Designation: D991 – 89 (Reapproved 2014)

Standard Test Method for Rubber Property—Volume Resistivity Of Electrically Conductive and Antistatic Products[1]

This standard is issued under the fixed designation D991; 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 the determination of volume resistivity of rubbers used in electrically conductive and antistatic products.

1.2 This test method assumes that the surface conductivity is negligible compared with the conductivity through the specimen.

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D3182 Practice for Rubber—Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets

D4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *rubber product, antistatic*—a rubber product sufficiently conductive to prevent a build-up of an electrical charge on the surface and sufficiently insulating to prevent an electrical hazard.

3.1.1.1 *Discussion*—Generally, antistatic rubber products are considered to have a resistance of 10^4 to 10^8 Ω.

3.1.2 *rubber product, conductive*—a rubber product having an electrical conductivity of sufficient magnitude that might be considered an electrical or thermal hazard.

3.1.2.1 *Discussion*—Generally, conductive rubber products are considered to have a resistance of less than 10^4 Ω at 120 V.

3.1.3 *volume resistivity*—the ratio of the electric potential gradient to the current density when the gradient is parallel to the current in the material.

4. Significance and Use

4.1 The electrical behavior of rubber products used in particular applications is important for a variety of reasons such as safety, static changes, current transmission, etc. This test method is useful in predicting the behavior of such rubber products.

5. Apparatus

5.1 *Electrode Assembly*—The electrode assembly (Fig. 1) shall consist of a rigid base made from an electrically insulating material having a resistivity greater than 10 TΩ·m (for example, hard rubber, polyethylene, polystyrene, etc.) to which a pair of current electrodes and a pair of potential electrodes are fastened in such a manner that the four electrodes are parallel and their top surfaces are in the same horizontal plane. Another pair of current electrodes identical with the first pair shall be fastened to a second piece of insulating material so that they can be superimposed on the specimen directly above the first pair. The current electrodes shall have a length at least 10 mm (0.4 in.) greater than the specimen width, a width between 5 and 8 mm (0.2 and 0.3 in.), and a height uniform within 0.05 mm (0.002 in.) between 10 and 15 mm (0.4 and 0.6 in.). The potential electrodes shall have a length and height equal to the current electrodes and shall be tapered to an edge having a radius of 0.5 mm (0.02 in.) maximum at the top surface. The distance between the potential electrodes shall not be less than 10 mm (0.4 in.) nor more than 66 mm (2.6 in.) and shall be known within ±2 %. The current electrodes shall be equidistant outside the potential electrodes and separated from them by at least 20 mm (0.8 in.). The electrodes shall be made from

[1] This test method is under the jurisdiction of ASTM Committee D11 on Rubber and is the direct responsibility of Subcommittee D11.10 on Physical Testing.

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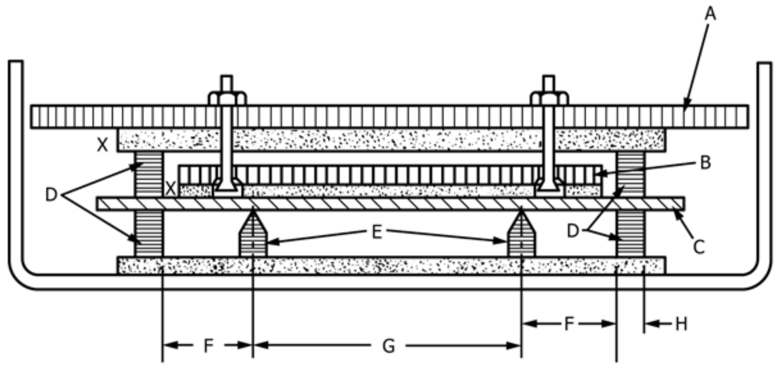
[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.



A — Mass for applying contact force between current electrodes and specimen
(300 N/m times specimen width in meters) (Note 1)
B — Mass for applying contact force between potential electrodes and specimen
(60 N/m times specimen width in meters) (Note 2)
C — Specimen
D — Current Electrodes
E — Potential Electrodes
F — Distance between current and potential electrodes (20 mm minimum)
G — Distance between potential electrodes (see Note 2in Section 9) depends on specimen size.
H — Width of current electrode, 5 to 8 mm (0.2 to 0.3 in.)
X — Insulation

NOTE 1—For a specimen 150 mm (6 in.) wide, mass is approximately 4.5 kg (10 lb).
NOTE 2—For a specimen 150 mm (6 in.) wide, mass is approximately 0.9 kg (2 lb).

FIG. 1 Electrode Assembly

a corrosion-resistant metal such as brass, nickel, stainless steel, etc. Insulation resistance between electrodes shall be greater than 1 TΩ.

5.2 *Resistance-Measuring Device*—Resistance may be measured by any electrical circuit that enables the current through the current electrodes and the potential across the potential electrodes to be measured within 2 %. Suitable devices for measuring current are: (*1*) a precision milliammeter, or (*2*) potential measurement across a reference resistor (resistance value known within 2 % in series with the current electrodes. Suitable devices for measuring potential are: (*1*) a galvanometer having a sensitivity of 1 µA or less per scale division in a null-voltage circuit; (*2*) an electrostatic voltmeter having a d-c resistance greater than 19 TΩ; or (*3*) an electrometer such as a multirange voltmeter having an input d-c impedance greater than 0.1 TΩ (Note 1). In any case, the current through the potential electrodes during measurement must be less than 1 % of that through the current electrodes. A stable source of d-c potential shall be provided that can be adjusted to limit the power dissipated in the specimen between potential electrodes to approximately 0.1 W. Because of the large range of resistances covered by conductive and antistatic rubbers, separate equipment for measuring resistances above and below approximately 50 000 Ω is generally desirable.

NOTE 1—Schematic diagrams of a typical apparatus that have been found to be satisfactory are shown in Figs. X1.1 and X1.2.

5.3 *Electrode Contacts*—Masses shall be provided to produce a uniform contacting force across the width of the specimen of approximately 300 N/m (4.5 kg (10 lb)) on the standard sheet, 150 mm (6 in.) wide, by the current electrodes and 60 N/m (0.9 kg (2 lb)) on the standard sheet, 150 mm (6 in.) wide, by the potential electrodes.

6. Specimens

6.1 *Size*—The width of the specimen shall be between 10 and 150 mm (0.4 and 6 in.) and the length shall be between 70 and 150 mm (2.8 and 6 in.). The width shall be uniform within ±1 %. The thickness of cut specimens is specified in 6.3. Molded specimens are specially prepared as described in 6.2 and therefore have a thickness of 2.0 ± 0.2 mm (0.08 ± 0.008 in.).

6.2 *Molded Specimen*—Standard sheets prepared in accordance with Practice D3182 may be used, provided the surface of the uncured rubber is kept free of soapstone or other contamination, and the surface of the vulcanized sheet is not contaminated with mold lubricant. To avoid surface contamination and minimize distortion of specimen prior to test, sheets may be molded between sheets of moisture-sensitive cellophane, which can be readily removed after brief immersion in warm water. After removing the cellophane, the surface of the sheet should be patted dry, taking care not to bend or stretch the sheet.

6.3 *Cut Specimen*—The specimen shall be cut from a product that has not been buffed or abraded. Surfaces of the specimen shall be cleaned if necessary by rubbing with Fuller's earth and water, washing with distilled water, and drying in air. The specimen shall be uniform in thickness within ±5 %, not more than 6.6 mm (0.26 in.), and if possible, not less than 2 mm (0.08 in.) thick. Care shall be taken to avoid distortion of the specimen during preparation.

7. Conditioning

7.1 The time between vulcanization and testing shall be not less than 16 h nor more than 4 weeks for molded specimens. Products shall be tested within 2 months after receipt by the customer.

7.2 Specimens cut from products or molded specimens that have been inadvertently distorted shall be annealed in air for 3 h at 23 ± 2°C (73.4 ± 3.6°F) to remove strains or other effects of handling.

7.3 Specimens shall be conditioned for at least 16 h and tested at a temperature of 23 ± 2°C (73.4 ± 3.6°F) and a maximum relative humidity of 65 %. Molded specimens can be conditioned in a desiccator. Specimens annealed at room temperature may be stored in a closed container during the conditioning period.

8. Procedure

8.1 After conditioning, place the specimen in the electrode assembly, taking care to avoid flexing or distortion. The identification portion of standard sheets shall be normal to the calender grain and shall not be in contact with, nor lie between, the current electrodes.

8.2 Adjust the current through the specimen after connection to the d-c source so that the power dissipation in the specimen between potential electrodes is approximately 0.1 W. The following values should not be exceeded for the maximum current in the specimen for various potentials across the potential electrodes:

Potential	Current, mA
3	50
6	25
10	15
30	5
75	2
150	1
300	0.5

8.3 As soon as the current has stabilized, in a maximum time of 5 s, measure the potential difference across the potential electrodes and the current through the current electrodes to the nearest 1 % of the respective values.

8.4 Measure the thickness and width of the specimen.

8.5 Make the measurements on three specimens.

9. Calculation

9.1 Calculate the volume resistivity as follows for each specimen:

$$\rho = Vwdk/Il \quad (1)$$

where:

ρ = volume resistivity, Ω·m,
V = potential difference, V, across potential electrodes,
I = current, A, through the current electrodes,
w = width of specimen,
d = thickness of specimen,
l = distance between potential electrodes,
k = factor depending on units in which, w, d, and l are measured; that is, k is 0.001 if w, d, and l are in millimetres and 0.0254 if they are in inches.

TABLE 1 Type 1 Precision for Log(ρ)

NOTE 1—Only two laboratories participated in the program for these results.

Material	Mean Level	Within Laboratory[A]			Between Laboratory[A]		
		S_r	r	(r)	S_R	R	(R)
1	3.392[B]	0.065	0.184	5.4	0.329	0.931	27.4
2	4.855	0.132	0.374	7.7	0.577	1.63	33.6

[A] S_r = within laboratory standard deviation.
r = repeatability (in measurement units).
(r) = repeatability (in percent).
S_R = between laboratory standard deviation.
R = reproducibility (in measurement units).
(R) = reproducibility (in percent).
[B] Tabulated values (as used for analysis), $\log_{10}(\rho)$.

NOTE 2—If l is made 64.5 mm (2.54 in.) and w and d are measured in inches, the equation becomes:

$$\rho = 0.01\ Vwd/I \quad (2)$$

9.2 Report the median value for the three specimens as the volume resistivity.

10. Report

10.1 Report the following information:

10.1.1 Temperature during conditioning and test,

10.1.2 Relative humidity during conditioning and testing,

10.1.3 Size of specimen,

10.1.4 Current through specimen in amperes,

10.1.5 Voltage across potential electrodes, and

10.1.6 Volume resistivity in ohm-metres, kilohm-metres, or megohm-metres.

11. Precision and Bias[3]

11.1 These precision and bias statements have been prepared in accordance with Practice D4483. Refer to Practice D4483 for terminology and other testing and statistical concepts.

11.2 Because of the special nature of this test and its lack of widespread use in the industry, a limited interlaboratory Type 1 test program was used to assess precision. Two materials (rubber compositions) of different volume resistivity in the form of cured sheets were prepared in one laboratory and sent to the other participating laboratory. Both laboratories were experienced in this testing.

11.3 In each laboratory the cured rubber sheets were measured for volume resistivity on two days, on each day by two different operators. The within laboratory variation, therefore, contains an "operator" and "day" component of variation.

11.4 A test result is the median value of three measurements of volume resistivity.

11.4.1 Table 1 gives the precision results. Due to the wide range of volume resistivity values that are possible (10–1000 fold variation) the analysis was conducted using the (base 10) logarithms of the (test result) volume resistivity, ρ.

[3] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D11-1030.

11.4.2 The rather large between laboratory variation indicates the difficulty frequently experienced with this measurement by experienced laboratories and operators.

11.4.3 *Bias*—In test method statistical terminology, bias is the difference between an average test value and the reference or true test property value. Reference values do not exist for this test method since the value or level of the test property is exclusively defined by the test method. Bias, therefore, cannot be determined.

APPENDIX

(Nonmandatory Information)

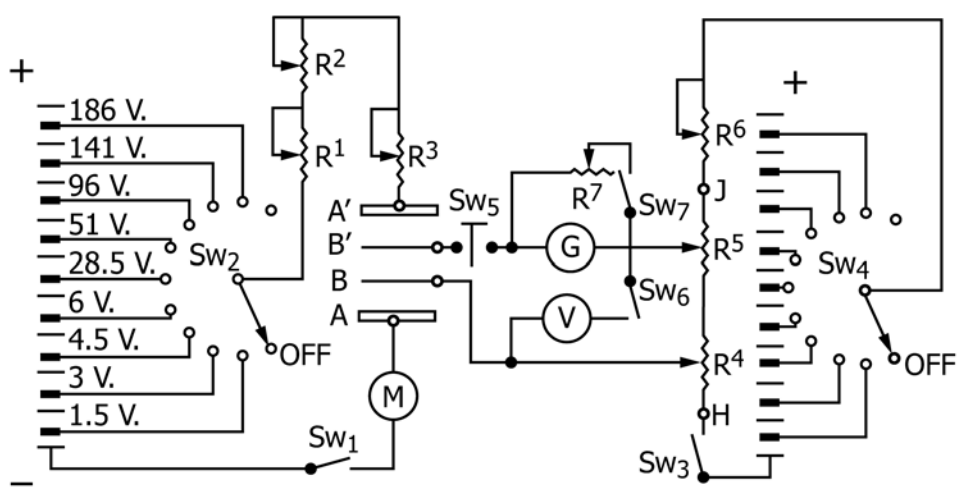
X1. CIRCUIT DIAGRAMS AND EXPLANATORY MATERIAL

X1.1 With switch Sw_1 closed and the milliammeter set at 0–15 mA, turn the rotary switch Sw_2 to develop current with maximum values as follows:

Switch Contacts	Maximum Current, mA
1–4	15
5–6	3
7–9	1

Fine adjustment of current can be accomplished by resistances R_1, R_2, and R_3.

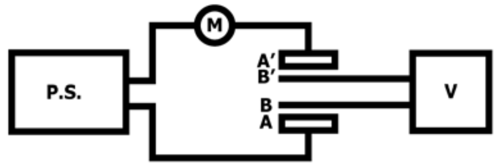
X1.2 With switch Sw_3 closed and rotary switch Sw_4 swung to approximate position, or one or two contacts less than Sw_2, close switch Sw_7, set R_7 for minimum resistance (least sensitive position for galvanometer), and then close switch Sw_5. For null balance (zero reading on galvanometer), adjust R_4, R_5, and R_6 and increase the sensitivity of the galvanometer by increasing R_7, eventually opening switch Sw_7 to eliminate R_7 altogether. Close switch Sw_6 to read voltage. It is desirable to limit the wattage dissipated in the sample to 0.1 W between voltage electrodes. This condition is satisfied by the product of volts times milliamperes being not greater than 100.



A and *A'*—Current electrodes.
B and *B'*—Voltage electrodes.
Sw_1, Sw_3, Sw_6, and Sw_7—On-off toggle switches.
Sw_2 and Sw_4—Single-pole, 11-contact radio type rotary selector switches.
Sw_5—Normally open momentary contact switch.
Source of Voltage—Two banks of dry cells each consisting of four 1½-V cells, and four 45-V "B" batteries—one connected at 22½ V.
M—Milliammeter, Weston D-C. Model 430, ranged 0–0.15, 1.5, 15 mA scale divisions 150; or equivalent milliammeter.
G—Galvanometer, having a sensitivity of 1 µA per scale division.
V—Voltmeter, Vacuum Tube Voltohmist, Electronic Designs Model 100, Electronic Designs, Inc., New York City; or equivalent performance vacuum tube or solid state voltmeter. If desired, a multi-range d-c. voltmeter with a sensitivity of 1000Ω/ V or better may be used. For protection of this voltmeter, it is suggested that a two "gang" 11-contact rotary selector switch be substituted for Sw_4 and the resistance multipliers for the voltmeter be connected to the proper points on the second set of switch contacts. In this case switch Sw_6 could be eliminated.
R_1, R_2, R_4, and R_5—2-W, 0–10 000-Ω potentiometers, Mallory wire wound or equivalent.
R_3 and R_6—2-W, 0–5000-Ω potentiometers, Mallory wire wound or equivalent.
R_7—2-W, 0–3000-Ω potentiometer, Mallory wire wound or equivalent.

NOTE 1—Where it may be desirable to extend the range of this equipment, more batteries may be added. Caution must be exercised to prevent electrical shock.

FIG. X1.1 Resistance-Measuring Device—Special Null Voltage Circuit



A and *B* — Current electrodes.
B and *B* — Voltage electrodes.
M — Milliammeter, Weston D-C. Model 430, ranged 0-0.15, 1.5, 15 mA scale divisions 150; or equivalent.
V — Voltmeter, multirange with input resistance of at least 100 M or input current of less than 1. For example, Gould Alpha IV Digital Multimeter, Keithly 616 Digital Electrometer, Penril Corp. Data Tech. Model 30L; or equivalent.
P.S. — Variable, regulated, D.C. power supply to provide up to 200 VDC. For example, EICO 1030, Hope Electronics PS-200-IEM, Kepco Inc. ABC 200M, Veepco Instruments Inc. (Lambda) LP-415-FM; or equivalent. For samples requiring under 30 volts supply voltage, a lower voltage supply such as EICO 1032 may be used.

FIG. X1.2 Alternative Circuitry