Helios

Project HELIOS - High Energy Lithium-Ion Storage Solutions (www.helios-eu.org)

(A 3 year project, supported by the European Commission, to study and test the comparative performances of various lithium-ion automotive traction batteries)

'Initial Cycling & Calendar Ageing Test Procedures and checkup tests for High Energy Li-Ion battery cells'

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Abstract

This document provides the full details of the cycle test and calendar life test procedures to be undertaken on the cells under examination throughout the project.

The cells are built from four different chemistries, NCA/graphite, NMC/graphite, LFP/graphite, Mn-spinel oxide/graphite and have all been prepared to the same specification. Although this specification is aimed at the Plug-in HEV vehicle application, the test procedures have been arranged for testing under both Electric Vehicle and Plug-in HEV conditions.

In addition to the test procedures themselves, also provided are the details of pre-test checks with cell conditioning and the 3 different performance checks to be carried out at appropriate intervals during testing.

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1. Introduction :

This report provides the full details of the test procedures to be undertaken on the cells under examination throughout the project. There are two types of test described, cycle test and calendar life test, for each the pre-conditioning actions are described followed by the actual test cycles or life test conditions, as appropriate.

The report provides five different types of cycling profiles, however not all cycling profiles need to be applied.

The total test durations are presented and then the performance characterisation checks to be carried out at various intervals. Three types of checks are given – Full, Short and Quick – and the intervals when each are to be carried out are described. The Full checks cover measurements of capacity, internal resistances, pulse power, impedance and Open Circuit Voltage (OCV). The Short and Quick checks concentrate on capacity and internal resistances.

The test procedures cover battery applications for both Electric Vehicles and Plug-in Hybrid Electric Vehicles and will hold 18 months.

Care is taken throughout to keep the test conditions within cell manufacturers' recommendations and to maintain a close control of cell temperatures.

2. Definition and abbreviations :

Charge:

In this document, charging current/power is given as a negative value.

Charge voltage limit:

The maximum charge voltage allowed by the manufacturer.

C rate:

Discharge or charge current, in amperes, numerically expressed in multiples of the rated capacity in Ah. For example, the 2C discharge current for a battery rated at the 2-h discharge rate is

$$\frac{C_2(Ah)}{0.5(h)} = I^*_{0.5}(A)$$

Note that the value $I^*_{0.5}$ is not the actual value of discharge of the battery in ½ hour. The relevant value of the current at different discharge regimes can be evaluated from the diagram formed by the experimental points.

Constant power discharge:

When the power rate for the constant power discharge is not supplied by the manufacturer, the power discharge rate is conventionally computed as follows:

$$P^*_h = \frac{E_h}{h}[W]$$

where: E_h is the energy given by the battery during the constant current discharge at h hours; h is the theoretical discharging time.

Note that P^*_h is not the actual value of discharge of the battery in h hours. The relevant value of the power at different discharge regimes is evaluated from the diagram formed by the experimental points.

Charge voltage limit, CVL:

The maximum allowed voltage during charge given by the manufacturer. The value is depending on the current applied and the duration of the current pulse.

Cycle:

A discharge (not necessarily full) followed by a complete charge. In order to comply with common usage, also the discharge composed by the repetition of a specific discharge or discharge/charge profile is called "cycle" (e.g. ECE-15 cycle).

Depth of Discharge (DOD):

The ratio of the quantity of electricity removed from a battery on discharge to its rated capacity (if these data are not available from the manufacturer, the results obtained during parameter tests will be assumed as a reference).

Discharge:

In this document, discharging current/power is given as a positive value.

Discharge voltage limit, DVL:

The greater of the minimum voltage limit at I_{max} and 2/3 of the nominal voltage given by the manufacturer.

Driving profile:

There exist a variety of standard driving profiles, e.g. for standard measurements of the emission of exhaust gases. Some profiles are derived from real road drives like the FTP 72 while other profiles, like the ECE cycles, are based on statistical data.

EIS

Electrochemical Impedance Spectroscopy

End of Life:

State of the lithium-ion battery cell that signals that its performance no longer is sufficient for its intended purpose. End-of-life criteria will be defined by the HELIOS partners.

EODV

End Of Discharge Voltage. Cut off voltage for continuous discharge, given by the manufacturer.

EV:

Electric vehicle. Vehicle is propelled by an electric motor and the source of energy for propulsion is electric energy.

FCT:

Full Characterization Test

Imax:

The maximum current allowed by the manufacturer. The value differs depending on if it is charge or discharge current and also the duration of the current pulse (e.g. continuous, 30 sec, 10 sec or other)

PHEV:

Plug-In Hybrid Electric Vehicle. Vehicle is propelled by an internal combustion engine and an electric motor. The source of energy for propulsion is electric energy and chemical energy (fuel).

OCV:

Open Circuit Voltage: Voltage of the battery cell without external load

QCT:

Quick Characterization Test

Qdch/C2:

The accumulated capacity discharged from a battery cell divided by the C_2 capacity.

Ragone graph:

A graph, where the discharged energy is plotted as a function of the discharge power. Energy is plotted on the y-axis and power on the x-axis.

Rated Capacity (Ah):

The specification of the capacity in Ampere-hours obtained from the battery cell discharged by a constant current in one hour to the end-of-discharge condition, usually the cut-off voltage, given by the manufacturer. If the 1C capacity obtained during testing differs more than 3% from the manufacturer´s specification, this should be clearly stated in the test report and the actual value should be used as Rated Capacity.

Reference Temperature (T_{ref}):

30 ± 2 °C

RMS current:

Root Mean Square values of the current. This value is obtained by calculating the square root of the average of the square values of the currents in a drive profile.

Room temperature:

23 ± 2 °C

SCT:

Short Characterization Test

State of Charge (SOC):

SOC= 100% - DoD

3. Initial Cycling & Calendar Ageing Test Procedures

3.1. Pre-conditioning cycles

3.1.1. Purpose

The cells under test shall be conditioned by performing some electrical cycles, before starting the real testing sequence, in order to ensure an adequate stabilization of the cell performance.

3.1.2. Test procedure

The procedure shall be the following:
The test shall be performed at RT.

The discharges shall be performed at 1C or at a different current if suggested and/or used by the cell supplier. The charging shall be performed according to the standard charge procedure.

Three consecutive preconditioning cycles shall be performed.

At end of discharge the cell voltage shall not go below the minimum voltage recommended by the supplier (the minimum voltage is the lowest voltage under discharge without irreversible damage).

The cells shall be considered as "preconditioned" when the discharged capacity during two consecutive discharges does not change by a value greater than 3 % of the rated capacity. If this condition cannot be met, two additional cycles shall be performed and the result shall be stated in the test report. The cells may be used for further tests although they do not fulfil the pre-conditioning requirements due to the prototype state of the cells.

3.2. Standard cycle (SC)

Purpose

The purpose of the standard cycle (SC) is to ensure the same initial condition for each test of the cells. A standard cycle (SC), as described below, shall be performed prior to each test.

Test procedure

General

The standard cycle (SC) shall be performed at T_{ref} (30°C). The SC shall comprise a standard discharge (SDCH), followed by a standard charge (SCH).
For all performance tests the time interval between the end of a SC and the start of a new test shall be not longer than 3 hours, otherwise the SC shall be repeated.

3.3. Standard discharge (SDCH):

Discharge rate:

1C or other specific discharge regime according to the specifications given by the battery supplier.

Discharge limit:

According to the specifications given by the battery supplier.

In cases where cells are connected in series, the voltage threshold (discharge voltage limit provided by the manufacturer) must be applied to each cell of the cell-string. In addition to the voltage termination, there should be a time limit as well as temperature limit (usually 60°C). Furthermore, a voltage threshold for safety reasons is recommended. In case this safety voltage limit is exceeded, discharge should be interrupted automatically.

3.4. Standard charge (SCH) :

To ensure that the test objects are in the same initial conditions and handled according to the manufacturer’s instructions for optimized use, the standard charge procedure, described below, must be used when the cells are charged.

The different test objects have different chemistries and safety limits, hence there are charge procedures for high energy nickel based, manganese based, nickel-manganese-cobalt based or iron phosphate based cells.

In cases where cells are connected in series, the voltage thresholds (charge voltage limits provided by the manufacturer) must be applied to each cell of the cell-string. In addition to the voltage and current terminations of the steps, there should be a time limit for each step as well as temperature limit (usually 60°C). Furthermore, a voltage threshold for safety reasons is recommended. In case this safety voltage limit is exceeded, charge should be interrupted automatically.

High energy cells Apply a charging current of 1C until the CVL is reached; continue charge at constant voltage until the current is below C/20 or 1h is reached; followed by a rest of 30 minutes. Continue the cycling or conduct a parameter check-up.

The CVL is given by the cell manufacturer.

Note: If the current of the standard charge procedure differs very much from the manufacturer´s instructions and the results of the complete test of the cell will be questionable, the current should be altered.

Table 1 : Standard charge profile

Step	High power cells and modules
Constant current charge at	1C
CVL is reached	
Constant voltage charge until current is below	C/20 or 1h is reached
Rest	30 min

3.5. Calendar life test

To be able to separate the effects of aging and the effects of cycling on the cells, storage tests at elevated temperatures are run in parallel with the cycling tests.

The cells have different SOC and are placed in a climatic chamber with a certain temperature. The duration of the test is normally six months. The voltage of each cell will be checked and noted at least once a week. It is not recommended to continually load the cells by measurement instruments.

The different temperatures used in the test are **45 and 60°C**.

The cells will be stored at **100% SOC**. The LMO cells shall be also stored at 20% SOC.

Parameter check-ups are performed every six weeks and also before the storage test starts. The parameter check-ups are performed at the reference temperature $\mathbf{T_{ref}}$. The cells are cooled down to reference temperature and discharged with 1C to the end of discharging voltage followed by a standard charge.
After the parameter check-up, a standard charge is performed, followed by a 1C discharge to the SOC the particular cell is tested at. The cell is then placed in the climatic chamber again and the calendar life test continues.

Data deliverables

Above the data deliverables included in the parameter check-up test, following data should be reported:
The voltage versus time from the weekly voltage controls.
Remaining capacity after storage period (“self discharge”) vs. storage time

3.6. Cycle life test

Purpose

Additionally to the ageing caused by storage (i.e. time, temperature), the energy throughput has a significant influence on the battery life.
The ageing profiles shall consider the real conditions during driving. That means that the applied C rates and SOC swing shall cover the vehicle demands in a proper way. Also, the usable SOC range shall be covered by the cycling profile.

Test procedure

EV & PHEV type profile

This profile is representing the battery cell usage within an EV application or the usage during the EV part of a PHEV application.

Preparation

It is necessary to keep the cell temperatures in a range of ± 3°C within the test temperatures (e.g. 30°C and 45°C).
Temperature control: A thermo couple should be attached to the cell and be used to control the chamber temperature to ensure the temperature accuracy as described above. It may be useful to apply air ventilation to the cells for cooling. However, all test institutes need to apply the same method.

Two cycle profiles are defined. One is the “dynamic discharge profile A”, where the amount of discharged energy is significantly lower than the “dynamic discharge profile B”. The profiles are shown in Figure 1 and Figure 2 and the current – time profiles are shown in Table 4 and in Table 5.
For EV application the cycle test is performed applying the “dynamic discharge profile B” and the current values shown in Table 5.
For PHEV application the cycle test is performed by applying the “dynamic discharge profile A”, but with a much higher average power than for the EV application. The profile is shown in Figure 3 and the current – time profiles is shown in Table 6.

The SOC range is defined as follows:
For EV application, the cycle test shall be performed between 100% and 20% SOC.
For PHEV application, the cycle test shall be performed between 95% and 25% SOC.

Table 2 : SOC range to be used

SOC Range	EV application	PHEV application
Max SOC	100 %	95%
Min SOC	20%	25%

The cycle test shall be started from the Max SOC according to Table 2 performing the relevant dynamic discharge profile A or B until the SOC reaches the Min SOC or the cell voltage reaches the lower voltage limit specified by the cell supplier. Within

the next step, the battery system shall be charged by applying a standard charge (SCH) to the upper limit of SOC.
This sequence of dynamic discharge profiles including charging shall be repeated for 2 weeks. Then, a quick characterization test is performed at cycling temperature and the cycling is continued. A short characterization test shall be performed every 6 weeks at 30°C and a full characterization test shall be performed every 5-6 months before post mortem analysis.

Table 3 : Sequence of Check-Ups

Quick characterization test	Short characterization test	Full characterization test
Week 2	week 0	Month 5-6
Week 4	week 6	Month 10-12
…….	Week 12	Month 15-18
After 2 months, decide to go on or to change the frequency	……	

Test profiles for cycle life test EV application

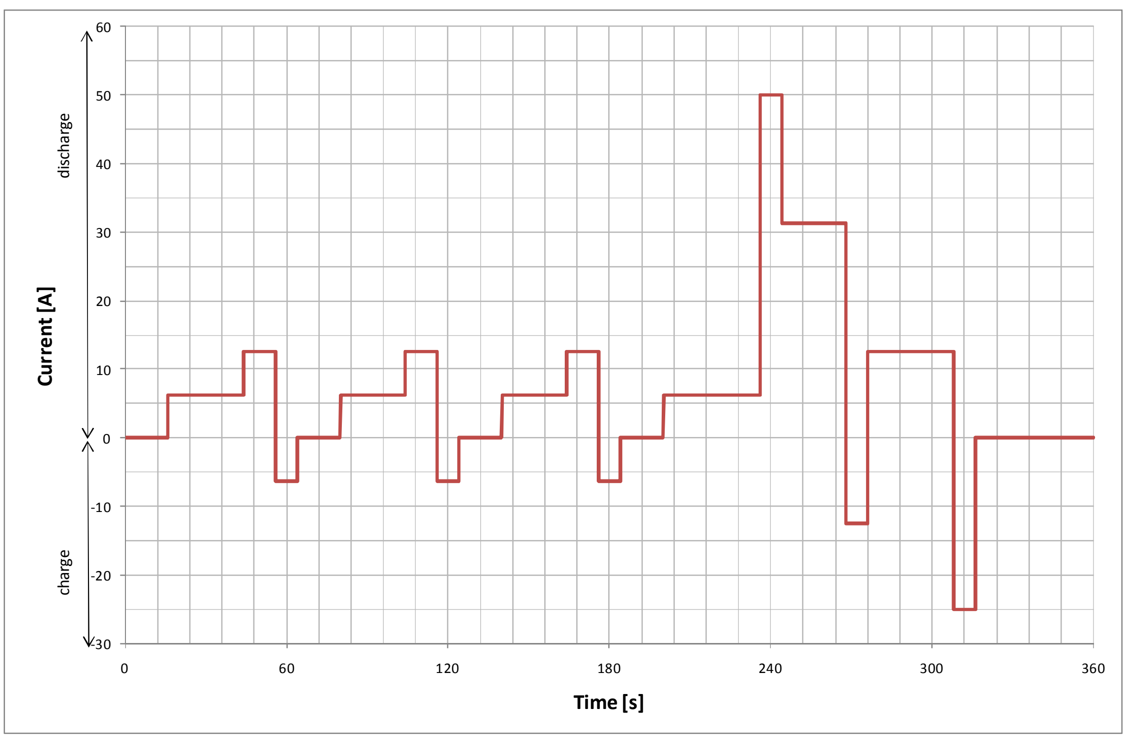


Figure 1 : Current profile for cycle life test — Dynamic discharge profile A

Table 4 : Times and power profile — Dynamic discharge profile A

Step	Time increment [s]	Time cumulative [s]	Current C rate	Current [A]
1	16	16	0	0
2	28	44	0,15	6,25
3	12	56	0,3	12,5
4	8	64	-0,15	-6,25
5	16	80	0	0
6	24	104	0,15	6,25
7	12	116	0,3	12,5
8	8	124	-0,15	-6,25
9	16	140	0	0
10	24	164	0,15	6,25
11	12	176	0,3	12,5
12	8	184	-0,15	-6,25
13	16	200	0	0
14	36	236	0,15	6,25
15	8	244	1,2	50
16	24	268	0,75	31,25
17	8	276	-0,3	-12,5
18	32	308	0,3	12,5
19	8	316	-0,6	-25
20	44	360	0	0

In this profile, the max. current shall be 50A which will equal a C-rate of approximately 1.2. The RMS current in this profile is 13.5A or C/3.

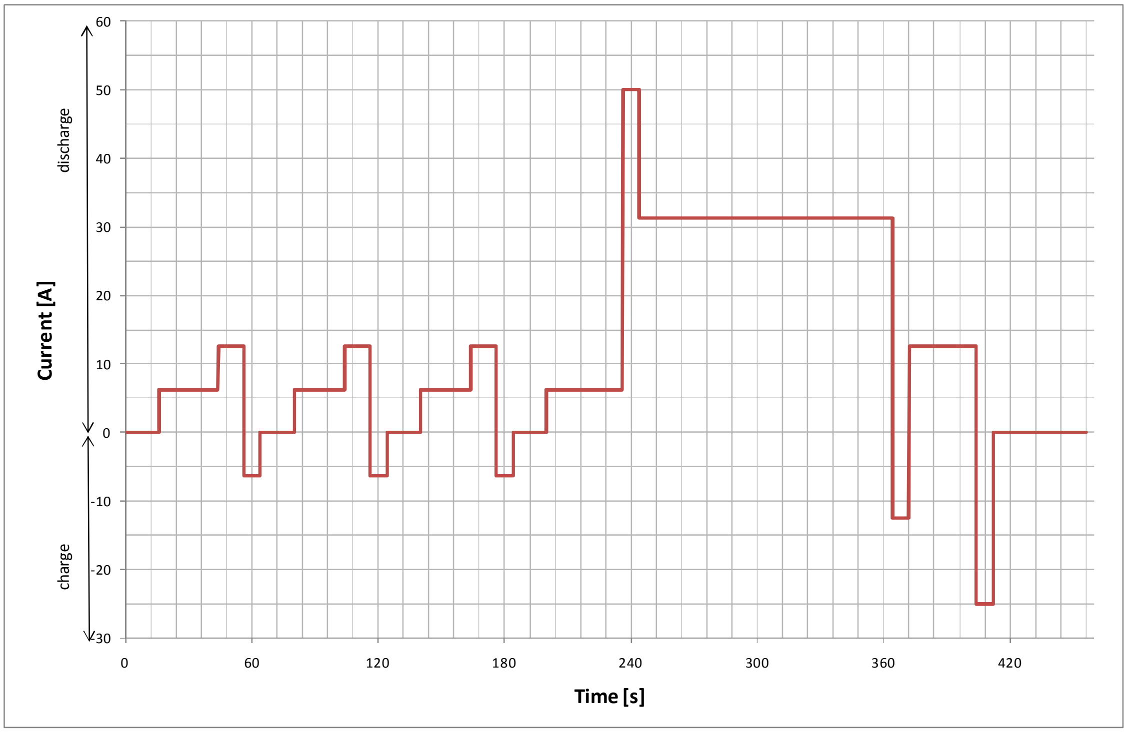


Figure 2 : Power profile for energy cycle test — Dynamic discharge profile B

Table 5 : Times and power profile — Dynamic discharge profile B

Step	Time increment [s]	Time cumulative [s]	Current C rate	Current [A]
1	16	16	0	0
2	28	44	0,15	6,25
3	12	56	0,3	12,5
4	8	64	-0,15	-6,25
5	16	80	0	0
6	24	104	0,15	6,25
7	12	116	0,3	12,5
8	8	124	-0,15	-6,25
9	16	140	0	0
10	24	164	0,15	6,25
11	12	176	0,3	12,5
12	8	184	-0,15	-6,25
13	16	200	0	0
14	36	236	0,15	6,25
15	8	244	1,2	50
16	120	364	0,75	31,25
17	8	372	-0,3	-12,5
18	32	404	0,3	12,5
19	8	412	-0,6	-25
20	44	456	0	0

In this profile, the max. current shall be 50A which will equal a C-rate of approximately 1.2. The RMS current in this profile is around 19A or C/2.

Test profiles for cycle life test PHEV application

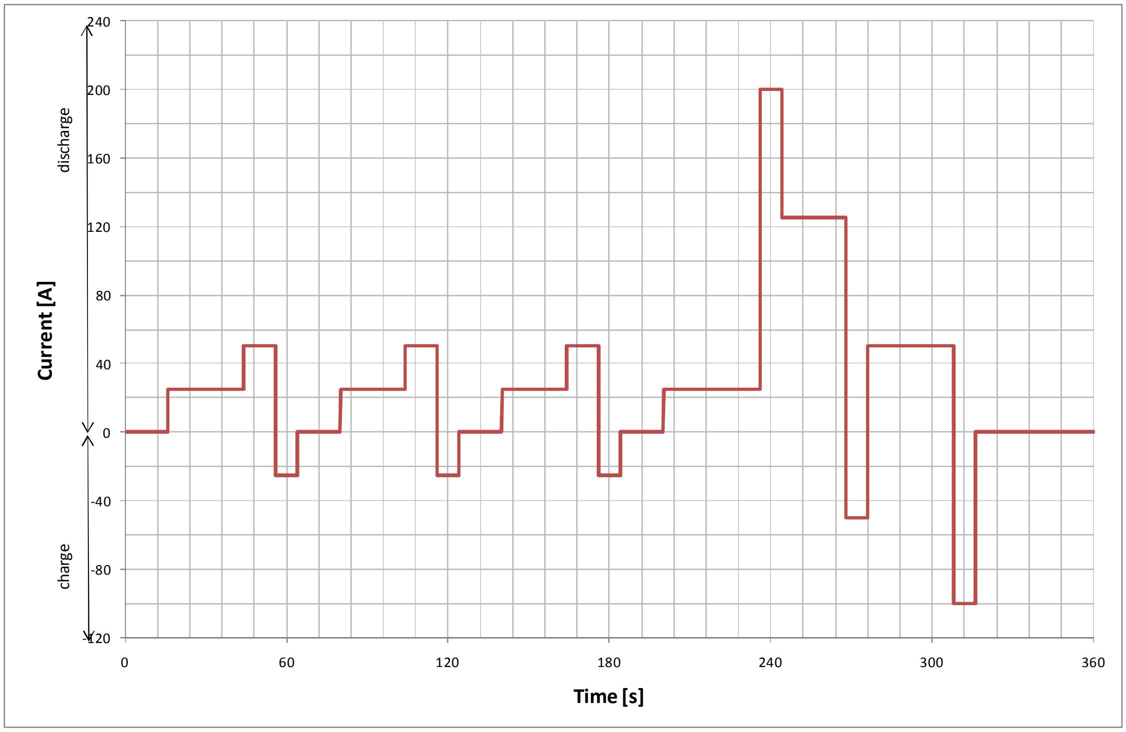


Figure 3 : Current profile for cycle life test — Dynamic discharge profile A

Table 6 : Times and power profile — Dynamic discharge profile A

Step	Time increment [s]	Time cumulative [s]	Current C rate	current [A]
1	16	16	0	0
2	28	44	0,625	25
3	12	56	1,25	50
4	8	64	-0,625	-25
5	16	80	0	0
6	24	104	0,625	25
7	12	116	1,25	50
8	8	124	-0,625	-25
9	16	140	0	0
10	24	164	0,625	25
11	12	176	1,25	50
12	8	184	-0,625	-25
13	16	200	0	0
14	36	236	0,625	25
15	8	244	5	200
16	24	268	3,125	125

Step	Time increment [s]	Time cumulative [s]	Current C rate	current [A]
17	8	276	-1,25	-50
18	32	308	1,25	50
19	8	316	-2,5	-100
20	44	360	0	0

In this profile, the max. current shall be 200A which will equal a C-rate of approximately 5. The RMS current in this profile is around 54A or 1.35 C.

Monitoring and data logging

All available voltage, current and temperature sensor data shall be monitored and logged. The amount of stored data may be reduced by logging only during selected (critical) parts of the test sequences.

Conditions

– Temperatures: The test shall be performed at 30°C and at 45°C in a temperature chamber with adequate safety equipment.
– It may be useful to apply air ventilation to the cells for cooling. However, all test institutes need to apply the same method.
– During cycling, the test bench shall assure that no cell limits will be exceeded, by achieving voltage limits as specified by the supplier. The current has to be reduced automatically to avoid any abuse operation.

4. Performance characterization

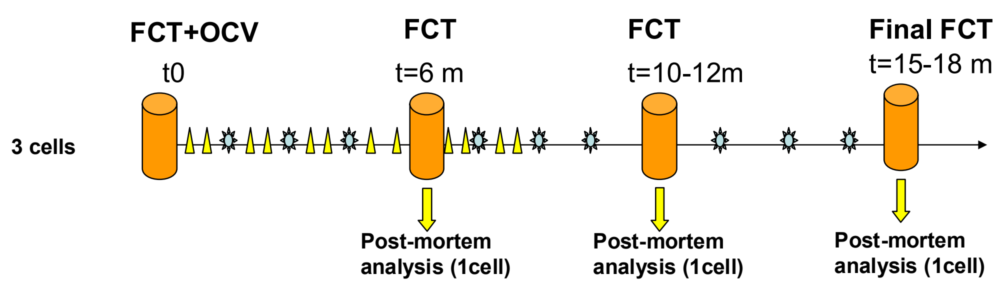
Purpose :

Determine the procedures to evaluate the parameter check up: capacity, internal resistances, pulse power, impedance and Open Circuit Voltage (OCV).
Characterizations are the same for the 2 different test profiles (EV and PHEV)

3 main types of check up will be performed:

- Full Characterization Test (**FCT**) : long & complete check up
- Short Characterization Test (**SCT**) : intermediate & short check up
- Quick characterization test (**QCT**) at cycling temperature

The Different characterizations can be performed according to the following schedule:



= FCT (Full Characterisation Test)
= SCT (Short Characterisation Test) every 6 weeks
QCT (Quick Characterisation Test) every 2 weeks

Description of the characterization Tests to evaluate the evolution of the main characteristics of the cells (according to the conditions of tests and the chemistry used).

4.1. Full Characterization Test (FCT)

FCT is a complete check up and will be performed before testing sequences (at t0) and on cell removed before post-mortem analysis (at 5-6, 10-12 or 15-18 months).

FCT is decomposed in 7 steps:

1. Initial or Interruption of cycling test
2. Discharge (to End Of Discharge Voltage): *procedure for adjusting SOC: starting from the fully* charge *status.*
3. Temperature equilibration

4. Capacity measurement
5. Internal Resistance pulse
6. Electrochemical Impedance Spectroscopy
7. Continue cycling or calendar test

Furthermore, **the SOC is related to the reference value (measured at Tref and at beginning of life)**

Duration : 4-5 days / cell (or total if 3 channels are available).

We're going now to describe each step:

1. General
The Full Characterization Test will be performed on each cell, before the beginning of the test (t0) or on used cells after interruption of testing sequences and before post-mortem analysis.

2. Discharge:
discharge rate = C/3 to End Of Discharge Voltage @ 30°C (reference Temperature)

3. Temperature equilibration (Tref = 30°C)
If necessary but the time of temperature equilibration should not exceed 2H

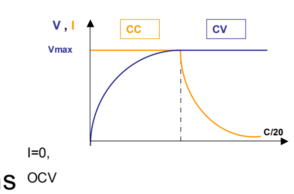
4. Capacity measurement

- Description :
The aim of these measurements is to determine the influence of the discharge rate and temperature on the cell capacity.

- test conditions :
  - Charge current (1C at 30°C or C/5 at 0°C)
  - 2 temperatures : 0°C & 30° C
  - 3 Discharge currents (C/3, C, 2C or Imax)
  - Rest period = 30 minutes or Ti < T < Ti + 2°C

- Procedure :

After temperature stabilization,



- Charge 1C rate to upper voltage limit (upper voltage limit being maintained until current decreases to C/20 rate (limit in time = 1H)) and according to the supplier recommendations

- Discharge C/3, 1C & 2C or Imax rate to lower voltage limit @ 30°C

- The measurements ( ) must be done twice. Only the 2nd measurement will be taken into account.

@ 2 temperatures : 0°C (C/5) , RT= 30°C

Discharge (1C) < 1H	Full charge (1C) 1H	Rest 30 mn	Full discharge (C/3)	Rest 10 mn	Full charge (1C)	Rest 30 mn	Full discharge (C/3)	Rest 10 mn
			Capacity measurement				2nd Capacity measurement	
	Full charge (1C) 1H	Rest 30 mn	Full discharge (1C)	Rest 10 mn	Full charge (1C)	Rest 30 mn	Full discharge (1C)	Rest 10 mn
	Full charge (1C) 1H	Rest 30 mn	Full discharge (2C or Imax)	Rest 10 mn	Full charge (1C)	Rest 30 mn	Full discharge (2C or Imax)	Rest 10 mn

- some spare cells will be measured **@ -20°C**@ t0 only (charge : C/5 and discharge 1C)

Full charge (C/5)	Rest 30 mn	Full discharge (1C)	Rest 10 mn	Full charge (C/5)	Rest 30 mn	Full discharge (1C)	Rest 10 mn

- some other spare cells will be measured in **quick charge conditions** @ 30°C (charge @ 2C & discharge @ 1C)

Full charge (2C)	Rest 30 mn	Full discharge (1C)	Rest 10 mn	Full charge (2C)	Rest 30 mn	Full discharge (1C)	Rest 10 mn

- Expected results:

Datas to be notified in the test report are:
current, voltage, capacity, energy density, Energy (Wh cumulated during the charge / discharge), cell and room temperature

- Expected graphs:

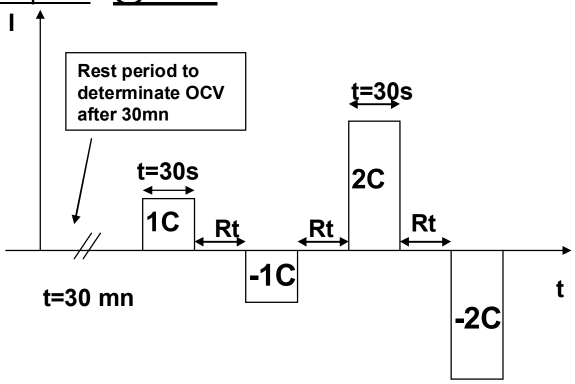
- Capacity = f(T, Crate)
- Wh = f (W) (Ragone) (taken from the CC discharge not constant power discharge required)
- Voltage = f(discharged capacity-Ah) at constant current, for each Temperature (with ref = OCV)

5- Internal Resistance pulse

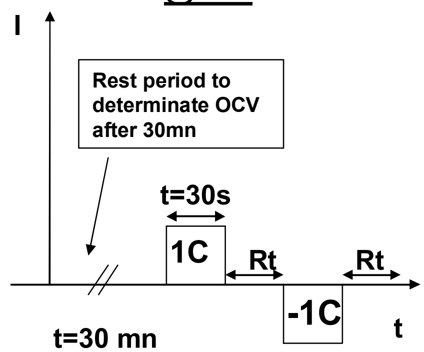
- Test conditions:
- 2 Temperatures : 0°C and 30°C
- Charge at C/5 @ 30°C for 9 different SOCs (x = 90 then p80…20,10%)

- Charge at C/5 @ 0°C for 9 different SOCs (x = 90,70, 50, 30,10%) @ t0 and Charge at C/5 @ 0°C for 1 SOC = 50% @t=6-12-18 months.
- Rest period (Rt) = 15mn (ideally and 10 mn minimum)

Description : **@ 30°C**



@ 0°C



- Procedure:

- Complete discharge
- OCV measurement during the initial 30 mn rest period
- Charge at C*/5 @ different SOCs
- Pulse duration : 30s
- Sampling datas : 100ms for first 2 s, then 1 sec : 3,4,5,…9,10, 11, 12,13,14, 15,…., 30 (every sec) during the pulse
- then1,2,3,4,5,…9,10, 15, 20, 25, 30, 60,120 s (f=1Hz) after the pulse (Rt) (TBC with simulation needs)

C* :Using initial / standard capacity @ 30°C(reference temperature) and SOC = 100%, BOL.

Warning: Peak current must be stopped immediately if the low voltage limit or high voltage limit is reached.

Step		Time	Current
1	SOC = X – OCV measure	30 min	0
2	SOC = X	30s	1C
3	SOC = X	10-15 mn	0
4	SOC = X	30s	-1C
5	SOC = X	10-15 mn	0
6	SOC = X	30s	2C
7	SOC = X	10-15 mn	0
8	SOC = X	30s	-2C
9	SOC = X	10-15 mn	0
	SOC = Y – OCV measure	30 mn	0
	SOC = Y	30s …..	….

Four different values of resistance will be calculated as follows:

Time (s)	0	1	2	…	10	30	x
Voltage (V)	U_0	U_1	U_2	…	U_{10}	U_{30}	U_x

Ohmic discharge resistance : pour ΔT = x sec,

$$R\Delta T,\text{discharge} = \frac{U_{31} - U_{30}}{I_{pk}}$$

Overall internal discharge resistance :

$$IR_{\text{1C or 2C discharge}} = \frac{U_{OCV} - U_x}{I_{pk}}$$

Ohmic charge resistance :

$$R\Delta T,\text{charge} = \frac{U_{31} - U_{30}}{I_{pk}}$$

Overall internal charge resistance :

$$IR_{\text{1C or 2C charge}} = \frac{U_{OCV} - U_x}{I_{pk}}$$

The peak power is calculated by 2 ways :

* Real power (after 2,5,10, 15 & 30) : $\mathbf{P_{xs,discharge} = (U_{OCV} - R\, I_{pk}) * I_{pk}}$
with I_{pk}= peak current at t

* Max theorical power : $\mathbf{P_{max,\ thdischarge}} = \dfrac{(U_{OCV} - U_{min}) * U_{min}}{R_{(SOC,I)}}$

- <u>Expected results : (@</u> 1,2,10,15,30,45,60s)

- Discharge power for pulses at 20s= f (SOC,T, I_{pk}) with I_{pk}= peak current at t
- Charge power for pulses at 20s = f (SOC,T, I_{pk})
- Discharge resistance for pulses at 20s = f (SOC,T, I_{pk})
- Charge resistance for pulses at 20s = f (SOC,T, I_{pk})
- OCV = f(SOC, T)

<u>- Expected graphs :</u>

- Discharge & charge Resistances = f(I, t,T) for each SOC
- Discharge & charge Power = f(SOC,T, I)
- OCV = f(SOC, T)

6- Electrochemical Impedance Spectroscopy (EIS)

- <u>Description</u> :
The impedance test will be performed on each cell, at different states of charge.

- Test conditions :
  - 1 temperature : 30°C
  - 3 SOCS (90,50 & 10%),
    other SOC's reached with Current= 1C + Rest period = 1H
  - frequency range between : 5kHz – 10mHz,
  - frequency distribution : 8-10 points/decade,
  - 2-3 measures / frequency

- Procedure

see the document "Impedance measurements" proposed by ISEA

Helios_Impedance_Measurements_isea.do

Caution : take into account the length of wires (inductance's value) and take care this length is the same for all the labs

- Expected results : Im(Z), Re(Z)
- Expected graphs :

- Im(Z) = f(Re(Z) ; SOC)

Plot only Im(Z) = f(Re(z)) for: SOC = 10, 50 & 90% @ 30°C

7- Discharge (1C discharge to the required SOC-level) and Continue cycling, calendar test or send the cell to LRCS for post-mortem analysis.

4.2. Short Characterization Test (SCT)

To be performed every 6 weeks @ 30°C+ rest period = 2h

1. **Procedure: Capacity & Energy**

After temperature stabilization,

-Charge 1C rate to upper voltage limit (upper voltage limit being maintained until current decreases to C/20 rate (limit in time for the constant voltage phase = 1H)

- Discharge 1C to lower voltage limit

- the measurement of capacity must be done twice. Only the 2nd measurement will be taken into account.

Full charge (1C) 1H	**Rest 30 mn**	**Full discharge (1C)**	**Rest 10 mn**	**Full charge (1C)**	**Rest 30 mn**	**Full discharge (1C)**	**Rest 10 mn**

- Expected results:

Current, voltage, capacity, energy density, Energy (Whc), instantaneous Energy, Instantaneous power, cell temperature and room temperature

- Expected graphs:

- Capacity = f(T, C)
- Wh = f (W) (Ragone)
- Voltage = f(discharged capacity-Ah) at constant current, for each Temperature (with ref = OCV)

2. Internal Resistance pulse (SOC=90-50-20%)

Frequency: to be performed every 6 weeks @Tref = 30°C

- Test conditions:
  - 1 Temperature: 30°C
  - Charge at C*/5 for 3 SOCs (x = 90, 50 & 20%)
  - Rest period (Rt) = 15mn (ideally, 10 mn minimum)
  - OCV measurement

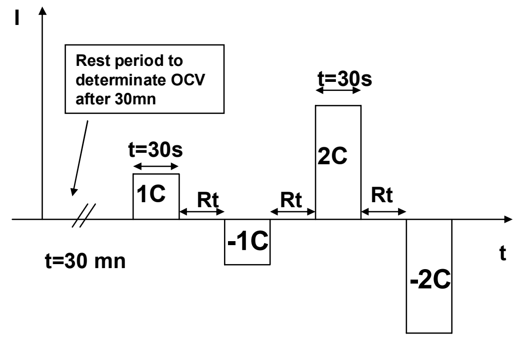
C* :Using initial / standard capacity @ 30°C(reference temperature) and SOC = 100%, BOL.

- Procedure:
  - Complete discharge
  - OCV measurement during the initial 30 mn rest period
  - Charge at C/5 @ 3 different SOCs
  - Pulse duration : 30s
  - Sampling datas : 100ms for first 2 s, then 1 sec : 3,4,5,…9,10, 11, 12,13,14, 15,…., 30 (every sec) during the pulse
  - then1,2,3,4,5,…9,10, 15, 20, 25, 30, 60,120 s (f=1Hz) after the pulse (Rt) (TBC with simulation needs)

Warning: Peak current must be stopped immediately if the low voltage limit or high voltage limit is reached.

Step		Time	Current
1	SOC = 20% – OCV measure	30 min	0
2	SOC = 20%	30s	1C
3	SOC = 20%	10-15 mn	0
4	SOC = 20%	30s	-1C
5	SOC = 20%	10-15 mn	0

6	SOC = 20%	30s	2C
7	SOC = 20%	10-15 mn	0
8	SOC = 20%	30s	-2C
9	SOC = 20%	10-15 mn	0
	SOC = 50% – OCV measure	30 mn	0
	SOC = 50%	30s …..	….
	………		



- Expected results & graphs:

- Discharge power for pulses at 20s= f (SOC,T, I_{pk}) with I_{pk}= peak current at t
- Charge power for pulses at 20s = f (SOC,T, I_{pk})
- Discharge resistance for pulses at 20s = f (SOC,T, I_{pk})
- Charge resistance for pulses at 20s = f (SOC,T, I_{pk})
- OCV = f(SOC, T)

4.3. Quick Characterization Test (QCT)

@ Cycling temperature + rest period = 2h

To be performed every 2 weeks (after 2 months, discussion to decide to go on or to change the frequency of this Quick check up).

1. **Procedure: Capacity & Energy**

After temperature stabilization,

-Charge 1C rate to upper voltage limit (upper voltage limit being maintained until current decreases to C/20 rate (limit in time for the constant voltage phase = 1H)

- Discharge 1C to lower voltage limit

- the measurement of capacity must be done twice. Only the 2nd measurement will be taken into account.

Full charge (1C) 1H	**Rest 30 mn**	**Full discharge (1C)**	**Rest 10 mn**	**Full charge (1C)**	**Rest 30 mn**	**Full discharge (1C)**	**Rest 10 mn**

- Expected graphs:

  - Capacity = f(T, C)
  - Wh = f (W) (Ragone)
  - Voltage = f(discharged capacity-Ah) at constant current, for each Temperature (with ref = OCV)

2. Internal Resistance pulse (SOC=50%)

- Procedure:

  - Complete discharge
  - OCV measurement during the initial 30 mn rest period
  - Charge at C/5 @ SOC =50%
  - Pulse duration : 30s
  - Sampling datas : 100ms for first 2 s, then 1 sec : 3,4,5,…9,10, 11, 12,13,14, 15,…., 30 (every sec) during the pulse
  - Then 1,2,3,4,5,…9,10, 15, 20, 25, 30, 60,120 s (f=1Hz) after the pulse (Rt) (TBC with simulation needs)