

UL 9540A Test Method for Evaluating Thermal Runaway Fire Propagation in Cell Energy Storage Systems, Third Edition

Cell Level Test Report Model V6.0 "Prussian Blue Cell"

Prepared by UL LLC for Natron Energy, Inc. Issued: December 23, 2019 Revised: July 8, 2020

Project Number: 4789109222

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Revision notes:

January 14, 2020

- Page 17, Figure 16 was incorrectly labeled;
- Page 24, The hydrogen, carbon monoxide, carbon dioxide, and THC quantification represents 100% of the gas mixture, not just hydrogen & carbon dioxide.

July 8, 2020 (ULProject# 4789542457)

- The V6.0 cell capacity rating has been increased from 4.3Ah to 4.6Ah. The increased capacity is a result of the end point cutoff voltage discharge specification being reduced from 1.0V to 0V. This change in end point voltage does not affect the test results as the cell was tested at 100% SOC and the charge voltage rating has not changed. No additional testing considered necessary to update the report to reflect the new capacity rating. The test results portion of this report will not be modified to reflect the new capacity because at the time of the test, this was the recorded value. This revision is to be consistent
- with the UL1973 certification of the V6.0 cell under UL file number MH63828.
- Page 4, updated nominal capacity from 4.3Ah to 4.6Ah
- Page 5, updated nominal capacity from 4.3Ah to 4.6Ah

Introduction

UL defines thermal runaway as follows:

THERMAL RUNAWAY – The incident when an electrochemical cell increases its temperature through self-heating in an uncontrollable fashion. The thermal runaway progresses when the cell's generation of heat is at a higher rate than the heat it can dissipate. This may lead to fire, explosion and gassing.

Scope

The test methodology in this document evaluates the fire characteristics of a cell energy storage system that may undergo thermal runaway.

The data generated will be used to determine the fire and explosion protection required for an installation of a cell energy storage system intended for installation, operation and maintenance in accordance with the International Fire Code (IFC), the Fire Code, NFPA 1, the National Electrical Code, NFPA 70, the National Electrical Safety Code (NESC), IEEE C2, NFPA 855, and other energy storage system codes, and the manufacturer's installation instructions.

Summary of UL 9540A Test Results



*Cell surface temperature measured during test #6.

For each of the 4 test methods implemented to induce thermal runaway, no thermal runaway was observed with the Natron Energy, Inc model V6.0 cell under test.

UL 9540A Test Report for Natron Energy, Inc.

Cell Energy Storage Description

Cell Energy Storage System Configuration

Table 1 – Product details

| Cell | | |
|--------------------------|--|--|
| Manufacturer | Natron Energy, Inc | |
| Model Number | V6.0 | |
| Chemistry | Sodium Ion | |
| Electrical Ratings | 1.56V 4.6Ah | |
| Dimensions | 194 mm x 246 mm x 5.1 mm | |
| Cell Weight | 305g | |
| Construction Description | Pouch | |
| UL Certifications | ANSI/CAN/UL 1973, BBGA2/8 File MH63828 | |
| | | |
| Figure 1 – Photo of cell | | |

Cell Level Test Description

Sample Preparation

Six batteries were prepared according to the requirements specified in UL 9540A Section 6.1.

Cell Conditioning and Stabilization (Section 8.1.4)

The batteries were subjected to a cycle of pre-charge, discharge, charge, discharge, and then final charge. The batteries were charged to 100% state of charge (SOC) per the manufacturer's instructions, summarized in Table 2. Charging profiles are included in Figures 2.

| Charging | | Disch | arging |
|----------------|---------------|----------------|--------|
| Current (CC) | 80 A | Current (CC) | 200 A |
| Voltage (CV) | 1.81 V | Cutoff Voltage | 1 V |
| Cutoff Current | Not Specified | | |
| Charging Time | 6 minutes | | |

The ambient temperature of the space during cell conditioning was maintained within 25 ± 5 °C (77 ± 9 °F) and $50\pm25\%$ RH.

Final charge capacity for each cell is presented in Table 3. Individual charge/discharge profiles are included in the Figures 2 & 3 below.

| UL Cell Number | Natron Cell Number | Test Number | Nominal Final Charge (Ah) |
|----------------|-----------------------|-------------|------------------------------|
| 1 | C14514 | 1 | 4.3 |
| 2 | C14511 | 2 | 4.3 |
| 3 | C13935 | 3 | 4.3 |
| 4 | C14375 | 4 | 4.3 |
| 5 | C14512 | 5 | 4.3 |
| 6 | C13914 | 6 | 4.3 |

Table 3 – Final cell charge capacities

The batteries were charged per manufacturer specifications. At a room ambient of 25°C, charging voltage is 1.81V, at 80A. When the 6 minute charge time was reached, charging was stopped.







Test Instrumentation

Each initiating cell was instrumented with 5 Type K thermocouples according to the layout illustrated in Figures 4-5.

| TC # | Location |
|------|------------------------|
| TC 1 | Top Middle of Cell |
| TC 2 | Bottom Middle of Cell |
| TC 3 | Side of Cell |
| TC 4 | Positive Terminal |
| TC 5 | Expected Vent Location |

| Table 4 – [•] | TC Locations |
|------------------------|--------------|
|------------------------|--------------|



Figure 4 – Initiating thermocouple locations



Figure 5 – Additional view of thermocouple locations

Thermal Runaway Methodology

The propensity for a cell to experience thermal runaway was examined through several different methodologies covering mechanical, thermal, and electrically induced events. The methods and parameters below were intended to place the most stress on the cell to attempt inducing thermal runaway. Thermal runaway is defined as the incident when an electrochemical cell increases its temperature through self-heating in an uncontrollable fashion. The thermal runaway progresses when the cell's generation of heat is at a higher rate than the heat it can dissipate. This may lead to fire, explosion and gassing.

A summary of the evaluated thermal runaway methodologies is included in Table 5.

| Test | Stress | Methodology | Test Parameters |
|------|------------|------------------|--|
| 1 | Electrical | Short Circuit | Direct short across positive and negative terminals |
| | | | |
| | | | |
| 2 | Thermal | External Heating | 4-7°C/min with no holding temperature |
| 3 | Electrical | Overcharge | Constant current, voltage increased 1V/min |
| 4 | Mechanical | Nail Penetration | Through external casing from short side through internal |
| | | | cell |
| 5 | Electrical | Overcharge | Constant current, voltage increased 1V/min |
| 6 | Electrical | Overcharge | Gas Composition, same methodology as test 5 |

Table 5 – Thermal Runaway Methodologies

Cell Level Test Results

Test 1 - Demonstration of Thermal Runaway Propensity by Short Circuit

| Test Initiation Details | | |
|---------------------------|------------|--|
| Test Date | 2019/09/18 | |
| Test Start Time | 08:41 AM | |
| Initial Lab Temperature | 20.5°C | |
| Initial Relative Humidity | 46% RH | |

Table 6 – Test initiation details

Figure 6 shows the surface temperatures measured during the test, in which no venting nor thermal runaway was observed. The open circuit voltage prior to the start of the test was 1.77V. A contactor was closed to complete the circuit, at the initiation of the test. The current measured at the initial short circuit was approximately 796A. The current across the short was drastically decreasing through the test. No venting or thermal runway was observed based on visual observations and temperature measurements of the cell enclosure. All temperatures were reducing after the initial short circuit event. At the end of testing, when current was 0 and temperatures were returning to near ambient, at approximately 27 minutes, the contactor was opened.



Surface temperatures measured on cell



Figure 7 – Photograph of cell after test



Figure 8 – Photograph of cell after test

Test 2 - Demonstration of Thermal Runaway Propensity by External Heating

| Test Initiation Details | | |
|---------------------------|------------|--|
| Test Date | 2019/09/18 | |
| Test Start Time | 1:29 PM | |
| Initial Lab Temperature | 20.5°C | |
| Initial Relative Humidity | 46% RH | |

Table 7 – Test initiation details

Figure 9 shows the surface temperatures measured during the test, in which cell venting was observed through the seams of the cell pack but thermal runaway was not observed. The open circuit voltage of the cell prior to the test was 1.78V. Heaters were place on both faces of the cell. Kapton tape was used to secure the heaters to the cell. TC5, just outside of the heater on the case, was heated at a rate of 4-7°C per minute until the heater reached approx. 400C. At this time the heater failed causing the heater itself to catch on fire. The heater power supply was shut off and the cell temperatures were further monitored. There was no observance of fire from the cell internally.



Figure 9 – Surface temperatures and electrical parameters measured on cell

Smoking was observed approx. 50 minutes into the test when the TC1 was approx. 294°C. Since there was no uncontrolled increase in temperature, the heaters proceeded to increase at a rate of 4-7°C per minute.



Figures 10-12 - Photographs of the cell after the external heating test.



Test 3A - Demonstration of Thermal Runaway Propensity by Overcharge #1

| Test Initiation Details | | |
|---------------------------|------------|--|
| Test Date | 2019/09/19 | |
| Test Start Time | 08:18 AM | |
| Initial Lab Temperature | 21.0°C | |
| Initial Relative Humidity | 47% RH | |

Table 8 – Test initiation details

Figure 13 shows the surface temperatures measured during the test, in which cell venting was observed through the seams of the cell pack but thermal runaway was not observed. The open circuit voltage of the cell prior to the test was 1.73V. The power supply used to charge the cell was placed in a constant voltage mode, where the voltage was increased by 1V every minute. During the test, the cell enclosure breached and a flame was observed in the cell when the supply was at 26V. This occurred at approx. 26 minutes into the test. The power supply was left on for an additional 10 minutes after the initial breach was observed. When the power supply was turned off at approx. minute 35, the flames self extinguished. Due to this, a second heating test was performed under Test #5 shown later in the report where the power supply was immediately turned off upon observation of the cell enclosure breaching.



Figure 13 – Surface temperatures measured on cell



Figure 14 – Photograph of cell top after test



Figure 15 – Photograph of cell bottom after test

Test 4 - Demonstration of Thermal Runaway Propensity by Nail Penetration

| Test Initiation Details | | |
|---------------------------|------------|--|
| Test Date | 2019/09/19 | |
| Test Start Time | 01:01 PM | |
| Initial Lab Temperature | 21.0°C | |
| Initial Relative Humidity | 47% RH | |

Table 9 – Test initiation details

Figures 16 show the surface temperatures measured during the test, in which no cell venting nor thermal runaway was observed. The open circuit voltage prior to the start of the test was 1.77V. An 8 mm diameter nail, 125 mm long, was used to penetrate through the side of the cell in order to puncture through the entire cell. The nail increased in temperature slightly due to the friction of the nail as it passed through the cell. There was no change in status after nail penetration. The test was concluded after 38 minutes.



Figure 16 – Surface temperatures measured on cell



Figure 17 – Nail Penetration Setup



Figure 18 – Nail Entry and Exit © 2019 UL LLC. All Rights Reserved.

Test 5 - Demonstration of Thermal Runaway Propensity by Overcharge #2

| Test Initiation Details | | |
|---------------------------|------------|--|
| Test Date | 2019/09/19 | |
| Test Start Time | 02:03 PM | |
| Initial Lab Temperature | 22.5°C | |
| Initial Relative Humidity | 47% RH | |

| Table 10 – Test | initiation | details |
|-----------------|------------|---------|
|-----------------|------------|---------|

Figure 19 shows the surface temperatures measured during the test, in which cell venting was observed through the seams of the cell pack but thermal runaway was not observed. The open circuit voltage of the cell prior to the test was 1.76V. The power supply used to charge the cell was placed in a constant voltage mode, where the voltage was increased by 1V every minute. During the test, the cell enclosure breached and a flame was observed in the cell when the supply was at 32V. This occurred at approx. 35 minutes into the test. The power supply was immediately shut off after the initial breach was observed with flaming. When the power supply was turned off, the flames self extinguished.



Figure 20 – Photograph of cell top after test

Figure 21 – Photograph of cell bottom after test

Test 6 - Gas Composition

The gas composition test was conducted with the cell inserted into the cell gas composition test chamber and the chamber was sealed. The cell gas composition test chamber is an 82 L pressure vessel and is shown in Figures 22. The method used for inducing venting was overcharging. From all 5 tests, none resulted in thermal runaway. The external heating and overcharging did cause the cell to vent. Venting and flames were not present in the external heating test until the heater failed and caught fire. Once the heater power was disconnected, the cell ceased to increase in temperature and self extinguished. During the overcharge tests, the cell expanded in size and eventually vented. Upon venting, there was a flame observered in each case. The flame present seemed to be sustained due to the additional energy provided by the power supply, not the cell itself. Under both tests, the cell self extinguished within seconds of the power supply turning off. It was determined that turning off the power supply immediately after venting was observered was the more accurate method as to not supply the cell with external power. The overcharging #2 methodology was employed as was demonstrated in Test 5.

Figure 22 – Cell gas composition test chamber

Prior to initiating venting for the gas collection, the chamber's atmosphere was purged until a condition of less than 1% oxygen by volume, as shown in Figure 23. Following the purge of the chamber atmosphere, the chamber pressure was relieved to 0 psig, Figure 24.

Figure 23 – Oxygen concentration during purge of cell gas composition test chamber prior to start of test

Figure 24 – Pressure inside the cell gas composition test chamber before overcharging

|--|

| Test Initiation Details | | | |
|---------------------------|------------|--|--|
| Test Date | 2019/09/25 | | |
| Test Start Time | 01:23 PM | | |
| Initial Lab Temperature | 22.6°C | | |
| Initial Relative Humidity | 49% RH | | |

Table 12 – Gas composition results

| Venting for Gas Collection Results* | | | |
|---|--|--|--|
| Venting Time 19:46 | | | |
| Temperature on Top Middle Surface 83.44°C | | | |
| Thermal Runaway Time No thermal runaway, only venting | | | |
| Thermal Runaway Temperature No thermal runaway, only venting | | | |
| *These results are not a direct comparison with the results from Cell Tests 1-4, as this test was | | | |
| conducted inside a pressure vessel with an inert gas atmosphere. | | | |

Figure 25 shows the surface temperatures measured during the test, in which cell venting was observed, but no indication of thermal runaway was observed. As thermal runaway was not observed in any iteration of the tests 1-5, the intent of this gas composition testing is to measure components of the vent gas release, and not thermal runaway gas release.

Upon completion of the venting event, the internal pressure was approx. 4psi. In order to obtain the cell vent gas, an inert gas was added to the vessel to increase the internal pressure to a suitable value to allow for transfer to a gas bottle. The gas compositon shown in Table 14 does not include the inert gas added.

All of the gas events documented after the "Nitrogen Added" event mark in Figure 25 were not a result of the cell venting.

Table 13 – Gas composition test

| Gas Composition Test | : |
|--|--------|
| Volume of Gas Generated (NTP Conditions) | 77.5 L |

Gas that vented from the cell in Test 6 was collected and analyzed using gas chromatography. Table 14 summarizes the results of the types and volume fractions of gases identified. Though the gas chromatography measurements did not resolve specific C4 hydrocarbons, the elution of the gas sample through the gas chromatograph was effective in determining the bulk components of the vented cell gas. The hydrogen, carbon monoxide, carbon dioxide, and THC quantification represents 100% of the gas mixture within the limitation of 2 digits of precision.

| Gas | | Measured % |
|-----------------|-------------------------------|------------|
| Hydrogen | H ₂ | 35.70 |
| Carbon Monoxide | CO | 26.25 |
| Carbon Dioxide | CO ₂ | 24.59 |
| Methane | CH_4 | 4.36 |
| Ethylene | C_2H_4 | 3.91 |
| Ethane | C_2H_6 | 0.83 |
| Propylene | C_3H_6 | 2.57 |
| Propane | C ₃ H ₈ | 0.33 |
| Propadiene | C_3H_4 | 0.02 |
| - | C ₄ (Total) | 1.28 |
| Pentane | $n-C_5H_{12}$ | 0.11 |
| Isopentane | C_5H_{12} | 0.03 |
| Hexane | C_6H_{14} | 0.02 |
| Total | - | 100 |

| Table 14 – Components measured in vented cell ga | easured in vented cell gas |
|--|----------------------------|
|--|----------------------------|

Analysis of lower flammability limit (LFL), maximum pressure rise (P_{max}), burning velocity (S_u) of the cell venting gases was conducted using the methodologies specified in UL 9540A. Please refer to Appendix A for testing details. The results are as follows:

- LFL: 5.24%
- Pmax: 121.1 psig
- S_u: 85 cm/sec

Post Test Thermal Runaway and Re-Ignitions

Additional thermal runaway behavior or re-ignitions were not observed during post test observation, disassembly, and disposal of the sample.

Summary of Cell Test Results

Cell Vent and Thermal Runaway Results

A summary of cell venting times and temperatures, and thermal runaway time and temperatures are presented in Table 15.

| Test | Test Method | Venting Time (mm:ss) | Venting Temperature (°C) | Thermal Runway Time (mm:ss) | Thermal Runway Temperature (°C) |
|------|------------------|-------------------------|--------------------------------|-----------------------------------|---------------------------------------|
| 1 | Short Circuit | Not Observed | N/A | Not Observed | N/A |
| 2 | Heating | 64:05 | 476.14* | Not Observed | N/A |
| 3 | Overcharge #1 | 26:34 | 239.6** | Not Observed | N/A |
| 4 | Nail Penetration | Not Observed | N/A | Not Observed | N/A |
| 5 | Overcharge #2 | 35:55 | 81.85 | Not Observed | N/A |
| 6 | Gas Composition | 19:46 | 83.44 | Not Observed | N/A |
| | (Overcharge) | | | | |

Table 15 – Summary of measurements collected in Cell Level Tests 1 - 6

*The increased temperature was due to the heater being increased to 475°C

**The increased temperature was due to the sustained fire when the power supply for charging was left on.

Thermal runaway was not observed in any of the 5 tests, therefore repeat tests were not required.

Cell Venting Gas Analysis

The total amount of gas collected from the cell after venting was 77.5 L at Normal Tempreature and Pressure (NTP), over a period of approximately 4 minutes. Flammability properties were determined empirically:

- LFL: 5.24%
- Pmax: 121.1 psig
- S_u: 85 cm/sec

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