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Federal Consortium for Advanced Batteries Pre-application Battery Test Manual

REVISION 1

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FOREWORD

This battery test procedure manual was prepared for the United States Federal Consortium for Advanced Batteries (FCAB). It is based on broad technical needs across multiple applications and is developed to provide federal sponsors and battery end users more common sets of data for pre-application purposes. As defined here the protocols aim to facilitate and accelerate technology transfer of key concepts related to cell design, materials development and innovations in other areas related to advanced batteries. The manual is not meant to serve as a replacement for existing application specific manuals such as those developed for the United States Advanced Battery Consortium (USABC) or for other government purposes such as Military Specifications.

This testing protocol is not intended to determine the commercial viability of any technology that is being tested. Instead, it is prepared as a set of basic, minimum pre-application testing for pre-commercial and commercial, off-the-shelf (COTS) cells for developers interested in coordinating efforts with the United States Government or as part of contract deliverables. The tests are designed to provide basic information that could be used to construct a spider diagram including energy, power, life and thermal performance.

Due to the complexity and the rapid advancement of battery technology some of the procedures may require future revisions. As necessary revisions to this manual will be released. As in many battery test manuals and due to the pre-application status, the procedures described here refer to tests performed at the single cell level. Cell volume, weight and performance can be used to scale to multi-cell architectures.

The Federal Consortium for Advanced Batteries Advanced Battery Technology Transfer and Standards (ABTT&S) Task Group supported the development of the manual. Eric Dufek and Lee Walker from Idaho National Laboratory served as the primary authors for the manual.

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GLOSSARY and ACRONYMS

Beginning-of-Life (BOL) – the point at which characterization of the test article begins. The BOL HPPC is used to determine the Useable Energy Target or confirm the test article meets or exceeds Table 1 energy goals.

C/3 Rate [A]– a current corresponding to the manufacturer's rated capacity (in ampere-hours) for a three-hour discharge at BOL and 30°C between $Vmax_{100}$ and $Vmin_0$. For example, if the battery's rated capacity is 40Ah, then the C/3 rate is 13.3A. Alternatively, the current can be based on the operating capacity between $Vmax_{op}$ and $Vmin_0$.

Charge – any condition in which energy is supplied to the device rather than removed from the device. Charge includes both recharge and regen conditions. Charge is indicated in this manual as a negative value (from the perspective of the battery).

Default rest [h] – a fixed rest period determined at BOL, it is at least one hour or the time needed to achieve thermal and voltage equilibrium (*e.g.*, rate of change less than 1°C/hour or less than 5 mV/h).

Depth-of-Discharge (DOD) [%]– the percentage of a device's operating capacity (Ah) removed by discharge relative to a fully charged condition, normally referenced to a constant current discharge at the C/3 rate.

Device – a cell, module, sub-battery or battery pack, depending on the context. The generic term "device" is normally used in test procedures except where a specific type of device is meant. (Most test procedures are intended to apply to any of these types.)

Discharge – any condition in which energy is removed from the device rather than supplied to the device. Discharge is indicated in this manual as a positive value (from the perspective of the battery).

End-of-Life (EOL) – a condition reached when the device under test is no longer capable of meeting the targets. This is normally determined from HPPC Test results, and may not coincide exactly with the inability to perform the life test profile.

End of Test – a condition where life testing is halted, either because criteria specified in the test plan are reached, or because it is not possible to continue testing.

Fully Charged – the condition reached by a device when it is subjected to the manufacturer's recommended charge algorithm. In this manual, a device is considered "fully charged" at $Vmax_{op}$, though the manufacturer has the option to make $Vmax_{op}$ equal to $Vmax_{100}$.

Hybrid Pulse Power Characterization (HPPC) Test – a Reference Performance Test procedure that is used to determine the pulse power and energy capability under no-load conditions as a function of aging for direct comparison with the targets in a Gap Analysis.

Profile – a connected sequence of pulses used as the basic 'building block' of many test procedures. A test profile normally includes discharge, rest and charge steps in a specific order, and each step is normally defined as having a fixed time duration and a particular (fixed) value of current or power.

Recharge – a charge interval corresponding to the sustained replenishment of energy by a continuous current or power source (such as an engine-generator or off-board charger).

Reference Performance Test (RPT) – periodic interruptions during calendar and cycle life aging to gauge degradation in the test article. Degradation rates are established by comparing results from the

RPTs during life testing with respect to the initial RPT performed immediately prior to the start of life testing (usually referred to as RPT0).

Rest – the condition in which energy is neither supplied to the device nor removed from the device. Rest is indicated by zero current.

State- of-Charge (SOC) [%] – an estimate of the device charge capability expressed as a percentage of the BOL rated or operating capacity and typically reached by obtaining specified voltages.

Voltage limits [V] – numerous voltage limits are defined in the manual as follows:

$Vmax_{100}$ *[V]* - manufacturer's specified voltage and the basis for the rated capacity.

$Vmax_{op}$ *[V]* – corresponds to the upper end of the intended operating window, as specified by the manufacturer. This is the relevant upper voltage used in all testing unless otherwise specified and the basis for the operating capacity. This voltage cannot exceed 420 V (scaled) based on the established performance requirements.

$Vmin_{op}$ *[V]* – (optional) corresponds to the lower end of the intended operating window. It is a variable parameter that will generally decrease as the test article ages and the minimum value is typically specified by the manufacturer.

$V_{nominal}$ *[V]* – The nominal electrochemical voltage between $Vmax_{100}$ and $Vmin_{0}$. It is determined by the ratio between the total discharge energy and discharge capacity from the static capacity test.

Federal Consortium for Advanced Batteries Pre-application Battery Test Manual

1. PURPOSE AND APPLICABILITY

This manual defines a series of tests to characterize aspects of the performance or life behavior for advanced batteries. The general purpose is to provide the ability enable pre-application assessment of technologies for possible use across a range of different purposes in support of the assorted missions of the United States government. The aim is to provide technology developers a pathway to broadly highlight the performance of their technology to different government agencies while using more similar reporting methods to enable the agencies a more direct comparison across technologies.

While most battery test manuals have specific targets which technologies should strive to meet, this manual instead focus on providing commonality in reporting to provide discrete understanding of the operational boundary conditions for new and existing battery technologies. The manual is written to focus on cell level performance and to provide basic information related to the energy, power and rate capability, thermal performance, and life of the specific technology. These are the core components of a spider diagram used by many in the battery community for comparison of different technologies.

2. TEST PROCEDURES

2.1. General Test Conditions and Related Information

In general, testing is divided into three broad phases, characterization, life, and Reference Performance Tests (RPTs). Characterization testing establishes the boundary conditions for cell performance. The high-rate discharge capability test and the high-rate charge tests as well beginning-of-life (BOL) capacity tests are all characterization tests. Life testing establishes behavior over time at various temperatures, states of charge and other stress conditions and includes both cycle life and calendar life testing. Reference Performance Tests establish changes in the baseline performance and are performed periodically during life testing, as well as at the beginning- and end-of-life testing.

While not specifically defined, each of the tests performed should use multiple replicates. Typically replication of results using 5 to 7 different cells for each test would occur. When reporting data the number of replicates should be included. Either the average data (with error bars showing variation or confidence limits) or data from each replicate should be included.

2.1.1. Voltage Limits

There are several varieties of voltage limits including both electrochemical limits dictated by cell design and chemistry and operation limits. As a pre-application manual that is focused on near-commercial and commercial cells the focus here is on the operational limits for the specific cell designs. A more complete discussion of the voltage limits and their definition can be found in the USABC Electric Vehicle Battery Test Manual Rev. 3. In some instances the operational and electrochemical voltage windows may align with each other. This is especially true the earlier a technology is in the development cycle. Here, when a protocol describes a full charge or discharge the voltage that aligns with this condition is either $Vmax_{op}$ or $Vmin_{op}$. All tests should be conducted within the operating window between $Vmax_{op}$ and $Vmin_{op}$. When providing metadata associated with testing conditions and performance both $Vmax_{op}$ and $Vmin_{op}$ should be reported. Capacity should be reported based on the operational limits. As with the rated capacity, the operating capacity is defined during the initial characterizations tests and is fixed during aging. It is used to establish the device's depth-of-discharge (DOD) condition from a fully charged state at $Vmax_{op}$.

2.1.2. Temperature Control

Where possible it is strongly recommended that evaluation be performed under strict temperature control using environmental chambers or comparable methods. Typical temperature control should be to within ±3°C of the target test temperature. For the purposes of this manual, room temperature is within the range of 23-30°C. While a range in room temperature is given, the specific temperature at which characterization is performed should be reported as measured and all means possible, such as use of an environmental chamber, should be used to minimize temperature variation or drift even for room temperature tests.

When performing low or high temperature evaluations care needs to be used to ensure that the device is given proper time to reach an equilibrated temperature. This is typically done by changing the temperature of the environmental chamber and then allowing sufficient time (usually 2-16 hours) prior to starting a test. The exact length of time will vary based on the size of the device under test, the loading in the environmental chamber, and the rate of temperature increase or decrease.

2.1.3. Fixturing and Pressure Control

As necessary cells should be fixtured in a reproducible manner that minimizes variation between cells. For some cell designs reproducible fixturing includes the maintenance of targeted pressures. In these instances the fixturing pressure or pressure range should be noted in the metadata which complements the cell performance data.

2.1.4. Standard Charging Procedure

The standard charging procedure used for each cell should be established by the developer and used uniformly across the different tests described below. Exceptions are for the high-rate charge test. Any specifics for the charge procedure should be reported as part of the data. Examples of charge procedure would include the nature of the charge, voltage limits for specific charge steps and any limitations associated with specific steps such as a 1-hour limit on any constant voltage steps. Typical charging procedures for some chemistries including Li-ion cells would be a C/3 constant current charge (CC) followed by a fixed duration (time or current) constant voltage hold. Other procedures such as fixed voltage charge, ramped voltage charge or the use of thermal cut-offs should all be noted.

2.2. Rate Capability Across a Wide Temperature Range

This test measures the discharge rate capability of cells across a wide operating temperature range. The aim of the test is to characterize rate across a broad temperature range of interest. It is designed to show where discharge capacity falls to below 50% of the low-rate, room temperature performance for the cell. Such information is important to bound future operational windows and to provide information related to application specific thermal management needs. Since the aim is to identify the boundary conditions, the maximum rate investigated may vary for different technologies. The recommend temperature range of characterization is -20 to 60°C. It is recommended that characterization occur using at least 5 different temperatures. A typical sequence of temperatures is 25°C, 0°C, -20°C, 25°C, 40°C and 60°C. All charging should be near room temperature (23-30°C) using the technology specific charging procedure described in Section 2.1. Following charging, the temperature should be adjusted to the temperature of interest and the device should undergo a suitable soak period to allow thermal equilibration. Depending on the thermal mass of the cell and any associated fixture this typically ranges from 2 to 16 hours. Following thermal equilibration the cell is discharged at the desired rate and then brought back to the charging temperature. An appropriate thermal soak should occur prior to charging. The process is repeated across the full suite of discharge rates and temperatures.

At each temperature a minimum of five different rates should be investigated starting from low-rate discharge (e.g. C/10) and progressing to a high rate discharge that will vary based on the power capability of the cell. For most high energy cell designs maximum rates will be close to 1C while power designs may have rates in excess of 10C. The aim is that within the five rates investigated the test will identify where the discharge capability is no longer sufficient to provide 50% of the low rate, room temperature discharge capacity. Figure 1 shows an example of data reporting and performance across a broad temperature range.

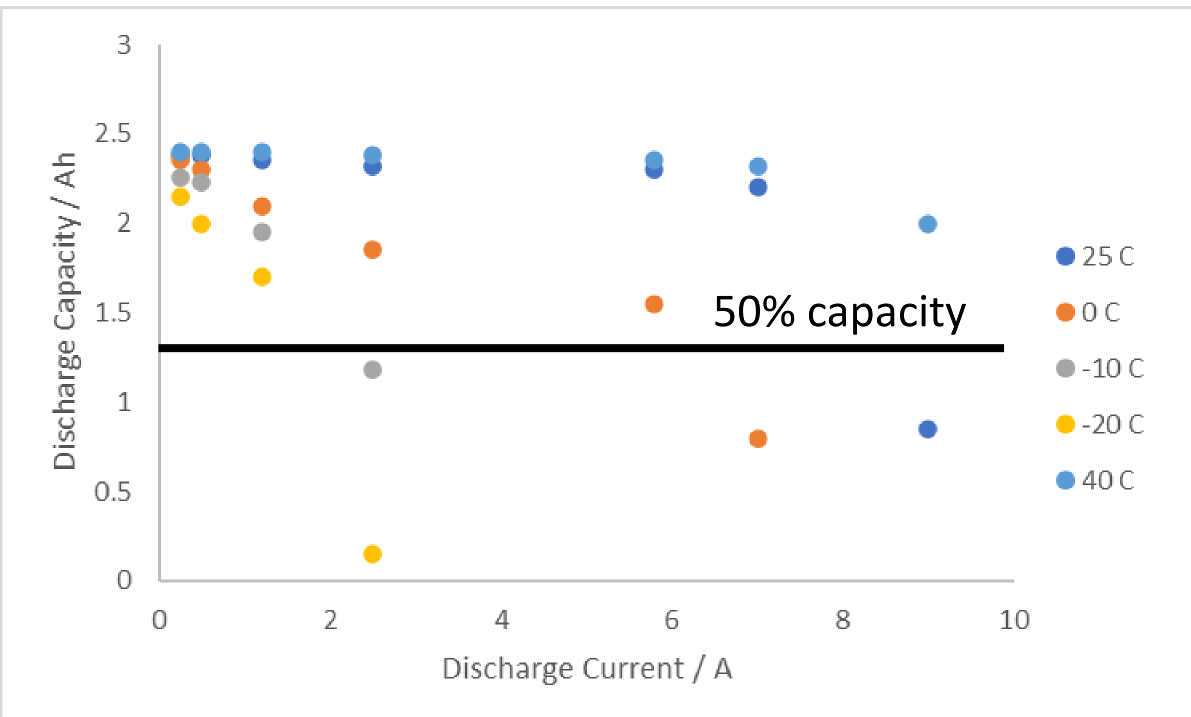

Figure 1: Discharge capacity as a function of discharge current at different temperatures. Data shown is an example for a 2.5 Ah cell. Note that for the present cell the 6C condition was not run at 25°C due to performance falling below the 50% capacity threshold. The rates shown for this example at 25°C nominally correspond to C/10, C/5, C/2, C/1, 2C, and 4C.

An example of the rate characterization process is shown below for a 0°C condition. Note that only the discharge occurs at the non-room temperature condition and that the beginning of each step requires a thermal soak. All charging should be to $Vmax_{op}$ and all discharging should be to $Vmin_{op}$.

1. Charge cell at 25°C using standard charge procedure
2. Discharge cell at 0°C at a C/10 rate
3. Charge cell at 25°C using standard charge procedure
4. Discharge cell at 0°C at a C/3 rate
5. Charge cell at 25°C using standard charge procedure
6. Discharge cell at 0°C at a C/1 rate
7. Charge cell at 25°C using standard charge procedure
8. Discharge cell at 0°C at a 2C rate
9. Charge cell at 25°C using standard charge procedure
10. Discharge cell at 0°C at a 4C rate
11. Charge cell at 25°C using standard charge procedure
12. Discharge cell at 0°C at a 6C rate
13. Charge cell at 25°C using standard charge procedure (optional)
14. Discharge cell at 0°C at a C/10 rate (optional)
15. Repeat steps 1-12 at a different test temperature.

Reported Data: Reported data includes the discharge capacity at each discharge step and at each temperature. Specific information related to temperature and rates should also be included and as available the discharge voltage as a function of capacity at each of the temperatures and rates should be included. If possible cell temperature rise during the discharge steps is also complimentary data.

Comparable Test Procedures: This test has close parallels with the static capacity variation of the Thermal Performance Test described in the USABC Electric Vehicle Test Manual Revision 3 [1].

2.3. High-Rate Charge Test

The High-Rate Charge test provides information on the charge acceptance capabilities of the test article based on a constant current charge from $Vmin_0$ over a 15-minute period. The test is intended to be performed at the beginning-of-life (BOL) though it may be repeated during cycle life aging or at the completion of other life tests. The developer should define and report the rate used for this test. The test sequence is as follows:

1. From a fully charged state at $Vmax_{op}$, discharge to $Vmin_{op}$ at an appropriate rate such as a C/3 rate.
2. Rest for 1 hour at open-circuit voltage conditions.

3. Charge at the specified high-rate constant current level for 15 minutes. If $Vmax_{op}$ is reached during the charge, taper the current to complete the 15 minute charge time.
4. If desired the developer can perform the High-Rate Charge test using multiple C-rates or other specific charge protocols.

Reported data: Data should include the voltage profile for both the charge and discharge as well as the total capacity attained during the fast charge. Where multiple rates are used for charging, the data for each rate should be included. It is advisable to also include a low-rate charge profile and capacity for reference. The temperature change of the cell during the fast charge is also valuable complimentary data.

Comparable Test Procedures: High-Rate Charge from the USABC Electric Vehicle Test Manual Revision 3 [1].

2.4. Room Temperature Cycle Life Test

The room temperature cycle life test aims to provide information on the performance of cells under a standard cycling protocol. Cells should be cycled through their full anticipated operational window during this test ($Vmin_{op}$ to $Vmax_{op}$). Ideally the discharge portion of the test should include some level of pulsing within each third of the state-of-charge (SOC) window. Though a constant current charge at a moderate rate such as C/3 discharge is also feasible. An alternative to performing pulsing during the cycle aging would be to perform periodic reference performance tests (RPTs) that incorporate methods to evaluate power performance across the full SOC window such as the hybrid pulse power characterization (HPPC) test. Testing should occur at room temperature as defined above and temperature of the testing should be reported.

General steps for the room temperature cycle life include:

1. Bring the cells to the appropriate temperature in a discharged state. Charge using the standard charge procedure to $Vmax_{op}$.
2. Once fully charged cells are typically rested between 0 and 1 hour. The total rest time and data acquisition during the rest is at the discretion of the developer and may depend on the cell chemistry and format
3. Discharge the cells at a defined rate (typically between a C/5 and 1C rate). If super-imposing a pulse these should be incorporated between 0-30% depth of discharge (DOD), 30-60% DOD and 60-100% DOD. Pulses should be at least 2x the standard rate used for the discharge and pulse length should be between 5-30 seconds. Discharge should be to the full $Vmin_{op}$.
4. Once discharged cells are typically rested between 0 and 1 hour. The total rest time and data acquisition during the rest is at the discretion of the developer and may depend on the cell chemistry and format.
5. Repeat steps 1-4 until the cells reach a defined point or display significant capacity fade.

Should the developer choose to use a RPT, these should be done on a fixed basis and performed at a prescribed temperature. The time duration between RPTs can be either based on time (*e.g.* 1 RPT per month) or cycle count (*e.g.* every 100 cycles).

Reported Data: Reported data from this test should include the cycle-by-cycle discharge capacity. The charge and discharge curves at distinct points across the aging should also be reported (from every 50-100 cycles). If RPTs are performed the RPT data should be reported.

Comparable Test Procedures: USABC DST, USABC CD profile [1].

2.5. Calendar Life Tests

This test evaluates cell degradation as a function of time at a specified temperature. It is not a pure shelf-life test as the devices are maintained at a high SOC during the test. The devices should be subjected to periodic RPTs.

While calendar life tests typically occur at a range of different temperatures and potentially different states-of-charge, this manual focus on three specific cases. High SOC at elevated temperature, high SOC at room temperature and an option for room temperature at a moderate SOC. Data from additional calendar life tests at other temperatures and SOCs may be reported at the discretion of the developer. Elevated SOC and temperature are commonly used to accelerate aging and thus shorten the time needed to obtain useful results. Cells to be tested may be included in a matrix of test variables such as temperature and SOC.

The outline of this test procedure for a particular cell is as follows:

1. The device is first fully charged at room temperature to $Vmax_{op}$ using the developer specific procedure from section 2.1.

2. If necessary, discharge to the target condition (*i.e.*, capacity removed or SOC) at 30ºC using the C/3 rate and rest for an hour. For the high SOC test the target condition is 80-100% SOC. For the moderate SOC test the target condition is 30-60% SOC.

3. Bring the cell to the target temperature at open-circuit condition and wait for the ambient temperature and voltage to stabilize (*i.e.*, 4 to 16 hours based on cell or pack mass).

4. Once at the desired SOC and temperature the cell should be held at that condition for the duration of the calendar life test. The majority of the test should be at an open circuit condition, but the SOC can be maintained at the target condition. If the SOC is being maintained it can be done by either having a periodic pulse profile (once per day) or a short duration voltage clamp (once per day) to ensure that significant self-discharge does not occur during the course of the test. When maintaining SOC the voltage prior to any charge or pulsing should be recorded and reported. Examples of the pulse per day can be found in the USABC Electric Vehicle Battery Manual Rev. 3 [1]. Capacities from pulses and charges should also be recorded and reported.

5. At specific intervals, return the cell to nominal temperature (*e.g.*, 30ºC), observe its open-circuit voltage after a 1-hr rest, and initiate a RPT. The RPT should include tests which capture both cell capacity (such as C/3 charge-discharge capacity) and power capability (measured using a HPPC, or a single pulsed discharge profile that aligns with test 2.4 or other method).

6. Repeat this test sequence until the cell reaches an end-of-test condition.

Calendar life tests should ideally be performed for the following conditions:

High State of Charge, Room Temperature (23-30°C, 80-100% SOC). This test is performed at or near room temperature (23-30°C) to understand the calendar life of cells. It should be performed at a high state of charge between 80 and 100%. If monthly loss is expected to be greater than 20% characterization should occur on a weekly basis.

High State of charge, elevated temperature (45-60°C, 80-100% SOC). The key difference between tests being that this test is performed at elevated temperature (between 45 and 60°C). As with the room temperature calendar test above, this test requires periodic performance evaluation using a RPT. Due to enhanced fade which may occur for some cell designs the elevated temperature RPTs should be performed at least monthly, but potentially on a weekly basis. If monthly loss is expected to be greater than 20% characterization should occur on a weekly basis.

Intermediate SOC, Room temperature - The difference between the above calendar life tests is that this test is performed at an intermediate SOC between 30 and 60%. As with the room temperature calendar test above, this test requires periodic performance evaluation using a RPT. Due to enhanced fade which may occur for some cell designs the elevated temperature RPTs should be performed at least monthly, but potentially on a weekly basis. If monthly loss is expected to be greater than 20% characterization should occur on a weekly basis.

Reported Data: Data should include the information from the RPTs including cell capacity at a specified charge-discharge rate and any information related to power capability. Where possible inclusion of the voltage profiles during the charge and discharge from the RPT should be included.

Comparable Tests: USABC Calendar Life Test at 30°C [1].

2.6. Reference Performance Tests

Reference performance tests are described in many of the previous tests. The purpose of RPTs is to capture specific points of data which can be tracked over the performance of a life test. Typically a RPT will contain components that provide information on both the cell energy and the power capability of the cell over aging. Examples of items included in RPTs would be constant current cycles at either a single or multiple rates (spanning C/20 to 1C) and HPPC tests.

3. References

[1] J. Christophersen, Battery Test Manual for Electric Vehicles, U. S. Advanced Battery Consortium, 2015, Rev. 3 INL/EXT-15-34184, https://doi.org/10.2172/1186745.