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Battery Test Manual for Baselining & Benchmarking Pre-commercial Cells

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The purpose of this manual is to document a series of battery performance testing procedures to standardize data collection and to promote data sharing and utilization across the U.S Navy and Marine Corps. Standardization of battery data collection is required to accurately assess and compare emerging battery technologies against one another. It is also critical to aid in the determination of technical readiness levels (TRLs) and manufacturing readiness levels (MRLs).

The experimental procedures contained within this test manual are largely adapted from those developed by Idaho National Laboratory under the direction of the Federal Consortium for Advanced Batteries (FCAB) for the purpose of sharing standardized data across government. The test procedures are for rechargeable (secondary) battery chemistries intended for the lithium-ion chemistry but can also be extended to other chemistries, such as sodium-ion batteries.

This manual can be utilized by Navy, Marine Corps, and other DoD battery researchers to guide battery data collection efforts to maximize the impact of their measurements. This manual provides a citable reference with unique DON document number to reference. It is a living document where revisions will be made periodically, based on user feedback and laboratory trials.

Table 1—Document Revisions

Date	Revision	Description	Author(s)
4 Aug 2022	1	Initial document	Corey T Love Dillon Hayman

BATTERY TEST MANUAL FOR BASELINING & BENCHMARKING PRE-COMMERCIAL CELLS

1 INTRODUCTION

Due to the wide array of battery form factors, chemistries and real-world applications, a uniform cell performance test methodology is needed to assess the unique capabilities of each cell-type and fit those capabilities to a suitable real-world application. Additionally, new prototype cells need to be effectively and fairly benchmarked against current commercial cell-types which necessitates standard practices during cell testing. This manual is intended to provide a battery testing methodology which can clearly and unbiasedly identify cell strengths and weaknesses across various cell types and chemistries. This manual should be employed to test rechargeable battery chemistries (Li-ion, Na-ion, etc.). Unless otherwise denoted, each test is to be run with 5 cell replicates to improve statistical reliability and build in redundancy to minimize gaps in data collection due to human error, instrument malfunction, etc.

2 INCOMING INSPECTION

The incoming inspection procedure is used to ensure only undamaged, pristine cells are utilized for performance testing. Measurements such as the top and bottom diameter, height, weight, DC resistance and Open-Circuit Voltage (OCV) should be recorded during the inspection. Cells which fall outside the tolerance ranges given in the manufacturer's specification sheet should be screened out and eliminated from further testing. A sample manufacturer specification sheet is provided in the Appendix for reference. Each cell should be visually inspected for signs of corrosion, physical damage, or pressure build-up and discarded if not in pristine condition. In addition to measuring initial physical and electrochemical properties, information from the manufacturer's specification sheet should be gathered to use in the test scripts. Table 2 shows the incoming inspection worksheet which lists data required to calculate currents required for cell charging and discharging. If a batch of cells has a quantity greater than 50 cells, a random selection of cells should be used to complete incoming inspection.

Manuscript approved October 3, 2022.

Table 2 — Incoming Inspection Worksheet for Cylindrical Cell Type

Physical and Electrochemical Properties	
Parameter	**unit**
top diameter	mm
bottom diameter	mm
height	mm
mass	g
DC resistance	mΩ
Open-Circuit Voltage (OCV)	V
Vendor Specification Sheet Information	
Parameter	**unit**
Nominal Capacity	Ah
Maximum Charge Voltage	V
Minimum Discharge Voltage	V
Constant-Voltage Current Cutoff	mA or h
Maximum Discharge Rate	A
Maximum Operating Temperature	°C
Minimum Operating Temperature	°C

3 REFERENCE PERFORMANCE TESTS

Reference performance tests (RPTs) are used to evaluate cell performance parameters at the beginning, end, and periodically throughout testing. RPTs are split into two categories: *Baseline Reference Performance Testing* and *Diagnostic Performance Testing*. Baseline tests are used to screen cells initially and provide a benchmark for comparison against future RPTs. Diagnostic RPTs are performed periodically throughout and at the end of testing. All RPTs are conducted in a temperature-controlled environment at 25°C, ±3°C. Both types of RPT utilize standard cycling, slow C/20 cycling, and hybrid pulse power characterization to assess cell performance parameters.

3.1 Standard Cycle

The standard cycling procedure will uses voltage limits (V_{min} and V_{max}) given by the cell manufacturer's specification sheet (Table 2). Prior to beginning cycling, cells should be thermally equilibrated to 25°C, ±3°C for a minimum of 3 hours. Cells are charged using a C/3, CC (Constant-Current), followed by a CV (Constant-Voltage) hold with a cutoff determined by the manufacturer's specifications sheet. Following a 1-hour rest, cells are discharged using a symmetric C/3, CC. A default 1 hour rest should be performed in between all charges and discharges.

3.2 C/20 Cycle

During each RPT, a fourth cycle is performed at a rate of C/20 for both the charge and discharge. Utilizing a slower rate enables collection of higher fidelity reaction information. Specifically, data from the C/20 cycle can be used to perform differential capacity analysis (dQ/dV) where each dQ/dV peak represents an electrochemical reaction. The location of peaks in the dQ/dV vs. voltage data from each RPT can be plotted and used to track reaction changes throughout testing.

3.3 Hybrid Pulse Power Characterization

Hybrid Pulse Power Characterization (HPPC) utilizes pulses of current to measure the charge and discharge resistances in the cell. HPPC is performed every 10% SOC (State-Of-Charge) ranging from 100% to 10%. The end condition used to determine when the SOC has decreased 10% is defined as when $1/10^{th}$ of the cell nominal capacity has been discharged. Starting from a discharged state, cells should be charged to V_{max} using a C/3, CC with a manufacturer recommended CV hold. After a 1-hour rest, cells are discharged with a 1C, CC pulse for 30 seconds. After a 40 second rest, cells are charged with a .75C, CC pulse for 10 seconds. Finally, a C/3, CC is used to discharge the cell to the next SOC. The HPPC procedure is repeated for up to 10 SOCs or until V_{min} is reached.

3.4 Baseline RPT

The baseline RPT defines a cell's baseline electrochemical behavior and is used for comparison against future RPTs after aging or performance testing. Additionally, if a cell's baseline cycling behavior is not within the tolerance ranges described in this manual and in the manufacturer's specifications sheet, then those cells are screened out of the study.

To access initial electrochemical behavior and screen out anomalies, up to 10 cycles are performed until cell discharge capacity is stable within ±1% for 3 consecutive cycles. Stability is defined as when the discharge capacity between cycles 1 and 2, 2 and 3, and 1 and 3 for any 3 cycles each measure within ±1%. If stability cannot be achieved by 10 cycles, the cell is screened out of the study. Additionally, the final averaged discharge capacity of the last 3 cycles should be within ±3.5% of the discharge capacity reported in the manufacturer's specifications sheet. The baseline RPT consists of standard cycling (up to 10 cycles), a slow C/20 cycle and hybrid pulse power characterization (1 cycle) as shown below. Once cells complete and pass the criteria for the baseline RPT they can be utilized in further tests (rate capability, room temperature cycle life, and calendar life).

Baseline RPT Procedure

1. 3-hour rest at 25°C 2. C/3, CC-CV charge to V_{max} 3. 1-hour rest 4. C/3, CC discharge to V_{min} 5. 1-hour rest	*Standard Cycle*
6. Repeat steps 2-5 until capacity is stable within ±1% for 3 cycles (up to 10 cycles)	
7. C/20, CC-CV charge to V_{max} 8. 1-hour rest 9. C/20, CC discharge to V_{min} 10. 1-hour rest	*C/20 Cycle*
11. Standard CC-CV charge to V_{max} 12. 1-hour rest 13. 1C, CC discharge for 30 seconds 14. 40-second rest 15. .75C, CC charge for 10 seconds 16. Standard C/3, CC discharge for $1/10^{th}$ of the cell nominal capacity 17. Repeat steps 8-12 until V_{min} is reached	*HPPC*

3.5 Diagnostic RPT

Diagnostic RPTs are performed periodically throughout and at the end of cell testing to compare changes in performance parameters to the baseline. Diagnostic RPTs use the standard cycling procedure for 3 cycles, a 4th cycle at a rate of C/20, and the HPPC procedure. Data is recorded for all steps every 2 mV of voltage change and/or every 30 seconds. Diagnostic RPT procedures are listed below and shown visually in Fig. 1.

Diagnostic RPT Procedure

1. 3-hour rest at 25°C
2. Standard CC-CV charge to V_{max}
3. 1-hour rest
4. C/3, CC discharge to V_{min}
5. 1-hour rest
6. Repeat steps 2-5 for 3 cycles
7. C/20, CC-CV charge to V_{max}
8. 1 hour rest
9. C/20, CC discharge to V_{min}
10. 1-hour rest
11. Standard CC-CV charge to V_{max}
12. 1-hour rest
13. 1C, CC discharge for 30 seconds
14. 40-second rest
15. 0.75C, CC charge for 10 seconds
16. Standard C/3, CC discharge for 1/10th of the cell nominal capacity
17. Repeat steps 8-12 until V_{min} is reached

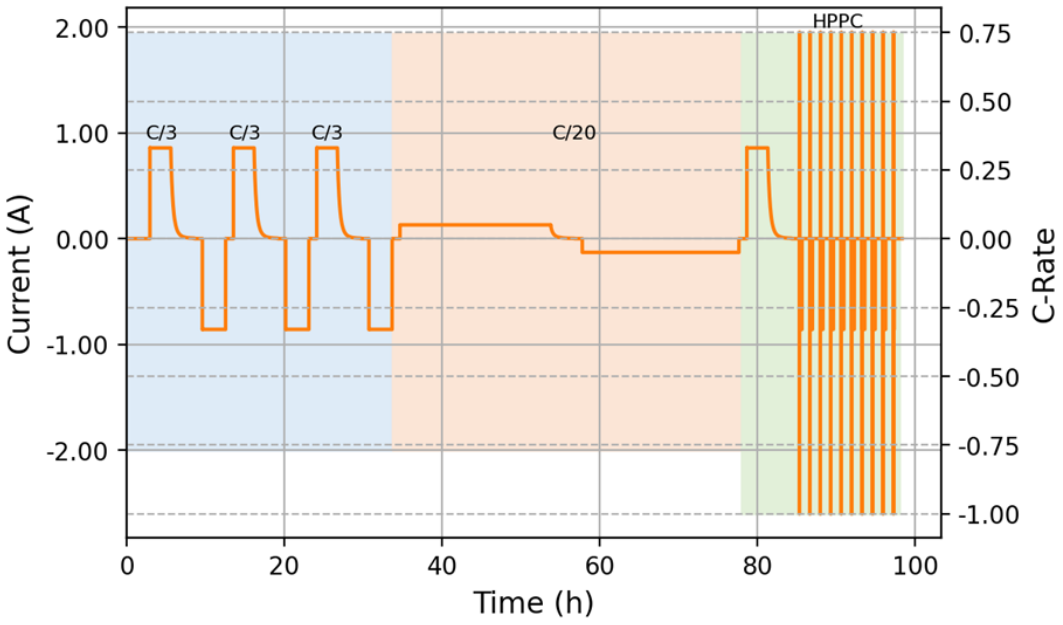

Fig. 1 —Schematic illustrating test protocol for a typical Diagnostic Reference Performance Test (RPT).

4 RATE CAPABILITY TEST

The rate capability test measures cell performance at various discharge rates and temperatures. RPTs should be performed before the start and at the end of the rate capability test. Before each discharge, cells will be charged to V_{max} using the standard charging procedure with the temperature maintained at 25℃, ±3℃. Cells should be given at least 3 hours to equilibrate at each temperature prior to testing. Cells or battery packs with large thermal mass may require more time to be thermally soaked. Once a cell is fully charged, it is equilibrated at the next target temperature and discharged at the next discharge rate. Cells are tested at 25℃, 0℃, minimum temperature (or -20℃), 10℃, and the max temperature (or 40℃). At each temperature, cells are discharged at C/10, C/5, C/2, 1C, and the max discharge rate as determined via the incoming inspection worksheet (Table 2). Data is recorded for all steps every 2 mV of voltage change and/or every 30 seconds. Rate Capability Test procedures are listed below and shown visually in Fig. 2.

Rate Capability Procedure

1. Perform Baseline RPT test
2. 3-hour rest at 25℃
3. Standard charge to V_{max}
4. 3-hour rest at next target temperature
5. Discharge cell to V_{min} at next target rate
6. Repeat steps 2-5 until all temperatures and rates are completed
7. Perform Diagnostic RPT test

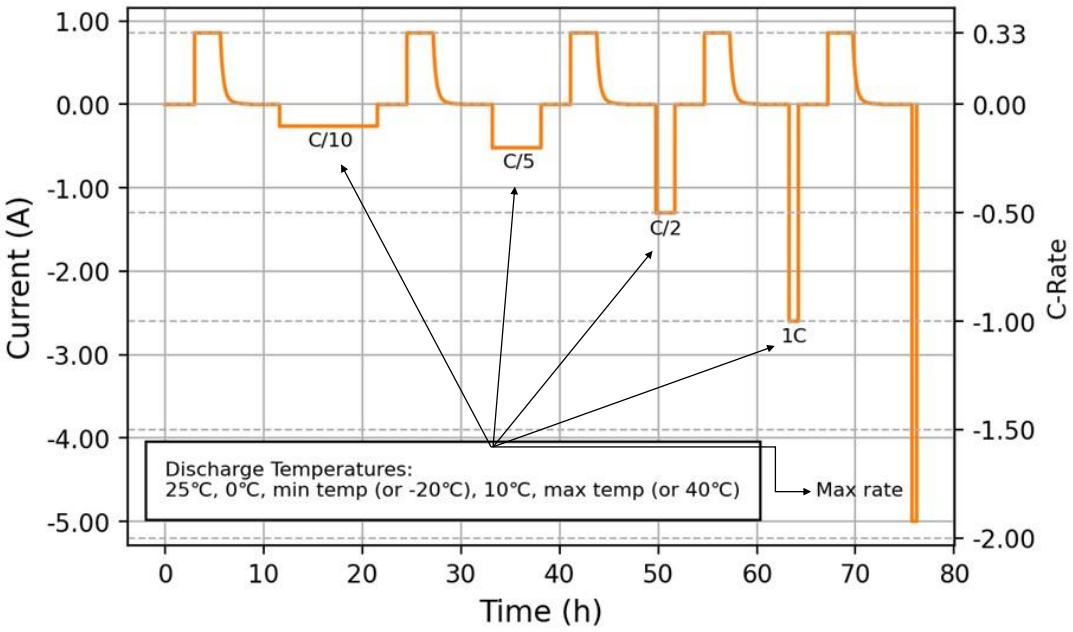

Fig. 2 — Schematic illustrating test protocol for Rate Capability Test for a single temperature (25°C).

5 ROOM TEMPERATURE CYCLE LIFE TEST

The room temperature cycle life test assesses cell capacity fade over its useful life. The EOL (End-Of-Life) condition is reached when cell capacity diminishes to 70% of the rated nominal capacity. Cells should be given a minimum of 3 hours to equilibrate and be maintained at 25℃, ±3℃ throughout the test. The cycling procedure is identical to the standard cycling procedure using a standard charge and discharge with default rests in between, as shown below. Data is recorded for all steps every 2 mV of voltage change and/or every 30 seconds. Room Temperature Cycle Life procedures are listed below and shown visually in Fig. 3.

Room Temperature Cycle Life Procedure:

1. Perform Baseline RPT Test
2. 3-hour rest at 25℃
3. Standard CC-CV charge to V_{max}
4. 1-hour rest
5. C/3, CC discharge to V_{min}
6. 1-hour rest
7. Repeat steps 3-6 for 100 cycles
8. Perform Diagnostic RPT test
9. Repeat steps 2-8 until cell reaches 70% RUL

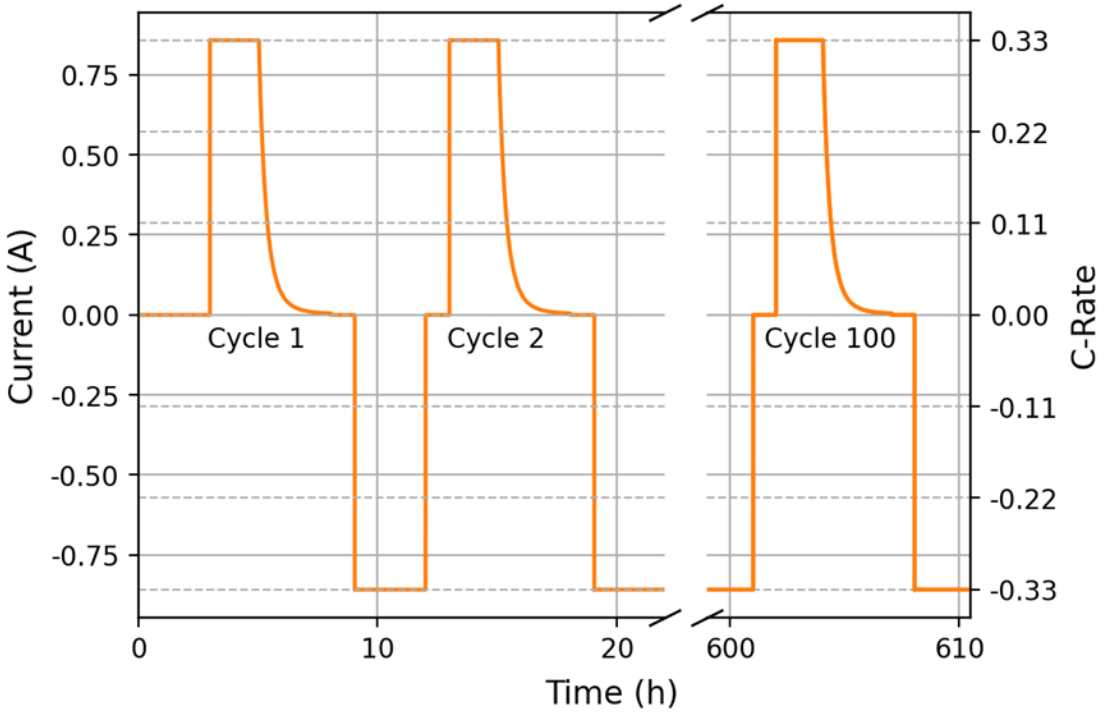

Fig. 3 — Schematic illustrating testing protocol for Room Temperature Cycle Life Test.

6 CALENDAR LIFE TEST

The calendar life test measures cell deterioration over time at a fixed SOC and temperature. A simplified calendar life test is provided herein. More sophisticated testing procedures are found outlined in the Federal Consortium for Advanced Batteries (FCAB) test manual listed in the Reference section of this report. The baseline RPT is performed first. Cells are then equilibrated to 25°C for 3 hours followed by a standard CC-CV charge to 100% SOC. After the completion of the standard charge, the cells can be removed from their battery test fixtures and stored in a 25°C environmental chamber. A diagnostic RPT is performed notionally every 30 days or 1-month to assess calendar aging. Data is recorded every 2 mV or every 30 seconds. Calendar Life Test procedures are listed below and shown visually in Fig. 4.

Calendar Life Test Procedure:

1. Perform Baseline RPT
2. 3-hour rest at 25°C
3. Standard CC-CV charge to V_{max}
4. Store cell at 25°C for approximately 30 days or 1-month
5. 3-hour rest at 25°C
6. Perform Standard CC Discharge
7. 1-hour rest
8. Perform the Diagnostic RPT procedure
9. Repeat steps 2-7 for the duration of the study

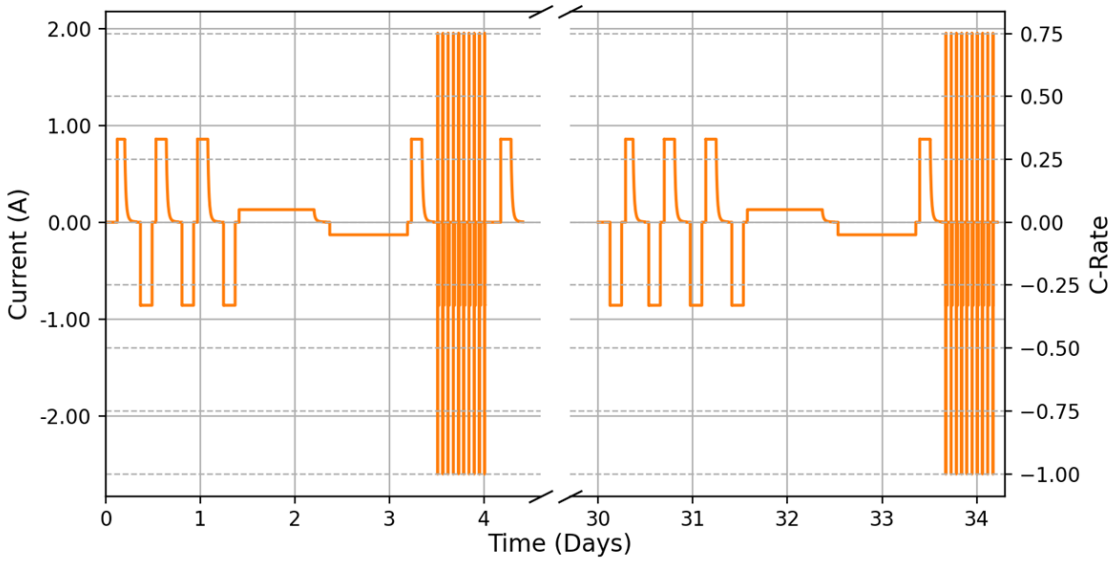

Fig. 4 —Schematic illustrating test protocol for Room Temperature Calendar Life Test.

REFERENCES

1. *Federal Consortium For Advanced Batteries Battery Test Manual, Revision 1*, INL/MIS-21-62073, August 2021

2. *USABC Electric Vehicle Battery Test Procedures Manual, Revision 3.1*, INL/EXT-15-34184, October 2020

3. *J. Christophersen, Battery Test Manual For Electric Vehicles, Revision 3*, INL/EXT-15-34184, June 2015

APPENDIX A – EXAMPLE DATA

1 Example Manufacturer Specification Sheet with Pertinent Data Highlighted in YELLOW.

-SAMSUNG SDI Confidential Proprietary –

Spec. No.	ICR18650-26F	Version No.	0.0

1. Scope

This product specification has been prepared to specify the rechargeable lithium-ion cell ('cell') to be supplied to the customer by Samsung SDI Co., Ltd.

2. Description and Model

2.1 Description Cell (lithium-ion rechargeable cell)

2.2 Model ICR18650-26F

3. Nominal Specifications

Item	Specification
3.1 Nominal Capacity	2600mAh (0.2C, 2.75V discharge)
3.2 Minimum Capacity	2550mAh(0.2C, 2.75V discharge)
3.3 Charging Voltage	4.2 ±0.05 V
3.4 Nominal Voltage	3.7V
3.5 Charging Method	CC-CV (constant voltage with limited current)
3.6 Charging Current	Standard charge: 1300mA Rapid charge : 2600mA
3.7 Charging Time	Standard charge : 3hours Rapid charge : 2.5hours
3.8 Max. Charge Current	2600mA(ambient temperature 25℃)
3.9 Max. Discharge Current	5200mA(ambient temperature 25℃)
3.10 Discharge Cut-off Voltage	2.75V
3.11 Cell Weight	47.0g max
3.12 Cell Dimension	Height : 65.00mm max Diameter : 18.40mm max
3.13 Operating Temperature	Charge : 0 to 45℃ Discharge: -20 to 60℃
3.14 Storage Temperature	1 year : -20~25℃(1*) 3 months : -20~45℃(1*) 1 month : -20~60℃(1*)

Note (1): If the cell is kept as ex-factory status (50% of charge), the capacity recovery rate is more than 80%.

7.1 Standard Charge

This "Standard Charge" means charging the cell with charge current 1300mA and constant voltage 4.2V at 25℃ for 3hours.

Fig. A1 — Example Manufacturer Specification Sheet

2 Sample Completed Inspection Worksheet

Physical and Electrochemical Properties	
Parameter	**Unit**
top diameter	18.24 mm
bottom diameter	18.22 mm
height	64.78 mm
mass	45.0 g
DC resistance, mΩ	22.47 mΩ
Open-Circuit Voltage (OCV)	3.714 V
Vendor Specification Sheet Information	
Parameter	**Unit**
Nominal Capacity	2.6 Ah
Maximum Charge Voltage	4.2 V
Minimum Discharge Voltage	2.75 V
Constant-Voltage Current Cutoff	3 h
Maximum Discharge Rate	5.2 A
Maximum Operating Temperature	60 °C
Minimum Operating Temperature	-20 °C

Fig. A2 — Completed Incoming Inspection Sheet

3 Reference Performance Test Data

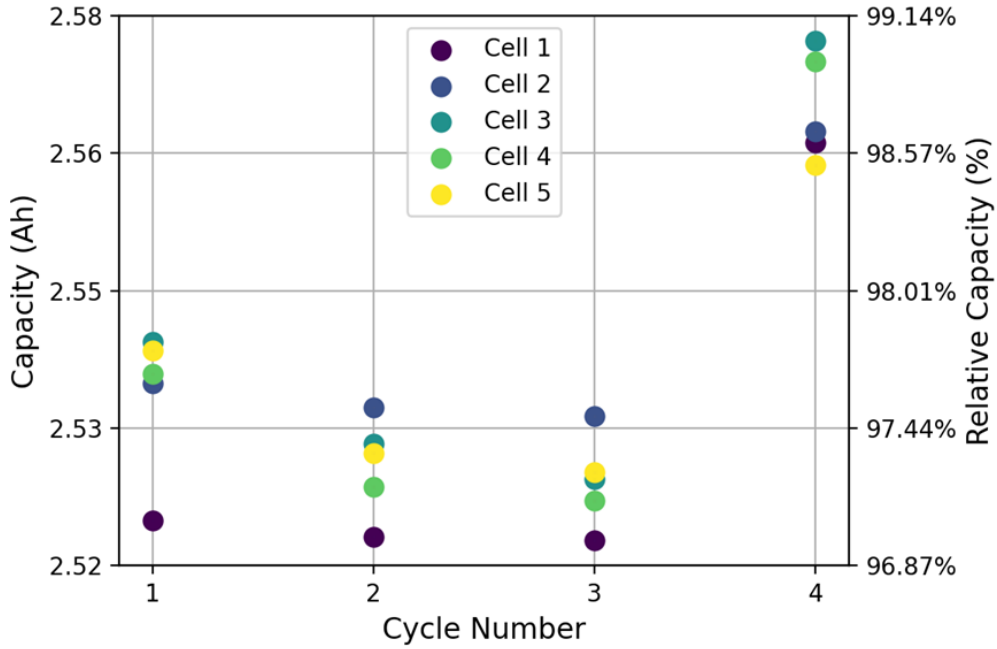

Fig. A3 — Capacity vs cycle during standard and slow cycling.

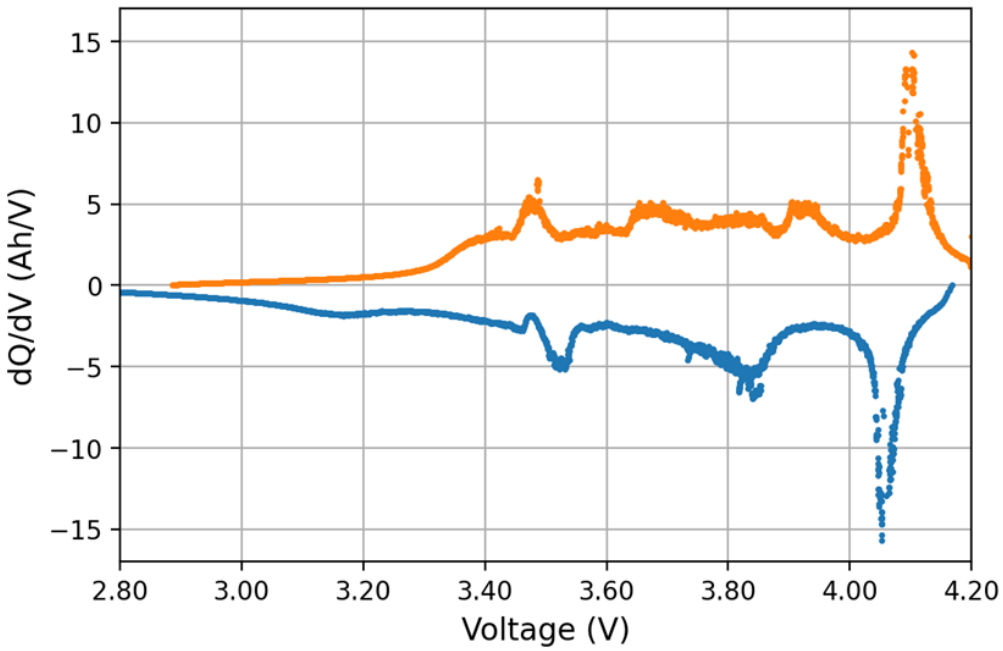

Fig. A4 — dQ/dV vs Voltage during slow charging and discharging.

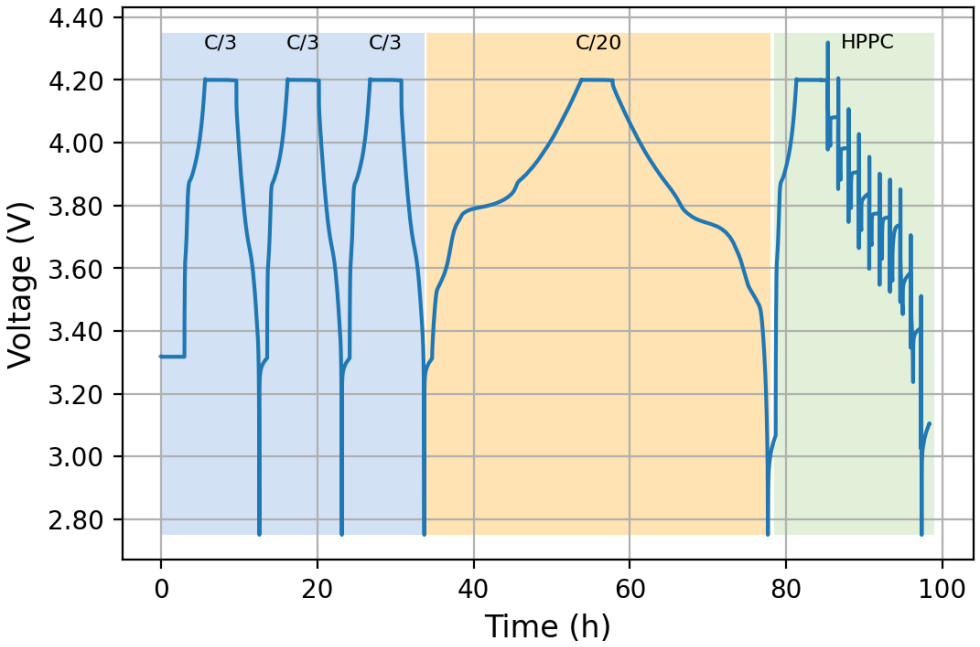

Fig. A5 — Voltage profile during a Reference Performance Test.

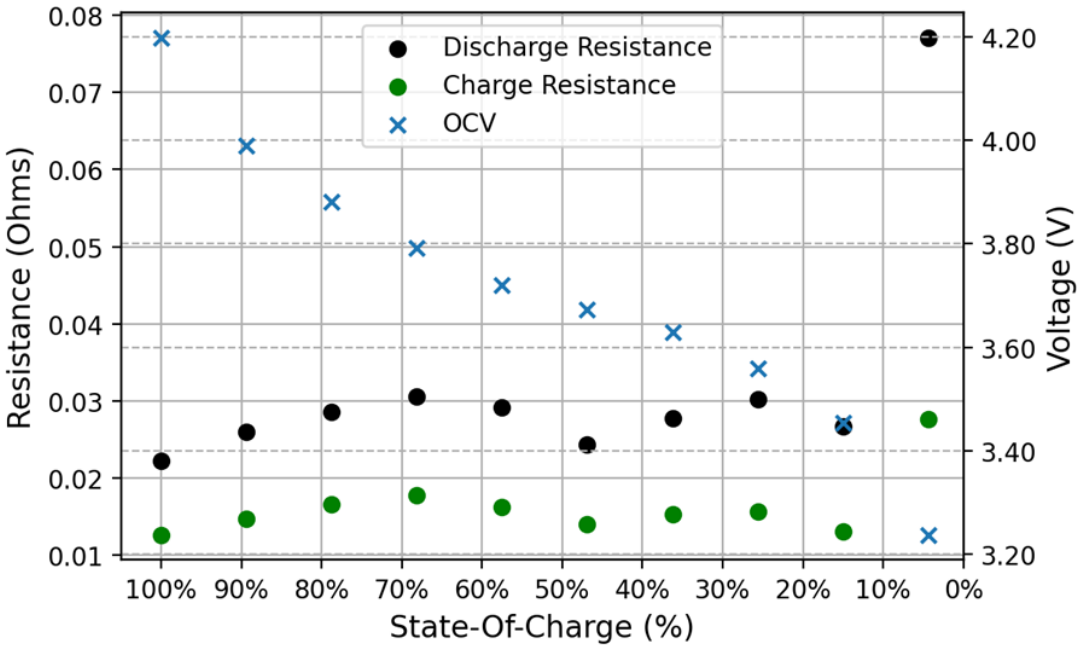

Fig. A6 — HPPC discharge and charge resistances at each SOC and OCV

4 Rate Capability Test Data

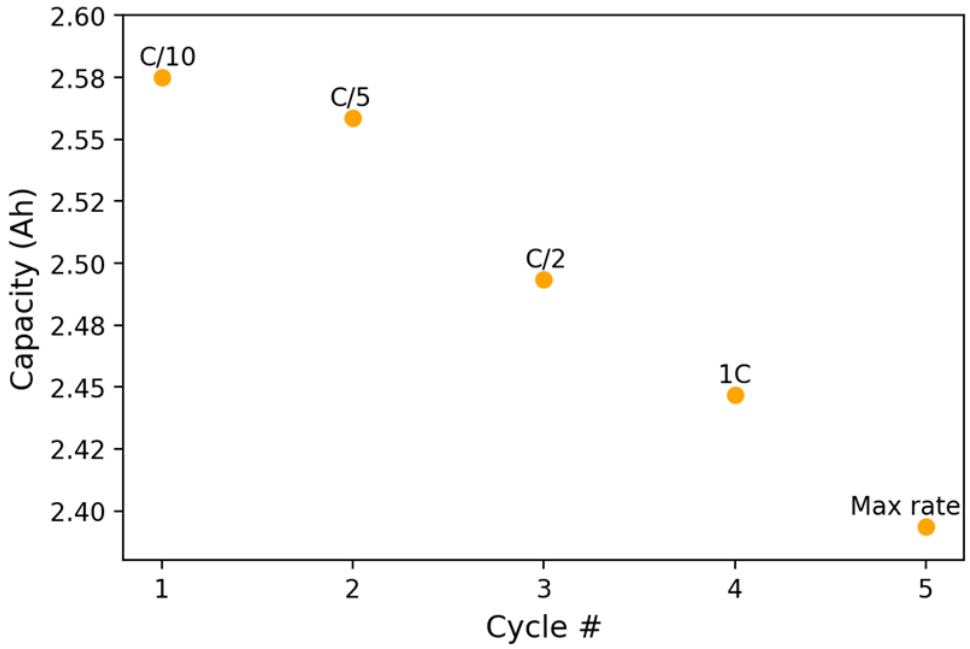

Fig. A7 — Capacity data from the rate capability test at a single temperature (25°C).

5 Room Temperature Cycle Life Test Data

Data to be included in next revision.

6 Calendar Life Test Data

Data to be included in next revision.