TESTING THE ELECTROSTATIC (ESD) PARAMETERS OF THERMOFORMED CONDUCTIVE AND LOW STATIC DISSIPATIVE MATERIALS FOR APPLICATIONS INCLUDING AUTOMOTIVE FUEL SYSTEMS (Proposed revisions to SAE J1645)

by
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INTRODUCTION

The evaluation of the electrostatic (ESD) characteristics of molded and thermoformed conductive materials, components and assemblies must be evaluated using three separate tests. First, the basic material must be certified as to having the correct electrostatic characteristics. Second, the components must be tested to confirm that the manufacturing process did not significantly alter the basic material electrostatic properties. Finally, the complete assembly must be tested to ensure that satisfactory bonding occurs between all components and between components and groundable point.

MATERIALS

Loaded, molded and thermoformed plastics are now replacing metal to fabricate components for ESD Safe applications such as automotive fuel systems. These materials consist of plastic resin filler with very high resistance properties loaded with a small percentage of conductive material such as carbon powder or fibers, stainless steel or other technologies. When formed, these materials exhibit either conductive or static dissipative properties as defined in ESD ADV1.0: Glossary of Terms. These materials have bulk resistance properties verses the surface only resistance properties found in many other ESD materials. When a voltage is applied either across or through the material, the dielectric of the filler breaks down and current flows from particle to particle. As the loading of conductive medium decreases the distance between conductive particles increases which then requires a higher voltage to break down the increased dielectric. At some point, the voltage required to measure continuity may develop carbon tracks that could permanently alter the material. Loaded thermoplastic materials is effective in establishing resistance over the range of $<10^3$ to 10^8 Ohms.

This characteristic causes these materials to become non-linear and voltage dependent. Hence, when attempting to measure resistance, different test voltages may give different results. **NOTE:** The series resistors incorporated in virtually all resistance meters are different from meter to meter may also contribute to measurement variations.

Loaded material is generally not adversely affected by humidity, as long as it is reasonable such as less than 75%. However, parts conditioned with fuel can cause the material to become humidity dependent.

RESISTANCE CHARACTERIZATION

Over the years many different resistivity or resistance values have been assigned to designate the various classifications of ESD material. Currently, ESD materials are classified by the ESD Association as follows:

	Conductive	**Dissipative**	**Insulative**
Surface Resistance	$<10^4$ Ohms	10^4 to $<10^{11}$ Ohms	$\geq 10^{11}$ Ohms
Volume Resistance	$<10^4$ Ohms	10^4 to $<10^{11}$ Ohms	$\geq 10^{11}$ Ohms

NOTE: Loaded thermoformed material should not be classified using surface resistivity! As a material becomes more bulk conductive the electrode ratio multiplier used to define surface resistivity in ohms per square (Ω/sq.) causes a significant error to be introduced as shown in Figure 1.

Material with *bulk* resistance characteristics, however, can be classified by specifying its ***volume resistivity***. This is simply done by multiplying the measured resistance by the area of the measuring electrode or material surface, whichever is smaller, and divided by the thickness. All values are in cm and volume resistivity is expressed in Ohms-cm.

$$p_V = A/t\ R_m \text{ Ohm-cm}$$

Increasing or decreasing the thickness of the material will change the actual resistance of the part having a specified volume resistivity. This is a common technique used in ESD products to achieve a particular resistance which is one parameter that determines how a part will dissipate a static charge. **NOTE:** The resistance/resistivity property of material does not predict whether the material will be low charging (antistatic) or not. The term “antistatic” as defined in the ESD Association (ESDA.org) Glossary of Terms ESD ADV 1.0 (2003) is the ability to resist tribocharging.

MEASUREMENT PARAMETERS

To ensure repeatable measurements it is necessary to specify all parameters that may have an effect on the measurement. These include:

1. A defined, repeatable test procedure.
2. Sample preparation.
3. Environmental conditions.
4. Sample profile.
5. Instrumentation incl. electrodes, setup & system verification tests.
6. A defined test voltage and electrification period (measuring time).
7. Documentation and reporting of data.

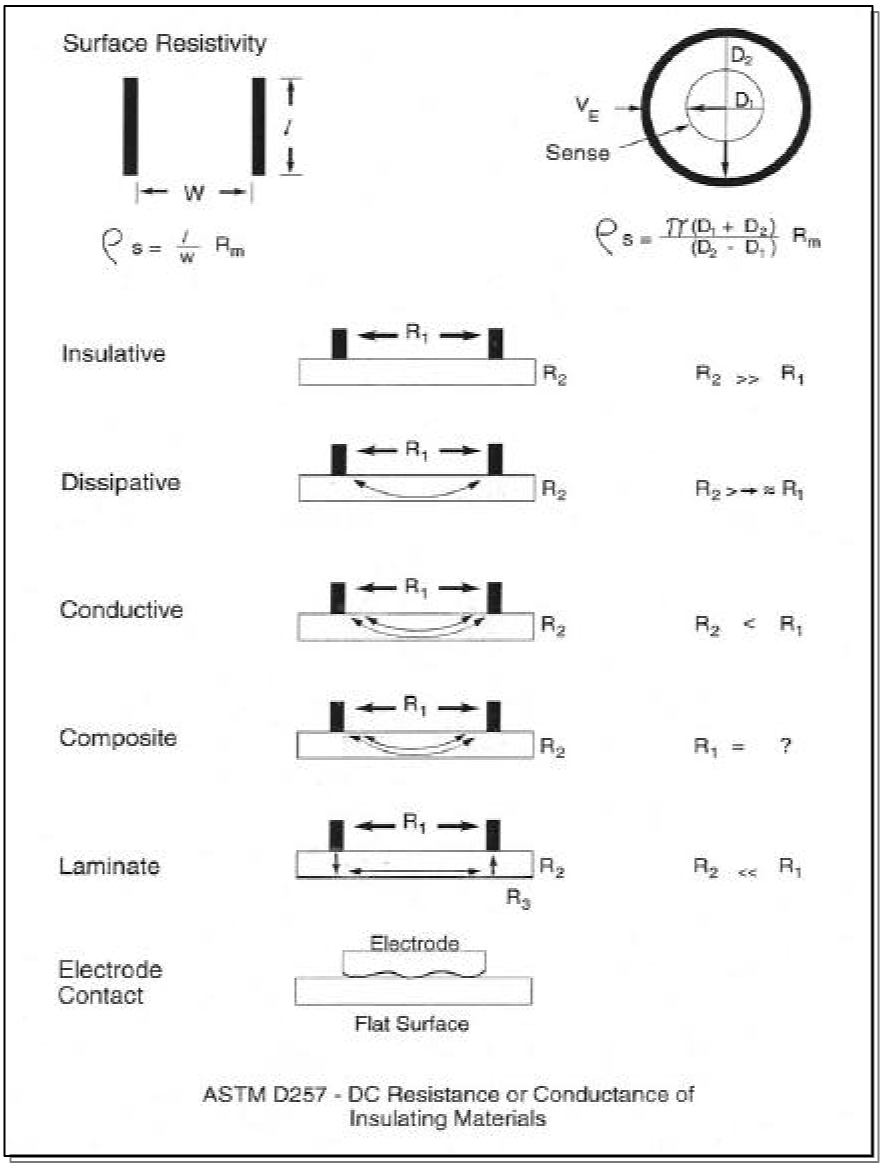
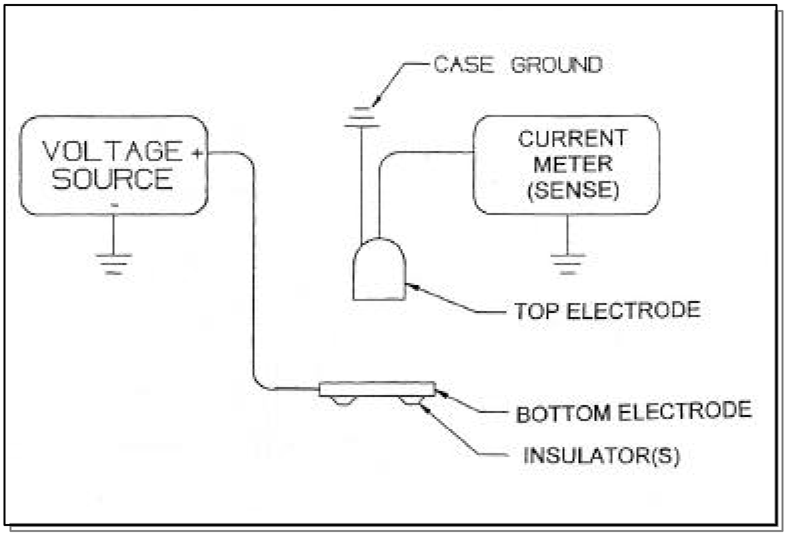


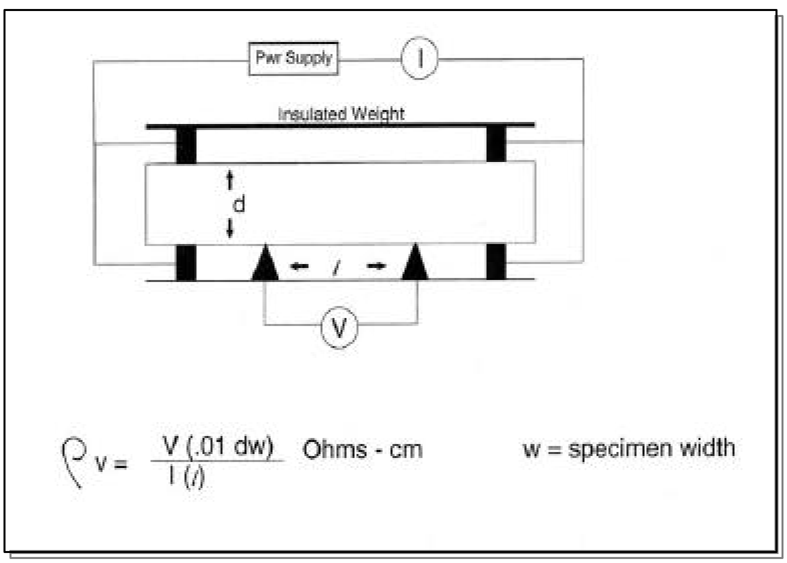
Figure 1

CERTIFYING MATERIAL

Sample plaques of loaded material are usually classified using volume resistance or volume resistivity using ESD STM 11.12 or ASTM D991, as shown in Figure 2. A second test, a modification of the standard static decay test in accordance with Mil-Std. 3010, Method 4046.1, is a way to verify adequate conductive component loading and the ability of the material to dissipate a 5000 Volt charge. Using both methods provides a good description of the dissipative properties of the material.



ESD STM 11.12



ASTM-D991

Figure 2

VERIFYING COMPONENTS AND ASSEMBLIES

Different ESD analysis procedures are required to verify components and assemblies because the test methods used for certifying material are not practical.

The single most important ESD characteristic is the ability to dissipate a static charge in a controlled manner to prevent the buildup of static electricity (static charge accumulation) or a spark discharge. This is defined by measuring the resistance of the material, components and finished product. In addition, the ability of the assembly to actually dissipate a static charge should be measured. This measurement takes into account both the resistance and capacitance of the system. Resistance tests utilize low voltage at a continuous current. Dissipation tests use a high voltage stored in the capacitance of the material which results in only a finite amount of available current.

RESISTANCE AND DISSIPATION LIMITS

Resistance Limits for Material

A material's resistance property must be compatible with the maximum resistance specified for the longest path being measured in the final fuel system assembly. Various standards define different resistance properties. Several of the most common are as follows:

NFPA 77, a standard for hazardous environments, references $1x10^{11}$ Ohm/sq. (a surface resistivity measurement) as the upper limit for sheet material. **NOTE:** The actual resistance is a function of the measuring electrode configuration used. For probes with a 10:1 conversion ratio, the upper limit would be $1x10^{10}$ Ohm.

UL 330, the standard for hoses used for dispensing flammable liquids, specifies $2.33\ x10^{5}$ Ohm-cm maximum.

The suggested resistance limit in the proposed SAE J1645 automotive fuel systems specification is a volume resistivity of $\leq 1x10^{6}$ Ohm-cm.

Virtually all specifications for material and/or ground paths (including wrist and heel straps) used in hazardous operations specify resistance in the 10^{3} to 10^{6} Ohm range.

Resistance Limits for Components and Assemblies

UL 330 specifies a maximum point-to-point resistance along the dissipating surface of hoses to be $\leq 7x10^4$ Ohms per foot.

Ground straps used for handling hazardous material usually incorporate a $5x10^4$ Ohm current limiting resistor.

Assemblies that consist of two or more components bonded together must compensate for any resistance as a result of bonding. It is recommended the upper resistance for assemblies be increased by one order of magnitude.

Dissipation Limits for Material

Static dissipation as measured in accordance with Mil-Std-3010, Method 4046.1 is usually referred to as “static decay”. As defined, this test is for material in the upper static dissipative range ($>10^8$ Ohms). However, following a defined test protocol enables this test to distinguish between satisfactory and unsatisfactory material.

The decay time measured from ±5000 Volts to the 1% cutoff point should be <0.10 seconds. In practice, decay times normally measure less than 0.03 seconds.

Dissipation Limits for Assemblies

The measurement of dissipation time is a function of both resistance and capacitance of the assembly.

The instrumentation recommended in the proposed SAE J1645 test procedure has a total capacitance of 32pf. The time for a material to dissipate 90% of its charge is 2.2τ (2.2RC time constant). With $R=1x10^6$ Ohms and $C=3.2x10^{-11}$ pf the maximum allowable dissipation time is theoretically 0.07 milliseconds. **NOTE:** ***Instrumentation used for measuring static dissipation generally cannot measure faster than 0.14 seconds (140 milliseconds).***

A practical upper limit for this measurement using commercially available instrumentation would be 0.50 seconds from 1000 to 100 Volts which is compatible with the NFPA 99 requirement.

INSTRUMENTATION

Resistance

The measurement of resistance for both certifying material and verifying components and assemblies require specific electrode configurations and resistance meters. These measurements cannot typically be made accurately with standard Digital Multimeters (DMMs), clips or probes.

Materials being measured are generally nonlinear and voltage dependent. For personal safety, when using test voltages of 100 Volts or more the maximum current allowed is 5 milliamps. ***Using a voltage source and current meter and then calculating the resistance (R=V/I) may enable the user to make measurements drawing >5ma.*** **The user should be aware that high voltage with high current poses a personnel safety hazard.** (Many instruments limit the maximum current to 2-3 ma.) The minimum resistances that can be measured at different test voltages with current limited to 5 ma are as follows:

100 V	2×10^4 Ohms
500 V	1×10^5 Ohms
1000 V	2×10^5 Ohms

STM 11.11 and 11.12 specify a concentric ring probe, shown in Figure 3, to measure the surface and volume resistance of planar material samples.

Figure 3

A modified version of the S11.11 probe *may* give more consistent results when measuring thermoplastic plaques. In lieu of conductive rubber electrodes, an array of spring loaded, gold plated pins with a 0.156" surface diameter are installed and the probe is mounted in a press to control applied pressure as shown in Figure 4.

Figure 4

ASTM D991 describes a completely different measurement system and test fixture. It is more complex, but is designed for these material types and would be a satisfactory alternative for certifying material. Figure five shows a typical ASTM D991 Test Fixture.

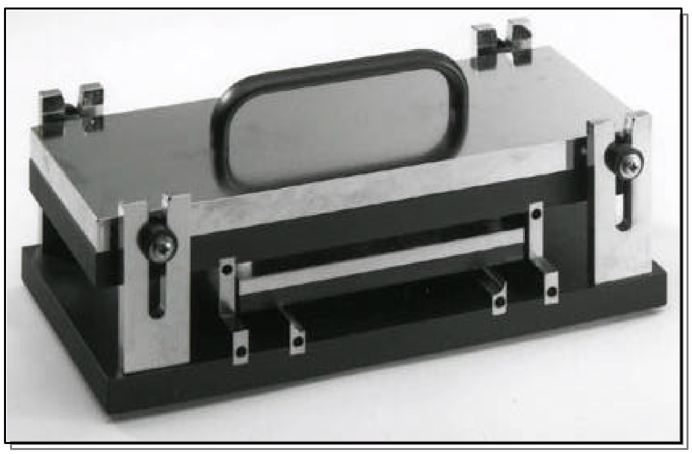


Figure 5

To measure point-to-point resistance of components and assemblies requires a special electrode design. ESD STM 11.13 describes a two-point probe to measure the outer surface of small parts. The contacts are 0.125" in diameter and spaced .25" apart. While this probe may be useable to measure the surface resistance of the components, it is not used for measuring the inside resistance of components or complete assemblies.

The recommended electrodes for testing in accordance with SAE J1645 are shown in Figure 5. The contact pads are .25"x.25" (6x6 mm) on the outside and .25"x.125" (6x3mm) on inside. The clamp applies approximately 10 lb (22kg) of force. The smaller (inside surface) electrode enables it to fit inside fittings and tubing as small as .25" (6mm) diameter.

Figure 6

Placing these electrodes a defined distance apart, such as 1.0" (2.5 cm) on sample plaques, may be an alternate way to test material.

NOTE: Certain plastic standards refer to conductive electrodes that are "painted" onto the sample. This configuration provides the lowest resistance measurement possible because the paint makes total surface contact and is not limited by less than 100% contact obtainable with all other types of electrodes. However, painted electrodes are not practical in measuring parts in a manufacturing or end user environment. In the real world application, parts are connected by mechanical contact. Utilizing a surface contacting electrode provides both a quick and easy measurement plus it also represents real world surface-to-surface contact.

Static Dissipation

When measuring static dissipation (static decay), the test method *must* be specified since several different static decay methods are available. Unfortunately, there is no direct correlation between the different methods. For loaded thermoformed material it is necessary to use two different test methods.

The first test method is based on Mil-Std. 3010, Method 4046.1. Following a defined test procedure, it is used to certify 3" x 5" plaques. The second test method uses a charged plate monitor to test assemblies.

TEST PROCEDURE FOR CERTIFYING MATERIAL

For material conformance, testing is performed on standard (flat) plaques.

Resistance

To measure surface and volume resistance of material, follow the procedures described in ESD STM 11.11 and 11.12. as modified below. The instrumentation must be capable of making measurements from $<1x10^3$ to $1x10^8$ Ohms. A typical component and assembly resistance measurement test set up is shown in Figure 6. Volume resistance measurements can be converted to volume resistivity as described in Appendix A of STM 11.12.

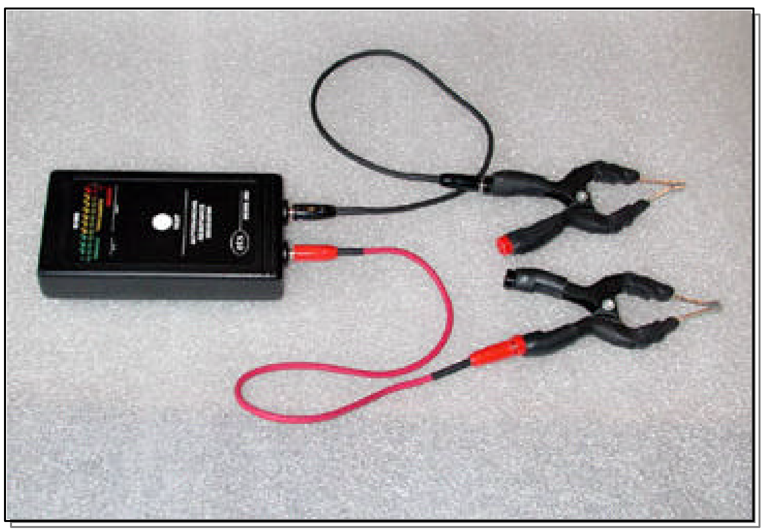


Figure 7

TEST PROCEDURE FOR COMPONENTS & ASSEMBLIES

a. Verify the test set up by measuring the 10 Megohm, 1% resistor at both 10 and 100 Volts.

b. Place the sample on an insulative surface.

c. Connect the electrodes to the ends of the component or to the ground point.

d. If the resistance meter being used allows manual selection of the test voltage, first take a measurement at 10 Volts and when at 100 Volts.

e. Measure and record the resistance after 5-seconds using both 10 Volts and 100 Volts. (J1645 currently specifies 100v and <100v).

f. Acceptance Criteria: The component is considered acceptable if either the 10 or 100 Volt resistance reading is less than the specified upper limit.

Static Decay

Static decay is performed in addition to resistance testing for new material (especially if stable resistance measurements cannot be obtained) and is specified for materials soaked in fuel. The defined test procedure takes into account initial charge, maximum accepted charge and decay time to 1% for new material and to 10% for fuel soaked material. Material meeting the defined criteria indicates that an adequate uniformly distributed conductive component is present.

STATIC DISSIPATION

Figure 8 shows a typical static dissipation test set-up.

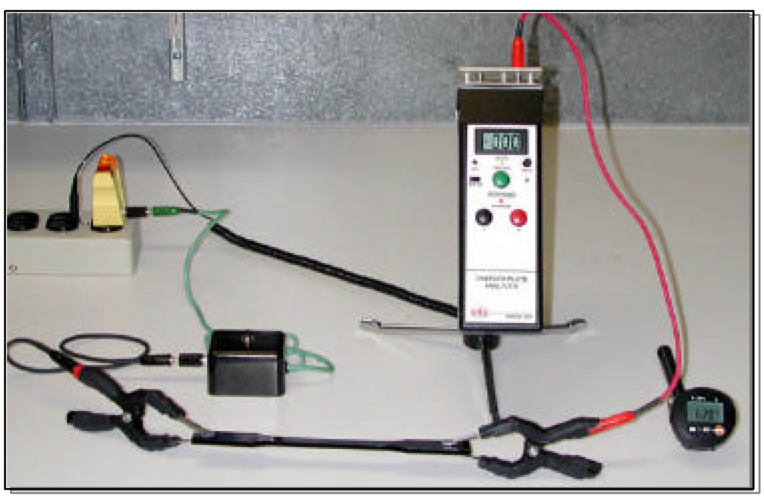


Figure 8

a. Prepare the samples to be tested in the same manner as described for resistance.

b. Verify the test set up by measuring the dissipation time for a 10 megohm, 1% resistor.

c. Set the measurement parameters for a >1000 Volt charge then connect the grounded electrode to the resistor. The decay time measured will be from 1000 to 100 Volts. All readings shall be ≤0.50 seconds.

d. Replace the resistor with the assembly.

e. Place the sample on an insulative surface.

f. Apply the charging voltage for approximately 2 seconds or when the display stabilizes at the reading greater than 1000 volts. Immediately ground the assembly by connecting the grounding electrode to the desired point. Repeat the measurement 3 times and record the results.

g. Acceptance Criteria: The dissipation time measured shall be <0.50 second.

REFERENCES

The following references are applicable for measuring conductive and low static dissipative material and components:

1. ASTM-D991-1989: Standard Test Method for Rubber Property – Volume Resistance of Electrically Conductive and Antistatic Products
2. UL 330: Hose and Hose Assemblies for Dispensing Flammable Liquids
3. ESD Association standards: ESD Association, 7900 Turin Rd, Bldg 3, Rome, NY 13440 (315-339-6937)

    a. ESDA Adv1.0: Glossary of Terms
    b. ESD STM 11.11-2001: Surface Resistance Measurement of Static Dissipative Planar Materials (To be reviewed & modified in 2005)
    c. ESD STM11.12-2000: Volume Resistance Measurements of Static Dissipative Planar Materials (To be reviewed & modified in 2005)
    d. ESD STM11.13-2004: Two-Point Resistance Measurement of Conductive, Dissipative and Insulative Items
    e. ESD TR 02-99: High Resistance Ohmmeters – Voltage Measurements
4. Military Specifications
    a. Antistatic Properties of Materials is now Mil Std 3010, Method 4046.1 (Formerly FTM 101C, Method 4046.1)
    b. Mil-PRF-81705D (Formerly Mil-B-81705D)

TEST RESULTS

The test procedures for measuring the resistance of components and assemblies, and static dissipation of assemblies were used to measure actual parts. Two different test instruments for each procedure were used. The following are the results obtained:

Part	**Resistance-Ohms**				**Dissipation**	
	Dr. Thiedig (Milli-TO-2)		ETS 872		EA3	ETS 204
	@10V &	@100V	@10V	@100V	Sec	Sec
10 Meg Resistor	10 Meg	9.96 Meg	9.9 Meg	10 Meg	0.13*	0.5*
Fuel Filter Assy.						
Hose-Hose (L)	5.0 Meg	1.6 Meg	4.7 Meg	2.0 Meg	0.13	0.5
Hose-Gnd Pt.	1.1 Meg	<1 Meg	330 Kilo	180 Kilo	0.13	0.5
Hose-Gnd Strap	2.2 Gig	8.1 Meg	1.9 Gig	25 Meg	0.13	0.5
Fuel Filter	140 Meg	22 Meg	23 Meg	3.5 Meg	0.13	0.5
Turn Electrodes 90°			24 Kilo	120 Kilo		
Hose w/Conn.	>1 Gig	>1 Terra	>1 Terra	>1 Terra		
10” Coated Tube	>1 Gig	>1 Terra	>1 Terra	>1 Terra		
Elbow Conn.	<1 Kilo	<1 Meg	180 ohms	<10 Kilo		
Test Strips						
PPS-1	1.1 Kilo	<1 Meg	2.2 Kilo	<10 Kilo		
PPS-2	<1 Kilo	<1 Meg	4.2 Kilo	<10 Kilo		
PPS-3	<1 Kilo	<1 Meg	8.0 Kilo	<10 Kilo		
POM-1	1.4 Gig u/s**	1.1 Meg	310 Meg	13 Meg		
POM-2	23 Meg u/s	420 Kilo	2.8 Gig	13 Meg		
POM-3	5.5 Meg u/s	<1 Meg	350 Kilo u/s	<10 Kilo		
POM-4	680 Meg u/s	7.4 Meg	2.5 Gig	60 Meg		

Notes:

* Dissipation times are the fastest times each CPM can measure. A modified Model 204 will measure to 0.15 seconds

** u/s denotes an unstable resistance measurement

Kilo = 10^{3}
Meg = 10^{6}
Gig = 10^{9}
Terra = 10^{12}

TEST INSTRUMENTATION

Instrumentation to perform the above measurements is available commercially. Please contact Electro-Tech Systems for additional information on probes and electrodes mentioned in this article at 215-887-2196 or visit our web site at www.electrotechsystems.com.

About the Author *-* ***Stanley Weitz****, President of ETS, Inc., earned his BSEE from Purdue University in 1961 along with graduate work at the University of Pennsylvania and Penn State University. He has worked for both the US Navy and industry in developing Advanced Airborne Antisubmarine Warfare Systems and has also developed automotive diagnostic systems. Mr. Weitz has worked as an Independent Consultant and was part owner of an electronic manufacturing company specializing in Medical Instrumentation. In 1976, Mr. Weitz founded Electro-Tech Systems, Inc. (ETS) which has grown to become a leader both in the static control and environmental control industries. Mr. Weitz is a member of a number of military and industry associations including the ESD Association, SAE, EIA, ASTM and NFPA and is directly responsible for many specifications and test procedures used to classify static control materials and products.*

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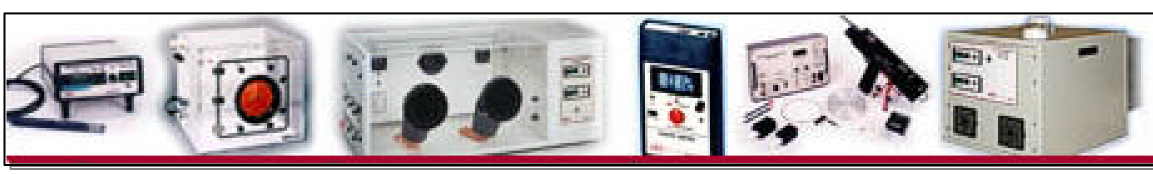
ETS Facility



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