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An American National Standard

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Standard Test Method for Measurement of Thermal Stability of Aviation Turbine Fuels under Turbulent Flow Conditions (HiReTS Method)[1,2]

This standard is issued under the fixed designation D 6811; 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.

1. Scope

1.1 This test method covers a laboratory thermal process,[3] using a specified apparatus for measuring the tendencies of aviation turbine fuels to deposit insoluble materials and decomposition products, such as lacquers, within a fuel system. This test method provides a quantitative result for fuel under turbulent flow conditions in 65 or 125 min.

1.2 The values stated in SI units are to be regarded as the standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

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

D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products[4]

D 4306 Practice for Aviation Fuel Sample Containers for Tests Affected by Trace Contamination[4]

E 128 Test Method for Maximum Pore Diameter and Permeability of Rigid Porous Filters for Laboratory Use[5]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *capillary tube, n*—a coated resistively heated stainless steel tube through which fuel is pumped and controlled to give a predefined constant fuel exit temperature.

3.1.2 *deposits, n*—oxidative products, such as lacquers, laid down predominantly at the fuel exit end (hottest), on the inside of the heated capillary tube.

3.1.3 *HiReTS, n*—high Reynolds number thermal stability.

3.1.4 *HiReTS Peak (P) number and Total (T) number, n*—the quantitative results of the test.

3.1.5 *tubeways, n*—plastic and metal tubes through which fuel flows during cleaning and the test.

4. Summary of Test Method

4.1 Fuel is pumped, at pressure, through an electrically heated capillary tube at a constant rate. The heating of the capillary tube is controlled to maintain a constant fuel temperature of 290 ± 3°C at the exit of the capillary tube. A flow rate of greater than 20 mL/min and the specified capillary bore of less than 0.300 mm ensures that turbulent flow is maintained (see Appendix X1) within the capillary. The formation of lacquers and fuel degradation products act as a thermal insulator between the cooler fuel and hotter capillary tube, resulting in an increase in temperature of the capillary tube which is measured at a number of positions by a contactless pyrometer. The HiReTS Total (T) number is displayed during and at the end of the test. The HiReTS Peak (P) number can be determined from analysis of the results.

5. Significance and Use

5.1 The thermal stresses experienced by aviation fuel in modern jet engines may lead to the formation of undesirable and possibly harmful insoluble materials, such as lacquers, on heat exchangers and control surfaces, that reduce efficiency and require extra maintenance.

5.2 Aircraft fuel systems operate mainly under turbulent flow conditions. Most large-scale realistic test rigs operate in the turbulent flow regime but fuel volumes are very large and test times are very long.

5.3 This test method tests fuel under turbulent flow (high Reynolds number) conditions, and it gives a quantitative result under standard operating conditions of 65 or 125 min. Continuous analysis of results during the test allows performance of the fuel to be monitored in real time thus enabling the test

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

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[2] This test method is being jointly developed with the Institute of Petroleum, where it is designated IP 482.

[3] This process is covered by a patent. Interested parties are invited to submit information regarding the identification of an alternative(s) to this patented item to the ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.

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

[5] *Annual Book of ASTM Standards*, Vol 14.04.

time to be reduced manually or automatically, if required.

5.4 The results of this test method are not expected to correlate with existing test methods for all fuels, since the test methods and operating conditions are different (see Appendix X2).

6. Apparatus (see Annex A1)

6.1 *General*—(See Fig. A1.2.) Fuel contained in the sample vessel is drawn through the sample filter by a pump. The temperature of the fuel is checked by the input fuel electronic thermometer. The fuel is pumped at a constant rate, at pressure set by the back pressure valve, through an electrically heated capillary tube which has a blackened outer surface to give a high thermal emissivity. The heating of the capillary tube is controlled to maintain a constant fuel temperature, as measured by the capillary exit electronic thermometer, at the exit of the capillary tube. The waste fuel is then cooled to a temperature of less than 20°C above ambient, as measured by the waste fuel electronic thermometer, before being discharged to a waste container. During the test, the temperature of the outside of the capillary tube is scanned, checked and recorded every 5 min at 12 points along the exit end of the capillary tube using a contactless pyrometer which is located on a computer-controlled elevating platform.

6.2 The thermal stability apparatus[6] and capillary tube[6] is specified in detail in Annex A1.

6.3 *Sparger*, of porosity 40 to 80 µm, which allows an air flow of approximately 1.5 L/min.

NOTE 1—The porosity of the sparger can be checked using Test Method E 128.

6.4 *Sample Filter*, 20-µm stainless steel.

6.5 *Aeration Dryer*, glass or other suitable transparent material, minimum height 250 mm, minimum diameter 50 mm, filled with dry calcium sulfate and cobalt chloride (see 7.4), which is used in conjunction with an air supply and the sparger (see 6.3) to aerate the test sample.

7. Reagents and Materials

7.1 *Heptane*, $CH_3{\cdot}(CH_2)_5{\cdot}CH_3$, technical grade 95 % purity, for cleaning the apparatus tubeways, and sampling vessels. (**Warning—**Extremely flammable; harmful if inhaled.)

7.2 *Trisolvent*, for cleaning sampling vessels. (**Warning—**Each of the components and the trisolvent is flammable; harmful if inhaled; irritating to skin, eyes and mucous membranes.) It consists of equal volumes of the following:

7.2.1 *Acetone*, $CH_3{\cdot}\ CO\ {\cdot}\ CH_3$, technical grade, 95 % purity.

7.2.2 *Toluene*, $C_6H_5{\cdot}\ CH_3$, technical grade, 95 % purity.

7.2.3 *Propan-2-ol*, $(CH_3)_2{\cdot}\ CH\ {\cdot}\ OH$, technical grade, 95 % purity.

7.3 *Cleaning Solvent*, technical grade, 95 % purity, for cleaning sampling vessels. (**Warning—**Extremely flammable; harmful if inhaled.) It consists of one of the following:

7.3.1 *2-methylpentane*.

7.3.2 *3-methylpentane*.

7.3.3 *2,2,4-trimethylpentane*.

7.4 *Drying Components*, to dry the air used for aeration and to indicate the absorption of water by changes from blue to pink color. Use a mix, by volume or weight of the following:

7.4.1 *Calcium Sulfate Anhydrous Powder*, $CaSO_4$(97 %).

7.4.2 *Cobalt Chloride Anhydrous*, $CoCl_2$(3 %) granules.

7.5 *Air*, 1.5 L/min for aeration of the test sample.

8. Sampling and Sample Containers

8.1 Obtain samples for testing in accordance with Practices D 4057 or D 4177, with the following additional requirements:

8.1.1 Containers shall be fully epoxy lined or made of polytetrafluoroethylene (PTFE). See Note 2 and Practice D 4306.

8.1.2 Prior to sampling, all containers and their closures shall be rinsed at least three times with the fuel being sampled.

8.1.3 Test samples as soon as possible after sampling.

NOTE 2—Test methods for measuring thermal stability are known to be sensitive to trace contamination during the sampling operation and from sample containers. New containers are recommended, but when only used containers are available, a thorough rinse with trisolvent (see 7.2) followed by cleaning solvent (see 7.1 and 7.3), and drying with a stream of air is recommended.

8.2 *Aeration of Test Sample*—Aerate the test sample, with dry air, through the sparger at an air flow rate of 1 to 2 L/min for 10 min.

8.3 *Sample Size*—Standard operating conditions are: 3 L for 13 scans (65–min test) and 5 L for 25 scans (125-min test).

9. Preparation of Apparatus

9.1 Prepare the instrument for operation in accordance with the manufacturer's instructions. (**Warning—**Installing and removing the capillary tube may result in exposure to fuel or solvent. It is recommended that impermeable gloves and safety glasses are worn.)

9.2 Remove the sample filter and inlet tubing and clean by rinsing with heptane and then by back flushing with heptane, and then refit.

9.3 Set the instrument in accordance with Table 1 and check that the correct standard operating conditions are in accordance with Section 10.

9.4 Inspect 40 mm of the blackened section at both ends of the capillary tube and reject the tube if any scratches, pinholes or cracks are deep enough to expose the capillary tube's bright metal surface.

TABLE 1 Standard Instrument Settings

NOTE 1—Tolerances for the instrument settings are given in Annex A1.

NOTE 2—The datum position is the bottom of the top bus bar connector. See Fig. A1.3.

NOTE 3—Touching the blackened section of the capillary tube should be avoided, especially within 50 mm of each end.

Distance from datum (see Fig. A1.3) to the first measurement position	1 mm
Number of capillary tube temperature measurement positions	12
Distance between individual measuring points	2.5 mm
Rate of scanning all capillary tube temperature measurement positions	12/h

[6] The equipment, as listed in the research report being prepared, was used to develop the precision statement. The apparatus and capillary tubes described in Annex A1 are both supplied by Stanhope-Seta, Chertsey, Surrey KT16 8AP, UK. To date no other equipment has demonstrated through ASTM interlaboratory testing the ability to meet the precision of this test. This is not an endorsement or certification by ASTM. A research report is being prepared.

9.5 Commence the pre-test by installing a new capillary tube and carrying out the following in accordance with the manufacturer's instructions.

9.6 Immerse the input tube and sample filter in heptane.

9.7 Clean the tubeways with heptane and reset the bypass valve to TEST.

9.8 Visually check the system for leaks. If a leak is found, abort the pre-test and vent the system using the bypass valve. Tighten or replace any leaking fittings, if necessary, and repeat 9.6.

9.9 Check the alignment and focus of the pyrometer and the straightness of the capillary tube in accordance with the manufacturer's handbook.

10. Standard Operating Conditions

10.1 *Fuel Test Temperature*, preset at 290°C or as specified in applicable specifications or as agreed upon between the parties.

10.2 *Fuel Flow Rate*, preset at 35 mL/min or as specified in applicable specifications or as agreed upon between the parties.

10.3 *Number of Capillary Scans*, preset as 13 (for a 65 min test) or 25 (for a 125 min test), or as specified in applicable specifications or as agreed upon between the parties.

NOTE 3—Each capillary scan takes 5 min to complete.

NOTE 4—Other test temperatures, besides 290°C, and flow rates, besides 35 mL/min, can be used but the precision may be affected.

11. Calibration and Standardization

11.1 Ensure that all of the manufacturer's instructions for calibrating, checking, cleaning, and operating the apparatus are followed.

11.2 Verify the performance of the temperature, flow, and pressure sensors at least every 6 months. The testing of fuels with poor thermal stability may necessitate more frequent equipment verification and cleaning.

12. Procedure

12.1 Immerse the input tube and clean filter into a clean sample vessel filled with fuel aerated as in 8.2. The inlet fuel temperature shall be between 15 and 30°C as measured by the input fuel electronic thermometer.

12.1.1 Set the bypass valve to TEST, and start the test.

12.2 Use the bypass valve to purge the tubeways with the fuel sample. Reset the bypass valve to TEST.

NOTE 5—The test can only start if all independent and computer-monitored safeguards have not detected a fault condition.

12.3 Visually check the system for leaks. If a leak is found, abort the test and vent the system by using the bypass valve. Tighten or replace any leaking fittings if necessary and repeat 12.3.

12.4 Close the capillary tube enclosure door, ensure that the bypass valve is set to TEST, and commence heating the capillary tube.

NOTE 6—The pyrometer commences measuring the temperature of the capillary tube at the required positions when the fuel exit temperature, from the capillary tube, has stabilized to the prescribed value.

12.5 At the end of the test remove the sample filter and inlet tubing and clean by rinsing with heptane and then by back-flushing with heptane, and then refit. Immerse the input tube and sample filter in heptane and clean the system in accordance with the manufacturer's instructions. When this procedure has been completed, vent the system using the bypass valve.

NOTE 7—Heptane draining out of the apparatus can be avoided by leaving a capillary tube installed or by capping the upper and lower unions, and ensuring that the bypass valve is in the TEST position.

12.6 The result of the test is automatically calculated. (See Section 13 for the derivation of the HiReTS Peak and Total numbers.)

13. Calculation of Result

13.1 The HiReTS Total number is the total of the difference between the minimum and final temperatures measured at the required positions, along the surface of the capillary tube, during the test.

$$\text{HiReTS Total number} = \delta T_1 + \delta T_2 + \delta T_3 + \delta T_4 + \delta T_5 + \delta T_6 + \delta T_7 + \delta T_8 + \delta T_9 \ldots\ldots + \delta T_n \quad (1)$$

where:

δT_n = difference between the minimum and final temperatures measured at Position n, °C during the test while the fuel exit temperature is maintained at a stable level.

13.2 The HiReTS Peak number is the largest of the differences between the minimum and final temperatures measured at any of the required positions, along the surface of the capillary tube, during the test.

13.3 Express the HiReTS Total and Peak results as whole numbers. Round up any fractions. Use the formats T65, T125, P65 and P125 to denote Total (T) or Peak (P) and the test time.

14. Report

14.1 Report the source, type, and identification of the material tested, plus the date tested.

14.2 Report the result of the test (see 13.3) and the number of capillary scans. Refer to this test method and report any deviation, by agreement or otherwise, from the procedure specified.

14.3 Record the capillary tube identification.

14.4 The report form given in Appendix X3 can be used for standard and non-standard tests.

15. Precision and Bias [6]

15.1 The precision of this test method, as determined by statistical analyses of interlaboratory results, is as follows:

15.1.1 *Repeatability*—The difference between two test results obtained by the same operator with the same apparatus under constant operating conditions, on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following only in one case in twenty.

- r P65 (Peak number with a 65–min test) = $2.006x^{0.667}$
- r P125 (Peak number with a 125–min test) = $2.249x^{0.667}$
- r T65 (Total number with a 65–min test) = $3.123x^{0.72}$
- r T125 (Total number with a 125–min test) = $1.322x^{0.9}$

where:

x = average of results being compared.

15.1.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would, in the long run, in the normal correct operation of the test method, exceed the following only in one case in twenty.

R P65 (Peak number with a 65–min test) = $3.077x^{0.667}$
R P125 (Peak number with a 125–min test) = $2.895x^{0.667}$
R T65 (Total number with a 65–min test) = $5.008x^{0.72}$
R T125 (Total number with a 125–min test) = $1.667x^{0.9}$

where:

x = average of results being compared.

15.1.3 The precision applies only to HiReTS Peak numbers up to 200 and to HiReTS Total numbers up to 1900, higher results can be measured but the precision may not apply.

15.1.4 *Bias*—This test method has no bias because the result of the test is defined only in the terms of this test method.

16. Keywords

16.1 capillary; deposits; lacquers; Reynolds number; thermal stability; turbine fuel; turbulent flow

ANNEX

(Mandatory Information)

A1. THERMAL STABILITY APPARATUS

A1.1 *High Reynolds Number Thermal Stability Tester (HiReTS)*,[5] (see Figs. A1.1 and A1.2).

A1.1.1 *Capillary Exit Electronic Thermometer*, capable of reading to 0.1°C with a minimum accuracy of 2°C.

A1.1.2 *Input Fuel Electronic Thermometer*, capable of reading to 1°C with a minimum accuracy of 2°C.

A1.1.3 *Waste Fuel Electronic Thermometer*, capable of reading to 1°C with a minimum accuracy of 2°C.

A1.1.4 *Pump/Fuel Flow Rate Controller*, capable of pressurizing the system to 10 MPa and of controlling the fuel flow through the capillary tube between 20 and 50 mL/min ± 1 mL/min, and displayed with a resolution of 1 mL/min.

A1.1.5 *Back Pressure Valve*, set to give a system pressure of 2.0 MPa ± 10 %.

A1.1.6 *Waste Sample Cooler*, capable of cooling the fuel to less than 20°C above ambient temperature.

A1.1.7 *Pressure Relief Valve*, set to 8.0 MPa ± 10 % for safety purposes.

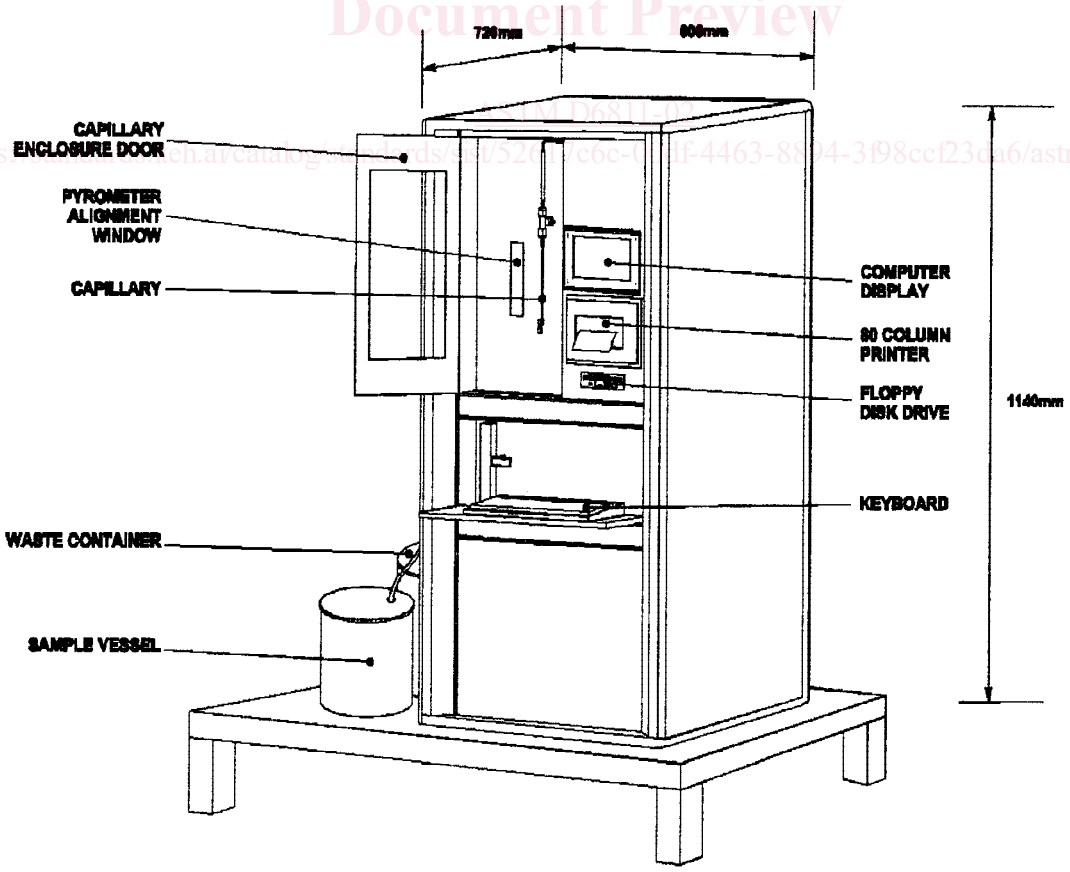

FIG. A1.1 HiReTS Tester