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An American National Standard

Standard Test Method for Determination of Water Separation Characteristics of Aviation Turbine Fuel by Small Scale Water Separation Instrument¹

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1. Scope Scope*

- 1.1 This test method covers a procedure to rate the ability of aviation turbine fuels to release entrained and emulsified water when passed through a water-coalescing filter.
- 1.2 Results are expressed as a Water Separation Index (WSI).
- 1.3 The values stated in SI units are to be regarded as standard.
- 1.3.1 Exception—Units in WSI are included.
- 1.4 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.
- 1.5 This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

2. Referenced Documents

2.1 ASTM Standards:²

D1655 Specification for Aviation Turbine Fuels

D2550 Method of Test for Water Separation Characteristics of Aviation Turbine Fuels (Withdrawn 1989)³

D3602 Test Method for Water Separation Characteristics of Aviation Turbine Fuels (Withdrawn 1994)³

D3948 Test Method for Determining Water Separation Characteristics of Aviation Turbine Fuels by Portable Separameter

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

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

D6300 Practice for Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products, Liquid Fuels, and Lubricants

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.J0.05 on Fuel Cleanliness.

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

³ The last approved version of this historical standard is referenced on www.astm.org.



D7224 Test Method for Determining Water Separation Characteristics of Kerosine-Type Aviation Turbine Fuels Containing Additives by Portable Separometer

3. Terminology

- 3.1 Definitions:
- 3.1.1 *surfactant*, *n*—*in petroleum fuels*, surface active material (or surface active agent) that could disarm (deactivate) filter separator (coalescing) elements so that free water is not removed from the fuel in actual service.
 - 3.1.1.1 Discussion—

Technically, surfactants affect the interfacial tension between water and fuel, which affects the tendency of water to coalesce into droplets.

- 3.2 Definitions of Terms Specific to This Standard:
- 3.2.1 *sonicator*, n—a device that applies ultrasonic sound energy to the test specimen.
 - 3.2.1.1 Discussion—

The sonicator is used to emulsify the water and aviation fuel.

3.2.2 *water separation index (WSI)*, *n*—a numerical rating indicating the ease of separating water from fuel by coalescence. 3.2.2.1 *Discussion*—

A high WSI indicates a fuel that separates water easily and is relatively free from surfactants.

4. Summary of Test Method

4.1 A fixed volume of test specimen is poured into the test beaker. The apparatus is purged with the test specimen. A precise amount of water containing a specific dye is added to the test beaker. The test specimen and dyed water are emulsified using a sonicator. The resulting emulsion is passed at a constant rate directly to the detector, which is sensitive to the dye, to measure a reference value. The emulsion is then passed at the same constant rate to the detector by means of a filter cartridge that is designed to remove entrained water. Readings from the detector are taken. The water separation characteristic, the Water Separation Index (WSI), is calculated from the reference value and the detector readings. Results range from 0.0 WSI to 100.0 WSI. A high value such as 100.0 WSI indicates a test specimen that coalesces water easily and that the test specimen is relatively free of surfactants.

5. Significance and Use

- 5.1 This test method provides an indication of the presence of surfactants in aviation fuel. Like Test Methods D2550, D3602, D3948, and D7224, this test method can detect carryover traces of refinery treating residues in fuel as produced. In addition, these test methods can detect surface active substances added to or picked up by the fuel during handling from point of production to point of use. Certain additives can affect the WSI. Some of these substances affect the ability of filter separators to separate free water from the fuel.
- 5.2 The small scale water separation tester has a measurement range from 0.0 WSI to 100.0 WSI.

Note 1—WSI values greater than 100.0 WSI can be caused by a reduction in the light transmittance (see A1.1.5) of the test specimen due to material that was removed during the testing process.

5.3 This test method was developed so refiners, fuel terminal operators, pipelines, and independent testing laboratory personnel can rapidly and precisely measure for the presence of surfactants, with a minimum of training, in a wide range of locations.

6. Apparatus

- 6.1 *General*—The apparatus, as detailed in Annex A1, comprises a test beaker, test beaker holder, sonicator, filter cartridge, specific dye detector, integral computer, automatic solenoid valves, pumps, solvent container, waste container, particulate sieve, and temperature probe.
- 6.2 Pipet, single use, disposable, of suitable size.
- 6.2.1 The single use disposable pipet is used to adjust the volume of test specimen in the test beaker to 220 mL \pm 10 mL.

7. Reagents and Materials

- 7.1 Dyed Water⁴—Proprietary liquid containing water and a controlled amount of specific marker dye.
- 7.2 Reference Materials:
- 7.2.1 Dispersing Agent—Toluene solution containing 1 mg/mL of solid (100 % dry) bis-2-ethylhexyl sodium sulfosuccinate.
- 7.2.2 *Reference Fluid Base*—A surfactant-free aviation turbine fuel that is used to verify proper operation and is prepared in the manner detailed in Appendix X1 and with a WSI by this test method of 97.5 WSI to 100.0 WSI.
- 7.2.3 Reference Fluids—For checking operational performance consisting of dispersing agent (7.2.1) added to reference fluid base (7.2.2) in concentrations, on a volume basis, ranging from 0 mL/L to 0.8 mL/L, in 0.1 mL/L increments. Reference fluids may be made up in situ in the graduated beaker on an ad-hoc basis, or supplied pre-made up in a suitable container. Typical values are shown in Table 1.
- 7.3 Cleaning Materials, technical grade.
- 7.3.1 Isopropyl Alcohol.
- 7.4 Filter Cartridge, see A1.1.11, individually packed.
- 8. Sampling and Test Specimen Preparation
- 8.1 Unless otherwise agreed, samples shall be taken in accordance with Practice D4057 or Practice D4177.
- 8.2 Collect the sample directly in a suitable container of a minimum volume of 1000 mL.

Note 2—The test method is known to be sensitive to trace contamination, for example from sampling containers or transfer glassware. For recommended sampling containers, refer to Practice D4306.

- 8.2.1 Epoxy-lined containers shall be visually inspected to ensure that the lining has not been damaged and that the containers are not dented. /standards.iieli.ai/catalog/standards.i
- 8.3 Do not pre-filter the sample as the filter media can remove the surfactants that the test method is designed to detect. If the test fuel contains particulate, then allow such materials to settle out before sub sampling.
- 8.4 Special precautions concerning sampling techniques are discussed in Appendix X2. Wipe the container outlet thoroughly with a clean, lint-free wipe. Take extreme care when pouring the sample directly into the test beaker to avoid contamination from the top of the container.
- 8.5 If the sample for test is not within the temperature range 18 °C to 29 °C, then allow the sample to stand until it is within this range.

TABLE 1 Reference Fluid Dispersing Agent Concentrations

Concentration (mL/L)	WSI
0	100.0
0.2	97.6
0.4	79.8
0.6	75.4
0.8	68.8

⁴ The sole source of supply of the dyed water known to the committee at this time is Stanhope-Seta/D-2 Incorporated, Falmouth, MA. 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, which you may attend.



9. Preparation of Apparatus

- 9.1 General—Follow the manufacturer's instructions for the correct set up, verification, calibration, and operation of the apparatus.
- 9.2 Location of Apparatus—Locate the apparatus on a solid surface in a well-ventilated area.
- 9.3 Filter Cartridge—At the beginning of each test, replace the filter cartridge and wipe the sample inlet tube, thermometer probe, and sonicator with a clean, lint-free wipe.
- 9.4 Test Beaker—Before each test, ensure that the test beaker is clean and dry.

Note 3—To avoid contamination, it is recommended to use a new test beaker for each test.

- 9.5 Cleaning Solvent—Check visually that there is sufficient isopropyl alcohol available in the solvent reservoir.
- 9.6 *Dyed Water*—Check visually that there is sufficient dyed water in the water reservoir to cover the inlet tube.
- 9.7 Switch on the apparatus in accordance with the manufacturer's instructions.
- 9.8 Purge the dyed water into a waste container to remove trapped bubbles of air in accordance with the manufacturer's instructions.

10. Calibration, Verification and Standardization

- 10.1 Follow the manufacturer's instructions for verification and calibration of the mechanical and electronic systems in the apparatus.
- 10.2 Verification:
- 10.2.1 Flow Rate—Verify that the flow rate is 25 mL/min to 30 mL/min, at least once every six months according to the manufacturer's instructions. If the flow rate is not correct, follow the manufacturer's instructions to diagnose and adjust.
- 10.2.2 Water Volume—Verify that the correct volume of dyed water (0.060 mL \pm 0.005 mL) is dispensed following the manufacturer's instructions.
- 10.2.3 *Temperature Probe Calibration*—Verify the temperature at 20 °C following the manufacturer's instructions. Recalibrate the temperature probe if the temperature measurement is not within 1 °C.
- 10.2.4 Overall Operation—Check the overall operation of the apparatus using a reference fluid containing 0.4 mL/L of dispersing agent, and with reference fluid containing 0 mL/L dispersing agent as required for quality control or at least every six months. Test the reference fluid following the procedure detailed in Section 11. Confirm that the value is between 75.0 WSI and 85.0 WSI for the 0.4 mL/L dispersing agent and between 97.5 WSI and 100.0 WSI for the 0 mL/L dispersing agent.

11. Procedure

- 11.1 Refer to the diagram for a description of the apparatus (Appendix X1).
- 11.2 Load a new filter cartridge into the holder.
- 11.3 Wipe the sonicator and inlet tube using a lint-free wipe.
- 11.4 Gently tumble the test specimen in its original container end over end five times.

- 11.5 Pour 220 mL \pm 10 mL of test specimen into a clean test beaker—if required, adjust the volume with a single use disposable pipet.
- 11.6 Put the test beaker into the test beaker holder on the apparatus.
- 11.7 Insert the inlet tube, sonicator and temperature probe—if required, wait for the temperature to be between 18 °C and 29 °C.
- 11.8 Press Start on the apparatus. The following steps occur automatically.
- 11.9 The test specimen is pumped into the apparatus to purge the previous sample.
- 11.10 An aliquot of dyed water, $0.06 \text{ mL} \pm 0.01 \text{ mL}$, is added to the test specimen in the test beaker.
- 11.11 The sonicator cycles on and off for 3 min \pm 0.5 min to form a test specimen/dyed water emulsion.
- 11.12 The emulsion is pumped to the detector and on to the waste container. The detector reading is stored every second.
- 11.13 The detector is cleaned with isopropyl alcohol.
- 11.14 The emulsion is pumped through the filter cartridge and detector and then to the waste container.
- 11.15 The detector readings are stored continuously and the test is completed after 5.6 min \pm 0.2 min.
- 11.16 The result is automatically calculated and displayed.

Note 4—Users are advised to check that there is less than 50 mL (typical) left in the beaker at the end of the test.

12. Calculation

12.1 The Water Separation Index (WSI) is automatically calculated by an algorithm (Version 1.0) in the apparatus to the nearest 0.1.

13. Report

- 13.1 The test report shall contain at least the following information:
- 13.1.1 A reference to this standard.
- 13.1.2 All details necessary for complete identification of the product tested.
- 13.1.3 The result of the test (see Section 12) to the nearest 0.1.
- 13.1.4 Any deviations, by agreement or otherwise, from the procedures specified, and
- 13.1.5 The time and date of the test.

14. Precision and Bias

- 14.1 *General*—The precision values given in 14.2 and 14.3 were derived from a 2015 laboratory study that used eight instruments and individual operators at a single location, to test 16 samples in duplicate and in random order. The calculation method that converts individual detector readings into the final result was improved after the ILS. Further details are available in the research report.⁵
- 14.1.1 As the precision was determined from results obtained at a single location, the reproducibility <u>value</u> may not be comparable when results obtained at different times and locations are compared, due to sampling, shipping, storage, and environmental factors. In practice, two results obtained from different locations would be within test method precision if their absolute difference did not exceed the <u>published reproducibility.conditions.</u>
- 14.1.2 The precision was obtained by statistical examination of the laboratory study test results according to ISO 4259(ASTM Practice D6300) D6300 using ADJ6300 D2PP.
- 14.1.3 A single site interlaboratory study (ILS) was carried out for the following reasons: to eliminate sample degradation due to extended transit periods and unknown shipping conditions, and was approved by the subcommittee.
- Note 5—Due to known sample degradation from transportation and shipping conditions, in lieu of standard reproducibility, it is only possible to determine the "intermediate precision" of this test method as specified in Form and Style for ASTM Standards. To determine precision of the test method without the precision being impacted by property changes, it is necessary to bring the operators and apparatus to a single location to conduct the ILS, thus alleviating the need to ship ILS samples. As required in Practice D6300, for the precision calculation, each unique operator/apparatus combination is treated as a 'laboratory'. The precision value calculated is labeled as 'reproducibility, single site' and is a form of intermediate precision.
- 14.2 *Repeatability*—The difference between successive test results obtained by the same operator with the same apparatus under constant operating conditions on nominally identical test material would, in the normal and correct operation of the test method, exceed the value below only in one case in 20:about 5 % of the time (one case in 20 in the long run):

Repeatability =
$$0.35 \cdot (105.66 - x)$$
 WSI (1)

where:

- x = the average of the results being compared.
- 14.2.1 See Table 2 for a tabular illustration and Appendix X3 for a graphical illustration of this relationship.
- 14.3 <u>Reproducibility—Reproducibility, Single Site—</u> The difference between two test results independently obtained by different operators using different apparatus on nominally identical test material <u>at the same location</u> would, in the normal and correct operation of the test method, exceed the value below only in one case in 20: about 5 % of the time (one case in 20 in the long run):

Reproducibility (R) =
$$0.41 \cdot (105.66 - x)$$
 WSI (2)

Reproducibility, Single Site =
$$0.41 \cdot (105.66 - x)$$
 WSI (2)

where:

x = the average of the two results being compared.

TABLE 2 Calculated Values for Repeatability and Reproducibility for Typical WSI Values

WSI	Calculated Repeatability (WSI)	Calculated Reproducibility (WSI)
100.0	2.0	2.3
90.0	5.5	6.4
80.0	9.0	10.5
70.0	12.5	14.6
60.0	16.0	18.7

⁵ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1843. Contact ASTM Customer Service at service@astm.org.

- 14.3.1 See Table 2 for a tabular illustration and Appendix X3 for a graphical illustration of this relationship.
- 14.4 Bias—The procedure in this test method has no bias because the value of WSI is defined only in terms of this test method.

15. Keywords

15.1 aviation fuel; AVTUR; surfactant; water separation; WSI

ANNEX

(Mandatory Information)

A1. APPARATUS DETAILS

- A1.1 General—The apparatus⁶ as shown is self contained and operates automatically to measure the water separation index (WSI).
- A1.1.1 Sonicator, 40 kHz Ultrasonic mixer to emulsify the water and test specimen.

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A1.1.2 Test Specimen Pump, with the capacity to pump up to 40 mL/min ($\pm 1 \text{ mL/min}$) with the ability to be controlled to a flow rate of 25 mL/min to 30 mL/min to pump the test specimen into the apparatus.

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A1.1.3 Water Pump, 5 μL (±1 μL) per revolution—to dispense the dyed water into the test specimen.

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- A1.1.4 Solvent Pump, with the capacity to pump up to 40 mL/min (±1 mL/min) to dispense solvent to clean the detector.
- A1.1.5 *Specific Dye Detector*, the detector measures the total fluorescence of a dye. The detector is tuned to be sensitive to the specific target dye contained in the applied dyed water.
- A1.1.6 Solenoid Valves, to divert flow through the filter cartridge.
- A1.1.7 Waste Container, to collect the test specimen and cleaning solvent after measurement.
- A1.1.8 Solvent Container, to dispense solvent to the apparatus.
- A1.1.9 *Temperature Probe*, platinum resistance thermometer to measure the sample temperature, capable of measuring temperature within 0.5 °C.

⁶ The sole source of supply of the apparatus, beaker and filter cartridge, known to the committee at this time is Stanhope-Seta/D-2 Incorporated, Falmouth, MA. 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 which you may attend.