Designation: D4308 – 13

An American National Standard

Standard Test Method for
Electrical Conductivity of Liquid Hydrocarbons by Precision Meter[1]

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 (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the U.S. 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:*[2]

D150 Test Methods for AC Loss Characteristics and Permittivity (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.

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

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

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[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.J0.04 on Additives and Electrical Properties.

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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**

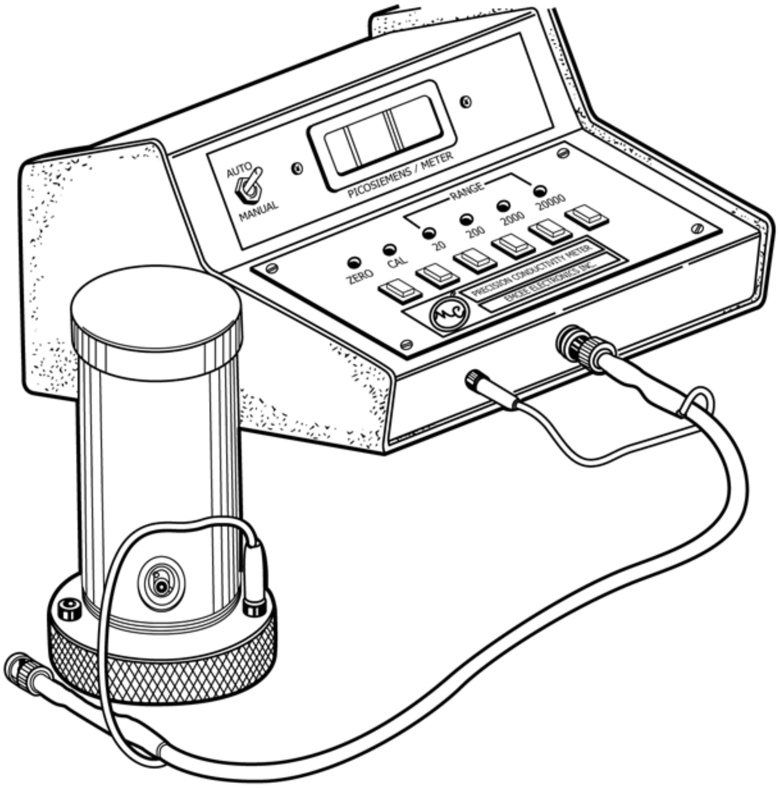

FIG. 1 Precision Conductivity Meter

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 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, 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[5,6] 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[5,7] 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

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

[4] 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.

[5] 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.

[6] 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.

[7] 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.

tube with the activated silica gel while tapping to achieve 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 ±5 % at the temperature of calibration.[8]

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 at least three 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.

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 ± 0.01 pS/m after 3 s. If readings exceed ±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 ± 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:

$$C_A = (C_S - C_E)/(k - 1) \quad (2)$$

where:

k = dielectric constant of the hydrocarbon at test temperatures.

10.2.3 Calculate the cell constant as follows:

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[8] A standard, such as cyclohexane, with certified dielectric constant, may be obtained from the National Bureau of Standards, Washington, DC 20234.

FIG. 2 Cleaned Cell Attached to Meter

$$K = 8.854/C_A \qquad (3)$$

10.2.4 The cell constant of a new KSLA cell is 1.0. Because of its configuration, the cell constant cannot change significantly unless there is gross damage.

11. Procedure

11.1 Attach the cleaned cell to the meter as shown in Fig. 2.

11.1.1 Depress the ZERO switch, the digital reading should indicate the same value recorded in 10.1.2.

11.1.2 Depress the CAL switch, the digital reading should indicate 1000 ± 5.

11.1.3 Rinse the cleaned cell three times with the sample, empty completely, then fill the outer chamber until sample overflows into the center receptacle. (Alternatively, the outer chamber can be filled by pipet with 100 mL of sample).

NOTE 7—Allow static charges generated by handling the sample to dissipate. If the sample is expected to have a conductivity below 1 pS/m, allow 15 min waiting time before reading.

11.1.4 If the sample conductivity is known, select the corresponding range position. When the conductivity is unknown, first check the fuel on a 2000 pS/m range position, then read in a lower scale if appropriate.

11.1.5 Using the AUTO mode, depress the appropriate RANGE switch. In the AUTO mode, the reading is stabilized after 3 s and held on display for 9 s. Record the pS/m value.

11.1.6 Repeat readings can be taken after a 1 min delay.

11.1.7 Remove the cell cover and measure the temperature of the test samples to the nearest 1°C with a clean thermometer.

12. Calculation

12.1 If the zero error in 10.1.2 was greater than ±0.01 and the zero of the meter was not adjusted, the readings should be corrected by subtracting any positive zero error or adding any negative zero error. For example:

Sample reading (11.1.5)	1.67
Uncorrected zero error (10.1.2)	+ 0.03
Calculated reading	1.64

13. Report

13.1 The report is to include the following:

13.1.1 The calculated conductivity of the sample in pS/m after correcting for the zero reading. Note MANUAL or AUTO mode.

13.1.2 Temperature of sample.

NOTE 8—It is recognized that the electrical conductivity of a fuel varies significantly with temperature and that the relationship differs for various types of aviation and distillate fuel. If it is necessary to correct conductivity readings to a particular temperature, each laboratory would have to establish this relationship for the fuels and temperature range of interest.

14. Precision and Bias[9]

14.1 The precision of this test method obtained by statistical examination of test results in the range between 0.1 and 2000 pS/m by operator/instrument pairs at a common test site is described in 14.2 and 14.3.

NOTE 9—The data used to determine the precision of this test method were obtained using the auto mode.

14.2 *Repeatability*—The difference between successive test results obtained by the same operator with the same apparatus under constant operating conditions on identical test samples

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[9] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1170.

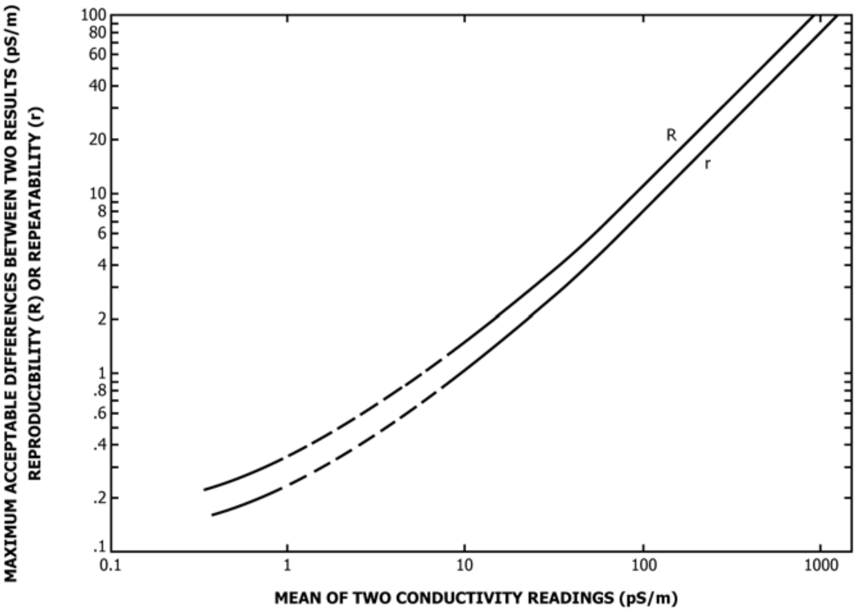

FIG. 3 Variation of Repeatability and Reproducibility with Conductivity Level

would, in the long run, in the normal and correct operation of the test method, exceed the values in Fig. 3 in only one case in twenty.

14.3 *Reproducibility*—The difference between two single and independent results obtained by different operators working at the same location on identical test materials would, in the long run, exceed the values in Fig. 3 only in one case in twenty.

14.3.1 In the event of a dispute or concern regarding shipped sample conductivity, it is recommended that operators come to the bulk hydrocarbon storage site to measure conductivity on freshly obtained samples according to the cited procedure. This ensures that samples identical to bulk storage are tested by either or both parties and the precision data in Fig. 3 shall apply (Note 10).

NOTE 10—In 1987, a test program using Test Method D2624 was carried out to investigate reproducibility of results when samples are shipped between laboratories.[10] While repeatability values were similar to those from common site testing, it was concluded that adequate reproducibility values were not obtained due to changes in conductivity of samples during shipment and storage. The same possibility of conductivity change would occur for samples tested according to Test Method D4308.

14.4 *Bias*—Since there is no accepted reference material suitable for determining the bias of the procedure in Test Method D4308 for measuring conductivity, bias cannot be determined.

ANNEX

(Mandatory Information)

A1. WARNING STATEMENTS

A1.1 Toluene

A1.1.1 Keep away from heat, sparks, and open flame.
Keep container closed.
Use with adequate ventilation.
Avoid breathing of vapor or spray mist.
Avoid prolonged or repeated contact with skin.

A1.2 n-Heptane

A1.2.1 Keep away from heat, sparks, and open flame.
Keep container closed.
Use with adequate ventilation.
Avoid prolonged breathing of vapor or spray mist.
Avoid prolonged or repeated skin contact.

A1.3 Aviation Turbine Fuel (Jet A or A-1)

A1.3.1 Keep away from heat, sparks, and open flame.
Keep container closed.
Use with adequate ventilation.
Avoid breathing vapor or spray mist.
Avoid prolonged or repeated contact with skin.

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[10] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1235.

A1.4 Aviation Turbine Fuel (Jet B)

A1.4.1 Keep away from heat, sparks, and open flame.

Keep container closed.

Use with adequate ventilation.

Avoid buildup of vapors and eliminate all sources of ignition, especially non-explosionproof electrical apparatus and heaters.

Avoid breathing vapor or spray mist.

Avoid prolonged or repeated contact with skin.

SUMMARY OF CHANGES

Subcommittee D02.J0.04 has identified the location of selected changes to this standard since the last issue (D4308–12) that may impact the use of this standard.

(1) Modified 8.3.

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