SAE International

SURFACE VEHICLE RECOMMENDED PRACTICE

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Recommended Practice for Determining Material Properties of Li-Battery Separator

RATIONALE

As the market for Li-battery continues to grow, due to the evolution of motive and stationary power applications, new separator concepts are being proposed for incorporation into the batteries. There are a variety of properties that could be measured and a variety of methodologies to perform testing. This Recommended Practice (RP) provides a set of test methods for the characterization of the Li-battery separator's properties, which if used consistently across different materials, will facilitate the comparison of the properties of Li-battery separator.

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1. SCOPE

This SAE RP provides a set of test methods and practices for the characterization of the properties of Li-battery separator.

The test methods in this RP have been grouped into one of three categories:

1. Manufacturing parameters: Minimum set of separator properties to be measured
2. Chemistry/Customer specific parameters: Properties that are dependent on the application, customer needs and/or requirements, manufacturing process etc. This RP will include the current best practice methodologies for these tests, with an understanding that the best practice methodologies are evolving as more information is learned.
3. R&D parameters: Properties that are dependent on the application, customer needs and/or requirements, manufacturing process etc. The methodologies in this 3rd section are under development and have not yet achieved broad application.

It is not within the scope of this document to establish criteria for the test results, as this is usually established between the vendor and customer.

2. REFERENCES

2.1 Applicable Document

The following publications form a part of this specification to the extent specified herein. Unless otherwise specified, the latest issue of SAE publications shall apply.

2.1.1 SAE Publications

Available from SAE International, 400 Commonwealth Drive, Warrendale, PA 15096-0001, Tel: 877-606-7323 (inside USA and Canada) or 724-776-4970 (outside USA), www.sae.org.

SAE J1715 Hybrid Electric Vehicle (HEV) & Electric Vehicle (EV) Terminology

2.1.2 American Society for Testing and Materials (ASTM)

Available from ASTM International, 100 Barr Harbor Drive, P.O. Box C700, West Conshohocken, PA 19428-2959, Tel: 610-832-9585, www.astm.org

ASTM D149-09	Dielectric Breakdown Voltage and Dielectric Strength of Solid Electrical Insulating Material at Commercial Power Frequencies", 2009
ASTM D618-00	Standard Practice for Conditioning Plastics for Testing
ASTM D726-94	Standard Test Method for Resistance of Nonporous Paper to Passage of Air
ASTM D882-09	Standard Test Method for Tensile Properties of Thin Plastic Sheeting
ASTM D1204-08	Standard Test Method for Linear Dimensional Changes on Nonrigid Thermoplastic sheeting or Film at Elevated Temperature
ASTM 2103	Standard Specifications for Polyethylene Film and Sheeting
ASTM D2873-94	Standard Test Method for Interior Porosity of Poly (Vinyl Chloride) (PVC) Resins by Mercury Intrusion Porosimetry.
ASTM F316-03	Standard Test Method for Pore Size Characteristics of Membrane Filters by Bubble Point and Mean Flow Pore Test
ASTM D3755-97	"Dielectric Breakdown Voltage and Dielectric Strength of Solid Electrical Insulating Materials under Direct Voltage Stress", 2004, are recommended
ASTM D5947-06	Standard Test Methods for Physical Dimensions of Solid Plastic Specimens
ASTM D6287 - 09	Standard Practice for Cutting Film and Sheeting Test Specimens

2.1.3 Japan Industrial Standard (JIS)

Available from Japanese Standards Association, 4-1-24 Akasaka, Ninato-ku, Tokyo, 104-8440 Japan; Telephone: 03-3588-8002; Fax: 03-3583-0462; http://www.jsa.or.jp/default_english.asp

JIS P 8117	Paper and board - Determination of air permeance and air resistance (medium range) - Gurley method

2.1.4 United States Publications

NASA/TM-2010-216099: "Battery Separator Characterization and Evaluation Procedures for NASA's Advanced Lithium-ion batteries", May 2010.

USABC "Procedure for determining shutdown temperature of battery separators" (http://www.uscar.org/guest/article_view.php?articles_id=86)

UL2591, "Investigation for Battery Separator", Issue 2, March 2009

ORNL/TM-2012/247 ("Standard Test Method for Fatigue Evaluation of Polymeric Separators for Li-ion Cells", E. Lara-Curzio and T.M. Trejo, 2011 is recommended

3. DEFINITIONS

Except as noted below, all definitions are in accordance with SAE J1715.

4. SAMPLE PREPARATION

Except where indicated, the tests in this recommended practice are to be performed at standard laboratory atmosphere, defined in ASTM D618-00 (Standard practice for Conditioning Plastics for Testing, Section 3).

Some of the test techniques discussed in the following section require preparation of samples with an accurate surface area. Preparation of such a sample is described in ASTM D6287-09 (Standard Practice for Cutting Film and Sheeting Test specimens)

In all cases, samples should be conditioned (e.g., dried) using the manufacturer's recommended practice

Except where indicated, the tests of this recommended practice are to be performed on a dry separator. It is possible that the addition of a solvent or electrolyte impact the performance of the separator. Users of this RP are urged to consider the interaction of the separator and solvent and perform tests on wet separators when deemed necessary, such as when a significant change in behavior is suspected to occur. The safety issues related with handling of electrolytes should be considered before doing the tests.

5. MANUFACTURING PARAMETERS

5.1 Thickness (μm)

In order to accurately and repeatedly measure thickness, some degree of pressure is applied to the film.

The acceptable pressure to be applied to the film during thickness measurement is determined by the film's porosity, composition and compressibility.

NOTE: Compression modulus of the material should be taken under consideration when selecting the pressure to be applied when measuring thickness. The pressure which yields the smallest degree of film compression is to be selected.

Thickness measurements are described in 2 separate ASTM documents, whose main difference is the pressure to be applied:

ASTM D5947-06: Standard Test Methods for Physical Dimensions of Solid Plastic Specimens

ASTM D2103-08: Standard Specifications for Polyethylene Film and Sheeting

The user is tasked with selecting the best method based on the criteria described above.

Basis Weight (grams / cm^2)

Sample preparation: Preparation of the sample area is critical to accurate determination of the basis weight - repeatable surface area is necessary. A sufficient amount of sample needs to be prepared to yield a recordable value on the balance.

Equipment specification: Balance accurate to 4 decimal places (± 1mg)

Sample size: Surface area of at least 225 cm^2 (equivalent of 15 x 15 cm)

Measurement environment: standard laboratory atmosphere (see Section 3)

Measurement frequency: measure each sample 3 times, and then average.

Number of samples: ≥ 3

5.2 Air Permeability (measure of time required for 100mL of air to pass through film).

The method as described in JIS P 8117 ("Paper and board – Determination of air permeance and air resistance (medium range) - Gurley method", 2009), is recommended with the following clarification

- for rapid elution times, the head fitting may need to change to a smaller footprint (0.645cm^2 = 0.1 sq inch). This smaller head fitting increases elution time to improve measurement resolution.
- normal head = 1 sq inch (= 6.45 cm^2)

In the event that the user does not have equipment to perform Gurley measurement, the alternative burette method, as described in ASTM D726 - 94 ("Standard Test Method for Resistance of Nonporous Paper to Passage of Air") may be used, with the following clarification

- The user must be able to transform the burette method result into an equivalent Gurley measurement result. The user is encouraged to measure Gurley and empirically determine the correlation factor.

5.3 Porosity

5.3.1 Calculated porosity

Requirements: Thickness (3.2), basis weight (3.3) and specific gravity (sg).

$$Porosity(\%) = \left(1 - \frac{BasisWeight}{Thickness * SpecificGravity}\right) * 100\%$$

5.3.2 Absorption method

NOTE: This method is recommended for materials of composite compositions where density is not known. Absoprtion techniques will measure porosity from blind pores and through pores.

Method:

- The sample is to be conditioned per manufacturer's recommendation.

- A liquid capable of wetting the substrate is to be used, preferably an oil or an alcohol, although the latter is more susceptible to evaporation

- Cut (minimum dimension of 10cm x 10cm or equivalent) and weigh the sample (m_{dry_sep}).

- Immerse the sample in the liquid for a minimum of 5 minutes. Remove the sample from the liquid.

- Fold the sample multiple times so that the exterior surface represents <10% of the total sample volume (purpose of folding is explained below).

- After absorption, excess liquid resides on the surface of the sample. To minimize the impact of this residual liquid, it is recommended to fold the sample into multiple layers and after absorption, to compress the sample to remove excess liquid from the inner layers (apply pressure as per thickness measurement). After compression, the excess liquid residing on the exterior surface should be gently blotted, where the blotting material is pre-dampened and has large pore size to prevent wicking of the liquid from the separator pores to the blotting material.

- Re-weigh the sample (m_{wet_sep}). Since any extra mass comes from the addition of liquid, whose density is known, pore volume is calculated by dividing the change in mass by the density of the liquid ($\rho_{electrolyte}$). This pore volume divided by the total sample volume yields porosity.

$$PoreVolume = \frac{m_{wet\_sep} - m_{dry\_sep}}{\rho_{electrolyte}}$$

5.3.3 Liquid intrusion method

NOTE: This method is recommended for materials where voids / impenetrable pore/ completely enclosed pore are suspected.

A Porosimeter is to be used. Porosimeters are based on the principle of either liquid intrusion into pores or liquid extrusion from pores. Porosimeters are instruments that are capable of measuring pore volume and pore volume distribution. Liquid intrusion porosimeters measure porosity from through pores and blind pores. Liquid extrusion will measure porosity only from through pores.

The porosimeter (intrusion method) and the material under investigation will determine which solvent is to be used (see below).

Intrusion method:

- The solvent must be non-wetting / contact angle > 90^{o}.

- Water intrusion is preferred since the solvent is innocuous. However it may not be possible to use water since not all separator materials are hydrophobic.

- Mercury intrusion: method as specified in ASTM D 2873 – 94 is recommended.

    - The user is cautioned that there is anecdotal evidence that mercury may chelate with polyolefin; reaction of the mercury with silicates. The user is encouraged to investigate further before choosing this method.

    - There is also a possibility that the pore structure is damaged during the mercury intrusion method as high pressures are used for the measurement. The user is encouraged to investigate further before choosing the method.

- NOTE:
  - Mercury intrusion porosimetry will give volume in cc/g. A specific gravity is still required to obtain a porosity unless bulk density and skeletal density measurements are also taken
  - Since mercury intrusion porosimetry is a volume based technique, multiple layers of separator are likely to be required. The gap between the layers will then be measured as a pore size and this factor should be eliminated from all calculations.

- Some equipment which is known to perform the testing as described above:
  - Water Intrusion porosimeter (Aquapore) (http://www.pmiapp.com/products/water_intrusion_porosimeter.html)
  - Mercury intrusion porosimeter (http://www.pmiapp.com/products/mercury_porosimeter.html)

6. CHEMISTRY/CUSTOMER SPECIFIC PARAMETERS

All of the tests described in the following sections include a recommendation on the key property to be measured after testing. However, in some cases, the test conditions may result in unintended changes in the separator. To fully comprehend the impact of a test on the separator, the user is encouraged to consider how best to evaluate the separator's performance.

6.1 Pore size

6.1.1 Intrusion techniques

Intrusion techniques are recommended. Refer to 4.4.3 for more information on intrusion methods

Intrusion techniques measure a distribution of both through and blind pores based on volume.

6.1.2 Porometer/capillary flow porometry

NOTE: this technique measures only the 'through' pores in the sample

A Porometer is to be used. The pores in the sample are spontaneously filled with a wetting liquid. Pressure of a non-reacting gas on one side of the sample is slowly increased to remove liquid from the pores and permit gas flow through the pores. Measured differential pressures and flow rates of gas through wet and dry conditions of the sample are used to compute maximum and median pore size, pore size distribution. Determination of the minimum pore size depends on the maximum pressure that can be applied by the instrument. (Refer to equipment manufacturer's manual for information on how the pore diameters are computed).

- The solvent must wet the sample
- Some equipment which is known to perform the testing as described above
- Porous Materials Capillary flow porometer (http://www.pmiapp.com/products/capillary_flow_porometer.html)

Measurement environment: per instrument guidelines

Number of samples: ≥ 5

6.2 Tensile strength

Tensile properties of the material should be determined using the methods described in ASTM D882 – 09: Standard Test Method for Tensile Properties of Thin Plastic Sheeting.

6.3 Shrinkage

6.3.1 Unrestrained Shrinkage

NOTE: material should be conditioned per the manufacturer's recommendation.

6.3.1.1 Oven method

Generally, the methods as described in ASTM D1204-08: "Standard test method for linear dimensional changes on non-rigid thermoplastic sheeting or film at elevated temperature", are recommended with the following exceptions

Equipment specification:

- Oven should be suitable for controlling test temperatures within +/-1 ºC in the temperature range of 80 ºC to 260 ºC.
- Thermometer/thermocouple should be graduated in 0.5 °C increments and suitable for the test temperature used.

Sample preparation:

- Measure sample dimensions
- Samples may be hung in the oven (no paper necessary), in which case the separator should not be exposed to temperatures near the melting point.
- If samples are placed in paper, samples should be mounted in silicone paper (Griff Paper and Film, Item No. 03313-001, Rolls: 8 inch wide, Description: 42#S2S Release Liner), not just heavy paper sheets. Talc is not recommended

Measurement conditions:

- Test temperature should be the manufacturer's recommended drying temperature or temperature specified by the battery manufacturer.
- Conditioning time at test temperature and post-test cooling are to be determined in conjunction with the manufacturer. Conditioning time is affected by sample composition.
- Additional tests at temperatures at least 20% above the manufacturer's recommendation are recommended to determine the tolerance of the material to fluctuation in the process

Compare the dimensions to the initial values.

Number of samples: ≥ 3

6.3.1.2 DMA method

Generally, the methods as described in NASA/TM-2010-216099: "Battery Separator Characterization and Evaluation Procedures for NASA's Advanced Lithium-ion batteries", May 2010, 3.3, are recommended with the following exceptions:

- The pre-load force is equipment and separator material dependent. Users should use the minimum pre-load. The 0.001N pre-load force, per the NASA method, may be appropriate but higher force may be needed to keep the separator taut and/or to get repeatable results.
- The force on the sample should be maintained at the pre-load level during testing (in the NASA document, the force increases)
- The isotherm temperature and duration are customer and application specific. The temperature and duration set by the NASA procedure are considered to be a good starting point.

Number of samples: ≥ 3

6.3.2 Restrained Shrinkage

6.3.2.1 DMA with load

Generally, the methods as described in NASA/TM-2010-216099: “Battery Separator Characterization and Evaluation Procedures for NASA’s Advanced Lithium-ion batteries”, May 2010, 3.3, are recommended with the following exceptions:

- The pre-load force is equipment and separator material dependent. (NOTE: 0.001N, per NASA may be appropriate, but higher force may also be needed to keep separator taut and/or to get repeatable results).

- The force to be applied during the temperature sweep is dependent on the material under test. The 0.01N force specified in the NASA procedure is considered a good starting point.

6.3.2.2 Dimensional stability

NOTE: The objective of this test is to evaluate the propensity of the separator to crack/tear when confined, as the separator would be in a cell, and exposed to elevated temperature.

The method, as described in UL 2591 (“Investigation for Battery Separators”, Issue 2, March 2009) 8.1 (Dimensional stability), is recommended with the following exceptions and clarifications:

Test temperature

- At a minimum the test temperature should be 130 °C (per UL 2591), which is a characteristic temperature for shutdown.

- Temperatures > 130 °C should be considered for separators which do not demonstrate shutdown.

Test time/duration

- The sample must be exposed to the test temperature for a minimum of 1 hr.

Sample measurement

- Per UL2591, the post-test sample should be examined for damage such as tears, cracks.

- As necessary, additional tests, such as thickness, pore size, porosity, air permeability, and tensile strength, could be performed before and after heat treatment.

6.3.3 Local Shrinkage / thermal penetration

This test aims at determining the resilience of the separator to local heating as a result of particle penetration.

There are currently no known methods. Some methods under discussion include (various load and/or temperature conditions) and may be available at the time of reading

6.4 Shutdown

The method as described in USABC “Procedure for determining shutdown temperature of battery separators” is recommended. The method can be found at: http://www.uscar.org/guest/article_view.php?articles_id=86.

6.5 Resilience to puncture during cell manufacturing

The method, as described in UL 2591 ("Investigation for Battery Separators", Issue 2, March 2009) 7.2 (Puncture strength), is recommended with the following exceptions and clarifications:

Penetration speed:

- The recommended range for penetration speed is between 25 mm/min and 50 mm/min.

6.6 Separator wetting

6.6.1 Wettability

To result in a functional battery, the electrolyte must wet the separator; hence, the wettability of the separator in a given electrolyte is a key parameter.

All of the tests described below are to be performed in a glove box or dry room; 25°C ± 5°C.

6.6.1.1 Visual check

For a traditional polyolefin, the wettability of the separator can be judged visually. The separator is to be placed in contact with electrolyte. The separator is deemed to be wettable if it becomes clear.

NOTE: Separators with inorganic particles may not become clear when placed in contact with electrolyte. In this case, wettability is determined by dropping electrolyte on the separator and confirming that it does not remain pooled on the surface of the separator

6.6.2 Rate of wetting - NASA method

A semi-quantitative method to visually measure wetting is detailed NASA/TM 2010-216099 ("Battery separator characterization and evaluation procedures for NASA's advanced lithium-ion batteries", May 2010) 1.4.

6.6.3 Rate of wetting - Wicking method

- Samples should be 1 cm wide (fits within graduated cylinder) and 15 cm long.
- Make 1mm marks down the length of one edge of the separator.
- Secure a strip of the marked material in a test stand so that it hangs vertically. A small weight could be used to help the sample hang vertically.
- Quickly and smoothly lower the sample into an electrolyte reservoir until 1cm of the sample is below the meniscus.
- Record the time needed for the median height of the wetting front of the electrolyte to reach each mm mark.
- Record the final height reached after 10 minutes.
- Number of samples: >5. Samples should consistently be prepared to be in the machine direction or transverse direction, since wetting rates may differ for the different directions.
- Plot average wicking height versus time. The slope of this graph will yield the wicking rate.
- NOTE:Sample width and length may vary, however they should be consistent across different samples.
- NOTE:The user is encouraged to measure rate of wicking in both machine and transverse directions.

6.7 Chemical Stability

Pre-requisite: Characterize degradation of the electrolyte as a function of temperature and/or time. This is necessary in order to later judge whether the separator is chemically inert in the electrolyte.

If the separator, after chemical stability testing, is to be characterized for properties such as tensile strength, air permeability, puncture resistance, porosity, etc., then these values must first be determined on separator material that has not undergone chemical stability testing.

Select the electrolyte based on customer recommendation, application needs

Preparation

- Sample geometry: approximately 3 cm by 10 cm
- Separator should be conditioned per manufacturer's recommendation.
- Measure the dimensions and weight of the separator sample.
- Characterize the separator, as needed, to obtain comparison values.
- Use a clean and dry test vial.

Sample preparation

- Place the sample on a piece of pre-cut Teflon sheet which is slightly longer than the longest dimension of the sample. Record the mass of the Teflon + sample.
- Use a dropper or syringe to apply the electrolyte solution dropwise until the separator is completely wet. The quantity of electrolyte to be added is to be based on either visual confirmation that the separator is completely wet and/or addition of a sufficient quantity of electrolyte to fill all of the porosity in the film.
- Record the weight of the Teflon + sample + electrolyte so that the exact electrolyte weight/volume can be calculated if necessary.
- Quickly roll up the separator in the Teflon and insert into the clean, dry vial. One more drop of electrolyte should be added to the vial to account for evaporative loss. The vial should be tightly capped and placed on an oven-proof tray.

Testing

- Place the sample-containing vial in the pre-heated oven at the desired test temperature. Testing should occur in the range between -50 °C to 70 °C. At high temperatures, the user should consider and apply appropriate safety precautions for storage of the separator/electrolyte samples at high temperature; handling of the samples after they have cooled off (gaseous products generated as a result of testing and/or reactivity of residual electrolyte and salt when exposed to air.)
    - The temperature range is selected based on the USABC Battery Goals (http://www.uscar.org/guest/tlc/3/Energy-Storage-TLC) regarding cell survival range, which specifies that cells must survive 12 hour exposure to temperatures ranging from -46 °C to +66 °C without loss of functionality once returned to normal operating temperature.
- Test sample is to remain at temperature at least 12 h or longer as prescribed by the manufacturer.
- Sample is removed from temperature chamber and allowed to cool.
- Remove the sample from the vial and then carefully extract the separator from the Teflon.
- Visually inspect the separator to make sure it is still fully wetted.
- Rinse the separator in three separate containers with a large excess volume (100-1000 excess by weight of the separator) of IPA and lay flat to dry on a clean piece of Teflon sheet.
- Recondition at the temperature and humidity of the initial measurement (minimum of 12 h).

Measurement

- Visually inspect the separator and electrolyte; Measure separator dimensions and weight and compare to initial values.
- Measure other properties as necessary.
- NOTE:If degradation is found to occur per the inspections above, spectroscopy or other methods could be used (as recommended in NASA/TM-2010-216099: “Battery Separator Characterization and Evaluation Procedures for NASA’s Advanced Lithium-ion batteries”, May 2010) to help identify/clarify degradation mechanism.

NOTE: Testing of the stability of the separator as a function of voltage and temperature is described in 6.1.

6.8 Skew

Ideally, when a roll of separator is unwound and laid flat, the edges should not show bowing (called skew). In practice, skew is normally observed. In extreme cases, skew can lead to misalignment between the electrode and separator.

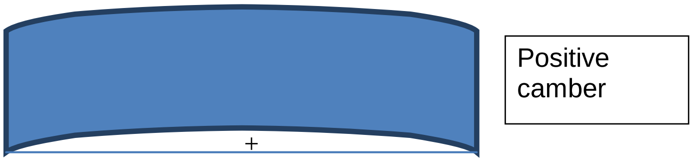
NOTE: This method is considered to have poor gauge repeatability and reproducibility (GRR). The wider the film, the more difficult it is to get the sample flat, leading to poorer GRR

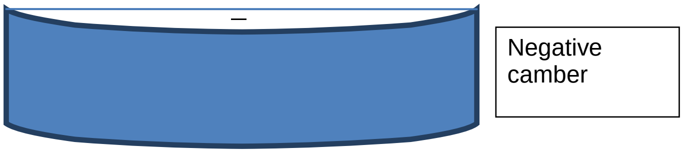
Preparation

- The separator should be sampled from the machine direction. Measurements should always be taken in the same orientation from each roll (i.e., coming off the top of the roll or the bottom of the roll).
- Sample length should be at least 1m. Ideally, the sample length is 3-4 m. Longer length results in improved resolution.
- A straight line reference with less than 0.5 mm deviation per meter is required. The straight reference line may be based on a ruler, the edge of the granite block table, a stretched string, a laser beam or other.

Testing

- Place the separator film flat on a surface large enough to accommodate the full width and length of each sample section.
- Place the concave side (if any) along one edge of a straight line while smoothing the sample so that it lies flat with no wrinkling, bunching, folding, etc. over the entire surface of the sample.
    - NOTE: The work surface may be a lab table covered with a glass sheet, a stainless steel table which may help alleviate static, a granite table with known surface flatness, etc.
    - The two ends of the sample section should touch the straight line, leaving any gap between the sample and the straight line in the middle.
    - Locate the maximum deviation from linearity along the edge and measure this deviation from the straight line to the edge of the separator film.
- Place the convex edge along one edge of a straight line.
    - The two ends of the sample section should touch the straight line.
    - The straight line will be on the sample and the maximum deviation from the line to the convex edge of the sample should be measured and recorded.
    - The maximum deviation of any 1 meter section should be recorded in mm per linear meter as above, and the average, range and standard deviation should also be recorded.





- If there is more than one deviation from the straight line down any meter length, the average should be recorded.

Number of samples ≥ 3

Skew results measured from samples of different lengths should not be compared.

6.9 Melt Integrity

6.9.1 Melt integrity (no compression)

This property allows the user to infer the temperature at which the separator material will reach a sufficiency low viscosity to allow the electrodes to contact.

The method in NASA/TM-2010-216099: “Battery Separator Characterization and Evaluation Procedures for NASA’s Advanced Lithium-ion batteries”, May 2010, 3.5, is recommended with the following exceptions:

- The pre-load force is equipment and separator material dependent. The smallest force possible which keeps the sample taut should be applied.
- This minimum force is to be maintained during the temperature sweep.
- At a minimum, the test should run from 30°C to 200°C. Users should consider whether a higher upper temperature limit is appropriate based on the composition of the separator.

6.9.2 Melt integrity (with compression)

In this variation, force is applied to the separator in order to determine how melt integrity is impacted.

The method in NASA/TM-2010-216099: “Battery Separator Characterization and Evaluation Procedures for NASA’s Advanced Lithium-ion batteries”, May 2010, 3.5, is recommended with the following exceptions:

- The pre-load force is equipment and separator material dependent. The same foot pressure as that applied for thickness measurement is recommended.
- The pre-load force should be maintained during the temperature sweep.
- At a minimum, the test should run from 30°C to 200°C. Users should consider whether a higher upper temperature limit is appropriate based on the composition of the separator.

6.10 Melt Temperature (DSC method)

Generally, the methods as described in NASA/TM-2010-216099: “Battery Separator Characterization and Evaluation Procedures for NASA’s Advanced Lithium-ion batteries”, May 2010, 4.1, are recommended with the following considerations:

At a minimum, the melt temperature of the dry separator should be measured.

The addition of electrolyte to the separator may cause depression of the melting point or solubilization of the separator. If the user considers that either of these or other conditions occur in the presence of electrolyte, DSC testing of the separator should be performed with electrolyte.

- Select the electrolyte based on customer recommendation, application needs and/or compatibility with separator.
- Only add an amount of electrolyte sufficient to fill the pores without excess.
- The electrolyte is volatile and could lead to increased pressure which may impact test results. In the extreme case, gases generated from electrolyte would also lead to pan venting.

6.11 Impact of separator on Ion Conductivity/Electrical Resistance

In a Li-ion battery, the lithium salt in electrolyte offers a means for movement of the lithium ions between electrodes. The separator, added between the electrodes in order to provide electrical isolation, has a measurable porosity, tortuosity and thickness. All of these properties of the separator may impact the ability of the ions to move between the electrodes.

The user should define an electrolyte to be used for conductivity testing. Conductivity results can only be compared for samples prepared using the same electrolyte formulation. The selection of electrolyte is to be defined by the user, based on the desired application and performance. The conductivity of the electrolyte may be measured by a conductivity meter.

Pre-requisite: The resistance of the electrolyte is to be measured separately.

To measure the effective conductivity of the separator/electrolyte, a blocking cell is recommended (coin cell is not recommended because of the pressure variation from sample to sample).

- Condition the separator by placing it in standard laboratory atmosphere (refer to Section 3) for at least 6 h.
- Cell electrodes: The electrodes must be chemically inert and resist the absorption of ions from the material under study (e.g., Stainless steel (SUS); Ni-alloy, Li,).
- Apply same pressure as used for thickness measurement (3.1).
- Option 1: flooded electrolyte test cell, where the distance between the electrodes remains fixed; 1 layer of separator is inserted between electrodes (pressure is not applied)
- Option 2: multiple layers of separator, multiple electrodes
    - Build cell with 1 separator layer. Measure resistance (R1).
    - Add 2nd separator layer to cell. Measure resistance (R2). Resistance of the separator (Rsep2)= R2-R1.
    - Add 3rd separator layer to cell. Measure resistance (R3). Resistance of the separator (Rsep3)= R3-R2.
    - Add 4th separator layer to cell. Measure resistance (R4). Resistance of the separator (Rsep4)= R4-R3.
    - Average Rsep2, Rsep3 and Rsep4 to get the separator resistance.

NOTE: The results of this test are meaningful only if the separator is completely ‘wetted’. The user should determine what conditions are necessary, prior to this test, in order to achieve complete separator ‘wetting’.

Test equipment

- For fixed frequency measurement: LCR meter (http://en.wikipedia.org/wiki/LCR_meter) (Inductance (L), Capacitance (C), Resistance (R)) is used to measure impedance.
- For measurement that sweeps frequency: Electrochemical impedance spectroscopy

Measurement environment:

- At a minimum, testing should be performed at a temperature consistent with standard laboratory atmosphere (refer to Section 3). For results to be comparable, the temperature must be controlled to at least within ±0.5 °C.
- Testing may also be conducted at temperatures ranging from -30°C to 80°C to determine the impact of temperature. The range of temperatures to be measured is dependent on the material, user and the application.

Measurement

- Place the sample in a temperature controlled environment. Let the sample equilibrate at temperature.
- Perform electrochemical impedance spectroscopy test.
    - The procedure imposes a controlled voltage gradient across the thickness of the sample and analyzes the current flow.
    - It is essential that the specimen have uniform thickness and assured contact with both faces of the test cell.
    - Typical operating parameters for EIS:
        - AC signal amplitude of 5 mV
        - Option 1 (preferred): Frequency range: 0.1 to 100 kHz could be employed and will fully characterize the material
        - Option 2 (screening): A single frequency measurement can be performed to ‘screen’ the material. The frequency is selected so that $Z_{img} = 0$ (the impedance = resistance). Typically, this frequency is >1kHz.

Number of samples: ≥ 3

Data analysis

If the blocking cell, as described by option 1, is used, the user will need to correct the measured resistance to compensate for the displacement of the electrolyte by the addition of the separator. The distance between the electrodes = L; the cross sectional area of the separator = A; the thickness of the separator = T. The resistivity of the electrolyte = $\rho_{electrolyte}$.

- $R_{blank} = R_{electrodes} + R_{\underline{electrolyte1}}$

  $= R_{electrodes} + \rho_{electrolyte} L/A$

- $R_{sample} = R_{electrodes} + R_{separator} + R_{\underline{electrolyte2}}$

  $= R_{electrodes} + R_{separator} + \rho_{electrolyte}(L-T)/A$

- $R_{sample} – R_{blank} = R_{separator} - \rho_{electrolyte} T/A \rightarrow R_{separator} = R_{sample} – R_{blank} + \rho_{electrolyte} T/A$

This correction factor is not necessary if a blocking cell, as described in option 2, is used.