Designation: D7148 – 13

An American National Standard

Standard Test Method for Determining the Ionic Resistivity (ER) of Alkaline Battery Separator Using a Carbon Electrode in an Electrolyte Bath Measuring System[1]

This standard is issued under the fixed designation D7148; 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 covers the pretreatment, test conditions, apparatus, and procedure to determine the ionic resistivity, commonly referred to in the battery industry as electrical resistance (ER) of an alkaline battery separator immersed in an electrolyte of 40 % potassium hydroxide (KOH).

1.2 The values stated in SI units are to be regarded as the standard.

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:*[2]

D1711 Terminology Relating to Electrical Insulation

2.2 *Battery Council International:*

Standard Test Method for Determining the Electrical Resistance of a Battery Separator Using a Palico Measuring System, Section 3.3b[3]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *barrier resistance (RB), n*—the resistance of the bath with a solid, nonporous sheet of alkaline resistant, electrical insulation that separates the electrodes.

3.1.2 *bath resistance (RC), n*—the resistance of the bath without the specimen (separator).

3.1.3 *bath resistance (RT), n*—the total electrical resistance of the bath and a separator specimen.

3.1.4 *battery separator, n*—an ion-permeable, nonconductive material that prevents electrical contact between electrodes of opposite polarity.

3.1.5 *electrolyte, n*—a 40 % potassium hydroxide solution.

3.1.6 *ionic resisitivity (ER), n*—the product of a change in electrical resistance times an area.

3.1.6.1 *Discussion*—The area is the aperture area divided by the number of separators between the electrodes. The change in resistance is the difference, in ohms, of the electrical resistance with and without the seperator(s). The SI units for ER are ohms-metre^2 but the customary practice in the battery industry is to report the ER in units of ohms-cm^2. It should be noted that this terminology is not in conflict with Terminology D1711.

3.2 *Symbols:*

3.2.1 ER—a symbol, peculiar to the battery industry, denoting that characteristic of a sheet material that is related to the rate of transfer of ions through the interstices of a porous sheet immersed between two carbon electrodes in an aqueous electroyte.

4. Summary of Test Method

4.1 This test method detects small changes in ohmic resistance between carbon electrodes immersed in an alkaline electrolyte with and without separator material between the carbon electrodes. This change is related to the rate of transfer of ions through a separator material. The ER is calculated from the fixed aperture area. The sensitivity and resolution of the apparatus detects electrical resistance changes of ±0.1 %.

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[1] This test method is under the jurisdiction of ASTM Committee D09 on Electrical and Electronic Insulating Materials and is the direct responsibility of Subcommittee D09.19 on Dielectric Sheet and Roll Products.

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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.

[3] Available from Battery Council International, 330 N. Wabash Ave., Ste. 2000, Chicago, IL 60611.

5. Significance and Use

5.1 The ER of a battery separator is a standard measurement used by separator and battery manufacturers for quality control purposes and separator selection.

5.2 Separator ER and the separator's interaction with the electrolyte, that is resistance to wetting or flow, will contribute to the internal resistance of the battery and this can potentially limit the electrical output of a battery. The ER determination is a tool for battery manufacturers to use in design, material selection, and performance specifications.

5.3 The change in the bath electrical resistance imparted by a separator is affected by the porosity, thickness, and tortuousity of the pore structure of the separator, the wettability of the separator to the electrolyte, and the temperature and concentration of the electrolyte.

5.4 Incomplete wetting or saturation of the pore structure limits the lowest ER value obtainable from a separator structure. Separators are pretreated to assure that the specimen being tested has been adequately wetted out. A separator that is not fully wetted out (saturated) will give a higher ER.

5.5 This test method is intended to give a rapid and repeatable measurement that approximates the change in ER that could happen when the separator is used in a battery.

6. Apparatus

6.1 Stainless steel container or equivalent to accommodate the separator test samples.

6.2 Hot plate or other heating device suitable to boil water.

6.3 Stainless steel screen.

6.4 Glass or plastic separator tank for presoaking separators in battery electrolyte.

6.5 Interval timer.

6.6 Safety glasses.

6.7 Thermometer.

6.8 Alkaline resistant gloves, that is neoprene latex or polyethylene gloves.

6.9 Plastic tongs for handling KOH-wetted separators.

6.10 ER bath with a fixed aperture area (32 cm^2 is standard) that puts a uniform current flux through a fixed area of the separator sample for measurement. A drawing of an ER bath is included in Annex A1.

6.11 A.C. resistance meter or bridge with sufficient sensitivity and resolution to measure ±0.1 % change in the ER bath resistance in the presence of potentials generated in the ER bath.

7. Reagents and Materials

7.1 Distilled or deionized water.

7.2 Reagent grade KOH.

8. Hazards

8.1 Safety goggles and face shield, lab coat, and rubber apron and gloves must be worn during KOH dilution operations. Enclose the glass-mixing bottle in a plastic container of sufficient capacity to contain the solution in case of bottle breakage. To prevent excessive heating, add concentrated KOH slowly to water with adequate agitation to produce thorough mixing. The diluted KOH is sufficiently corrosive at both ambient and elevated temperatures that careful handling is necessary to prevent injury or damage.

8.2 The hazard associated with mixing KOH with water is that it generates a lot of heat as it dissolves; this has the potential to lead to spattering. A concentrated KOH solution will erode skin and eyes. Avoid these hazards by wearing safety glasses, face shield, and protective gloves, and immediately rinsing with water if any skin contact occurs. (If any eye contact occurs, flush the eyes with water for 15 min and then get medical attention.)

9. Sampling, Test Specimens, and Test Units

9.1 Select samples representative of the separator material to be tested.

9.2 Use a specimen size of at least 120 by 120 mm and no larger than what will fit in the bath.

9.3 If specimens require identification, use an alkaline resistant marker. Mark an area of the specimen that will not interfere with the ion transfer through the separator.

10. Preparation of Apparatus

10.1 Fill the ER bath with prepared battery electrolyte (40 ± 1 % KOH solution) to a level above the aperture opening (12 to 25 mm below the top of the ER bath). Adjust the bath's temperature to 25 ± 5°C .

10.2 Connect the electrodes to the resistance meter control unit using alkali-resistant wire and hardware.

10.3 Turn on the resistance meter. Allow the equipment to stabilize for at least 20 min. If the meter includes a standard reference resistor (place in "Standby" mode and "Standard" scaling on the resistance meter control unit and set to the appropriate resistance range scale for a 0.200-Ω resistor reading). Unless the meter reading is within ±0.1 % of the value of the resistor, service will be required.

10.4 If available, check the resistance standard for the system outlined in 11.4. If this agrees within ±1 %, proceed to Section 13.

10.5 Periodically, or when problems occur or changes are made to the system, test the stability, polarization, current leakage, and standardization using the maintenance procedures set forth in Section 11.

11. Testing and Maintenance of Equipment

11.1 Stability of Equipment

11.1.1 Check the resistance of the ER bath (Place the meter in "Operate" mode and "Standard" scaling and press "Reset." Set the appropriate resistance range to give the maximum meter reading without going off scale.).

11.1.2 Agitate the electrolyte to make certain that the temperature and concentration are uniform.

11.1.3 Ensure the bath resistance (RC) is constant within ±0.5 % at a given temperature and electrolyte concentration.

11.1.4 If readings are inconsistent, proceed to 11.2 and 11.3 for polarization and leakage determination.

11.2 Polarization of Equipment

11.2.1 Polarization affects the reading of any resistance measuring system differently. Follow manufacturer instructions.

11.2.2 For the resistance measuring system, place the resistance unit in "Operate" mode and "Standard" scaling and press "Reset."

11.2.3 Set the appropriate resistance range to give the maximum meter reading (RC) without going over scale.

11.2.4 Press "Test Offset." If the reading is higher than midrange of the set value then the polarization is too high.

11.2.5 If the polarization is above the maximum, allow 30 min for stabilization or discharge, or both. Then repeat measurement.

11.2.6 If the polarization persists, change the electrolyte and clean the electrodes.

11.3 Leakage Current

11.3.1 If the test bath has warped or cracked, it can have leakage currents, and potentially affect the ER value measured. Check the bath periodically, whenever the electrolyte is changed or if the bath is moved by using the following procedure:

11.3.1.1 Insert a thin solid sheet of alkali-resistant solid flexible nonporous plastic (such as polyethylene) that has the same dimensions as the separator specimen. Close the gate snugly against the plastic sheet. Adjust the resistance range of the meter to obtain the fullest scale reading possible and record as the barrier resistance (RB). It is possible that the reading will rise after the gate is closed. Wait until the readings are constant.

11.3.1.2 Remove the barrier sheet and measure the bath resistance (RC).

11.3.1.3 If the ratio of RC/RB is greater than 0.07, bath maintenance or replacement is required. In the ER bath, this ratio can be reduced by sealing the leaks or cracks in the partitions. A bath with sealed partitions is harder to clean but is more stable electrically and mechanically.

11.4 Standardization

11.4.1 If available, insert a porous plastic sheet that has been standardized for the ER bath into the test area and close the slide. Read the bath resistance with the sheet in place (RT).

11.4.2 Remove the sheet without moving the slide and read the resistance of the bath (RC) without moving the slide.

11.4.3 Ensure that the difference between these two readings times the aperture area is equal to the measured standard within ±1 % corrected for temperature in 14.1.

11.4.4 In the resistance measuring system, it is possible to set the meter to "Scaled" mode and to press the "Preset" switch with the plastic standard in the test area with a closed slide. When the standard is removed, ensure that the reading on the meter equals the standardized value within ±1 % when corrected for temperature.

11.4.5 Clean the surfaces of the bath and the surroundings after each use to remove spilled electrolyte on the surfaces and connections. Ensure that the resistance meter is far enough from the KOH bath to prevent fumes from corroding the meter. Check and if necessary adjust the electrolyte to the proper concentration before each use. If polarization contaminants begin to affect the reading, replace the electrolyte. To prevent cracking or crazing of the plastic bath, avoid adding hot electrolyte or mixing concentrated alkali in the bath.

11.4.6 If the bath must be dried for shipment, storage, or another reason, pour the electrolyte out and refill the bath with deionized water. Allow it to stand for 24 h. Then drain it and allow it to dry at room temperature. This will minimize the chance of cracking or crazing of the bath's plastic walls.

12. Specimen Conditioning

12.1 Use specimen as received. If samples are hydrophobic, use a prewetting procedure.

13. Procedure

13.1 Measure the temperature of the electrolyte in the ER bath. If the temperature is not within 25 ± 5°C, adjust the bath temperature.

13.2 Remove the pretreated specimens from the separator storage tank and insert the specimens into the test area of the ER bath.

13.3 Tap the separator specimens to remove any air bubbles clinging to the surfaces.

13.4 Carefully close the gate against the separator, so that the separator can be removed without moving the gate. Measure the total resistance of the bath and the separators. Record as RT. If the resistance drifts, wait until the unit fluctuates over the same range and then record the mean value of the range. For the resistance meter's control unit, press the "Reset" switch and then the "Preset" switch. Repeat if the meter does not zero out.

13.5 Remove the specimen and remeasure the resistance of the bath (RC) with no separators. Important: do not move the gate. For the resistance meter control unit with the "Preset" option, the reading after removing the separators directly gives the difference (RT-RC) used in the calculations in 14.1.

13.6 If the resistance meter controls uses a "Scaled" setting, the appropriate aperture area is automatically multiplied times the meter reading. If the unit is in the "Standard" mode, aperture calculations shall be made manually

13.7 Repeat this procedure for the remaining specimens.

14. Calculation or Interpretation of Results

14.1 ionic resistivity (ER)

$$\text{ionic resistivity}\ (ER) = (RT - RC) \times \frac{\text{Aperture Area}}{\text{No. of separator specimens}} \tag{1}$$

where:

RC = the bath resistance obtained in 13.5, and
RT = the total resistance of the bath and separators obtained in 13.5.

14.2 If the resistance meter control unit uses the "Scaled" setting and the "Preset" feature, the appropriate aperture area is automatically multiplied times the reading and the calculation is simply:

$$\text{ionic resistivity}\ (ER) = \text{Meter Reading} \times \frac{CF}{\text{No. of separator samples}} \tag{2}$$

14.2.1 Units for ER are (ohms) × area (cm^2) and are typically reported as units of "ohm-cm^2."

14.2.2 Report the pretreatment method used, such as "20-min soak" or "boiled" and the aperture size of the ER bath and the number of separator samples tested.

15. Report

15.1 Report the following information:

15.1.1 Ionic resistivity (ER) in ohm-cm^2.

15.1.2 The bath's KOH concentration and the pretreatment method used, if any.

16. Precision and Bias

16.1 The precision of this test method is based on an interlaboratory study of ASTM D7148 -07 Standard Test Method for Determining the Ionic Resistivity (ER) of Alkaline Battery Separator Using a Carbon Electrode in an Electrolyte Bath Measuring System, conducted in 2010. Two laboratories participated in this study. Each of the two labs reported duplicate test results for three different materials, taking resistivity measurements in both 100 cm^2, and in.^{2} areas. Every "test result" reported represents an individual determination. Except for the use of only two laboratories, and a limited number of materials, Practice E 691 was followed for the design and analysis of the data; the details are given in ASTM Research Report No. D09-1038[4]

16.1.1 *Repeatability limit (r)*—Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the "*r*" value for that material; "*r*" is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory.

16.1.1.1 Repeatability limits are listed in Tables 1 and 2.

16.1.2 *Reproducibility limit (R)*—Two test results shall be judged not equivalent if they differ by more than the "*R*" value for that material; "*R*" is the interval representing the critical difference between two test results for the same material, obtained by different operators using different equipment in different laboratories.

16.1.2.1 Reproducibility limits are listed in Tables 1 and 2.

16.1.3 The above terms (repeatability limit and reproducibility limit) are used as specified in Practice E 177.

16.1.4 Any judgment in accordance with statements 16.1.1 and 16.1.2 would normally have an approximate 95 % probability of being correct, however the precision statistics obtained in this ILS must not be treated as exact mathematical quantities which are applicable to all circumstances and uses. The limited number of materials tested and laboratories reporting results guarantees that there will be times when differences greater than predicted by the ILS results will arise, sometimes with considerably greater or smaller frequency than the 95 % probability limit would imply. Consider the repeatability limit and the reproducibility limit as general guides, and the associated probability of 95 % as only a rough indicator of what can be expected.

16.2 *Bias*—At the time of the study, there was no accepted reference material suitable for determining the bias for this test method, therefore no statement on bias is being made.

16.3 The precision statement was determined through statistical examination of 24 results, from two laboratories, on three materials. These three materials were described as the following:

Part 1: Commercial Type of Cellulosic Separator—Typical of AAA/AA Size Alkaline Cell

Part 2: Commercial Type of Cellulosic Separator—Typical of C/D Size Alkaline Cell

Part 3: Non-Woven PP Based Material

To judge the equivalency of two test results, it is recommended to choose the material closest in characteristics to the test material.

TABLE 1 Resistivity (mohm/100 cm^2)

Material	Average	Repeatability Standard Deviation	Reproducibility Standard Deviation	Repeatability Limit	Reproducibility Limit
	$\bar{x}$	S_r	S_R	r	R
Part 1	0.0620	0.0039	0.0039	0.0109	0.0109
Part 2	0.1250	0.0043	0.0101	0.0121	0.0282
Part 3	0.1453	0.0116	0.0116	0.0325	0.0325

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[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D09-1038. Contact ASTM Customer Service at service@astm.org.

TABLE 2 Resistivity (milliohm/in.^{2})

Material	Average	Repeatability Standard Deviation	Reproducibility Standard Deviation	Repeatability Limit	Reproducibility Limit
	$\bar{x}$	S_r	S_R	r	R
Part 1	4.0000	0.2500	0.2500	0.7000	0.7000
Part 2	8.0625	0.2795	0.6495	0.7826	1.8187
Part 3	9.3750	0.7500	0.7500	2.1000	2.1000

17. Keywords

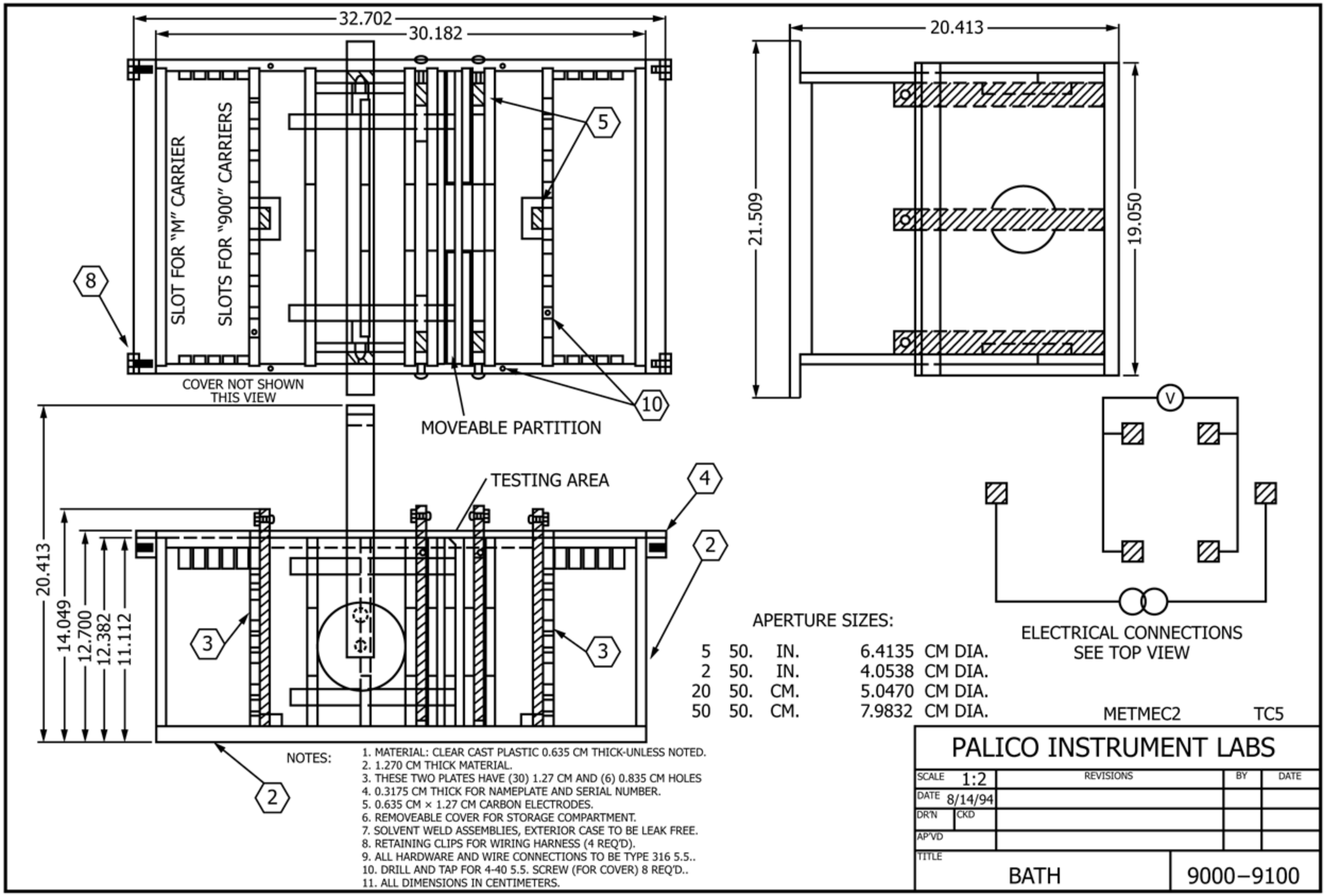
17.1 alkaline; battery; ER; ionic resistivity; separator

ANNEX

(Mandatory Information)

A1. ANNEX

A1.1 **ER BATH** (See Fig. A1.1.)



NOTE 1—Palico address: 6189 Fieldstone Ct., Stacy, MN 55079, U.S.A.

FIG. A1.1 ER Bath

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