Designation: D3241 – 16a

An American National Standard

Designation 323/16

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.

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 deposition values stated in SI units shall be regarded as the referee value.

1.4 The pressure values stated in SI units are to be regarded as standard. The psi comparison is included for operational safety with certain older instruments that cannot report pressure in SI units.

1.5 No other units of measurement are included in this standard.

1.6 **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.

1.7 *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.

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,

[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 July 1, 2016. Published July 2016. Originally approved in 1973. Last previous edition approved in 2016 as D3241 – 16. DOI: 10.1520/D3241-16A.

[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**

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 (VTR),* the tuberator described in Annex A1.

6.2.2 *Interferometric Tube Rater (ITR)*—the tuberator described in Annex A2.

6.2.3 *Ellipsometric Tube Rater (ETR)*—the tuberator described in Annex A3.

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) or other self-indicating drying agent 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:

[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, D]	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] D3241/IP 323 Thermal Stability is a critical aviation fuel test, the results of which are used to assess the suitability of jet fuel for aviation operational safety and regulatory compliance. The integrity of D3241/IP 323 testing requires that heater tubes (test coupons) meet the regulations of D3241 Table 2 and give equivalent D3241 results to the heater tubes supplied by the original equipment manufacturer (OEM).
[B] 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.
[C] 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.
[D] 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.

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.

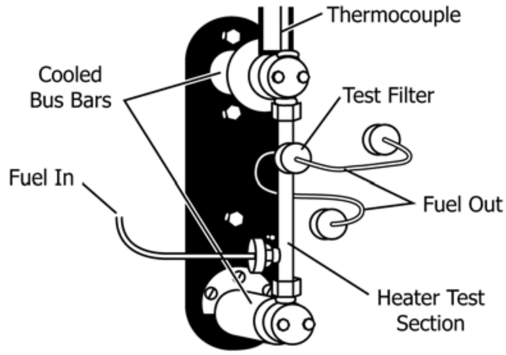


FIG. 1 Standard Heater Section, Essential to All D3241 Test Instruments

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 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 Rate the deposits on heater tube in accordance with Annex A1, Annex A2, or Annex A3 as directed by the specification referencing this method.

12.1.1 When a specification allows multiple rating techniques, the method providing deposit measurements in SI units is preferred.

12.1.2 When the rating techniques do not agree, the method providing measurements in SI units shall be regarded as the referee.

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.

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

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.

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.

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:*

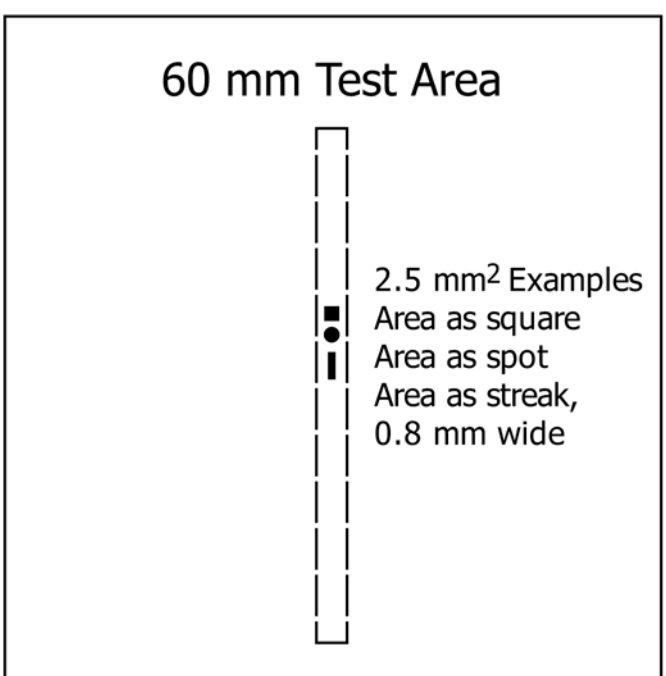


FIG. A1.1 Defect Areas

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 by this method was evaluated by the subcommittee and is reported in RR:D02-1786.[7]

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.

[7] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1786. Contact ASTM Customer Service at service@astm.org.

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:D02-1786[8] 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 nanometers, 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*[9]—with two reference deposits of known and traceable thickness made with silicon dioxide on silicon ($Si + SiO_2$). See Fig. A2.2.

[8] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1786. Contact ASTM Customer Service at service@astm.org.

[9] 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

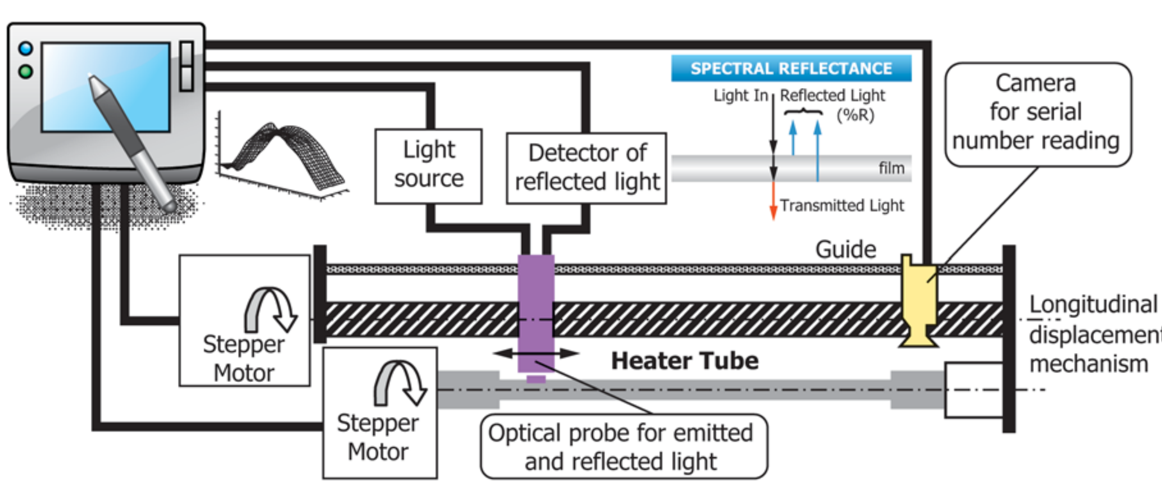


FIG. A2.1 Interferometric Apparatus—General Principle

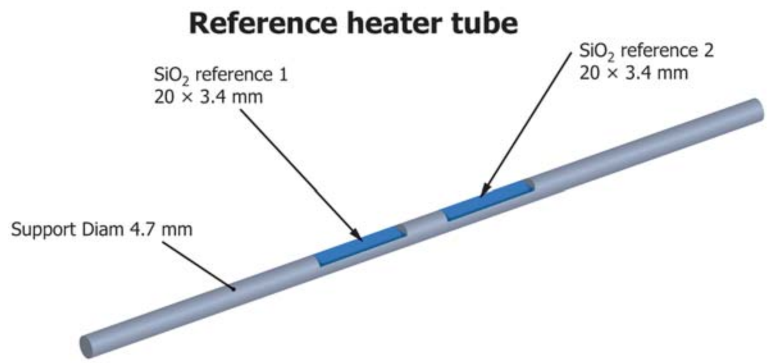


FIG. A2.2 Reference Heater Tube

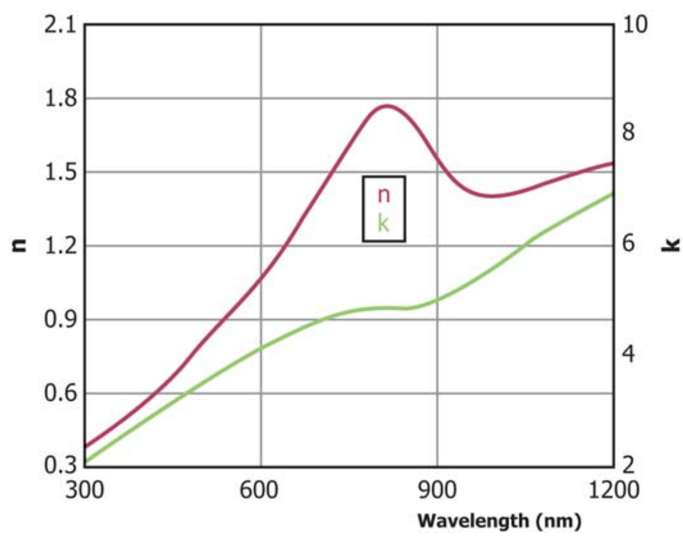


FIG. A2.3 n and k Values of the Heater Tube

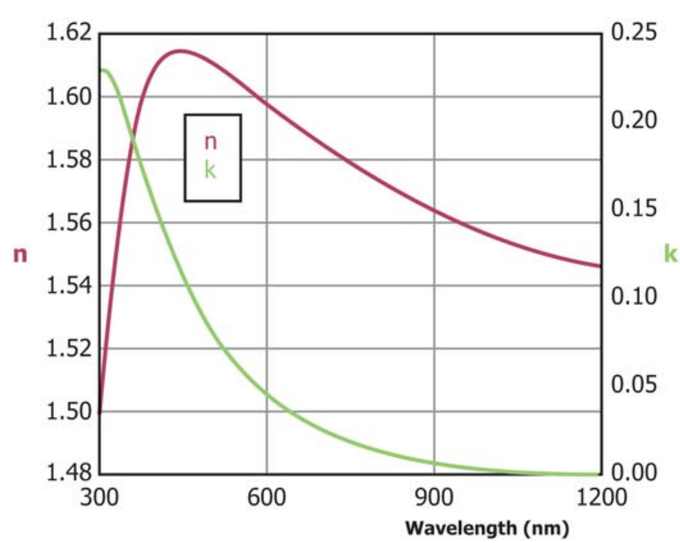


FIG. A2.4 n and K Values of the Heater Tube

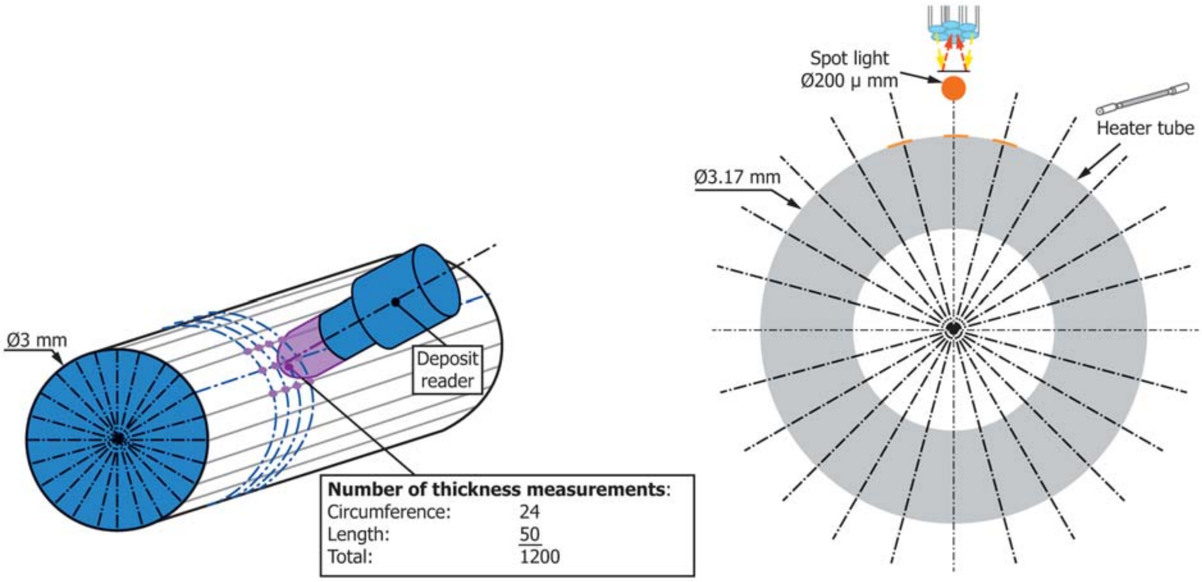


FIG. A2.5 Circumferential Resolution

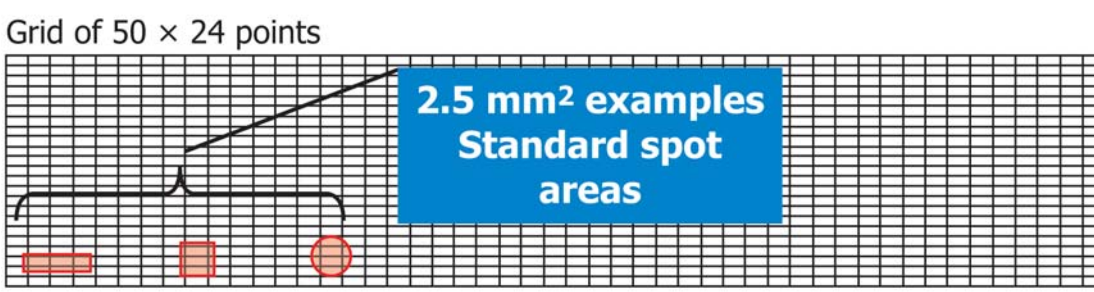


FIG. A2.6 Standard Spot

A2.6 Apparatus

A2.6.1 *Deposit Rater:*[10]

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

[10] 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.

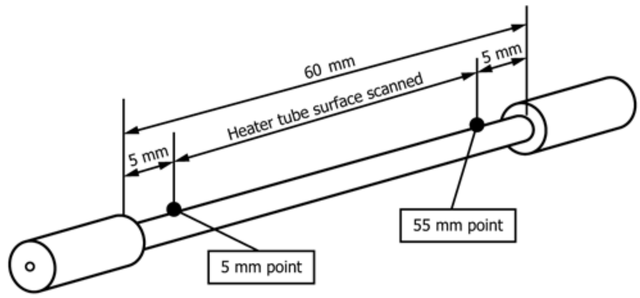


FIG. A2.7 Description of Heater Tube Scanned Surface

on the instrument and during the operation. Any malfunction will be automatically indicated.

A2.8.2 *Standard Operating Conditions:*

A2.8.2.1 *Heater Tube (Substrate)*—refractive index (n) and absorption coefficient (k) values are automatically selected by the computer software using the relationship shown in the graph below (Fig. A2.3).

NOTE A2.5—The value of n and k index are dependent on the metallurgy and surface roughness of the heater tube under examination. The values on the graphs here above are appropriate for aluminum heater tubes used in D3241.

A2.8.2.2 *Deposit Film*—refractive index (n) and absorption coefficient (k) values are automatically calculated by the computer software using the relationship shown in the graph below (Fig. A2.4).

NOTE A2.6—This represents the average value of refractive index of deposit after D3241 test procedure.

NOTE A2.7—It is assumed that the film is optically transparent to the wavelength of UVVIS light used in the DR 10, currently 200 nm to 1100 nm.

NOTE A2.8—This method is not applicable to heater tubes coated with Langmuir-Blodgett films of cadmium behenate. Consequently, heater tubes coated with Langmuir-Blodgett films of cadmium behenate cannot be used for instrument verification. Behenate films are anisotropic and do not relate to deposits. They are sub-layered with metals separated by fatty chains which creates multiple reflection for each sub layer. In other words, the Lanmuir-Blodgett films of cadmium behenate do not create any interference with spectral reflectance technique when the emitted light is normal to the deposit surface which is the case with the DR 10.

A2.8.2.3 *Circumferential Resolution*—(number of points measured on the heater tube circumference), 24 points (Fig. A2.5).

A2.8.2.4 *Longitudinal Resolution*—(number of points measured on a length of 50 mm), 50 points (Fig. A2.6).

A2.8.2.5 *Heater Tube Scanned Surface*—the instrument shall be set up to measure the thickness between the 5 mm and the 55 mm points. See Fig. A2.7.

A2.9 Baseline Calibration

A2.9.1 Heater tube surface finishes vary slightly between brands due to variations in polishing techniques. Therefore, a baseline calibration shall be performed using a new heater tube. The heater tube shall have been visually inspected according to section A2.11.2. Baseline calibration shall be performed prior to initial use of the instrument and whenever there is a change in heater tube brand

A2.9.2 During this phase, the instrument records and memorizes the reflected light spectrum from 12 different points on the new heater tube (Fig. A2.8).

A2.9.3 The typical reflected spectrum is calculated by averaging the 12 points and it is saved as the baseline. This baseline is then used to determine the interferences wave length generated by the deposit.

A2.9.4 To perform this calibration, refer to the operation manual delivered with the instrument, the duration of this phase is approximately three minutes.

A2.10 Apparatus Verification

A2.10.1 On a regular basis, a minimum of once per week, verify the operation of the apparatus by measuring a reference heater tube (A2.5.1). To do so, refer to the operation manual delivered with the instrument. The results shall be within ±5 % of the known thickness. If the determined values are not within the acceptable limits, return the apparatus to the manufacturer for recalibration.

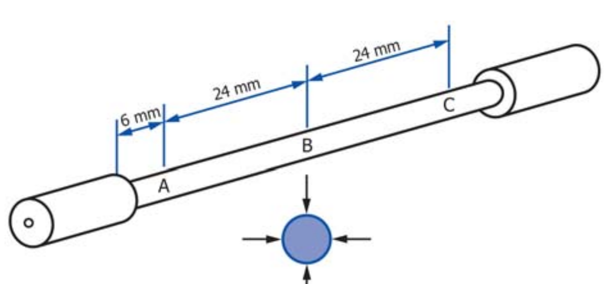


FIG. A2.8 Baseline Calibration Points

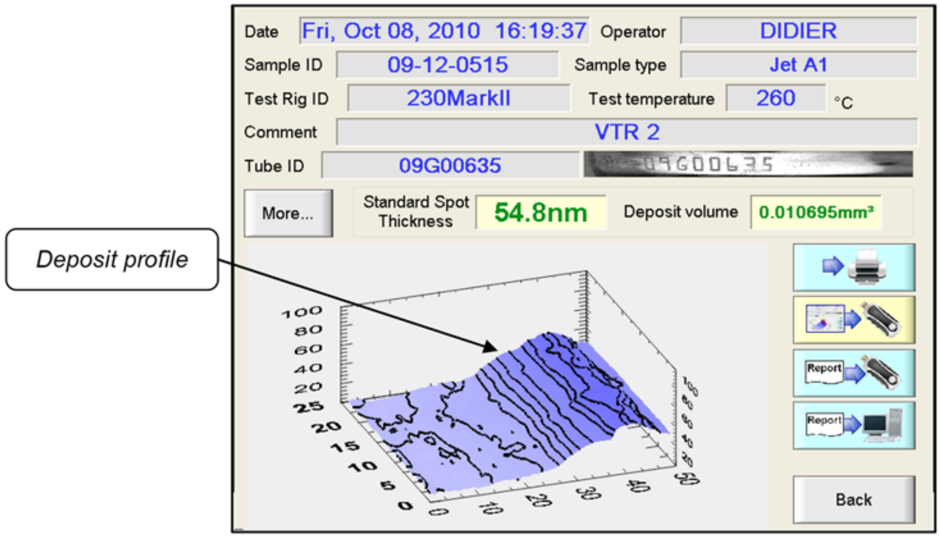


FIG. A2.9 Reporting Example

A2.11 Heater Tube Inspection

A2.11.1 Handle the heater tube carefully and do not touch the center section at any time.

A2.11.2 Visually examine the heater tube for straightness by rolling the heater tube on a flat dust-free surface and noting the gap between the flat surface and the center section. Heater tubes with a bent center section are not suitable for rating by the DR 10.

A2.11.3 Examine the heater tube, without magnification, in laboratory light for visible mechanical scratches on the centre section of the heater tube. If such defects are visible, discard the heater tube.

A2.12 Procedure

A2.12.1 To start a test, refer to the operation manual delivered with the instrument.

A2.12.2 Once the jet fuel sample details in addition with the heater tube details are entered, the thickness measurement is automatically initiated.

NOTE A2.9—The rotation and the positioning of the heater tube has two functions. The first one for traceability of the test, the instrument magnifies the serial number and takes a picture of the heater tube serial number. Second, it positions the heater tube on the same point of the circumference to ensure that the sequence of thickness measurements is started from the same point on the circumference. Consequently, for verification, when the test is repeated on the same heater tube, it facilitates the comparison of the deposit profiles.

A2.12.3 At the end of the measurement, the reported values are displayed and recorded, as shown in Fig. A2.9.

A2.12.4 Remove the rated heater tube and return it to its original container.

A2.13 Reporting

A2.13.1 Report the following:

A2.13.1.1 Standard spot deposit thickness to the nearest 1 nm.

A2.13.1.2 Deposit volume to the nearest 0.00001 mm^3 or "N/A" when the volume is not reported.

NOTE A2.10—In some cases, the apparatus is unable to precisely determine the thickness at one or several of the 1,200 measured points. When this situation occurs, because safety is involved with D3241 test, the apparatus does not extrapolate the thickness from the surrounding points. It simply reports "N/A" (Non Applicable) for the point in question. If the number of reported "N/A" points exceeds 10, then the Deposit Volume of is reported as "N/A". However, the apparatus still reports the standard spot thickness which is based on the average of the thickest points in a 2.5 mm^2 area. This reported standard spot is calculated with all the points for which the thickness was determined.

In some cases, when the apparatus reports a "N/A Deposit Volume", the heater tube in question is visually rated (VTR) as an abnormal deposit. No correlation can be established between "N/A Deposit Volume" reporting and abnormal visual rating. It was observed with the results of the ILS conducted in 2011 (Research Report RR:D02-1786) that when a "N/A Deposit Volume" was reported, it was associated with thick deposits and it is expected to be typical.

A2.14 Test Report

A2.14.1 The test report shall contain at least the following information:

A2.14.1.1 Sufficient details for complete identification of the fuel and the heater tube tested;

A2.14.1.2 A reference to this standard;

A2.14.1.3 The result of the test (see A2.12);

A2.14.1.4 Any deviation, by agreement or otherwise, from the procedure specified;

A2.14.1.5 The date of the test.

A3. RATING OF D3241 HEATER TUBES—ELLIPSOMETRIC METHOD

A3.1 Scope

A3.1.1 This annex describes a procedure for the ellipsometric rating of heater tubes produced by Test Method D3241—Thermal Oxidation Stability of Aviation Turbine Fuels.

A3.1.2 The final result from this rating procedure is an absolute measurement of the deposit thickness on the heater tube that can provide a basis for judging the thermal oxidative stability of the fuel sample. For aircraft fuel systems performance, deposit thickness is a useful parameter.

NOTE A3.1—If this procedure is to be used to rate the tube after the D3241 test, the new tube may be examined in an Ellipsometeric Tube Rater (ETR) to establish a base line or condition of satisfactory starting quality.

A3.2 Terminology

A3.2.1 *Definitions of Terms Specific to This Standard:*

A3.2.1.1 *deposit profile*—three-dimensional representation in terms of deposit thickness along and around the length of the heater tube test section.

A3.2.1.2 *deposit thickness*—the thickness of deposit present on the heater tube substrate surface expressed in nanometers (nm).

A3.2.1.3 *ellipsometry*—a technique used for measuring the optical properties of surfaces (refractive index and absorption coefficient) based on changes in the polarized state of light upon reflection from the surface.

A3.2.1.3.1 *Discussion*—In the presence of a thin transparent layer, with a known refractive index and absorption coefficient, ellipsometry can also be used to provide film thickness information.

A3.2.1.4 *maximum deposit thickness*—the maximum thickness of an average 2.5 mm^2 deposit present on the D3241 heater tube surface, expressed in nanometers, nm.

A3.3 Summary of Test Method

A3.3.1 An ellipsometric apparatus[11] is used to rate the deposits on the D3241 heater tube. The computer-driven software analyses the ellipsometric data and the maximum deposit thickness of an average 2.5 mm^2 is derived and displayed.

A3.4 Significance and Use

A3.4.1 The final tube rating is a direct measure of the thickness of the degraded fuel deposited on the tube. This rating is a basis for judging the thermal oxidative stability of the fuel sample.

A3.5 Reagents and Materials

A3.5.1 Ellipsometer reference tubes, on which layers of deposits of known thickness of 30 nm, 80 nm, 130 nm and optical constants and know position have been generated using a controlled deposition process (for example, the Langmuir-Blodgett multilayer deposition technique, atomic layer deposition, and/or chemical vapor deposition).[12]

A3.6 Apparatus

A3.6.1 Ellipsometric Tube Rater (ETR) is comprised of a suitable laser light source, optical detector for measuring the ellipsometric parameters, tube handling assembly and computer-driven software for analyzing the ellipsometric data. It is designed to combine the ellipsometric principles with specific heater tube handling.

A3.6.2 The thickness measurement is performed over the length of the heater tube, from 5 to 55 mm of the total 60 mm length, as designed by Test Method D3241.

A3.7 Apparatus Preparation

A3.7.1 Install the apparatus in accordance with the manufacturer's instructions. Ensure satisfactory function of the ellipsometric device in terms of alignment and optical diagnostics. If any malfunction is indicated refer to the manufacturer's instructions.

A3.7.2 Standard operating conditions.

A3.7.2.1 Substrate refractive index setting 2.65.

NOTE A3.2—The value of refractive index selected is dependent on the metallurgy and surface roughness of the D3241 heater tube under examination. The value 2.65 is appropriate for aluminum tubes used in D3241. See the ETR operating instructions for detailed requirements for other metallurgies.

A3.7.2.2 Substrate absorption coefficient setting (k) – 9.40.

NOTE A3.3—The value of absorption coefficient is dependent on the metallurgy of the D3241 heater tube under examination. The value of minus 9.40 is appropriate for aluminum tubes used in D3241. See ETR operating instructions for detailed requirements for other metallurgies.

A3.7.2.3 Deposit film refractive index setting 1.45.

NOTE A3.4—This represents the average value of refractive index based on many organic films.

A3.7.2.4 Deposit film absorption coefficient 0.00.

NOTE A3.5—It is assumed that the film is optically transparent to the wavelength of the laser light used in the ETR, currently 1550 nm.

A3.7.2.5 Resolution (number of circumferential points), 24 points.

A3.7.2.6 Resolution (number of longitudinal points), 50 points.

NOTE A3.6—The ETR has the capability to measure up to a maximum of 100 points along the length of the tube and up to 360 equally spaced points around the circumference of the tube.

[11] 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.

[12] The sole source of supply of the apparatus (reference tube kits) known to the committee at this time is Falex Corporation, 1020 Airpark Dr., Sugar Grove, Ill 60554. 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.

A3.7.2.7 The ETR records the maximum average 2.5 mm^2 deposit thickness in nanometers, nm. When using the test resolution of 24 × 50 points, the 2.5 mm^2 area is defined 6 points in a row or any set of 3 consecutive pairs of points. The manufacturer includes an algorithm that automatically identifies all 6 point combinations, calculates the average thickness of each of these combinations, selects the maximum average 6 point combination, displays the maximum average and shows the location of the maximum on the deposit graph.

A3.8 Apparatus Verification

A3.8.1 Verify the operation of the ellipsometric apparatus by measuring Ellipsometer reference tubes (A3.5.1). The results shall be within the ±5 % of the known thickness and ±1 mm of the known position. If the determined values are not within the acceptable limits contact the manufacturer for re-calibration service.

A3.9 Heater Tube Inspection

A3.9.1 Handle the heater tube carefully and do not to touch the center portion at any time.

A3.9.2 Visually 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. Heater tubes with a bent center section are not suitable for rating by the ETR.

A3.10 Procedure

A3.10.1 Run the instrument in accordance with the manufacturer's operating instructions.

A3.10.2 At the end of the measurement procedure, record the maximum average 2.5 mm^2 deposit thickness in nanometers, nm. When using the test resolution of 24 × 50 points, the 2.5 mm^2 area is defined 6 points in a row or any set of 3 consecutive pairs of points. The manufacturer includes an algorithm that automatically identifies all 6 point combinations, calculates the average thickness of each of these combinations, selects the maximum average 6 point combination, displays the maximum average and shows the location of the maximum on the deposit graph.

A3.11 Reporting

A3.11.1 Report the:

A3.11.1.1 The D3241 heater tube reference number;

A3.11.1.2 The maximum average 2.5 mm^2 deposit thickness in nm;

A3.11.1.3 The deposit volume in cm^3;

A3.11.1.4 Sufficient details for complete identification of the fuel and the heater tube tested;

A3.11.1.5 A reference to this standard;

A3.11.1.6 Any deviation, by agreement or otherwise, from the procedure specified; and

A3.11.1.7 The date of the test.

A4. EQUIPMENT

A4.1 Test Instrument

A4.1.1 The instrument described in this annex is the Aviation Fuel Thermal Oxidation Stability Tester that is used to test the thermal oxidation stability of turbine fuel. There are five instrument models which will be described. All provide a means to pump the sample once through the test system across the metal test coupon and through a test filter. There are means to control and measure coupon temperature, system pressure, and pressure drop across the filter, and methods of control and measurement vary with each instrument model. Mechanism for pumping is positive displacement using a gear pump or piston pump.

A4.2 Test Details

A4.2.1 *General Description*—This instrument uses a fixed volume of jet fuel that has been filtered, then aerated to provide a sample saturated with air. During the test, fuel is pumped at a steady rate across a heated aluminum tube which is maintained at a relatively high temperature, typically 260°C, but higher under some specifications. The fuel, saturated with oxygen from the aeration, may degrade on the hot aluminum heater tube to form deposits as a visible film. Also, the degraded materials of the fuel may flow downstream and be caught by the test filter. Both the increase in differential pressure across the test filter and the final heater tube rating are used to determine the oxidative stability of the fuel.

A4.2.2 *Fuel System*—Freshly filtered and aerated fuel is initially placed in a reservoir, then circulated once through the apparatus to a spent sample receptacle. Motive force for the sample is a positive displacement pump that will maintain flow at 3.0 mL/min and overcome any tendency of initial filter blockage from affecting the flow rate. Deviation of 10 % in flow rate is permitted. If filter blockage becomes severe, the bypass valve located before the test filter can be opened in order to finish the test. Then, any deposit on the heater tube can be evaluated based on a complete test.

A4.2.2.1 The heart of the test system is the tube-in-shell heat exchanger, or test section, which holds the test coupon and directs flow of fuel over it. It is important for the heater tube to be aligned correctly in the heater test section as shown in Fig. A4.1. This component is critical to consistent results and is a common component in all models.

A4.2.2.2 There are some other points regarding the fuel system that deserve mention:

(1) Fresh fuel is filtered immediately out of the reservoir through 0.45-μm membrane filter paper before entering the heater test section;

(2) The heater tube is sealed in the heater test section by elastomer O-rings (Fig. A4.2);

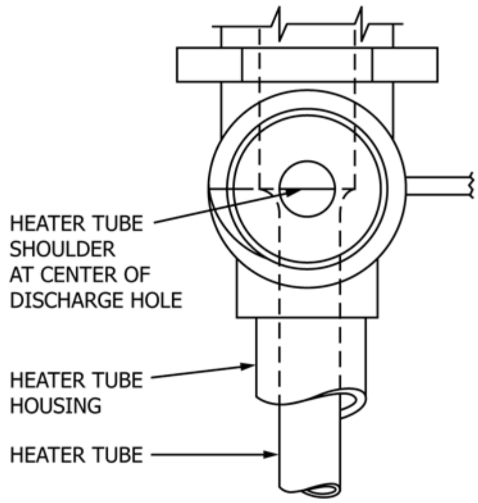


FIG. A4.1 Alignment of Heater Tube

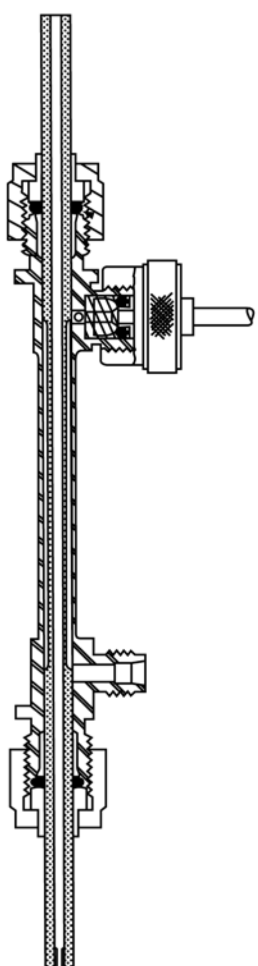


FIG. A4.2 Assembly Drawing of Heater Tube Test Section

(3) The test filter is of stainless steel of 17 µm rated porosity. If this filter causes an increase in differential pressure, an alarm will sound (normally at 125 mm Hg) alerting the operator. Bypass of the filter can then be accomplished if desired;

(4) Models 202, 203, and 215 use a single fuel reservoir with a floating piston to separate the fresh fuel (in bottom) and spent fuel (on top). Models 230 and 240 use two reservoirs, one for fresh fuel and one for spent;

(5) Flow of fuel in all models can be monitored by visually counting drops of flow. Models 230 and 240 also allow volumetric measure of flow with time, which is considered the most accurate flow measure.

A4.2.2.3 Diagrams of fuel flow though the three main configurations are shown in Fig. A4.3.

A4.2.3 *Heating/Temperature Control System*—The heater tube is resistively heated by the conductance of high amperage, low voltage current from a transformer through the aluminum tube. The heater tube is clamped to relatively heavy, water cooled current conducting bus bars, which increase in temperature relatively little.

A4.2.3.1 The temperature controller in all instrument models serves as indicator and controller. In automatic mode, the controller provides a source of steady heat during the test, varying the power as necessary to maintain the target (setpoint) temperature. In manual mode, the controller provides temperature indication only. Temperature range of operation is from ambient to a maximum of about 350°C.

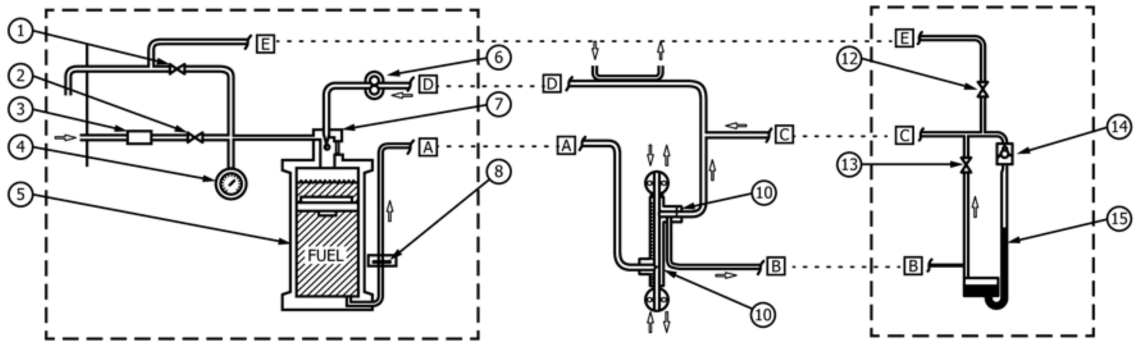
A4.2.3.2 Critical to temperature control is the thermocouple and its position. The thermocouple itself must be calibrated to ensure acceptable accuracy. The position of the tip must be carefully placed so the temperature reading during automatic control is the maximum (the hottest spot) for the heater tube. A simple mechanical positioning system allows easy and accurate placement of the thermocouple.

A4.2.3.3 A diagram of the basic heating system is shown in Fig. A4.4.

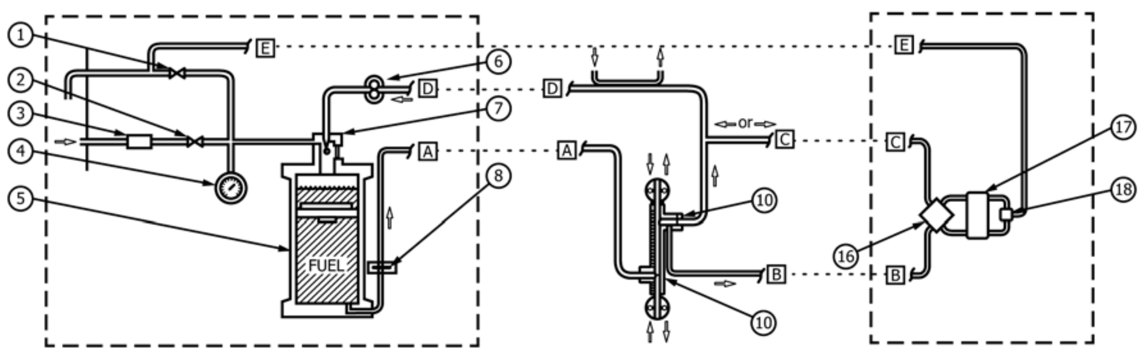
A4.2.4 *Cooling System*—In the normal operation of the instrument, some cooling is necessary to remove heat going into the bus bars by conduction from the hot heater tube. Cooling water is circulated through each bus bar using either laboratory tap water (Models 202, 203, and 215) or an internally circulated and radiator cooled liquid system (Models 230 and 240). The only precautions with these systems is to monitor them to be sure they are working and to avoid use of coolants that contain contaminants or salts that may eventually foul the system.

A4.2.5 *Pressurization*—At the temperature of a normal test, jet fuel would typically boil at the temperature of the heater tube. This would prevent accurate temperature control and interfere with natural deposit formation. Therefore, the system must be operated under a total pressure of about 3.45 MPa (500 psi). This pressure level is accomplished in each model by either using nitrogen gas (Models 202, 203, and 215) or a hydraulic piston pump (Models 230 and 240) to produce the high pressure needed.

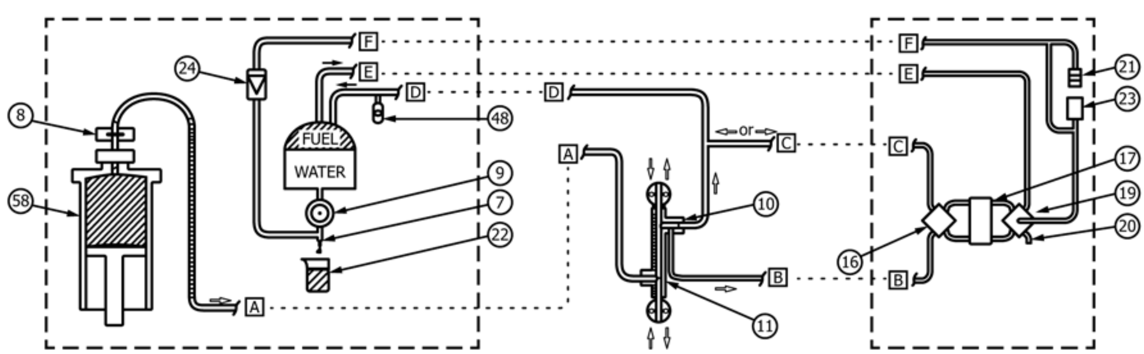
A4.2.5.1 A pressure gauge or transducer is used to measure and allow monitoring of the total system pressure. Of particular note is that the gas pressurized systems are run closed after pressurization, whereas the hydraulically pressurized systems have a relief valve through which the fluid passes in a constant leak throughout the test. For the relief valve control to operate



203 Type—pneumatically pressurized/gear pump/mercury manometer/standard test section



215 Type—pneumatically pressurized/gear pump/differential transducer/standard test section



230 Type—hydraulically pressurized/syringe pump/differential transducer/standard test section

1. Nitrogen Bleed Valve
2. Nitrogen Pressurize Valve
3. Pressure Limiter
4. Pressure Gauge
4a. Absolute Pressure Gauge (Transducer)
5. Fuel Reservoir with Piston and Seal
5a. Hydraulic Fuel Reservoir
6. Const. Speed Metering Pump
7. Drip Flow Indicator
8. Membrane Prefilter
9. Pressure Regulator
10. Test Filter
11. Standard Heater Tube Test Section
12. Manual Bleed Valve
13. Filter Bypass Valve
14. Float Check Valve
15. Manometer
16. 4–way Transducer Bypass Valve
17. Liquid Full Differential Transducer
18. 3 Way Vent (Bleed) Valve
19. 5 Way Vent (Bleed) Valve
20. Plugged Outlet
21. Air Trap Jar
22. Spent Liquid
23. Accumulator
24. Check Valve

FIG. A4.3 Fuel System Schematics

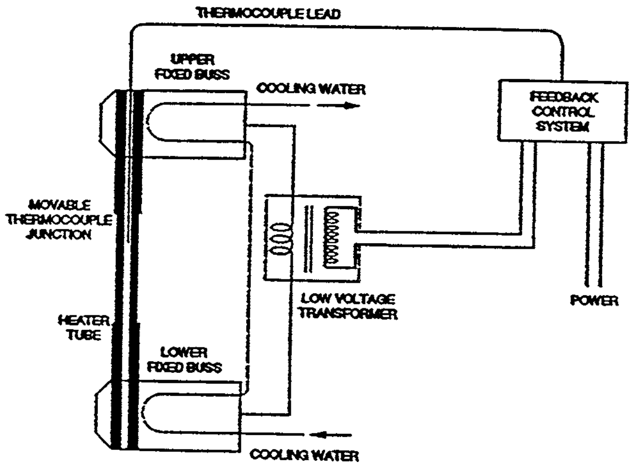


FIG. A4.4 Heater Tube and Temperature Control Schematic

uniformly for any fuel, a displacement cell is used where the spent fuel enters the top displacing water out the bottom and through the relief valve. Since the valve sees only water, the valve works consistently.

A4.2.6 *Differential Pressure Measure*—There are two instrument configurations used in the models to measure differential pressure (ΔP) across the test filter as products of fuel degradation are caught by the filter during the test. Models 202 and 203 (before 1984) use a mercury manometer with a possible strip chart ΔP recording option. Models 215, 230, and 240 use an electronic ΔP transducer. Details of how these two methods are included in the fuel schematic can be seen in the diagrams under Fuel System (see Fig. A4.3).

A4.2.6.1 Proper use of these differential measuring devices requires two special actions: bypass and air bleeding. The first allows the fuel flow to bypass the filter whenever that action becomes necessary. The second is used to remove air or nitrogen that at times may become trapped in the cell chambers. The manometer output is read as the height of the column of mercury; the transducer output must be displayed digitally.

A4.2.6.2 The manometer system, by nature, includes a bias due to the presence of fuel instead of the usual air over the mercury. This changes the value of pressure expressed in terms of column height of mercury such that a result about 6 % higher than true occurs. The transducer is not subject to this error, so in order to have manometer and transducer models read the same, a 6 % bias is added to the transducer so it gives the same value as a manometer.

A4.2.6.3 When operated, the ΔP measuring device employed must be zeroed under actual flow conditions at the start of the test. This is because a small pressure drop is created across the system when fuel is flowing. Zeroing the transducer or manometer at the beginning of the test compensates for the flow.

A4.2.7 *Differential Pressure Measurement Standardization*—The ΔP measurement accuracy can be checked by a technique of reading the pressure created by a column of known density fluid on each side of the ΔP cell. The details for doing this are included as part of each operating manual for the particular model instrument. This standardization is really a verification that the ΔP cell is operating correctly and is not meant to be a true calibration of the cell. Calibration must be done by the manufacturers of the cell if such action is suggested based on the results of the standardization.

A4.2.8 *Thermocouple Calibration*—It is important for the thermocouple to be accurate. To ensure this value, a method of calibration against known melting points is used and is described in detail in the appropriate User Manual for each instrument. With the first models, only pure tin was used as the indicator metal. Starting with Models 230 and 240 the use of two metals, pure tin at 232°C and pure lead at 327°C, was initiated to define two points surrounding the normal range used with the instrument. Also, an ice-water mixture is used to establish a 0°C low reference point.

A4.2.8.1 The principle used with melting point of metals is to immerse the thermocouple tip in the melted metal, then allow the metal to cool. As the metal goes through its freeze point, the temperature reading will hesitate momentarily indicating the known point for the metal.

A4.2.8.2 The difference between the known metal freezing value and the displayed temperature becomes a correction for setting test temperatures. For example, using tin with a known freeze temperature of 232°C (see Fig. A4.5), if the temperature noted at the time the metal froze was higher than 232°, then this would indicate the thermocouple was reading high by the difference indicated and the applied correction would be to lower any test temperature by this same amount. Where two metals and ice water (low point) are used, the principle is the same but the correction is calculated and applied automatically by the internal computer.

A4.2.9 *Fuel Aeration System*—All models have means to aerate the sample prior to testing. Without the presence of oxygen in the sample, a proper test is not achieved. Filtered, dry air is metered through the sample at about 1.5 L/min rate for 6 min. This 9 L of air ensures 97 % saturation of the sample.

A4.2.10 *Elapsed Time Measurement*—There are various methods of timing the test depending on the model. The elapsed time indicator is normally the basis used, but in some models, the timing of the ΔP data collection is done with a different timer. Since these two timers may not be exactly the same, the last data point may be lost if the test stops before the

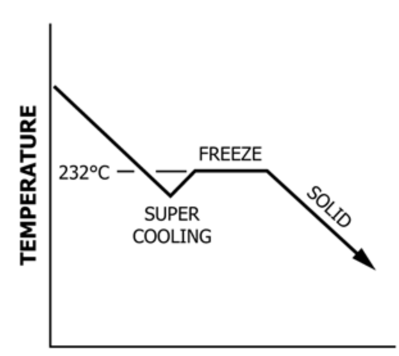


FIG. A4.5 Freezing Characteristics of Tin

last timed data point. The user manuals for the various instrument models cover techniques for avoiding loss of data points.

A5. WARNING STATEMENTS

A5.1 Acetone

A5.1.1 Keep away from heat, sparks, and open flame.

A5.1.2 Keep container closed. Use with adequate ventilation.

A5.1.3 Avoid buildup of vapors and eliminate all sources of ignition, especially nonexplosion-proof electrical apparatus and heaters.

A5.2 Toluene

A5.2.1 Avoid prolonged or repeated breathing of vapor or spray mist.

A5.2.2 Use only with adequate ventilation.

A5.2.3 Eye irritation and dizziness are indications of overexposure.

A5.2.4 Do not take internally.

A5.2.5 Swallowing may cause injury, illness, or death.

A5.2.6 Avoid prolonged or repeated contact with skin.

A5.2.7 Do not get in eyes.

A5.2.8 Can produce toxic vapors on contact with flames, hot glowing surfaces, or electric arcs.

A5.3 Iso-propanol (2-propanol)

A5.3.1 Keep away from heat, sparks, and open flame.

A5.3.2 Keep container away from heat, sparks, and open flame.

A5.3.3 Keep container closed.

A5.3.4 Use with adequate ventilation.

A5.3.5 Avoid buildup of vapors and eliminate all sources of ignition, especially nonexplosion-proof electrical apparatus and heaters.

A5.3.6 Avoid prolonged breathing of vapor or spray mist.

A5.3.7 Avoid prolonged or repeated skin contact.

A5.4 n-Heptane

A5.4.1 Keep away from heat, sparks, and open flame.

A5.4.2 Keep container closed.

A5.4.3 Use with adequate ventilation.

A5.4.4 Avoid prolonged breathing of vapor or spray mist.

A5.4.5 Avoid prolonged or repeated skin contact.

A5.5 Compressed Gases (Nitrogen)

A5.5.1 Keep cylinder valve closed when not in use.

A5.5.2 Do not enter storage areas unless adequately ventilated.

A5.5.3 Always use a pressure regulator.

A5.5.4 Release regulator tension before opening cylinder.

A5.5.5 Do not transfer to cylinder other than one in which gas is received.

A5.5.6 Do not mix gases in cylinders.

A5.5.7 Never drop cylinder.

A5.5.8 Make sure cylinder is supported at all times.

A5.5.9 Stand away from cylinder outlet when opening cylinder valve.

A5.5.10 Keep cylinder out of sun and away from heat.

A5.5.11 Keep cylinder from corrosive environment.

A5.5.12 Do not use cylinder without label.

A5.5.13 Do not use dented or damaged cylinders.

A5.5.14 For technical use only.

A5.5.15 Do not use for inhalation purposes.

A5.6 Aviation Turbine Fuel (Jet B, See Specification D1655)

A5.6.1 Keep away from heat, sparks, and open flames.

A5.6.2 Keep container closed.

A5.6.3 Use with adequate ventilation.

A5.6.4 Avoid breathing vapor or spray mist.

A5.6.5 Avoid prolonged or repeated contact with skin.

A5.7 Aviation Turbine Fuel (Jet A or A-1, See Specification D1655)

A5.7.1 Keep away from heat, sparks, and open flame.

A5.7.2 Keep container closed.

A5.7.3 Use with adequate ventilation.

A5.7.4 Avoid buildup of vapors and eliminate all sources of ignition, especially nonexplosion-proof electrical apparatus and heaters.

A5.7.5 Avoid breathing vapor or spray mist.

A5.7.6 Avoid prolonged or repeated contact with skin.

A5.8 Mercury

A5.8.1 Do not breathe vapor.

A5.8.2 Keep container closed.

A5.8.3 Use with adequate ventilation.

A5.8.4 Do not take internally.

A5.8.5 Cover exposed surfaces with water if possible, to minimize evaporation.

A5.8.6 Do not heat.

A5.8.7 Keep recovered mercury in tightly sealed container prior to sale or purification.

A5.8.8 Do not discard in sink or in rubbish.

APPENDIXES

(Nonmandatory Information)

X1. INSTALLATION, MAINTENANCE, SPECIAL CHECKS

X1.1 Laboratory Installation Requirements

X1.1.1 The tester should be placed on a level laboratory bench, allowing a 200 to 300 mm wide bench area in front of the tester. Ready access to the rear of the tester should be provided for routine maintenance and service requirements. Ensure that the vent on top or side of the instrument cabinet is not obstructed during installation or use. Adequate ventilation should be provided, and proper procedures for handling solvents and hydrocarbons should be used. A constant voltage transformer may be required by early versions of the instrument. Single-phase electrical power, 115 V-60 Hz-15 Amp or optional 220 V-50 Hz-8 Amp with a ground outlet is required.

X1.1.2 For pneumatic models, a nitrogen supply bottle with a suitable regulator capable of supplying 3.45 MPa should be placed conveniently and connected with 3.2-mm diameter tubing to the tester. A suitable 6.4-mm diameter line needs to be connected from the WATER INLET connection to a 200 to 700 kPa water supply and a 6.4-mm diameter line needs to be connected from WATER DRAIN to a drain having a minimum capacity to receive 80 L/h.

X1.2 AutoCal Calibrator Metal Replacement

X1.2.1 The tin (and lead, if used) in the well of the AutoCal Calibrator must be replaced whenever the quantity is below minimum or when contaminated.

X1.2.2 To remove the metal, install the AutoCal Calibrator inverted between the upper fixed bus and the lower floating bus.

X1.2.3 Place a paper tissue or rag under the well to catch the molten metal.

X1.2.4 Apply power to the AutoCal Calibrator as during normal calibration, and at same time gently tap the well until all molten metal has dropped out.

X1.2.5 Remove and install the AutoCal Calibrator in upright position and refill with new metal. The proper amount of tin for one filling is about 1.5 to 1.9 g, and for lead about 3.3 to 4.7 g.

X1.3 Thermocouple Replacement and Position Adjustment

X1.3.1 The thermocouple used for measuring and controlling the temperature of the heater tube may have to be replaced at intervals due to damage or failure. If not of the simple plug in type, remove the thermocouple, loosen the thermocouple clamp, support clamp, and thermocouple connections on back of the temperature controller.

X1.3.2 Install a new thermocouple reversing the steps used to remove old thermocouple. Replace and tighten screws as required. If applicable, when tightening the Allen screw of the thermocouple clamp, the tip of the thermocouple must be flush with top of upper fixed bus when position indicator is set at the reference mark.

X1.3.3 Check for proper thermocouple indexing under actual test operating conditions.

X1.4 Heater Tube Temperature Profile

X1.4.1 If it is desired to measure the heater tube temperature profile, do so after the first hour of the test or before significant ΔP occurs. Follow the procedure in the user manual for the particular model.

X1.5 Flow Rate Check

X1.5.1 *Gear Pump Instruments (Models 202, 203, 215)*—Check the flow by observing the drip in the drip housing and measuring the time for 20 drops. The time should be 9 ± 1 s.

X1.5.2 *Syringe Pump Instruments (Models 230, 240):*

X1.5.2.1 *Drip Rate Method*—Check the flow by observing the drip from the drip assembly and measuring the time for 20 drops. The time should be 19 ± 1 s.

X1.5.2.2 *Volumetric Method*—Check the flow by measuring the time it takes to collect 15 mL of fuel in a graduated cylinder. That time should be between 4 min, 33 s and 5 min, 33 s.

X1.5.2.3 For Syringe Pump Instruments, the volumetric method is the referee.

NOTE X1.1—Drip rate counts start with zero (drop 0, drop 1, drop 2… drop 20) to get an accurate measure of the time required for 20 drops.

X1.6 Filter Bypass Valve Leakage Check (Models 202, 203, and 215 only)

X1.6.1 Obtain a used filter and plug the upstream side with any fast-drying glue, such as industrial adhesive. Install this filter together with any heater tube in the test section.

X1.6.2 Circulate clean filtered fuel at 3.45 MPa with MAN BYPASS valve in the open position (no heat applied).

X1.6.3 After steady flow is observed in the sight glass (20 drops in 9.0 ± 1.0 s), close the MAN BYPASS valve and simultaneously start a stopwatch. Observe the time required for the ΔP to reach 100 mm. Immediately open the MAN BYPASS valve to resume normal fuel flow.

X1.6.4 If the time measured to reach 100 mm ΔP is equal to or less than 60 s, the MAN BYPASS valve and the fuel pump meet normal performance requirements.

X1.6.5 The time required for the ΔP to exceed 100 mm can be quite short; in some equipment the increase may occur almost instantly depending on pump condition and system details. Such a rapid rise in ΔP is acceptable and considered to be within the range of expected and normal operation.

X1.6.6 If the time measured to reach 100 mm ΔP exceeds 60 s, either the filter bypass valve is leaking or the fuel metering pump performance is unsatisfactory. In this case, the fuel metering pump performance should be checked to determine if the pump or filter bypass valve needs to be replaced.

X1.7 Fuel Metering Pump Check (Gear Pumps Only)

X1.7.1 Install a plugged filter, used heater tube, and establish normal fuel flow.

X1.7.2 After steady flow is established, adjust the MAN BYPASS valve to maintain a steady ΔP of 50 mm.

X1.7.3 Measure the time with a stopwatch for 20 drops flow rate as observed in the sight glass.

X1.7.4 The time for a properly performing fuel pump is 9.0 ± 1.0 s for 20 drops fuel flow rate. Pumps that measure above 10 s should be replaced.

X1.7.5 After installing a new pump, repeat the pump check.

X1.7.6 If low flow persists, clean all lines and fittings from the test filter through the metering pump to the fuel reservoir with tri-solv. Replace lines as necessary. Repeat pump check.

X1.8 Maintenance Manual

X1.8.1 A maintenance manual is available[5] that provides additional maintenance information, such as the electrical schematic (also available on inside of back door of instrument cabinet). Complete details for operating the instrument models are contained in the user manuals for each instrument.

X2. DETERMINATION OF BREAKPOINT

X2.1 Definition

X2.1.1 *Breakpoint*—In Test Method D3241, *breakpoint* is the highest control temperature at which the fuel meets tube rating and ΔP specification requirements.

X2.1.1.1 *Discussion*—This definition of breakpoint describes the highest pass temperature for a fuel. Note that some published papers have used the term breakpoint to describe the lowest fail temperature, which is the (x + 5)°C temperature referred to below.

X2.2 Determination of Breakpoint

X2.2.1 The breakpoint can be derived by conducting a series of tests of different control temperatures to arrive at the temperature, x°C, at which the fuel meets both tube rating and ΔP specification requirements, and where a test at a control temperature of (x + 5)°C would result in a fail (that is, failure to meet tube rating or ΔP requirements). The temperature x°C would then be reported as the Test Method D3241 breakpoint.

SUMMARY OF CHANGES

Subcommitee D02.J0 has identified the location of selected changes to this standard since the last issue (D3241 – 16) that may impact the use of this standard. (Approved July 1, 2016.)

(1) Updated IP yeardate.

Subcommitee D02.J0 has identified the location of selected changes to this standard since the last issue (D3241 – 15$^{\varepsilon 2}$) that may impact the use of this standard. (Approved Feb. 1, 2016.)

(1) Added new footnote A to Table 2.

Subcommitee D02.J0 has identified the location of selected changes to this standard since the last issue (D3241 – 14a$^{\varepsilon 1}$) that may impact the use of this standard. (Approved July 1, 2015.)

(1) Added new subsection A3.11.1.3.