



Designation: **D7546 – 15** **D7546 – 24**

## Standard Test Method for Determination of Moisture in New and In-Service Lubricating Oils and Additives by Relative Humidity Sensor<sup>1</sup>

This standard is issued under the fixed designation D7546; 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 reappraisal. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reappraisal.

### 1. Scope\*

1.1 This test method covers the quantitative determination of water in new and in-service lubricating oils and additives in the range of 10 mg/kg to 100 000 mg/kg (0.001 wt./wt. to 10 % wt./wt.) using a relative humidity (RH) sensor. Methanol, acetonitrile, and other compounds are known to interfere with this test method.

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

1.3 **Warning**—Samples tested in this test method can be flammable, explosive, and toxic. Use caution when handling them before and after testing.

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 and health, 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:*<sup>2</sup>

[D4175 Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants](#)

### 3. Terminology

3.1 For definitions of terms used in this test method, refer to Terminology [D4175](#).

#### 3.1 Definitions:

3.1.1 For definitions of terms used in this test method, refer to Terminology [D4175](#).

### 4. Summary of Test Method

4.1 An aliquot of sample is heated to a temperature between 25 °C to 200 °C with 1 °C resolution. The sample is maintained at

<sup>1</sup> 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 [D02.96.02](#) on Chemistry for the Evaluation of In-Service Lubricants.

Current edition approved April 1, 2015; Feb. 1, 2024. Published June 2015; February 2024. Originally approved in 2009. Last previous edition approved in 2009 as [D7546 – 09](#); [D7546 – 15](#). DOI: [10.1520/D7546-15.10.1520/D7546-24](#).

<sup>2</sup> 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

a constant temperature for the duration of the test. Dry inert gas flows over the heated sample and carries the thermally evolved moisture past a relative humidity sensor. The sensor signal is integrated over time to provide a measurement of total mass of water in the sample.

4.2 The sample injection may be done either by mass or by volume.

4.3 This test method utilizes anhydrous compressed gas or ambient air passed through a desiccant to prevent contamination from moisture present in the atmosphere.

4.4 Viscous samples can be analyzed by preheating them to place them in a more fluid state allowing them to be drawn into a syringe, or by dissolving them in a compatible anhydrous solvent. Care should be taken to minimize time spent preheating samples to prevent moisture loss.

## 5. Significance and Use

5.1 Knowledge of the water content of lubricating oils, additives, and similar products is important in the manufacture, purchase, sale, transfer, or use of such petroleum products to help in predicting their quality and performance characteristics.

5.2 For lubricating oils, the presence of water can lead to premature corrosion and wear, an increase in the debris load resulting in diminished lubrication and premature plugging of filters, impedance to the effect of additives, and undesirable support of deleterious bacterial growth.

## 6. Interferences

6.1 Methanol and acetonitrile are known to interfere with the determination of moisture by this test method. These substances contribute to a high bias in the final results. More generally, some short-chained polar molecules mimic the effect of water at the RH sensor resulting in a positive interference. Strong polar solvents, such as n-methyl-pyrrolidone, can severely damage the RH sensor.

## 7. Apparatus

7.1 *Sample Injection Moisture Analyzer Apparatus:*

7.1.1 *Flow Regulator*, capable of maintaining the carrier gas flow rate within the manufacturer's specified conditions.

7.1.2 *Flow Meter*, capable of measuring the carrier gas flow rate within the manufacturer's specified conditions.

7.1.3 *Stainless Steel Sample Coil*, for heating the sample as it is transported from the sample inlet to the sump.

7.1.4 *Sample Coil Heater*, capable of maintaining the sample coil temperature within 1 °C of the programmed temperature between 25 °C and 200 °C.

7.1.5 *Sample Delivery System*, provides programmable variable speed injection of the sample into the coil.

7.1.6 *Sump*, allows for collection of the sample at the bottom of the sample coil during testing and discharge of the sample to a waste container after testing is completed.

7.1.7 *Manifold*, which provides:

7.1.7.1 A thermally stable port for mounting and operation of the relative humidity sensor.

7.1.7.2 Inlet and outlet ports for the carrier gas.

7.1.8 *Relative Humidity (RH) Sensor*, a capacitive sensing element that measures the relative humidity of the carrier gas.

7.1.9 *Microcontroller*, which provides:

7.1.9.1 Capability of integrating and converting the RH sensor signal.

7.1.9.2 Capability of controlling the temperature of the coil heater, and the sensor manifold.

7.1.9.3 Capability of controlling the speed of the sample delivery system.

7.1.10 *Balance*, external, with 1 mg precision for weighing sample.

#### 7.2 *Headspace Vial Moisture Analyzer Apparatus:*

7.2.1 *Flow Regulator*, capable of maintaining the carrier gas flow rate within the manufacturer's specified conditions.

7.2.2 *Flow Meter*, capable of measuring the carrier gas flow rate within the manufacturer's specified conditions.

7.2.3 *Sample Heating Chamber*, capable of maintaining the sample chamber temperature within 1 °C of the programmed temperature between 25 °C and 200 °C.

7.2.4 *Sample Delivery System*, transports vial into sample heating chamber.

7.2.5 *Manifold*, which provides:

7.2.5.1 A thermally stable port for mounting and operation of the relative humidity sensor

7.2.5.2 Inlet and outlet ports for the carrier gas.

7.2.6 *Relative Humidity (RH) Sensor*, a capacitive sensing element that measures the relative humidity of the carrier gas.

7.2.7 *Microcontroller*, which provides:

7.2.7.1 Capability of integrating and converting the RH sensor signal.

7.2.7.2 Capability of controlling the temperature of the sample heating chamber, and the sensor manifold.

7.2.7.3 Capability of controlling the speed of the sample delivery system.

7.2.8 *Balance*, external, with 1 mg precision for weighing sample.

## 8. Reagents and Materials

8.1 *Carrier Gas*, any dry inert gas including, but not limited to, dry air, nitrogen, helium, or argon.

8.2 *Water*, deionized.

#### 8.3 *For Sample Injection Moisture Analyzer:*

8.3.1 *Traceable Syringe*, traceable at the desired total mass of water, typically 500 µg (0.5 µL).

8.3.2 *Glass or Plastic Syringe*, 1 mL or 5 mL capacity.

8.3.3 *Needle*, 18 gauge or 22 gauge.

#### 8.4 *For Headspace Vial Moisture Analyzer:*

8.4.1 *Traceable 1 µL microcapillary*, traceable at the desired total mass of water, typically 1000 µg (1.0 µL).