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An American National Standard

Standard Test Method for Determination of D-C Resistivity of Writing Paper (Keithley Method)[1]

This standard is issued under the fixed designation D 4949; 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 describes the procedure for determination of both surface and volume (bulk) d-c resistivity of writing paper. This test method is specific for the Keithley 6105 resistivity adapter.

1.2 This test method has been developed as a special case for use on writing papers processed in high-speed nonimpact printers where resistivity control is critical. It is not in conflict with Test Methods D 257.

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

2. Referenced Documents

2.1 *ASTM Standards:*

D 257 Test Methods for D-C Resistance or Conductance of Insulating Materials[2]

D 585 Practice for Sampling and Accepting a Single Lot of Paper, Paperboard, Fiberboard, and Related Products[3]

D 644 Test Method for Moisture Content of Paper and Paperboard by Oven Drying[3]

D 645 Test Method for Thickness of Paper and Paperboard[3]

D 685 Practice for Conditioning Paper and Paper Products for Testing[3]

D 1749 Practice for Interlaboratory Evaluation of Test Methods Used with Paper and Paper Products[3]

E 122 Practice for Calculating Sample Size to Estimate, with a Specified Tolerable Error, the Average for Characteristic of a Lot or Process[4]

E 337 Test Method for Measuring Humidity with a Psychrometer (the Measurement of Wet- and Dry-Bulb Temperatures)[5]

3. Terminology

3.1 *Definitions:*

3.1.1 *surface resistivity*—the surface resistivity of a material is the ratio of the potential gradient parallel to the current along its surface to the current per unit width of the surface. Surface resistivity of a material is numerically equal to the surface resistance between two electrodes forming opposite sides of a square. The size of the square is immaterial.

3.1.2 *volume resistivity*—the volume resistivity of a material is the ratio of the potential gradient parallel to the current in the material to the current density.

4. Summary of Test Method

4.1 A paper specimen is placed between appropriate metal electrodes. An electrical voltage is applied for a fixed period of time and the resultant current between two electrodes is measured accurately. By using appropriate electrodes, surface and volume resistance may be measured separately. From this information, a calculation of both surface and volume resistivity can be made.

5. Significance and Use

5.1 Paper generally is considered to be a material having good electrical insulating characteristics; for example, relatively high resistivity when dry. However, in certain applications, control of electrical resistivity to within specified limits is an important function, and resistivity measurement techniques are required.

5.2 While it is possible to measure resistivity using a variety of instruments (Test Methods D 257), this test method is specific for the Keithley Model 6105 resistivity adapter.[6] The precision statement was based on the use of that instrument. Studies have shown that results will vary depending upon the type of resistivity adapter used and if reproducibility between different instruments in the same laboratory or between different laboratories is to be achieved, it is essential that the same model resistivity adapter be used in each determination.

5.3 The power supply and electrometer referred to in this method are manufactured by Keithley and the procedure

[1] This test method is under the jurisdiction of ASTM Committee D06 on Paper and Paper Products and is the direct responsibility of Subcommittee D06.92 on Test Methods.

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[2] *Annual Book of ASTM Standards*, Vol 10.01.

[3] *Annual Book of ASTM Standards*, Vol 15.09.

[4] *Annual Book of ASTM Standards*, Vol 14.02.

[5] *Annual Book of ASTM Standards*, Vol 11.03.

[6] Manufactured by Keithley Instruments, Inc., 28775 Aurora Rd., Cleveland, OH 44139.

describes their use. It is believed that comparable equipment manufactured by others would serve the same purpose. The precision statement was developed with Keithley equipment.

5.4 The control of resistivity is of special importance on high-speed nonimpact printing machines where that characteristic will affect printing, machine feeding and handling, or both. Resistivity is important in the image transfer function of a nonimpact printer or copier using the electrophotographic process. Resistivity is an important factor in controlling static buildup on high-speed nonimpact printers and copiers.

6. Apparatus

6.1 *Power Supply*—A d-c generator capable of producing up to 500 V, Keithley Model 247.[6]

6.2 *Metal Electrodes*—A set of circular metal electrodes with annular guard electrodes capable of measuring surface and volume resistivity, Keithley Model 6105 resistivity adapter.[4]

NOTE 1—Electrodes showing pitting or corrosion should be refinished or replaced.

6.3 *Electrometer*—A meter capable of measuring current as low as 10^{-14} A, Keithley Models 610C, 614, or 485.[6]

NOTE 2—Interconnecting wires and cables shall be shielded.

6.4 *Insulating Film*—A 9-cm (3.5-in.) square piece of 5 to 10 mil polyester insulating film or TFE-fluorocarbon for surface resistivity measurements only.

7. Preparation

7.1 For acceptance sampling, obtain the sample in accordance with Practice D 585.

7.2 When sampling for other purposes, use Practice E 122 as an alternative.

7.3 Identify and mark the felt and wire sides.

7.4 Precondition the sample at 10 to 35 % relative humidity and 22 to 40°C, and condition at 50 ± 2 % relative humidity and 23 ± 2°C (73.4 ± 3.5°F) as described in Practice D 685. After conditioning, determine the actual moisture content of the sample accurately by oven drying (Test Method D 644) or by another appropriate technique.

NOTE 3—The preconditioning step is especially important since the hysteresis effect, as explained in Practice D 685, can cause significant variation in the test results. Complete conditioning at 50 % relative humidity is also important.

7.5 Cut the sample into 9-cm (3.5-in.) square (approximately) specimens and handle with clean gloves or manually only at the edges to prevent contamination. From each test unit of the sample, prepare five test specimens for each side to be tested.

7.6 If measuring volume resistivity, determine the thickness of the test specimens in accordance with Test Method D 645.

7.7 Determine the actual temperature and relative humidity of the test environment according to Test Method E 337.

8. Procedure

8.1 *Instrument Calibration*:

8.1.1 Connect the power supply to the electrometer with a cable connector. Turn the power supply "ON'' and the electrometer to" OFF'' (stand-by) and allow 20 min warmup time.

8.1.2 Adjust the range dial on the electrometer to read" VOLTS,'' the multiplier dial to "100,'' the feedback switch to" NORMAL,'' and the meter knob to " + '' (positive).

8.1.3 Set the power supply output voltage switch to "0'' and the voltage dial to "90.'' Unlock the zero check knob on the electrometer and read the meter. Adjust the power supply voltage dial so the meter pointer reads "9.00'' on the top scale. Turn the electrometer zero check knob back to "LOCK,'' and the meter knob back to "OFF.''

8.1.4 Set the electrometer multiplier knob to "1.'' Turn the meter knob to "CENTER ZERO.'' The meter pointer should come to the center position within 10 s. If necessary, adjust the meter zero with the fine zero control; normally there is no need to use the medium zero control.

8.1.5 Turn the electrometer meter knob to "POWER OFF'' and the power supply switch to "OFF.'' These checks should be completed before each testing round.

8.1.6 Clean the electrodes with an alcohol-ether mixture or other suitable solvent, applied with a soft cloth pad.

8.2 *Surface Resistivity Test*:

8.2.1 Connect the power supply to the resistivity adapter (containing the metal electrodes) with a cable connector. Connect the adapter to the electrometer with the other connector. Open the adapter and place the short circuit plug over the bottom two jacks and insert the banana plug from the test weight assembly into the top "SURFACE'' jack.

8.2.2 Turn the power supply "ON'' and the electrometer meter knob to "OFF.'' Set the electrometer controls as follows:

8.2.2.1 *Meter Knob*—Set to " + '' (positive).

8.2.2.2 *Range Dial*—Set to "AMPERES.''

8.2.2.3 *Multiplier Knob*—Set to "10.''

8.2.2.4 *Feedback Switch*—Set to "FAST.''

8.2.2.5 *Zero Check Knob*—Set to "LOCK.''

8.2.2.6 If the approximate range of the sample to be tested is known, the ampere dial can be set to that range. Otherwise, start with the 10^{-1} setting and work down to the correct range.

8.2.3 Unless otherwise specified, set the power supply voltage dial at 90 ± 1 V.

8.2.4 Place the paper specimen on top of the electrodes in the adapter, with the side to be measured down. Place the 9-cm square of insulating polyester film or TFE-fluorocarbon on top of the specimen. Cover with the test weight assembly and close the adapter cover. The adapter has a built-in safety device that disconnects the electrodes from the power supply when the cover is open.

8.2.5 Unlock the zero check knob on the electrometer. Move the range dial along the ampere scale until the needle on the meter reaches the mid-range of the upper meter scale. At the end of 1 min electrification time, record the amperage reading to the nearest 0.05. After the reading is obtained, move the zero check knob on the electrometer back to the "LOCK'' position to protect the instrument circuitry.

8.2.6 To test the other side of the paper, repeat the procedure in 8.2.4 and 8.2.5 with a new specimen placed opposite-side down on the adapter electrodes.

8.2.7 At the conclusion of testing, turn the power supply power switch to "OFF'' and turn the electrometer meter knob

to "POWER OFF.'' Remove the specimen, replace the TFE-fluorocarbon sheet, and close and lock the adapter cover.

8.3 *Volume Resistivity Test*:

8.3.1 Connect the power supply to the resistivity adapter with a cable connector. Open the adapter and place the short circuit plug over the top two jacks. Insert the banana plug from the test weight assembly into the bottom "VOLUME'' jack.

8.3.2 Unless otherwise specified, set the power supply voltage at 500± 5 V.

8.3.3 Repeat procedures 8.2.2 through 8.2.7, except omit the polyester film or TFE-fluorocarbon sheet in 8.2.4. Place the wire side of the specimen down on the electrodes. Since volume resistivity is measured through the sheet, there is only one test per specimen.

9. Calculation

9.1 *Surface Resistivity (SR)*—Surface resistivity is determined by measuring current flow across a paper surface between a circular center electrode and an annular ring electrode. These electrodes are separated by a finite distance. From measured current flow surface resistivity can be calculated from the following adaptation of Ohm's Law:

$$SR = K_s \cdot \frac{V}{I} \tag{1}$$

where:

V = applied voltage,
I = current reading as measured by the electrometer, A, and
K_s = a cell constant that accounts for gap distance (53.4).

To simplify when V = 100, this equation can be modified to:

$$SR = \frac{5.34 \cdot 10^3}{I} \tag{2}$$

The resulting number is expressed in ohms or more correctly" ohms per square'' to indicate a surface measurement. Also report the side of the specimen tested (felt or wire).

9.2 *Volume Resistivity (VR)*—Volume resistivity can be calculated from current between electrodes placed at top and bottom sheet surfaces. Here specimen thickness must be taken into account. The resulting equation is as follows:

$$VR = \frac{K}{T} \cdot \frac{V}{I} \tag{3}$$

where:

V = applied voltage,
I = current reading as measured by the electrometer, A,
T = average specimen thickness, cm or in., and
K = a cell constant that accounts for electrode dimensions: K = 22.9 when using centimetres, K = 3.53 when using inches.

To simplify when V = 500, the equation can be modified to the following:

9.2.1 When using centimetres:

$$VR = \frac{1.145 \cdot 10^4}{TI} \tag{4}$$

9.2.2 When using inches:

$$VR = \frac{1.765 \cdot 10^3}{TI} \tag{5}$$

The resulting number is expressed in ohm-centimetres or ohm-inches.

10. Report

10.1 For each paper sample, at least five specimens should be tested and their average resistivity reported.

10.2 Report the temperature and relative humidity of the test environment and the voltage(s) at which measurements were made.

10.3 Report actual moisture content.

11. Precision and Bias [7]

11.1 *Precision*:

11.1.1 *Repeatability*:

11.1.1.1 *Surface Resistivity*—The repeatability standard deviation of the procedure for measuring surface resistivity in this test method is approximately 4.3×10^{10} and the 95 % repeatability limit is approximately 1.2×10^{11}.

11.1.1.2 *Volume Resistivity*—The repeatability standard deviation of the procedure for measuring volume resistivity in this test method is approximately 8×10^4 and the 95 % repeatability limit is approximately 2.4×10^5.

11.1.1.3 The repeatability values in 11.1.1.1 and 11.1.1.2 were determined on samples having a mean value for surface resistivity of 1×10^{12} and a mean value for volume resistivity of 1×10^6, respectively.

11.1.2 *Reproducibility*—It is not yet practible to provide an estimate on reproducibility of surface or volume resistivity as measures by this test method. Reproducibility estimates provided by sample exchange between pairs of laboratories indicates that reproducibility values will be approximately twice those for repeatability. This test method is specific to certain instrumentation, as described in 5.2, and finding the number of laboratories recommended by Practice D 1749 willing to participate in the reproducibility study has proven more difficult than anticipated. Work in this area continues.

11.2 *Bias*—The procedure in this test method has no bias because the values of surface and volume resistivity are defined in terms of the equipment and procedures in the test method.

12. Keywords

12.1 electrical resistivity; Keithly resistivity method; surface resistivity; volume resistivity; writing papers

[7] Data supporting this test are on file at ASTM Headquarters. Request RR:D06-1001.