Designation: D8148 – 22

Standard Test Method for Spectroscopic Determination of Haze in Fuels[1]

This standard is issued under the fixed designation D8148; 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 spectroscopic procedure for determining the level of suspended water and particulate contamination (haze) in liquid middle distillate fuels including those blended with synthesized hydrocarbons or biofuels.

1.1.1 An ordinal, whole-number, Instrument Haze Rating (IHR) from 1 to 6 and a Haze Clarity Index (HCI) from 50.0 to 100.0 are determined on a test specimen at a temperature of 22.0 °C ± 2.0 °C.

1.1.1.1 The stated precision only applies at this temperature.

1.1.1.2 Fuels analyzed at other test specimen temperatures may not be as precise.

1.2 The values stated in SI units are to be regarded as standard. Other units of measurement included in this standard are defined in Section 3.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *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]

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4175 Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants

D4176 Test Method for Free Water and Particulate Contamination in Distillate Fuels (Visual Inspection Procedures)

D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

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

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology D4175.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *check standard cuvette, n*—cuvette containing a certified solid semi-transparent material that is used to confirm instrument operation and calibration.

3.2.2 *clear-and-bright (also termed clean-and-bright), adj*—a condition in which the fuel is free of haze or cloudiness.

3.2.2.1 *Discussion*—This clear and bright definition does not consider the presence of visible water droplets or solid particulates and is restricted to the presence of visible haze or cloudiness in the sample.

3.2.3 *crown glass, n*—a type of optical glass having a low refractive index and low dispersion.

3.2.4 *cuvette transfer time, n*—a user-set timed event that limits the amount of time the operator has to prepare the test specimen and instrument for analysis.

3.2.5 *Haze Clarity Index (HCI), n*—a numerical value from 50.0 to 100.0 that indicates fuel clarity derived from spectroscopic measurements and an algorithm.

3.2.5.1 *Discussion*—The HCI values increase with sample clarity and ranges from 100.0 HCI (very clear and bright) to 50.0 HCI (very cloudy and opaque). Accordingly, a fuel with an HCI value of 90.0 has less haze than a fuel with an HCI value of 80.0. HCI can be used to evaluate haze intensity changes within a given IHR.

3.2.6 *Instrument Haze Rating (IHR), n*—an ordinal, whole-number from 1 to 6, that corresponds to haze ratings defined in Test Method D4176, Procedure 2 and is assigned to the test specimen based upon spectroscopic measurements and an algorithm.

3.2.7 *optically clear, adj*—transparent to light having wavelengths from 340 nm to 2500 nm.

[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 D02.14 on Stability, Cleanliness and Compatibility of Liquid Fuels.

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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.

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

3.2.8 *sample shaking time, n*—a user-set timed event that establishes the duration of sample agitation or swirling prior to beginning test specimen preparation.

3.3 *Abbreviations:*

3.3.1 *D*—depth

3.3.2 *H*—height

3.3.3 *HCI*—Haze Clarity Index

3.3.4 *IHR*—Instrument Haze Rating

3.3.5 *IR*—infrared

3.3.6 *LED*—light emitting diode

3.3.7 *NIR*—near-infrared

3.3.8 *W*—width

4. Summary of Test Method

4.1 The test unit (sample) is conditioned (allowed to heat or cool) to a test temperature of 22.0 °C ± 2.0 °C. **Warning—** Some laboratory analyzed fuel samples may not exhibit the presence of haze found when the sample was originally collected. See Appendix X1.

4.2 Proprietary optics, software, and calibration materials are used to provide a numerical Instrument Haze Rating (IHR) and Haze Clarity Index (HCI) of the fuel sample after a test specimen is placed into an optically clear cuvette and measured according to Beer-Lambert Law for percent transmittance and nephelometric principles for percent light scatter.

4.2.1 The IHR and HCI are obtained by comparison of the measurements in 4.2 to a previously prepared calibration curve and applied to an algorithm to obtain:

4.2.1.1 An ordinal instrument haze rating (IHR) from 1 to 6 corresponding to those described for Test Method D4176, Procedure 2.

4.2.1.2 A Haze Clarity Index (HCI) from 50.0 to 100.0 that may be used to evaluate haze intensity in general.

5. Significance and Use

5.1 It has long been the practice to include in fuel specifications a requirement that the fuel is clear and bright.

5.2 One primary cause for failure to meet this specification requirement is the occurrence of a cloudy or hazy appearance caused by suspended solid particulates or water or some combination of both.

5.2.1 This cloudiness or haze can range from barely visible to opaque.

5.3 This test method provides an objective analytical means for providing a haze rating that does not depend on subjective visual ratings that typically vary with operator and lighting conditions.

6. Apparatus

6.1 *Haze Rating Instrument,*[3] meeting the following specifications:

NOTE 1—All precision testing has been performed using Color Choice hz and Clarity Choice hz instruments. The precisions statistics provided in Section 16 will not apply to tests performed using other instrumentation.

6.1.1 The instruments are portable and self-contained units operating on an alternating current (ac) power source. Power cords are furnished for various voltages.

6.1.2 Both instrument models are identically sized and share the same control panel as shown in Fig. 1.

6.1.3 All of the controls are located in a push-button array on the control panel. Both instrument types may also be controlled by the touch screen or by computer mouse.

6.1.4 The spectrometer, nephelometric sensors, and test specimen temperature measuring device are located in the test specimen cuvette chamber.

6.1.4.1 *Spectrometer/Nephelometer,* consisting of a combination of a near-infrared (NIR) light-emitting diode (LED) light source and photodiodes positioned to measure transmission and scatter across the test specimen.

6.1.4.2 *Temperature Measuring Device,* non-contact infrared (IR) thermopile-based temperature sensor, with an accuracy of ±2.0 °C and a range of 4.0 °C to 38.0 °C.

6.2 *Check Standard Cuvette(s),*[3] one or more 10.0 mm W by 15.0 mm D by 38.0 mm H inside dimension crown glass cuvettes having four optically clear walls that contain a certified solid semi-transparent material (optional).

6.3 *Crown Glass Cuvette,* inside dimensions 10.0 mm W by 15.0 mm D by 38 mm H; optically clear on all four sides.

6.3.1 Cuvette dimensions are ±0.1 mm.

6.4 *Printer,* (optional).

[3] The sole source of supply for the apparatus, calibration materials and check standard cuvettes known to the committee at this time is Choice Analytical, Inc., 527 21st St., Suite 327, Galveston, TX 77550, www.choiceanalytical.com. 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. 1 Haze Apparatus Models

6.5 *Temperature-Controlled Bath,* of suitable dimensions and capable of controlling the sample container temperature within ±0.5 °C of the desired temperature for laboratory tests that require measurements to be made at a specific temperature (optional).

7. Reagents and Materials

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society,[4] where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 *Haze Calibration Set,* consisting of six calibration materials certified to correspond to the ordinal number 1 to 6 haze rating described by Test Method D4176, Procedure 2.

7.2.1 Haze Calibration Sets are stable for up to one year unopened.

7.2.2 Select proprietary quality control procedures are employed to ensure calibration set consistency.

7.3 *n-Dodecane,* $CH_3(CH_2)_{10}CH_3$ ≥99.0% purity.

7.4 Expendable materials needed to perform the test consist of the following:

7.4.1 Disposable dispensing pipet, approximate 5 mL capacity.

7.4.2 Lint-free optical lens wipes.

8. Hazards

8.1 Hazards are typical of those experienced when handling fuel. There are no additional hazards associated with this test method.

9. Sampling

9.1 Obtain a test unit (sample) in accordance with Practice D4057 or Practice D4177.

10. Preparation of Apparatus

10.1 Power-up (turn on) the apparatus and allow sufficient warm-up time as indicated by the instrument ready status message located on the apparatus home screen.

10.2 Follow manufacturer's recommendations to enter the Update Haze Settings mode.

10.2.1 Highlight each respective field and set sample shaking time to 15.0 s, cuvette transfer time to 45.0 s, and time between scans to 1000.0 ms using the on-screen numeric display.

10.2.1.1 Users may choose alternative sample shaking, cuvette transfer times, and between scan times to accommodate operator experience, sample characteristics, or other factors; however precision and bias data provided in Section 16 do not apply.

10.2.2 Activate the displayed Save button and upon confirmation that settings have been successfully saved, activate the Next or Home button to exit the Update Haze Settings mode.

11. Calibration

11.1 Obtain a temperature-conditioned (see 12.1) high purity dodecane (7.3) material and a temperature-conditioned (see 12.1) instrument manufacturer supplied haze calibration set (7.2).

11.2 Follow manufacturer's recommendations to enter the Haze Calibration mode.

11.2.1 Activate the Next button to display the calibration queue; Calibration Reference is highlighted.

11.2.2 Activate the Next button to display the analysis Start screen.

11.3 Analyze the dodecane as the reference material as follows:

11.3.1 Vigorously shake the dodecane sample to suspend any sediment or silt.

11.3.2 Carefully inspect the container to ensure all sediment and silt is suspended.

11.3.3 Briefly place the container on a stable surface and allow visible air bubbles to rise or dissipate (typically less than 30.0 s).

11.3.4 Once air bubbles have risen or dissipated, promptly activate the instrument by pressing the Start button (sample shaking time starts).

11.3.5 During the time allowed, gently shake or swirl the dodecane to ensure sediment or silt remains suspended in the sample.

11.3.6 Once sample shaking time has elapsed, as prompted by the instrument, cease agitation (cuvette transfer time starts).

11.3.7 The instrument automatically displays and counts down the cuvette transfer time.

11.3.7.1 Open the dodecane sample container and use a disposable dispensing pipette (7.4.1) to promptly rinse a clean cuvette (6.3) with the material to be tested.

11.3.7.2 Dispose of the rinse material and use the pipette to fill the cuvette to its 80 % to 90 % full capacity (approximately 5 mL of test specimen).

11.3.7.3 Clean all four cuvette optical surfaces to remove any smudges or liquids with a lint free wipe (7.4.2). Open the test compartment and place the test specimen into the instrument haze cuvette holder.

11.3.8 When the cuvette is installed and its testing chamber is closed within the allotted cuvette transfer time, the test specimen analysis is performed automatically.

11.3.9 Remove the cuvette and dispose of the test specimen.

11.4 Follow on screen instructions to analyze each of the calibration materials to create a 1 to 6 instrument haze rating calibration curve.

11.4.1 Following the reference material analysis, the calibration queue is displayed and calibration point one is highlighted.

[4] *ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

11.4.2 Activate the Next button to display the analysis Start screen.

11.4.3 Follow procedures described for the dodecane reference material in 11.3.1 – 11.3.9 to analyze the haze rating 1 calibration material.

11.4.4 Following the calibration point 1 analysis, the calibration queue is again displayed and calibration point 2 is highlighted.

11.4.5 Follow procedures described in 11.4.2 and 11.4.3 to analyze calibration point 2 and each of the four remaining haze rating calibration materials.

11.5 Following analysis of the haze reference and all six calibration materials, activate the displayed Next button to enter the calibration review screen.

11.6 Confirm that each of the 1 to 6 calibration points has an HCI value.

11.7 Highlight then activate the Calibration Accept button.

11.8 When the Calibration Accepted message is displayed, activate the Home button to exit the calibration mode.

11.9 Optionally, confirm instrument operation by analyzing one or more Check Standard Cuvettes (6.2) following procedures described in Appendix X2.

12. Conditioning

12.1 *Sample Conditioning*—Heat or cool the sample until its temperature has stabilized at 22.0 °C ± 2.0 °C.

12.1.1 Samples can be analyzed at other temperatures; however, the precision and bias statement described in Section 16 does not apply.

12.2 No instrument conditioning is required.

13. Procedure

13.1 Obtain a test sample using one of the procedures described in Section 9.

13.2 From the apparatus' Home screen, activate the Test button to enter the Test mode.

13.3 Follow displayed prompts to enter operator, single test, and sample information as required.

13.4 Follow procedures described in 11.3.1 – 11.3.9 for the dodecane reference material to analyze the sample.

13.5 Record the IHR result, HCI result, and test specimen temperature as reported by the instrument.

13.6 Activate the Test button to perform another analysis or the Home button to exit the test mode.

14. Report

14.1 Report the individual sample qualities as follows:

14.1.1 Instrument Haze Rating (IHR)—1, 2, 3, 4, 5, or 6.

14.1.2 Temperature of test specimen in degrees Celsius (°C).

14.1.3 Haze Clarity Index (HCI)—HCI values (50.0 to 100.0).

14.2 State that the results were obtained according to Test Method D8148.

15. Quality Control

15.1 Each day of use, following calibration or as prompted by apparatus on-screen instructions, perform the following:

15.1.1 With the apparatus in the test mode, select the reference test option and analyze the dodecane reference material as described in 11.3.

15.1.2 Following the dodecane reference test, activate the Test button to analyze the dodecane reference material as a sample; follow displayed prompts to enter operator, single test, and sample information.

15.1.3 Activate the Next button to display the Start analysis screen.

15.1.4 Follow procedures described in 11.3.1 – 11.3.9.

15.1.5 Haze rating analysis is performed automatically.

15.1.6 If IHR is >1, or, if HCI value is <98.0, the instrument is deemed to have failed the Quality Control requirement. Investigate for root cause.

16. Precision and Bias

16.1 Statistical analysis from two separate interlaboratory studies (ILSs) follow.

16.2 *Precision and Bias for Instrument Haze Rating (IHR)*—Because the IHR results are non-quantitative, no information is presented about either the precision or bias of this test method for the determination of IHR in the materials described in its scope.

16.2.1 An Interim Repeatability Study (Appendix X3) was completed to assess the within-operator rating consistency of IHR.[5]

16.2.1.1 For the ILS-1406, one laboratory conducted a pilot study and two laboratories provided data for the repeatability study pursuant to Practice D6300 guidelines, (that is, each laboratory used separate instrumentation and operators). Each laboratory received a total of 40 samples consisting of five randomized replicates taken from 8 bulk samples that included 4 fuel grades in duplicate. The bulk samples were from multiple sources and had Test Method D4176, Procedure 2, haze levels (1 to 6).

16.2.1.2 *Within-operator/Apparatus Rating Consistency*—For Haze ratings repeated 5 times by the same operator within a short interval of time using the same apparatus at the same location, the probability of no disagreement amongst the 5 ratings (that is, unanimous ratings for all 5 IHR) is approximately 80 %; the probability of no more than 1 disagreement amongst the 5 repeats is approximately 20 %.

16.3 *Precision and Bias for Haze Clarity Index (HCI)*[6]:

16.3.1 For the ILS-1726, testing was completed at one laboratory over a two-day period by 10 separate entities. Each entity consisted of a separate operator from separate companies assigned to one of ten separate analyzers. Each entity received a total of 20 samples consisting of randomized replicates taken

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1867. Contact ASTM Customer Service at service@astm.org.

[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-2028. Contact ASTM Customer Service at service@astm.org.

from 10 bulk samples that included five fuel types and a check standard control. The bulk samples were from multiple sources and had Test Method D4176, Procedure 2, haze levels (1 to 6). Results were collected and analyzed pursuant to Practice D6300 guidelines.

16.3.2 *Repeatability (r)*—The difference between two independent results obtained by the same operator in a given laboratory applying the same test method with the same apparatus under constant operating conditions on identical test material within short intervals of time would, in the normal and correct operation of the test method, exceed the following values about 5 % of the time (one case in 20 in the long run):

$$r = 0.24(X)^{0.5} \quad (1)$$

where X is the average of the two HCI results.

16.3.3 *Intermediate Precision (Between Operator/Apparatus Repeatability)*—The difference between two single and independent results obtained by different operators applying the same test method in the same laboratory using different apparatus on identical test material within short intervals of time would, in the normal and correct operation of the test method, exceed the following values about 5 % of the time (one case in 20 in the long run):

$$\text{Between Operator/Apparatus Repeatability} = 0.3726(X)^{0.5} \quad (2)$$

where X is the average of the two HCI results.

16.3.4 *Reproducibility*—Due to the instability of the measurand (HCI property) of this test method over time, it is not possible to determine reproducibility of this test method using traditional interlaboratory studies in strict adherence with Practice D6300.

16.3.5 *Bias for HCI*—No information is presented about the bias of this test method for measuring HCI because this property is defined and recognized only by this test method.

17. Keywords

17.1 cleanliness; haze; liquid fuels; spectroscopy

APPENDIXES

(Nonmandatory Information)

X1. HAZE TEMPERATURE HISTORY CONSIDERATIONS

X1.1 Haze formation and visual appearance for the presence of water are not ‘intrinsic properties’ of a fuel, but are a function of the temperature of the fuel and the temperature-history of the fuel. For example, a fuel can be hazy at the point of sampling, and when the sample warms up, the water induced haze can disappear, but when the sample is re-cooled to the original temperature, haze may not reappear. This is because water contained in the sample can form a film or droplets on the side of the container, or even a separate lower layer of water can be formed instead of a haze throughout the sample. When haze determination is providing important point of sale/transfer information, it may not be practical or proper to take a sample, store or ship, and later test the sample for the presence of haze under different conditions than those present at the time and temperature of sale or transfer.

X2. CHECK STANDARD CUVETTE TESTING

X2.1 From the Home screen, activate the Test button to enter the Test mode.

X2.2 Follow displayed prompts to enter operator, single test, and sample information as required.

X2.3 Activate the instrument by pressing the Start button (Shaking Time starts).

X2.4 Once sample shaking time has elapsed, the instrument automatically displays and counts down the cuvette transfer time.

X2.5 Clean all four cuvette optical surfaces to remove any smudges with a lint-free wipe (7.4.2).

X2.6 Open the test compartment and place the Check Standard Cuvette into the instrument haze cuvette holder.

X2.7 When the cuvette is installed and its testing chamber is closed within the allotted cuvette transfer time, the test specimen analysis is performed and displayed automatically.

X2.8 Instrument operation may be verified by confirming the following:

X2.8.1 An instrument haze rating (IHR) value of 1, 2, 3, 4, 5, or 6 is displayed and matches the certified instrument haze rating (IHR) value for the Check Standard Cuvette.

X2.8.2 A haze clarity index (HCI) value from 50.0 to 100.0 is displayed.

X2.8.3 A test specimen temperature that is within ±2.0 °C of the temperature at which the Check Standard Cuvette was stored is reported.

X2.9 Record the IHR result, haze clarity index (HCI), and test specimen temperature as reported by the instrument as required.

X2.10 Remove the Check Standard Cuvette and activate the Home button to exit the test mode.