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AnAmerican National Standard

# Standard Test Method for Electrical Conductivity of Liquid Hydrocarbons by Precision Meter<sup>1</sup>

This standard is issued under the fixed designation D4308; 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  $(\varepsilon)$  indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

## 1. Scope\*

- 1.1 This test method covers and applies to the determination of the "rest" electrical conductivity of aviation fuels and other similar low-conductivity hydrocarbon liquids in the range from 0.1 to 2000 pS/m (see 3.1.2). This test method can be used in the laboratory or in the field.
- 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—Mercury has been designated by many regulatory agencies as a hazardous material that can cause central nervous system, kidney and liver damage. Mercury, or its vapor, may be hazardous to health and corrosive to materials. Caution should be taken when handling mercury and mercury containing products. See the applicable product Material Safety Data Sheet (MSDS) for details and EPA's website—http://www.epa.gov/mercury/faq.htm—for additional information. Users should be aware that selling mercury and/or mercury containing products into your state or country may be prohibited by law.
- 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 practices and determine the applicability of regulatory limitations prior to use. For specific warning statements, see 7.1.1, 7.2, 8.3, and Annex A1.

### 2. Referenced Documents

2.1 ASTM Standards:<sup>2</sup>

D150 Test Methods for AC Loss Characteristics and Permit-

tivity (Dielectric Constant) of Solid Electrical Insulation D2624 Test Methods for Electrical Conductivity of Aviation and Distillate Fuels

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

E1 Specification for ASTM Liquid-in-Glass Thermometers

# 3. Terminology

- 3.1 Definitions:
- 3.1.1 *picosiemens per metre*, *n*—the unit of electrical conductivity is also called a conductivity unit (CU). A siemen is the SI definition of reciprocal ohm sometimes called mho.

1 pS/m = 
$$1 \times 10^{-12} \Omega^{-1} \text{ m}^{-1} = 1 \text{ cu} = 1 \text{ picomho/m}$$
 (1)

3.1.2 rest conductivity, n—the reciprocal of the resistance of uncharged fuel in the absence of ionic depletion or polarization. It is the electrical conductivity at the initial instant of current measurement after a dc voltage is impressed between electrodes.

## 4. Summary of Test Method

4.1 A sample of liquid hydrocarbon is introduced into a clean conductivity cell which is connected in series to a battery voltage source and a sensitive dc ammeter. The conductivity, automatically calculated from the observed peak current reading dc voltage and cell constant using Ohm's law, appears as a digital value in either a manual or automatic mode of meter operation.

#### 5. Significance and Use

5.1 The generation and dissipation of electrostatic charge in fuel due to handling depend largely on the ionic species present which may be characterized by the rest or equilibrium electrical conductivity. The time for static charge to dissipate is inversely related to conductivity. This test method can supplement Test Method D2624 which is limited to fuels containing static dissipator additive.

Note 1—For low-conductivity fluids below 1 pS/m in conductivity, an ac measurement technique is preferable to a dc test method for sensing the electrical conductivity of bulk fluid. This dc test method can be used at conductivities from 0.1 to 1 pS/m if precautions are observed in cell cleaning and sample handling. A waiting period of 15 min is required after

<sup>&</sup>lt;sup>1</sup> This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.J0.04 on Additives and Electrical Properties.

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<sup>&</sup>lt;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.



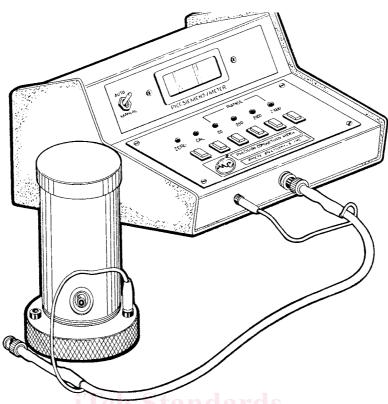


FIG. 1 Precision Conductivity Meter

filling the cell before measuring dc conductivities below 1 pS/m. A single-laboratory program was conducted comparing this test method with ac Test Methods  ${ t D150.}^3$ 

#### 6. Apparatus

- 6.1 *Conductivity Apparatus*—Components of the dc conductivity apparatus are shown in Fig. 1.4,5
- 6.1.1 The conductivity cell shown in Fig. 1 consists of an inner electrode and an outer electrode separated by an insulator. The outer electrode and cap provide a ground path and electrostatic (Faraday) shield.
- 6.1.2 The electrometer shown in Fig. 1 contains a battery which supplies a voltage to the cell and a bridge circuit which senses the flow of current and converts the output signal directly into conductivity units, that is, pS/m. A pushbutton selector allows selection of zero reading, calibration, and four range selections.
- 6.1.3 The cell and electrometer are connected by a triaxial cable as shown in Fig. 1.
- 6.2 *Thermometer*, general purpose type, having a range of 0 to 60°C (see Specification E1). Temperature measuring devices that cover the temperature range of interest, such as an ASTM 1C thermometer, or liquid-in-glass thermometers,

<sup>3</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1241.

thermocouples, or platinum resistance thermometers that provide equivalent or better accuracy and precision than ASTM 1C thermometers may be used.

# 7. Reagents

- 7.1 Cleaning Solvent, The following may be used:
- 7.1.1 Toluene-Isopropyl Alcohol Mixture—(Warning—Flammable. Vapor harmful. See Annex A1.1.) Mix two volumes of toluene and three volumes of isopropyl alcohol both of reagent grade and distill. Discard the first 20 % and last 5 % fractions.
- 7.2 n-Heptane—(Warning—Flammable. Harmful if inhaled. See Annex A1.2.) Prepare by percolating ASTM reference fuel grade n-heptane through silica gel<sup>5,6</sup> as follows:
- 7.2.1 Activate approximately 2000 g of 100 to 200 mesh silica gel by heating at 180°C for 24 h. Allow it to cool in a desiccator under nitrogen or in vacuum. Soak approximately 0.5 g of glass wool<sup>5,7</sup> for 24 h in clean n-heptane.
- 7.2.2 Take a tube of borosilicate glass having an inside diameter of 60 to 70 mm, a length 1500 mm, with a conically shaped lower end provided with a glass cock. Place a perforated porcelain disk (diameter 25 mm) in the lower end of the tube and put the soaked glass wool on top of the disk. Fill the tube with the activated silica gel while tapping to achieve

<sup>&</sup>lt;sup>4</sup> The sole source of supply of the apparatus, the KSLA Cell and Precision Conductivity Meter System, Emcee Model #1154, known to the committee at this time is Emcee Electronics, Inc., 520 Cypress Ave., Venice, FL 34285.

<sup>&</sup>lt;sup>5</sup> 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.

<sup>&</sup>lt;sup>6</sup> The sole source of supply known to the committee at this time is Code 923, from W. R. Grace & Co., Davison Chemical Division, Baltimore, MD 21202.

<sup>&</sup>lt;sup>7</sup> The sole source of supply of the apparatus, filtering fiber Pyrex Wool. Catalogue No. 3950, known to the committee at this time is Owens-Corning Fiber Glass Corp., Toledo, OH.

homogeneous filling. The silica gel layer will be approximately 1250 mm high. Wrap the column in black paper to exclude light.

7.2.3 Percolate *n*-heptane through the column at a rate of about 2 to 3 L/h. Discard the first 3 L. Never allow the column to run dry. The silica gel charge is sufficient for the percolation of 1000 L of *n*-heptane, provided the conductivity of the untreated *n*-heptane is below 1 pS/m.

Note 2—If the conductivity of the *n*-heptane after treatment, measured in accordance with Section 11 in a thoroughly cleaned cell, is higher than 0.1 pS/m, the treatment should be repeated.

7.3 *Hydrocarbon*, for calibration. The dielectric constant must be known to  $\pm 5$  % at the temperature of calibration.<sup>8</sup>

# 8. Sampling

- 8.1 The sample volume should be at least 0.7 L.
- 8.2 Use a clean epoxy-lined can, or a new glass bottle that has been rinsed successively with hot water, distilled water, acetone, and cleaning solvent then flush with dry nitrogen. Use only non-contaminating caps.

Note 3—Test method results are known to be sensitive to trace contamination from sampling containers. For recommended sampling containers refer to Practice D4306.

Note 4—Bottle samples should be *tested immediately*, since the glass surface tends to absorb from the fuel the conductive substances that the test method is intended to measure.

8.3 Rinse the container several times with portions of the hydrocarbon liquid to be sampled. (When testing diesel or aviation turbine fuels Jet A or A-1, **Warning**—Combustible. Vapor harmful. See Annex A1.3.) (When testing gasoline, aviation gasoline, or aviation turbine fuel Jet B, **Warning**—Extremely flammable. Harmful if inhaled. Vapors can cause flash fire. See Annex A1.4.) If possible, fill the container, let stand, then empty and refill. Avoid taking the sample for test by pouring from the container; pipet instead. The sample should be clean and bright when tested.

## 9. Preparation of Apparatus

- 9.1 Cleaning the Cell—The cleaning procedure to be used depends on the estimated conductivity of the sample to be tested.
- 9.1.1 For samples that are expected to exhibit conductivities below 1 pS/m, the KSLA cell should be completely disassembled, the parts cleaned and the cell reassembled using protective gloves.
- 9.1.1.1 Dismantle the cell by removing the loose battery cap, the outer electrode electrical connector and the bottom screw-on cap. Press the inner electrode towards the bottom of the outer electrode and remove the inner electrode TFE-fluorocarbon insulator assembly.
- 9.1.1.2 Each part of the cell should be rinsed thoroughly five times with cleaning solvent followed by rinsing with treated n-heptane. The parts should be dried with a stream of nitrogen gas.
- <sup>8</sup> A standard, such as cyclohexane, with certified dielectric constant, may be obtained from the National Bureau of Standards, Washington, DC 20234.

- 9.1.1.3 After reassembly, the cell should be rinsed with treated *n*-heptane.
- 9.1.1.4 After cleaning, check the cleanliness of the cell by measuring the conductivity of treated *n*-heptane in accordance with Section 11. The corrected value should be lower than 0.05 pS/m
- 9.1.2 For samples that are expected to exhibit conductivities above 1 pS/m, the KSLA cell still assembled should be rinsed five times with cleaning solvent, followed by rinsing with treated *n*-heptane. The cell should be dried with a stream of nitrogen gas.
- 9.1.2.1 After cleaning, check the cleanliness of the cell by measuring the conductivity of treated n-heptane in accordance with Section 11. The corrected value should be lower than 0.1 pS/m.
  - 9.2 Cleaning of Auxiliary Equipment:
- 9.2.1 Pipets used to transfer samples should be rinsed inside and outside with cleaning solvent using a non-contaminating squeeze bottle, then blown dry with clean, dry nitrogen. Thermometers should be similarly rinsed and maintained.

Note 5—If a cell has been used to test samples of high-conductivity, that is, more than 1000 pS/m, it should be disassembled for thorough cleaning. Very thorough cleaning may also be accomplished by placing the disassembled cell in a Soxhlet apparatus containing boiling toluene/ isopropyl alcohol for several hours.

Note 6—If testing is to be done on both low-conductivity (<1 pS/m) and high-conductivity (>1000 pS/m) samples, separate cells are recommended.

#### 10. Calibration and Standardization

- 10.1 Checking the Test Equipment:
- 10.1.1 Remove cell and cable from the meter.
- 10.1.2 Depress the 20 pS/m switch. The digital reading should indicate  $0.00 \pm 0.01$  pS/m after 3 s. If readings exceed  $\pm 0.01$  either adjust zero or record the zero error for calculating final report value.
- 10.1.3 Depress the calibrate switch. The digital reading should indicate  $1000 \pm 3$  pS/m.
- 10.1.4 If low battery indicator is displayed during measure or calibration, the internal batteries should be replaced.
  - 10.2 Checking the Cell Constant:
- 10.2.1 A check on the cell constant is necessary only if the cell has been damaged. Two capacitance measurements are required with a precision ac bridge. Make a rigid two-terminal connection between the cell assembly and the bridge. Measure the total capacitance,  $C_E$  (picofarad) of the empty assembly. Without disturbing the connection, add 100 mL of a hydrocarbon standard and measure the new total capacitance,  $C_S$  (picofarad) and the temperature in the cell. Alternatively, the cell can be sent to the manufacturer for recalibration.
- 10.2.2 Calculate the actual capacitance,  $C_A$ , of the empty cell as follows:

$$C_{A} = (C_{S} - C_{E})/(k-1) \tag{2}$$

where:

- k =dielectric constant of the hydrocarbon at test temperatures.
  - 10.2.3 Calculate the cell constant as follows: