Designation: D8545 – 23ε1

Standard Test Method for Determination of the Metal Deactivator Additive (MDA) (N,N'-disalicylidene-1,2-propanediamine) Content in Aviation Turbine Fuel by High Performance Liquid Chromatography (HPLC)[1]

This standard is issued under the fixed designation D8545; 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.

ε[1] NOTE—Editorially corrected 5.1 in February 2024.

1. Scope

1.1 This test method covers the determination of the metal deactivator additive (MDA) content of aviation turbine fuels. The specific MDA determined and used to develop this test method is N,N'-disalicylidene-1,2-propanediamine. Other MDAs have not been tested by this test method.

1.1.1 This test method specifically covers the determination of uncomplexed MDA content in aviation turbine fuel. MDA is a chelator of divalent metal ions, and the MDA-metal ion complexed species content of aviation turbine fuel will not be accounted for by this test method.

1.2 This test method is divided into two procedures: (*1*) Procedure A uses a semi-portable capillary-liquid chromatography system (Capillary-HPLC) that may be used in the field or laboratory; (*2*) Procedure B uses a standard laboratory version of liquid chromatography (Conventional-HPLC). Procedures A and B have separate precisions.

1.3 The test method has an interim repeatability determined in accordance with Practice D6300. Based on the mean values of the samples used in the interim repeatability study, Procedure A is applicable in the range of 0.50 mg⁄mL to 10.0 mg⁄mL; the range for Procedure B is 0.60 mg⁄mL to 9.6 mg⁄mL. Higher concentrations can be determined by dilution, but the precision of the test method has not been determined.

1.3.1 An extended interlaboratory study (ILS) will be conducted in the future to determine the full repeatability and reproducibility and the final applicable concentration ranges.

1.3.2 The test method applies to MDA in petroleum-based aviation fuels and Synthetic Aviation Fuels (SAF). However, for the interim precision, a petroleum-based aviation fuel was used. Future ILS will include petroleum-based and SAFs. The test method is applicable to aviation fuels conforming to Specification D1655.

1.4 Appendix X2 indicates other additives that have been verified to not interfere with the analysis of this test method.

1.5 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.6 *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.*

1.7 *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:*

D1655 Specification for Aviation Turbine Fuels

D4052 Test Method for Density, Relative Density, and API Gravity of Liquids by Digital Density Meter

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

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

D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

D6299 Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance

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 D02.04.0C on Liquid Chromatography.

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3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this test method, refer to Terminology D4175.

3.1.2 *aviation turbine fuel, n*—refined petroleum distillate, generally used as a fuel for aviation turbines.

3.1.2.1 *Discussion*—In this test method, aviation turbine fuel refers to petroleum-based aviation fuels and synthetic aviation fuels.

3.1.3 *metal deactivator additive (MDA), n*—a fuel additive that counteracts the effect of trace metals in the bulk fuel.

3.1.3.1 *Discussion*—Trace metals such as copper, cadmium, iron, cobalt and zinc may have an adverse effect on fuel thermal and storage stability as those metals can catalyze oxidation reactions that lead to degradation of the fuel.

3.1.3.2 *Discussion*—In this test method it refers to N,N'-disalicylidene-1,2-propanediamine; other commercially used MDAs have not been tested.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *capillary-HPLC, n*—high performance liquid chromatography using a capillary separation column with typical 1 mm or less internal diameter.

3.2.1.1 *Discussion*—In this test method capillary-HPLC is used in a semi-portable field applicable apparatus (Procedure A).

3.2.2 *conventional-HPLC, n*—high performance liquid chromatography with separation column with >1 mm internal diameter.

3.2.2.1 *Discussion*—In this test method such application usually is used with a laboratory apparatus and may not be portable (Procedure B).

4. Summary of Test Method

4.1 A fuel sample is filtered to remove small particulates. A fixed volume of the filtered test sample is injected into a calibrated high performance liquid chromatograph. An analytical column is used to separate the sample components of the test sample by polarity.

4.2 The analytical column is interfaced to a liquid chromatography detector where the MDA is detected by UV absorption as it elutes from the column. Uncomplexed MDA absorbs light at a maximum of approximately 315 nm. The electronic signal from the liquid chromatography detector is monitored continuously by a chromatography data system. The amplitudes of the signal (peak area) from the MDA are compared with peak areas obtained from previously measured external calibration standards in order to calculate the concentration of mg/L MDA present in the sample.

5. Significance and Use

5.1 Specification D1655 provides a maximum permissible concentration (5.7 mg/L) of MDA in aviation turbine fuel. This test method will allow the quantification of MDA in aviation turbine fuels. The MDA additive is used for fuel thermal stability control and to reduce fuel degradation caused by the presence of trace metals (copper in particular) in aviation fuels.

PROCEDURE A: CAPILLARY-HIGH PERFORMANCE LIQUID CHROMATOGRAPHIC SYSTEMS (SEMI-PORTABLE CAPILLARY-HPLC)

6. Apparatus

6.1 *High Performance Liquid Chromatograph (HPLC)*[2]—Any HPLC capable of column temperature control at 55 °C ± 1 °C and pumping an isocratic mobile phase at flow rates between 1 µL/min and 10 µL/min, with a precision better than 0.5 % and a pulsation of <1 % full scale deflection under the test method conditions.

6.2 *Ultraviolet Photometric Detector or Photometric Diode Array Detector (DAD)*—Capable of operation at 315 nm ± 5 nm.

6.3 *Manual or Automatic Sample Injection Valve*—Capable of injecting 2 µL to 3 µL, using either partial or full loop mode, with a repeatability ±1 %.

6.3.1 Both manual and automatic sample injection systems will, when used correctly, meet the repeatability requirements specified in 6.3.

6.4 *Chromatography Data System*—Any data system can be used, provided it is compatible with the liquid chromatography detector, has a minimum sampling rate of at least 8 Hz, and is able to measure peak areas and retention times and perform post-run data processing such as baseline correction, integration, calibration, and quantitation.

6.5 *Analytical Column*—GL Sciences Inertsil Diol Cat. No. 5020-11523, 3 µm particle size, 50 mm by 0.3 mm ID or equivalent. Other columns that resolve MDA from other fuel components as shown in Fig. 1 and meet all other quantitative and quality control requirements described in this method may be used.

6.6 *HPLC Column Oven*—Any suitable HPLC column oven (block heating or air circulating) capable of maintaining a constant temperature of 55 °C ±1 °C.

6.7 *Analytical Balance*—Accurate to ±0.00001 g (0.01 mg).

6.8 *Syringes*—100 µL or 25 µL such as Hamilton's 81075 and 80275 (gas tight syringes with 2 in. 22-gauge needles and blunt tips) or equivalent.

[2] The sole source of supply of the turnkey apparatus known to the committee at this time is Axcend Corp. 5252 N. Edgewood Dr., #185, Provo, UT 84604. 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.