposit Rating Apparatus*:

6.2.1 *Visual Tube Rater*, the tuberator described in Annex A1.

6.3 Because jet fuel thermal oxidation stability is defined only in terms of this test method, which depends upon, and is inseparable from, the specific equipment used, the test method shall be conducted with the equipment used to develop the test method or equivalent equipment.

[5] The following equipment, as described in Table 1 ~~and RR:D02-1395, RR:D02-1396, and RR:D02-1397, was used to develop this test method, as provided by PAC, 8824 Fallbrook Drive, Houston, TX 77064. This is not an endorsement or certification by ASTM International.~~ and RR:D02-1309, was used to develop this test method. The following equipment, as described in Table 1 and determined as equivalent in testing as detailed in RR:D02-1631, is provided by PAC, 8824 Fallbrook Drive, Houston, TX 77064. The following equipment, as described in Table 1 and determined as equivalent in testing as detailed in RR:D02-1728, is provided by Falex Corporation, 1020 Airpark Dr., Sugar Grove, IL, 60554-9585. This is not an endorsement or certification by ASTM International.

TABLE 1 Instrument Models

Instrument Model	~~User Manual~~	Pressurize With	~~Pump~~ Principle	Differential Pressure ~~B~~ by
202	~~202/203~~[A]	nitrogen	gear	Hg Manometer; No Record
203	~~202/203~~[A]	nitrogen	gear	Manometer + Graphical Record
~~215~~	~~215~~[B]	nitrogen	gear	Transducer + Printed Record
215	~~215~~[A]	nitrogen	gear	Transducer + Printed Record
~~230~~	~~230/240~~[C]	hydraulic	syringe	Transducer + Printout
230	~~230/240~~[A]	hydraulic	syringe	Transducer + Printout
~~240~~	~~230/240~~[C]	hydraulic	syringe	Transducer + Printout
240	~~230/240~~[A]	hydraulic	syringe	Transducer + Printout
~~230 Mk III~~	~~23hydraulic~~	~~dual piston (HPLC Type)~~	~~Transducer + Printout~~	
230 Mk III[B]	hydraulic	dual piston (HPLC Type)	Transducer + Printout	
~~F40 Mk III~~[D]	~~hydraulic~~	~~dual piston (HPLC Type)~~	~~Transducer + Printout~~	
F400[C]	hydraulic	dual piston (HPLC Type)	Transducer + Printout	

[A] ~~Available from ASTM International Headquarters. Request~~ RR:D02-13095.
[B] ~~Available from ASTM International Headquarters. Request~~ RR:D02-139631.
[C] ~~Available from ASTM International Headquarters. Request~~ RR:D02-1397.
~~[D] Available from ASTM International Headquarters. Request~~ RR:D02-16318.

TABLE 2 Critical Operating Characteristics of D3241 Instruments

Item	Definition	
Test apparatus	Tube-in-shell heat exchanger as illustrated in Fig. 1.	
Test coupons		
Heater tube [A,B,C]	Specially fabricated aluminum tube that produces controlled heated test surface; new one for each test	
Tube identification	Each heater tube may be physically identified with a unique serial number, identifying the manufacturer and providing traceability to the original material batch	
Tube metallurgy	6061-T6 Aluminum, plus the following criteria a) The Mg:Si ratio shall not exceed 1.9:1 b) The Mg_2Si percentage shall not exceed 1.85 %	
Tube dimensions	Dimension	Tolerance
Tube length, mm	161.925	±0.254
Center section length, mm	60.325	±0.051
Outside diameters, mm		
Shoulders	4.724	±0.025
Center section	3.175	±0.051
Inside diameter, mm	1.651	±0.051
Total indicator runout, mm, max	0.013	
Mechanical surface finish, nm, in accordance with ISO 3274 and ISO 4288 using the mean of four 1.25–mmmeasurements	50 ± 20	
Test filter [5]	nominal 17-µm stainless steel mesh filter element to trap deposits; new one for each test	
Instrument parameters		
Sample volume	600 mL of sample is aerated, then this aerated fuel is used to fill the reservoir leaving space for the piston; 450 ± 45 mL may be pumped in a valid test	
Aeration rate	1.5 L/min dry air through sparger	
Flow during test	3.0 ± 10 % mL/min (2.7 min to 3.3 max)	
Pump mechanism	positive displacement, gear or piston syringe	
Cooling	bus bars fluid cooled to maintain consistent tube temperature profile	
Thermocouple (TC)	Type J, fiber braid or Iconel sheathed, or Type K, Iconel sheathed	
Operating pressure		
System	3.45 MPa ± 10 % on sample by pressurized inert gas (nitrogen) or by hydraulically transmitted force against control valve outlet restriction	
At test filter	differential pressure (ΔP) measured across test filter (by mercury manometer or by electronic transducer) in mm Hg	
Operating temperature		
For test	as stated in specification for fuel	
Uniformity of run	maximum deviation of ±2°C from specified temperature	
Calibration	pure tin at 232°C (and for Models 230 and 240 only, pure lead at 327°C for high point and ice + water for low point reference)	

[A] The following equipment, heater tubes, manufactured by PAC, 8824 Fallbrook Drive, Houston, TX 77064, was used in the development of this test method. This is not an endorsement or certification by ASTM International.

[B] A test protocol to establish equivalence of heater tubes is on file at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1550.

[C] The following equipment, heater tube and filter kits, manufactured by Falex Corporation, 1020 Airpark Dr., Sugar Grove, IL, 60554-9585, was run through the test protocol in RR:D02-1550 and determined as equivalent to the equipment used to develop the test method. This test is detailed in RR:D02-1714. This is not an endorsement or certification by ASTM International.

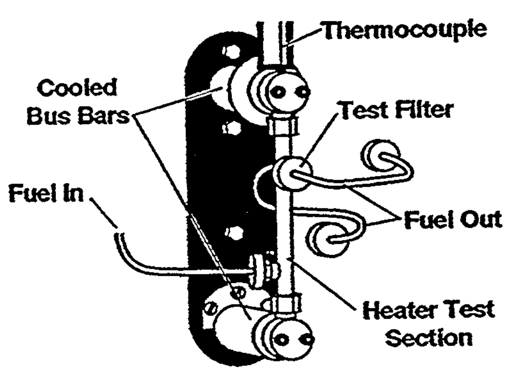

FIG. 1 Standard Heater Section, Essential to All D3241 Test Instruments

7. Reagents and Materials

7.1 Use distilled (preferred) or deionized water in the spent sample reservoir as required for Model 230 and 240 instruments.

7.2 Use methyl pentane, 2,2,4-trimethylpentane, or n-heptane (technical grade, 95 mol % minimum purity) as general cleaning solvent. This solvent will effectively clean internal metal surfaces of apparatus before a test, especially those surfaces (before the test section) that contact fresh sample. (**Warning** —Extremely flammable. Harmful if inhaled (see Annex A3).)

7.2.1 Use trisolvent (equal mix of acetone (*1*), toluene (*2*), and isopropanol (*3*)) as a specific solvent to clean internal (working) surface of test section only. (**Warning**—(*1*) Extremely flammable, vapors may cause flash fire; (*2*) and (*3*) Flammable. Vapors of all three harmful. Irritating to skin, eyes, and mucous membranes.)

7.3 Use dry calcium sulfate + cobalt chloride granules (97 + 3 mix) in the aeration dryer. This granular material changes gradually from blue to pink color indicating absorption of water. (**Warning**—Do not inhale dust or ingest. May cause stomach disorder.)

8. Standard Operating Conditions

8.1 Standard conditions of the test method are as follows:

8.1.1 *Fuel Quantity*, 450-mL minimum for test + about 50 mL for system.

8.1.2 *Fuel Pre-Treatment*—Filtration through a single layer of general purpose, retentive, qualitative filter paper followed by a 6-min aeration at 1.5 L/min air flow rate for a maximum of 1000 mL sample using a coarse 12-mm borosilicate glass gas dispersion tube.

8.1.3 *Fuel System Pressure*, 3.45 MPa (500 psi) ±10 % gauge.

8.1.4 *Thermocouple Position*, at 39 mm.

8.1.5 *Fuel System Prefilter Element*, filter paper of 0.45-µm pore size.

8.1.6 *Heater Tube Control Temperature*, preset as specified in applicable specification.

8.1.7 *Fuel Flow Rate*, 3.0 mL/min ± 10 %.

8.1.8 *Minimum Fuel Pumped During Test*, 405 mL.

8.1.9 *Test Duration*, 150 ± 2 min.

8.1.10 *Cooling Fluid Flow*, approximately 39 L/h, or center of green range on cooling fluid meter.

8.1.11 *Power Setting*, approximately 75 to 100 on non-computer models; internally set for computer models.

9. Preparation of Apparatus

9.1 *Cleaning and Assembly of Heater Test Section*:

9.1.1 Clean the inside surface of the heater test section using a nylon brush saturated with trisolvent material to remove all deposits.

9.1.2 Check the heater tube to be used in the test for surface defects and straightness by referring to the procedure in Annex A1.10. Be careful, also, to avoid scratching tube shoulder during the examination, since the tube shoulder must be smooth to ensure a seal under the flow conditions of the test.

9.1.3 Assemble the heater section using new items: (*1*) visually checked heater tube, (*2*) test filter, and (*3*) three O-rings. Inspect insulators to be sure they are undamaged.

NOTE 1—Heater tubes must not be reused. Tests indicate that magnesium migrates to the heater tube surface under normal test conditions. Surface magnesium may reduce adhesion of deposits to reused heater tube.

9.1.4 During assembly of heater section, handle tube carefully so as not to touch center part of tube. IF CENTER OF HEATER TUBE IS TOUCHED, REJECT THE TUBE SINCE THE CONTAMINATED SURFACE MAY AFFECT THE DEPOSIT-FORMING CHARACTERISTICS OF THE TUBE.

9.2 *Cleaning and Assembly of Remainder of Test Components*:

9.2.1 Perform the following steps in the order shown prior to running a subsequent test.

NOTE 2—It is assumed that the apparatus has been disassembled from previous test (see Annex A2 or appropriate user manual for assembly/disassembly details).

9.2.2 Inspect and clean components that contact test sample and replace any seals that are faulty or suspect especially the (*1*) lip seal on piston, and (*2*) O-rings on the reservoir cover, lines, and prefilter cover.

9.2.3 Install prepared heater section (as described in 9.1.1-9.1.4).

9.2.4 Assemble pre-filter with new element and install.

9.2.5 Check thermocouple for correct reference position, then lower into standard operating position.

9.2.6 On Models 230 and 240, make sure the water beaker is empty.

10. Calibration and Standardization Procedure

10.1 Perform checks of key components at the frequency indicated in the following (see Annexes or user manual for details).

10.1.1 *Thermocouple*—Calibrate a thermocouple when first installed and then normally every 30 to 50 tests thereafter, but at least every 6 months (see A2.2.8).

10.1.2 *Differential Pressure Cell*—Standardize once a year or when installing a new cell (see A2.2.6).
10.1.3 *Aeration Dryer*—Check at least monthly and change if color indicates significant absorption of water (see 7.3).
10.1.4 *Metering Pump*—Perform two checks of flow rate for each test as described in Section 11.
10.1.5 *Filter Bypass Valve*—For Models 202, 203, and 215, check for leakage at least once a year (see X1.6).

11. Procedure

11.1 *Preparation of Fuel Test Sample*:
11.1.1 Filter and aerate sample using standard operating conditions (see A2.2.9). (**Warning** —All jet fuels must be considered flammable except JP5 and JP7. Vapors are harmful (see A3.3, A3.6, and A3.7).)

NOTE 3—Before operating, see **Warning** in 6.1.1.
NOTE 4—Test method results are known to be sensitive to trace contamination from sampling containers. For recommended containers, refer to Practice D4306.

11.1.2 Maintain temperature of sample between 15°C and 32°C during aeration. Put reservoir containing sample into hot or cold water bath to change temperature, if necessary.
11.1.3 Allow no more than 1 h to elapse between the end of aeration and the start of the heating of the sample.
11.2 *Final Assembly*:
11.2.1 Assemble the reservoir section (see User Manual).
11.2.2 Install reservoir and connect lines appropriate to the instrument model being used (see User Manual).
11.2.3 Remove protective cap and connect fuel outlet line to heater section. Do this quickly to minimize loss of fuel.
11.2.4 Check all lines to ensure tightness.
11.2.5 Recheck thermocouple position at 39 mm.
11.2.6 Make sure drip receiver is empty (Models 230 and 240 only).
11.3 *Power Up and Pressurization* :
11.3.1 Turn POWER to ON.
11.3.2 Energize the ΔP alarms on models with manual alarm switch (Models 202, 203, and 215).
11.3.3 Pressurize the system slowly to about 3.45 MPa as directed in the User Manuals for Models 202, 203, and 215 (see also A2.2.5).
11.3.4 Inspect the system for leaks. Depressurize the system as necessary to tighten any leaking fittings.
11.3.5 Set controls to the standard operating conditions.
11.3.6 Use a heater tube control temperature as specified for the fuel being tested. Apply any thermocouple correction from the most recent calibration (see A2.2.8).

NOTE 5—The test can be run to a maximum tube temperature of about 350°C. The temperature at which the test should be run and the criteria for judging results are normally embodied in fuel specifications.

11.4 *Start Up*:
11.4.1 Use procedure for each model as described in the appropriate User Manual.
11.4.2 Some instrument models may do the following steps automatically, but verify that:
11.4.2.1 No more than 1 h maximum elapses from aeration to start of heating.
11.4.2.2 The manometer bypass valve is closed as soon as the heater tube temperature reaches the test level, so fuel flows through the test filter (see A2.2.6).
11.4.2.3 Manometer is set to zero (see A2.2.6).
11.4.3 Check fuel flow rate against Standard Operating Conditions by timing flow or counting the drip rate during first 15 min of test. (See X1.5.)

NOTE 6—When counting drop rate, the first drop is counted as drop 0, and time is started. As drop 20 falls, total time is noted.

11.5 *Test*:
11.5.1 Record filter pressure drop every 30 min minimum during the test period.
11.5.2 If the filter pressure drop begins to rise sharply and it is desired to run a full 150-min test, a bypass valve common to all models must be opened in order to finish the test. See appropriate User Manual for details on operation of the bypass system (see A2.2.2).
11.5.3 Make another flow check within final 15 min before shutdown (see 11.4.3 and accompanying note). (See X1.5.)
11.6 *Heater Tube Profile*—If a heater tube temperature profile is desired, obtain as described in X1.4.
11.7 *Shutdown*:
11.7.1 For Models 202, 203, and 215 only:
11.7.1.1 Switch HEATER, then PUMP to OFF.
11.7.1.2 Close NITROGEN PRESSURE VALVE and open MANUAL BYPASS VALVE.
11.7.1.3 Open NITROGEN BLEED VALVE slowly, if used, to allow system pressure to decrease at an approximate rate of 0.15 MPa/s.
11.7.2 Models 230 and 240 shut down automatically.

11.7.2.1 After shutdown, turn FLOW SELECTOR VALVE to VENT to relieve pressure.

11.7.2.2 Piston actuator will retreat automatically.

11.7.2.3 Measure effluent in drip receiver, then empty.

11.8 *Disassembly*:

11.8.1 Disconnect fuel inlet line to the heater section and cap to prevent fuel leakage from reservoir.

11.8.2 Disconnect heater section.

11.8.2.1 Remove heater tube from heater section carefully so as to avoid touching center part of tube, and discard test filter.

11.8.2.2 Flush tube with recommended general cleaning solvent (see 7.2) from top down. If the tube is grasped from the top, do not wash solvent over gloves or bare fingers. Allow to dry, return tube to original container, mark with identification and hold for evaluation.

11.8.3 Disconnect reservoir.

11.8.3.1 Measure the amount of spent fluid pumped during the test, and reject the test if the amount is less than 405 mL.

11.8.3.2 Discard fuel to waste disposal.

12. Heater Tube Evaluation

12.1 Visually rate the deposits on heater tube in accordance with Annex A1.

12.2 Return tube to original container, record data, and retain tube for visual record as appropriate.

13. Report

13.1 Report the following information:

13.1.1 The heater tube control temperature. This is the test temperature of the fuel.

13.1.2 Heater tube deposit rating(s).

13.1.3 Maximum pressure drop across the filter during the test or the time required to reach a pressure differential of 25 mm Hg. For the Model 202, 203 models, report the maximum recorded ΔP found during the test.

13.1.4 If the normal 150-min test time was not completed, for example, if the test is terminated because of pressure drop failure, also report the test time that corresponds to this heater tube deposit rating.

NOTE 7—Either the tube rating or the ΔP criteria, or both, are used to determine whether a fuel sample passes or fails the test at a specified test temperature.

13.1.5 Spent fuel at the end of a normal test. This will be the amount on top of floating piston or total fluid in displaced water beaker, depending on model of instrument used.

13.1.6 Heater tube serial number may be reported.

14. Precision and Bias

14.1 An interlaboratory study of oxidative stability testing was conducted in accordance with Practice E691 by eleven laboratories, using thirteen instruments including two models with five fuels at two temperatures for a total of ten materials. Each laboratory obtained two results from each material.[6]

14.1.1 The terms repeatability and reproducibility in this section are used as specified in Practice E177.

14.2 *Precision*—It is not possible to specify the precision of this test method because it has been determined that test method results cannot be analyzed by standard statistical methodology.

14.3 *Bias*—This test method has no bias because jet fuel thermal oxidative stability is defined only in terms of this test method.

15. Keywords

15.1 differential pressure; fuel decomposition; oxidative deposits; test filter deposits; thermal stability; turbine fuel

ANNEXES

(Mandatory Information)

A1. TEST METHOD FOR VISUAL RATING OF D3241 HEATER TUBES

A1.1 Scope

A1.1.1 This method covers a procedure for visually rating the heater tube produced by Test Method D3241.

A1.1.2 The final result from this test method is a tube color rating based on an arbitrary scale established for this test method plus two additional yes/no criteria that indicate the presence of an apparent large excess of deposit or an unusual deposit, or both.

[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1309.