Designation: D3241 – 14^ε1

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 U.S. Department of Defense.

ε[1] NOTE—A Research Report was added editorially to Annex A3 in August 2014.

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 **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 A5.

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

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

[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.J0.03 on Combustion and Thermal Properties.

Current edition approved May 1, 2014. Published July 2014. Originally approved in 1973. Last previous edition approved in 2013 as D3241 – 13. DOI: 10.1520/D3241-14E01.

[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**

3.1.2.1 *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]—Eight 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. (**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.

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 A5).)

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

[5] The following equipment, as described in Table 1 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	Pressurize With	Principle	Differential Pressure by
202[A]	nitrogen	gear	Hg Manometer; No Record
203[A]	nitrogen	gear	Manometer + Graphical Record
215[A]	nitrogen	gear	Transducer + Printed Record
230[A]	hydraulic	syringe	Transducer + Printout
240[A]	hydraulic	syringe	Transducer + Printout
230 Mk III[B]	hydraulic	dual piston (HPLC Type)	Transducer + Printout
F400[C]	hydraulic	dual piston (HPLC Type)	Transducer + Printout
230 Mk IV[D]	hydraulic	single piston (HPLC Type)	Transducer + Printout

[A] See RR:D02-1309.
[B] See RR:D02-1631.
[C] See RR:D02-1728.
[D] See RR:D02-1757.

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. An electronic recording device, such as a radio-frequency identification device (RFID), may be embedded into the heater tube rivet located at the bottom of the heater tube.	
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. This data may be stored on an electronic recording device, such as a RFID, embedded into the heater tube.	
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–measurements	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.

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:*

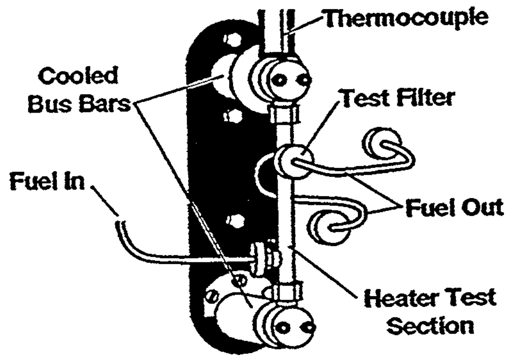

FIG. 1 Standard Heater Section, Essential to All D3241 Test Instruments

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 A4 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 A4.2.8).

10.1.2 *Differential Pressure Cell*—Standardize once a year or when installing a new cell (see A4.2.6).

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 A4.2.9). (**Warning** —All jet fuels must be considered flammable except JP5 and JP7. Vapors are harmful (see A5.3, A5.6, and A5.7).)

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 A4.2.5).

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 A4.2.8).

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 A4.2.6).

11.4.2.3 Manometer is set to zero (see A4.2.6).

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 A4.2.2).

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

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

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.

A1.2. Referenced Documents

A1.2.1 *Adjunct:*[4]
Color Standard for Tube Deposit Rating

A1.3. Terminology

A1.3.1 *abnormal*—a tube deposit color that is neither peacock nor like those of the Color Standard.

A1.3.1.1 *Discussion*—This refers to deposit colors such as blues and grays that do not match the Color Standard.

A1.3.2 *peacock*—A multicolor, rainbow-like tube deposit.

A1.3.2.1 *Discussion*—This type of deposit is caused by interference phenomena where deposit thickness exceeds the quarter wave length of visible light.

A1.3.3 *Tube Rating*—A ten-step discrete scale from 0 to >4 with intermediate levels for each number starting with 1 described as less than the subsequent number.

A1.3.3.1 *Discussion*—The scale is taken from the five colors—0, 1, 2, 3, 4—on the ASTM Color Standard. The complete scale is: 0, <1, 1, <2, 2, <3, 3,< 4, 4, >4. Each step is not necessarily of the same absolute magnitude. The higher the number, the darker the deposit rating.

A1.4. Summary of Test Method

A1.4.1 This test method uses a specially constructed light box to view the heater tube. The tube is positioned in the box using a special tube holder. Uniformity of the new tube surface is judged under the optimum light conditions of the box. Color of the tube is judged under light and magnification by comparing to the Color Standard plate slid into optimum position immediately behind the tube.

A1.5. Significance and Use

A1.5.1 The final tube rating is assumed to be an estimate of condition of the degraded fuel deposit on the tube. This rating is one basis for judging the thermal oxidative stability of the fuel sample.

A1.6. Apparatus

A1.6.1 *Heater Tube Deposit Rating Apparatus*—The colors of deposits on the heater tube are rated by using a tuberator and the ASTM Color Standard.

A1.7. Test Samples (Coupons)

A1.7.1 Handle the heater tube coupon carefully so as not to touch the center portion at any time.

NOTE A1.1—Touching the center of the coupon will likely contaminate or disturb the surface of the tube, deposit, or both, which must be evaluated in pristine condition.

A1.8 Standard Operating Conditions

A1.8.1 *Inside of Light Box,* opaque black.

A1.8.2 *Light Source,* three 30 W incandescent bulbs, clear, reflective type; all shall be working for optimum viewing.

A1.8.3 *Bulb Positions,* one above, two below, each directed toward tube holder and color standard.

A1.8.4 *Magnification,* 2×, covering viewing window.

A1.8.5 *Evaluators*—Use persons who can judge colors, that is, they should not be color blind.

A1.9. Calibration and Standardization

A1.9.1 No standardization is required for this test apparatus, but since the Color Standard is known to fade, store it in a dark place.

NOTE A1.2—The lifetime of the Color Standard is not established when continuously or intermittently exposed to light. It is good practice to keep a separate Standard in dark (no light) storage for periodic comparison with the Standard in regular use. When comparing, the optimum under the light conditions are those of the tube rating box.

A1.9.2 *Standardization of Rating Technique:*

A1.9.2.1 In rating a tube, the darkest deposits are most important. Estimate grades for the darkest uniform deposit, not for the overall average color of the deposit area.

A1.9.2.2 When grading, consider only the darkest continuous color that covers an area equal or larger than a circle of size one-half the diameter of the tube.

A1.9.2.3 Ignore a deposit streak that is less in width than one-quarter the diameter of the tube regardless of the length of the streak.

A1.9.2.4 Ignore spots, streaks, or scratches on a tube that are considered tube defects. These will normally not be present, since the tube is examined before use to eliminate defective tubes.

A1.10 Pretest Rating of Tubes

A1.10.1 Examine the tube without magnification in laboratory light. If a defect is visible, discard the tube. Then examine the center (thinner area) of the tube between 5 and 55 mm above the bottom shoulder using the Tuberator. If a defect is seen, establish its size. If it is larger than 2.5 mm^2, discard the tube. Fig. A1.1 provides an illustration of defect areas equivalent to 2.5 mm^2.

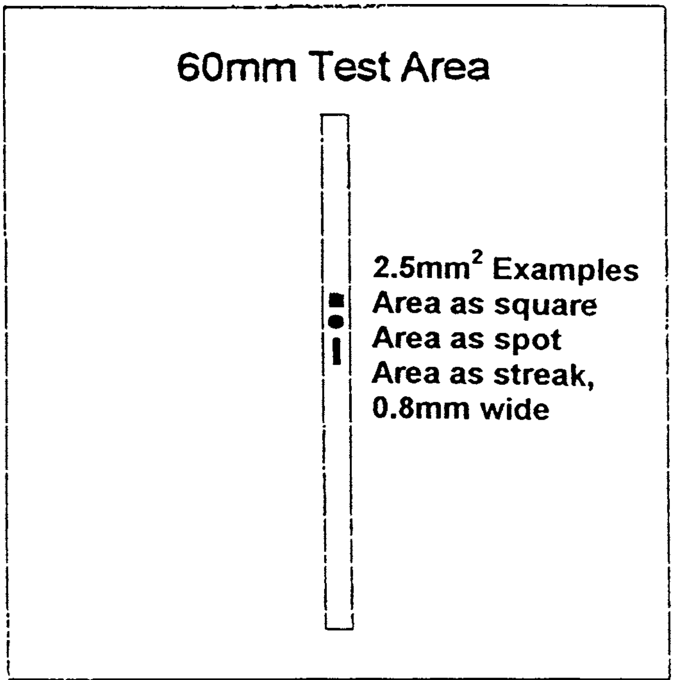

FIG. A1.1 Defect Areas

A1.10.2 Examine the tube for straightness by rolling the tube on a flat surface and noting the gap between the flat surface and the center section. Reject any bent tube.

A1.11. Procedure

A1.11.1 *Set Up:*

A1.11.1.1 Snap the upper end of the heater tube into the clamp of the holder for the heater tube.

A1.11.1.2 Push the heater tube against the stop of the holder for the heater tube.

A1.11.1.3 Slide the holder with the heater tube over the guide rod into the tuberator.

A1.11.1.4 Rotate the holder and position the heater tube such that the side with the darkest deposit is visible.

A1.11.1.5 Insert the ASTM Color Standard into the tuberator.

A1.11.2 *Evaluation:*

A1.11.2.1 On completion of the test, compare the darkest heater tube deposit color, between 5 and 55 mm above the bottom shoulder, with the ASTM Color Standard. Only rate a deposit if the area is greater than 2.5 mm^2 and the width of any streak or spot is greater than 0.8 mm. Fig. A1.1 provides an illustration of spots or streaks with an area equivalent to 2.5 mm^2.

A1.11.2.2 When the darkest deposit color corresponds to a color standard, that number should be recorded.

A1.11.2.3 If the darkest heater tube deposit color being rated is in the obvious transition state between any two adjacent color standards, the rating should be recorded as less than the darker (that is, higher number) standard.

A1.11.2.4 In the event the heater tube has deposits which do not match the normal Color Standard colors, use the following rules for rating. With reference to standard terms:

(*1*) If the deposit is peacock color, rate this as Code P, but also rate any deposit that shows normal deposit color; or

(*2*) If the deposit contains an abnormal color, rate this as Code A, but also rate any deposit that shows normal deposit color.

A1.11.3 Remove the rated heater tube and return to its original container.

A1.12. Report

A1.12.1 Report the numerical rating for the heater tube plus A or P, or both, with additional description, if applicable.

A1.12.1.1 When reporting the overall rating, report the maximum rating, and, if there are colors present that do not match the Color Standard, report these also.

A1.12.1.2 If there are only P or A, or both, deposits, report only these and do not attempt to estimate a numerical grade.

A1.12.2 *Examples:*

A1.12.2.1 *Example 1*—A heater tube has a maximum deposit falling between Color Standard Codes 2 and 3 with no other colors present. The overall tube rating would be less than 3.

A1.12.2.2 *Example 2*—The darkest deposit on a tube matches a Code 3, but there is also a peacock deposit present. The overall rating of the tube would be reported as 3P.

A1.12.2.3 *Example 3*—A heater tube has a deposit that matches Color Standard Code 1 and also has an abnormal deposit. The overall tube rating would be reported as 1A.

A1.13. Precision and Bias

A1.13.1 *Precision*—The precision of the procedure in Test Method D3241 for measuring tube deposit rating is being determined.

A1.13.2 *Bias*—The procedure in Test Method D3241 for determining tube deposit rating has no bias because the value of tube deposit rating is defined only in terms of the test method.

A2. TEST METHOD FOR THICKNESS DEPOSIT RATING OF D3241 HEATER TUBES—INTERFEROMETRIC METHOD

A2.1 Scope

A2.1.1 This annex describes a procedure for the interferometric thickness deposit rating in the range of 0 nm to 1200 nm of heater tubes produced by Test Method D3241—Thermal Oxidation Stability of Aviation Turbine Fuels.

A2.1.2 The final result from this rating procedure is an absolute measurement of the thickness and volume of deposit on the heater tube that provides a basis for judging the thermal oxidative stability of the fuel sample. For aircraft fuel systems performance, deposit thickness and volume are useful parameters.

A2.1.3 An interlaboratory study was conducted in October 2011 (see ASTM Research Report RR:D02-1774[7] for supporting data) involving 8 interferometric instruments and 117 heater tubes tested in duplicate. The interferometric procedure demonstrated objective rating.

NOTE A2.1—The particular technique used for this test method is called spectral reflectance.

NOTE A2.2—If this procedure is to be used to rate the heater tube after the thermal oxidation test, the new heater tube may also be examined by the same technique to establish a base line or condition of satisfactory starting quality.

A2.2 Terminology

A2.2.1 *Definitions of Terms Specific to This Standard:*

A2.2.1.1 *deposit*—film of oxidized product deposited on the test area of the heater tube after D3241 test procedure.

A2.2.1.2 *deposit profile*—three-dimensional representation of deposit thickness profile along and around the length of the heater tube test section.

A2.2.1.3 *deposit thickness*—the thickness of deposit present on the heater tube substrate surface expressed in manometers, nm.

A2.2.1.4 *deposit volume*—the volume of deposit present on the test section of the heater tube expressed in mm^3.

A2.2.1.4.1 *Discussion*—The deposit volume is derived by integration of the area under the deposit profile.

A2.2.1.5 *interferometry*—a technique used for measuring the optical properties of surfaces (refractive index and absorption coefficient) based on studying the pattern of interference created by their superposition. In the presence of a thin transparent layer called film, interferometry can also be used to provide film thickness information.

A2.2.1.6 *standard spot*—the mean thickness of the six thickest points in a 2.5 mm^2 area, as shown in Fig. A2.6, defined in section A1.11.2.1 of this test method.

A2.3 Summary of Test Method

A2.3.1 An interferometric apparatus, as shown in Fig. A2.1, is used to rate the deposit on the heater tube. The computer-driven software analyzes the interferometric data. The deposit thickness and deposit volume are derived and displayed.

A2.4 Significance and Use

A2.4.1 The final heater tube rating is a direct thickness and volume measurement of the degraded fuel deposited on the heater tube. This rating is one basis for judging the thermal oxidative stability of the fuel sample.

A2.5 Reagents and Materials

A2.5.1 *Reference Heater Tube*[8]—with two reference deposits of known and traceable thickness made with silicon dioxide on silicon ($Si + SiO_2$). See Fig. A2.2.

[7] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1774. Contact ASTM Customer Service at service@astm.org.

[8] The sole source of supply of the reference heater tube known to the committee at this time is AD systems (www.adsystems-sa.com), available from AD systems, P.A. Portes de la Suisse Normande, Allée de Cindais, 14320 Saint André sur Orne, France. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend

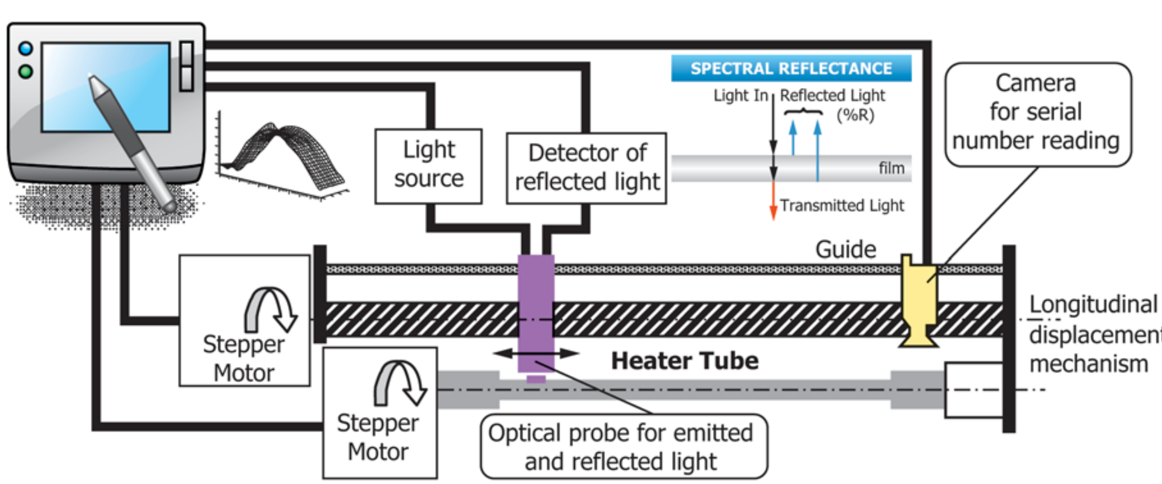

FIG. A2.1 Interferometric Apparatus—General Principle

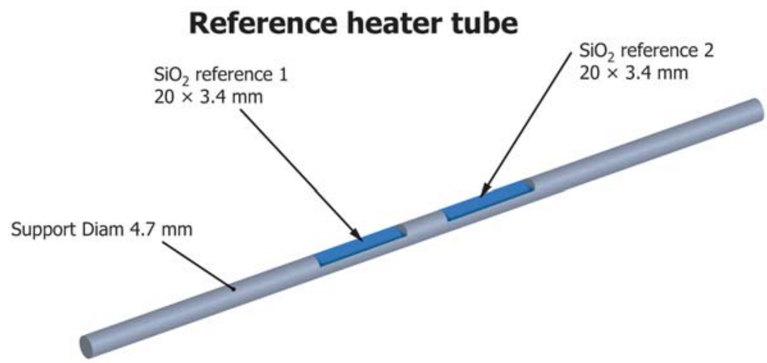

FIG. A2.2 Reference Heater Tube

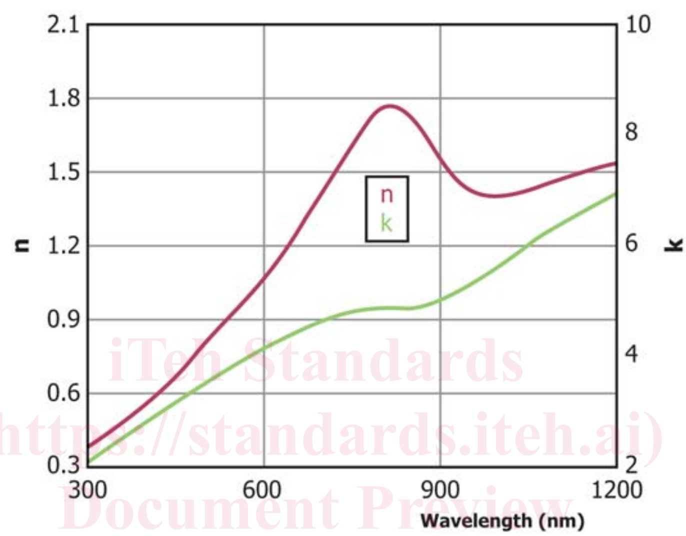

FIG. A2.3 n and k Values of the Heater Tube

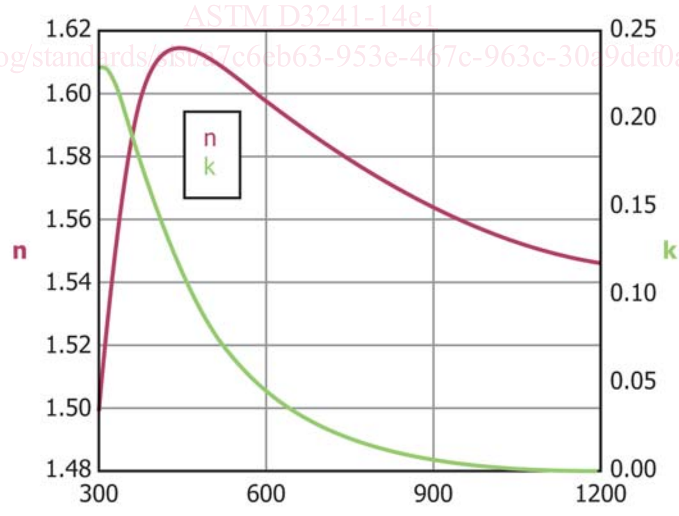

FIG. A2.4 n and K Values of the Heater Tube

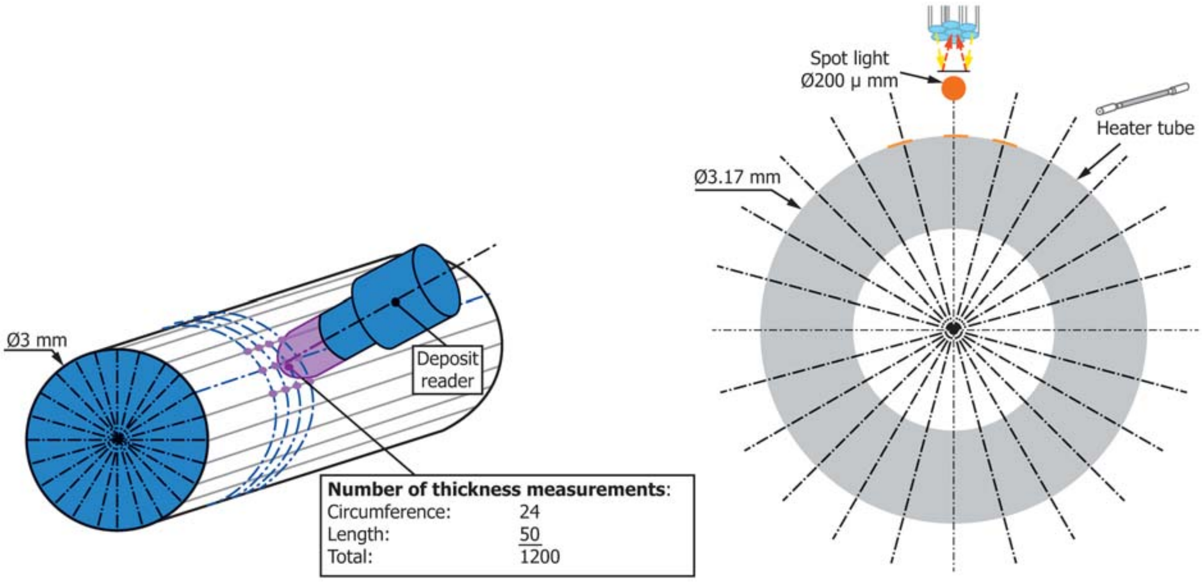

FIG. A2.5 Circumferential Resolution

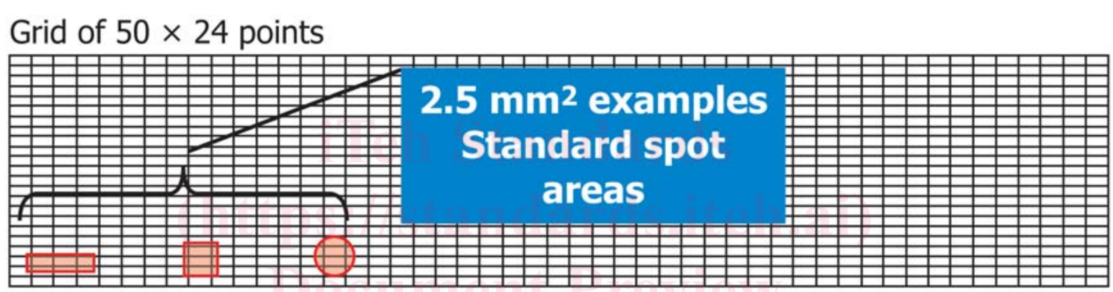

FIG. A2.6 Standard Spot

A2.6 Apparatus

A2.6.1 *Deposit Rater:*[9]

A2.6.1.1 Comprising of a suitable UVVIS light source (200 to 1100 nm), reflected light probe capable to generate a spot light of 200 µm diameter, detector of reflected light for measuring light interferences, heater tube handling assembly, heater tube rotating system, optical probe displacement system and computer-driven software for analyzing the interferometric data.

A2.6.1.2 The instrument must be capable to precisely and automatically displace the optical probe with the resolution defined in section A2.8.2.3.

A2.6.1.3 The instrument must be able to automatically rotate the heater tube with the resolution defined in section A2.8.2.4.

A2.6.1.4 The instrument, with its optical probe, must be able to automatically detect the edge of one of the two shoulders of the heater tube; the distance between these two shoulders is 60 mm.

A2.6.1.5 The instrument can measure the thickness over the whole length of the heater tube. However, this test method describes a procedure to measure the deposit thickness between the 5 mm and the 55 mm points located between the two shoulders of the heater tube, as defined in Test Method D3241 (Fig. A2.7).

A2.6.1.6 For the calculation of the film deposit thickness, the computer driven software must be able to automatically select against wavelength the appropriate refractive index value (n) and absorption coefficient value (k) for the substrate and the deposit film. These values are indicated in the graphs in sections A2.8.2.1 and A2.8.2.2.

A2.7 Test Samples (Heater Tube Coupon)

A2.7.1 Handle the heater tube coupon carefully so as not to touch the center portion at any time.

NOTE A2.3—Touching the center (thinner area) of the coupon will likely contaminate or disturb the surface of the heater tube, deposit, or both, which must be evaluated in pristine condition.

A2.8 Apparatus Preparation

A2.8.1 Install the apparatus in accordance with the manufacturer's instructions. If any malfunction is indicated refer to the manufacturer's instructions.

NOTE A2.4—Malfunctions are checked automatically when switching

[9] The sole source of supply of the deposit rater apparatus known to the committee at this time is AD systems (www.adsystems-sa.com), model DR 10 – Deposit Rater, available from AD systems, P.A. Portes de la Suisse Normande, Allée de Cindais, 14320 Saint André sur Orne, France. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.