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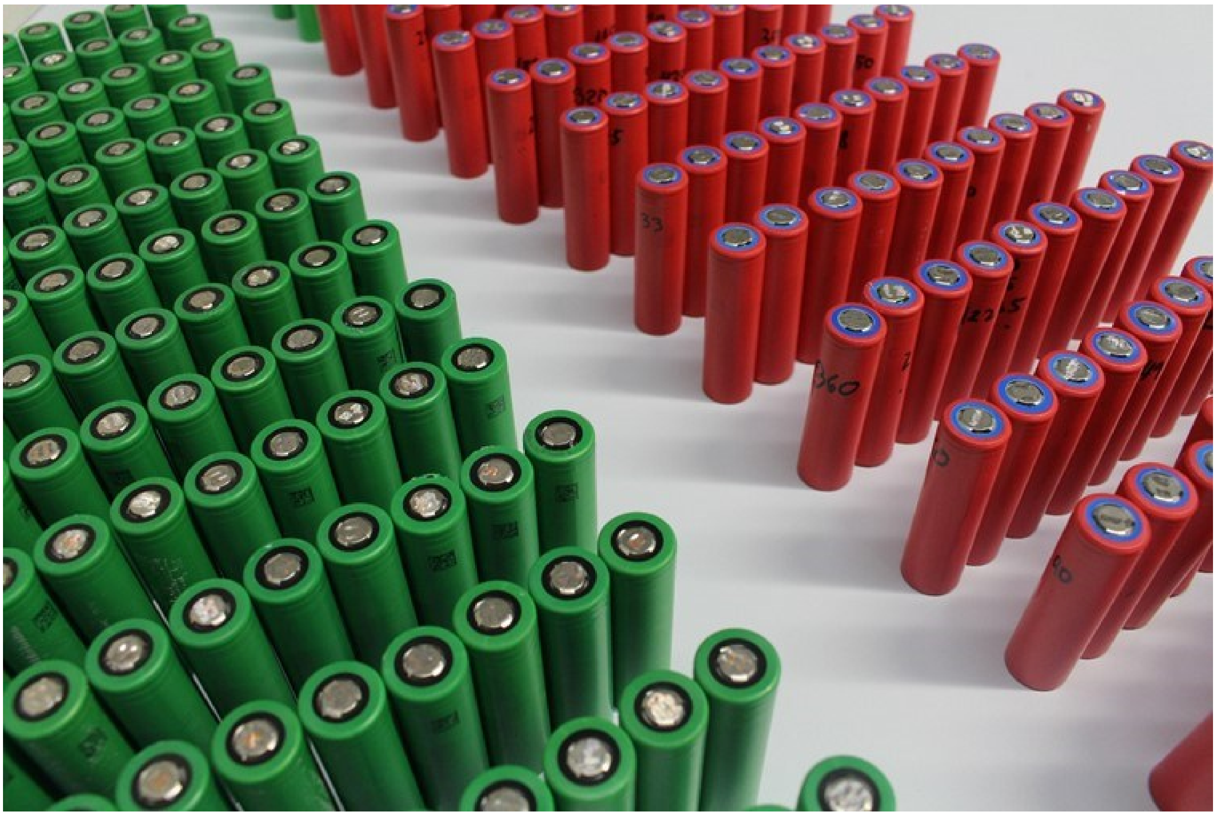
Federal Department of the Environment, Transport, Energy and Communications DETEC

Swiss Federal Office of Energy SFOE
Energy Research and Cleantech Division

Final report dated 19th May 2021

BAT4SEL - Battery Accelerated Testing for SEcond-Life

Methodology, testing and results



Source: CSEM SA - 2021

Date: 19th May 2021

Location: Neuchâtel

Publisher:
Swiss Federal Office of Energy SFOE
Energy Research and Cleantech
CH-3003 Bern
www.bfe.admin.ch

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SFOE contract number: SI/502046-01

The authors bear the entire responsibility for the content of this report and for the conclusions drawn therefrom.

Zusammenfassung

CSEM und Libattion haben sich im Projekt BAT4SEL zusammengeschlossen, um die Gültigkeit eines beschleunigten Testverfahrens zur Abschätzung der verbleibenden Kapazität (SoH) von gebrauchten Li-Ionen-Zellen zu überprüfen. Das Projektziel war eine statistische Quantifizierung der Genauigkeit der SoH-Schätzung von verschiedenen Verfahren und der erforderlichen Testzeit, um diese Schätzung zu erhalten. Um dieses Ziel zu erreichen stellte Libattion eine statistisch repräsentative Anzahl von Li-Ionen Zellen aus E-Bike-Anwendungen zur Verfügung. Die statistische Analyse basiert auf dem Test einer hohen Anzahl von Zellen von vier ausgewählten Li-Ionen Technologien, um aussagekräftige Indikatoren zu entwickeln, die mit der SoH-Entwicklung korreliert werden können. Die Testmethodik basierte auf zwei verschiedenen Untergruppen von Tests: (i) Diagnosetests, bei denen eine kleine, aber repräsentative Stichprobe von Zellen charakterisiert wurde, um bestehende Korrelationen zwischen Indikatoren und SoH zu finden und (ii) Validierungstests, bei denen eine größere Anzahl von Zellen getestet wurde, um die Protokolle durch Vergleich der geschätzten und gemessenen SoH-Werte zu validieren. Es wurden drei Haupttypen von Indikatoren verwendet: widerstandsbasierte Indikatoren, effizienzbasierte Indikatoren und Indikatoren basierend auf elektrochemischer Impedanzspektroskopie (EIS). Außerdem wurde die Korrelationsbewertung für drei verschiedene Testbedingungen verifiziert: (i) zufällige Spannungsbedingung (keine Regulierung des Ladezustands (SoC) der Zelle); (ii) Nennspannungsbedingung (Regulierung des SoC der Zelle); (iii) gefilterte Version der zufällige Spannungsbedingungen (Regulierung der Zellen außerhalb innerhalb bestimmten Spannungsintervalls).

Die Ergebnisse der Diagnosetests zeigten, dass es keinen Indikator gibt, der die anderen übertrifft,, und dass Experimente bei zufälliger Spannung selten gute Korrelationen liefern. Die Ergebnisse der Validierungstests zeigten, dass widerstandsbasierte Indikatoren im Durchschnitt einen Schätzfehler von unter 2,5 % liefern während effizienzbasierte Indikatoren einen stabilen Schätzfehler von etwa 2 % zeigen. EIS-basierte Indikatoren zeigen etwas höhere Schätzfehler, aber eine geringere Standardabweichung als die beiden anderen Methoden. Über alle Testbedingungen hinweg bringen alle Indikatoren eine signifikante Reduzierung der Testzeit von mindestens 70 %, bezogen auf den 5-stündigen Standard Entlade-Lade-Entlade-Zyklus. Im Falle von Tests bei zufälliger Spannung mit Filteroption benötigen widerstandsbasierte Indikatoren 8 Minuten (-97%), effizienzbasierte Indikatoren 19 Minuten (-93%), während EIS-basierte Indikatoren 45 Minuten (-85%) benötigen.

Es sollte beachtet werden, dass Unsicherheiten und größere Fehler durch die Testausrüstung eingeführt werden könnten. Im Rahmen des Projektes wurde nur High-End-Testausrüstung während der Diagnosephase verwendet, was eine hohe Genauigkeit und Präzision bei den Messungen und Korrelationen sicherstellt. Darüber hinaus führen schlechte Ausgangsbedingungen der Batteriezellen (z. B. tief entladene Zellen) zu größeren Standardabweichungen; aus diesem Grund lieferte der Ansatz der zufälligen Spannungsfilterung die zuverlässigsten Ergebnisse.

Zusammenfassend kann festgestellt werden, dass die effizienzbasierten Indikatoren den besten Kompromiss für eine präzise und schnelle SoH-Schätzung von den im Projekt untersuchten gebrauchten Li-Ionen-Zellen darstellen. Die Wahl des am besten geeigneten Indikators hängt jedoch davon ab, welcher Aspekt für den Endanwender am wichtigsten ist: Schätzgenauigkeit, Robustheit (konsistente Korrelationen zwischen den Zelltechnologien), Testzeit sowie auch die Anwendbarkeit auf den existierenden Maschinenpark.

Résumé

CSEM et Libattion se sont associés dans le projet BAT4SEL pour vérifier la validité d'une procédure de test accélérée pour estimer la capacité restante (SoH) des cellules Li-ion de seconde vie (c'est-à-dire les cellules Li-ion usées). L'objectif du projet était de donner une quantification statistique entre la précision de l'estimation du SoH des différentes procédures et le temps de test nécessaire pour obtenir cette estimation. Pour réaliser cette objectif, un nombre statistiquement représentatif d'échantillons provenant

d'applications de vélos électriques a été fourni par Libattion. L'ensemble de l'analyse statistique était basé sur le test d'un grand nombre de cellules de quatre technologies sélectionnées afin de développer des indicateurs significatifs qui peuvent être corrélés avec l'évolution du SoH. La méthodologie de test a été basée sur deux sous-ensembles de tests différents : (i) les tests de diagnostic, où un nombre limité d'échantillons représentatifs de cellules a été caractérisé pour trouver les corrélations existantes entre les indicateurs et le SoH et (ii) les tests de validation, où un plus grand nombre d'échantillons a été testé pour valider les protocoles en comparant les valeurs estimées et mesurées du SoH. Trois principaux types d'indicateurs ont été appliqués : les indicateurs basés sur la résistance, les indicateurs basés sur l'efficacité et les indicateurs basés sur la spectroscopie d'impédance électrochimique (EIS). De plus, l'évaluation de la corrélation a été vérifiée pour trois conditions de test différentes : (i) condition de tension aléatoire (aucune régulation du SoC de la cellule) ; (ii) condition de tension nominale (régulation de l'état de charge (SoC) de la cellule) ; (iii) tension aléatoire filtrée (régulation des cellules dans un intervalle de tension spécifique).

Les résultats des tests de diagnostic ont montré qu'il n'existe pas d'indicateur prévalant sur les autres et que les expériences à tension aléatoire fournissent rarement une bonne corrélation. Les résultats des tests de validation ont montré que les indicateurs basés sur la résistance fournissaient en moyenne une erreur d'estimation inférieure à 2,5% et que les indicateurs basés sur l'efficacité présentaient une erreur d'estimation stable autour de 2%. Les indicateurs basés sur le EIS ont montré des erreurs d'estimation légèrement plus élevées mais un écart-type plus faible que dans les deux cas précédents. Il convient de noter que les incertitudes et les erreurs plus importantes peuvent être dues à l'équipement de test et à l'état initial des cellules (par exemple, des cellules profondément déchargées). Dans toutes les conditions de test, tous les indicateurs permettent une réduction significative du temps de test d'au moins 70 %, par rapport au cycle standard de décharge-charge-décharge de cinq heures. Dans le cas de tests à tension aléatoire avec option de filtrage, les indicateurs basés sur la résistance nécessitent 8 minutes (-97%), les indicateurs basés sur l'efficacité nécessitent 19 minutes (-93%), tandis que les indicateurs basés sur le EIS nécessitent 45 minutes (-85%).

Il convient de noter que des incertitudes et des erreurs plus importantes peuvent être introduites par l'équipement de test. Dans le cadre du projet des équipements de test haut de gamme ont été utilisés au cours de la phase de diagnostic, ce qui a permis d'assurer une grande exactitude et précision des mesures et des corrélations. En outre, les mauvaises conditions initiales des cellules de la batterie (par exemple, des cellules profondément déchargées) introduisent un écart type plus important. Pour cette raison, l'approche de filtrage de tension aléatoire a fourni les résultats les plus fiables.

En conclusion, on peut observer que les indicateurs basés sur le rendement représentent le meilleur compromis pour une estimation précise et rapide du SoH des cellules Li-ion de seconde vie qui était analysé dans le cadre du projet. Cependant, le choix de l'indicateur le plus approprié à utiliser dépend de l'aspect de plus haute importance pour l'utilisateur final : la précision de l'estimation, la robustesse (c'est-à-dire les corrélations cohérentes entre les technologies de cellules), le temps de test et l'applicabilité aux machines de test déjà existantes.

Summary

CSEM and Libattion have partnered in the BAT4SEL project to verify the validity of an accelerated testing procedure to estimate the SoH of second-life Li-ion cells (i.e., used Li-ion cells). The project objective was to give a statistical quantification of the trade-off between the SoH estimation precision and the required testing time to obtain this estimation; for this reason, a statistically representative number of samples from e-bike applications has been provided by Libattion to CSEM. The whole statistical analysis was based on the test of a high number of cells of four selected technologies to develop meaningful indicators which can be correlated with the SoH evolution. The testing methodology has been based onto two different sub-set of tests: (i) Diagnostic tests, where a representative sample of cells have been characterized to find existing correlations between indicators and SoH and (ii) validation tests, where a larger number of

samples have been tested to validate the protocols by comparison of the estimated and measured SoH values. Three main type of indicators have been applied: resistance-based indicators, efficiency-based indicators and EIS-based indicators. Moreover, the correlation assessment has been verified for three different testing conditions: (i) random voltage condition (no regulation of cell’s SoC); (ii) nominal voltage condition (regulation of cell’s SoC); (iii) random voltage filtered (regulating cells inside a specific voltage interval).

The diagnostic tests showed that there is not an indicator prevailing on the others and that experiments at random voltage rarely provide a correlation. The results of validation tests showed that resistance-based indicators provided on average an estimation error below 2.5% and efficiency-based indicators showed a stable estimation error around 2%. EIS-based indicators showed slightly higher estimation errors (up to 3%) but lower standard deviation than in the two previous cases. Across all testing conditions all indicators bring a significant reduction on the testing time of at least 70%, with respect to the 5 hours standard discharge-charge-discharge cycle. In the case of tests at random voltage with filtering option, resistance-based indicators require 8 minutes (-97%), EE-based indicators require 19 minutes (-93%), while EIS-based indicators require 45 minutes (-85%).

It should be noticed that uncertainties and larger errors could be introduced by the testing equipment; only high-end test equipment has been used along the diagnostic phase which ensured high accuracy and precision on the measurements and correlations. Moreover, initial bad conditions of the battery cells (e.g., deeply discharged) do introduce larger standard deviation; for this reason, the random voltage filtering approach provided the most reliable results

In conclusion it can be observed that the efficiency-based indicators represent the best compromise for a precise and fast SoH estimation of second-life Li-ion cells. However, the choice of the most proper indicator to be used depends on which aspect is the most important for the final user: estimation accuracy, robustness (i.e., giving consistent correlations among cell technologies), testing time and machine applicability.

Main findings

1. BAT4SEL project was about aged cells but not about second-life cells (averaged SoH > 90%).
2. All tested indicators bring a significant reduction on the testing time of 70%, with peak up to 97% (i.e. 8 minutes test).
3. SoH validation measurements has shown biases (the SoH measurements performed at project partners were giving different values even though applied on the same cells).
4. The characterization process takes 1 month per technology, including testing protocol definition and result analysis.
5. The testing equipment might influence the results introducing uncertainties (mainly due to precision and accuracy of the battery tester and to the temperature at which tests are carried out).
6. Not all the battery technologies tested brought to positive results. One specific technology did not show any correlations between any of the chosen indicator and SoH.
7. Tests at random voltage condition rarely show correlations (only in 25% of the cases the correlation was found).
8. A pre-triage process to exclude cells outside a predefined voltage interval is a good practice.
9. There is not the best indicator for all. One should choose according to his/her needs regarding accuracy, robustness, testing time and testing machine applicability.

Contents

Abbreviations

AC	Alternate Current
BMS	Battery Management System
CSEM	Centre Suisse d'Électronique et de Microtechnique SA
CC	Constant Current
CV	Constant Voltage
DC	Direct Current
DoD	Depth Of Discharge
EE	Energy Efficiency
EIS	Electrochemical Impedance Spectroscopy
NCA	Lithium Nickel Cobalt Aluminium Oxide
NMC	Lithium Nickel Manganese Cobalt Oxide
OCV	Open Circuit Voltage
SoC	State of Charge
SoH	State Of Health
SoX	State Of X (where X stands for any cell state variable, e.g. charge, health, power, resistance)

1 Introduction

1.1 Background information and current situation

Every year, approximately 543’000 automotive batteries for traction (BEV and PHEV) enter the EU [1] which is almost 27 GWh by assuming a standard battery pack of 50kWh. Although uncertain, it is forecasted that in the year 2025, 27% of those batteries will have a second life in stationary applications, while the remaining 73% will be available to be recycled [2]. The EU commission, within the strategic action plan on batteries [3], announced endeavours to support second-life through a sustainable battery value chain and they suggest that full advantage should be taken of the Eco-design Directive framework [4]. However, this will depend on several factors/challenges, including: (i) the cost to remanufacture first-life batteries for storage applications, (ii) the value of sub-components and materials that could be extracted from first-life batteries, (iii) the regulatory barriers that defines which entity is responsible for the battery during second-life.
Point (iii) is a regulatory issue, not addressable in this project.
Point (i) needs improvements in disassembly procedure which should exploit automation (fostered by design for disassembly approaches [5]) and plug & play approaches for second-life remanufacturing. Point (i) is not investigated in this project.
Point (ii) instead needs procedures that qualify the residual energetic value of the cell and/or module to be then quantified economically for the second-life battery pack to be sold. Such procedures should be fast enough not to create high additional cost for the second-life manufacturing entity. Being an EV battery pack composed of thousands of cells, the assessment should not take more than minutes/cell. Point (ii) is investigated in this project.
As regards of point (ii), CSEM has already performed preliminary evaluation on a small set of cells (15 samples to quantify the remaining energy content in minutes rather than hours. The developed proxy has been shown to follow with a positive correlation factor the trend on the measured SoH. The estimated SoH from the proxy has been proved to bring to an acceptable error: $\bar{x}$ = 1.24%, σ = 0.94%.
As regards of point (ii), Libattion has partnered with Batrec Industrie AG, the national battery recycling company in Switzerland, to try in providing the swiss answer to the challenge. It has developed an innovative battery processing technique in which used e-bike batteries are selected for further reuse in other applications such as light electric vehicle and stationary energy storage systems.

1.2 Purpose of the project

To prove the feasibility of a fast testing procedure to assess SoH of lithium ion cells, there exists the need to perform a wider set of measurements with respect to the one already carried out at CSEM (from tens to hundreds) and to extend the approach other chemistries.
For these reasons, CSEM and Libattion has partnered in the BAT4SEL project (6 months project) to verify the validity of an accelerated testing approach via a statistically representative number of different Li-ion cells, i.e. to extend the approach to up-to-date chemistries (NMC/NCA new types) from e-bike applications provided by Libattion.

Before the project CSEM and Libattion has agreed on the followings:

- the number of cells should be greater than 100 for each tested chemistry.
- The number of chemistries should be greater than two, ideally three.
- Aging status of tested cells should be SoH < 85%.

1.3 Objectives

The whole results are based on two KPIs:

- The affordable testing time per cell:
  Target: 5-10 mins/cell
- The affordable estimation error on the SoH in terms of acceptable mean error $\bar{x}$ and standard deviation σ
  Target: $\bar{x}$ = 1.25%, σ = 1%

So the whole project outcome will be evaluated on the trade-off between the SoH estimation accuracy and the time required to assess SoH with standard equipment.

Implementation at CSEM focused onto different fast testing procedures to find the suitable proxy to satisfy the above KPIs: resistance tests, efficiency tests, EIS tests.

The a-priori plan of the project was the following:

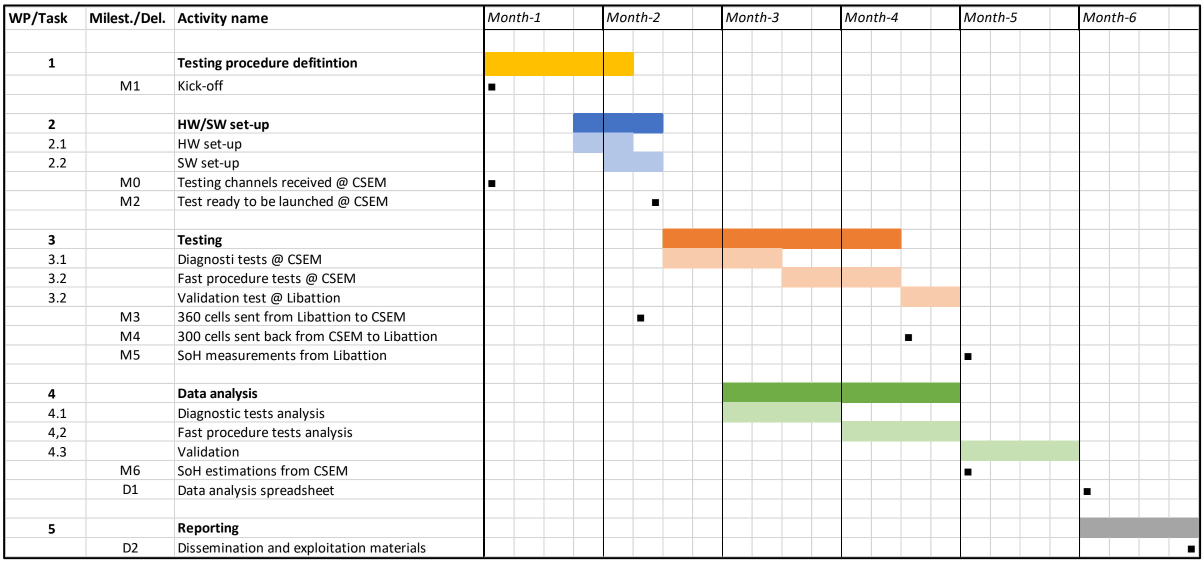


Figure 1 – Initial planning of project BAT4SEL.

A-posteriori, the plan has been respected. Few minor delays, linked to the 2020 Covid19 sanitary situation, delayed the data analysis task. Specifically, the impossibility to hold a project meeting caused a delay in the return of cells from CSEM to Libattion.

The following chapters of the report will detail the followings:

- Chapter 2: description of the testing facility
- Chapter 3: description of the testing methodology.
- Chapter 4 and Chapter 5: presentation of results.
- Chapter 6: Conclusions.
- Chapter 7: outlook and next steps.

2 Description of facility

The main tests have been performed in the 566-Energy Systems laboratory at CSEM. The laboratory is equipped with a battery tester and a thermostatic chamber. The specifications are given in the following:

- The battery tester: Biologic® BCS815 [6]. The tester is equipped with 4 racks of 8 channels, with a total number of 32 channels. 8 channels have been completely dedicated to this project, newly acquired from manufacturer. As regards the performance of the tester, each channel has a maximum current of 15A and maximum voltage of 9V. Besides its high control accuracy (0.01% FSD for the voltage and 0.015% FSD for the current, for each available range), BCS815 allows to run EIS (10kHz – 10mHz) with the same testing machine (i.e. without disconnecting samples and rely on EIS dedicated spectroscopes). All the connections are with 4 wires cables and dedicated cell holders (Biologic BH-1i) that allow the four-points measurement: The signal is less noisy because the power line and measurement line are separated. Each channel also includes the temperature measurement with a K-type thermocouple. The battery tester is connected to a computer and it is controlled by a dedicated software (BT-Lab®). This software allows to build specific testing protocols and to run the same on a singular channel or on multiple channels. This latter option is used to test several cells with the same characteristics or to parallelize a selected number of channels to increase the maximum allowed current (up to 120A).
- The thermostatic chamber: ATT-DM1200T [7]. It has an internal volume of 1200 litres that can be organized in two levels. The temperature can be adjusted in the interval -45°C up to +180°C; a low content of oxygen can be guarantee by constant injection of nitrogen. All the tests have been carried out at 20°C.

All the cells have been tested also at Libattion facilities, with proprietary equipment (Figure 3). These tests have been carried out to compare the results obtained in the two laboratories (i.e. CSEM and Libattion) and to validate the procedure under development. Further details in the following paragraphs.

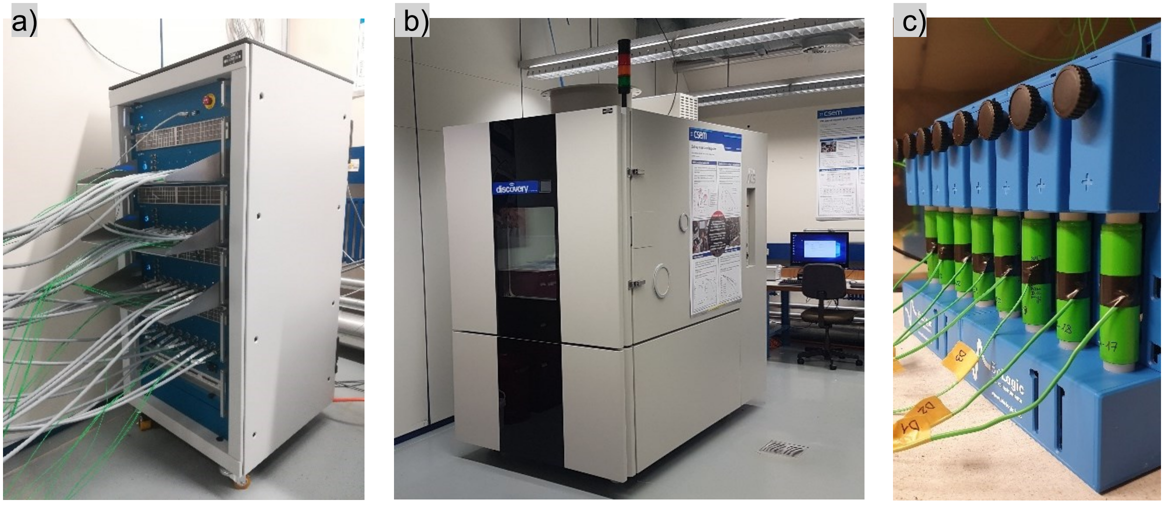


Figure 2 – Overview of equipment and cells in the Energy System laboratory at CSEM. a) Battery tester; b) Thermostatic chamber; c) Cells under testing inside the climatic chamber.

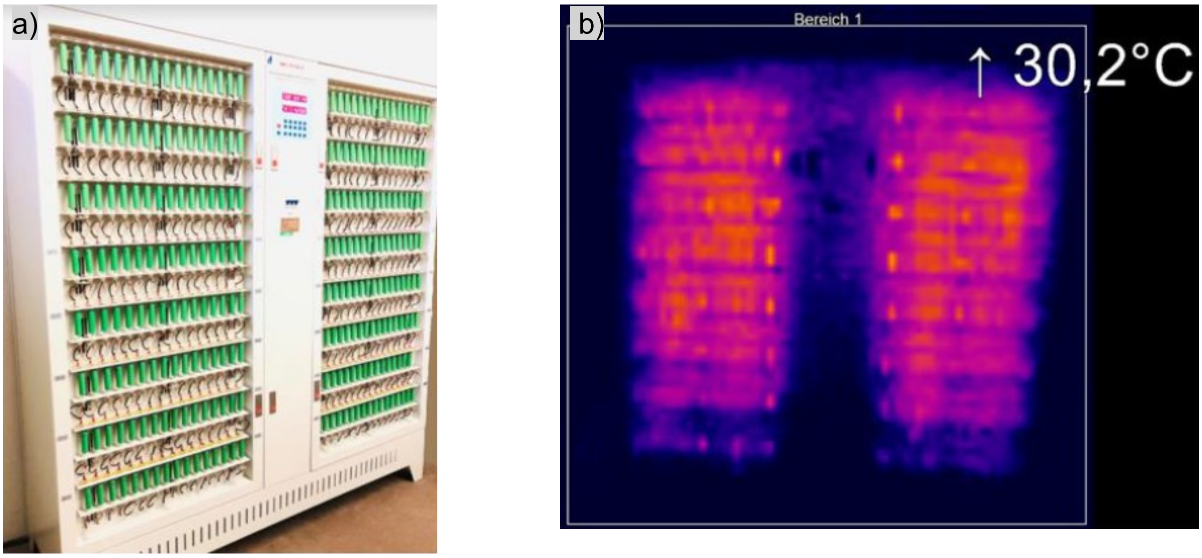


Figure 3 – Overview of equipment and cells at Libattion facilities. a) Battery tester; b) Image taken with thermo-camera to check the temperature of the cells. All the tests are performed at ambient temperature.

3 Procedures and methodology

Above all, a detailed planning of testing activity was needed throughout the project to ensure the project was respecting the initial planning of Figure 1. The planning was linked to the following variables:

- The total number of samples (i.e. cells) to be tested: 523 cells in the BAT4SEL project.
- The number of testing channels available: 8 channels in the BAT4SEL project.
- The time available for testing: 2 months in the BAT4SEL project (as per original plan of Figure 1).

In the next few sub-sections, details about the tested cells, the testing methodology and the different testing protocols will be presented.

3.1 Tested cells

All the Li-ion cells tested in this project are cylindrical cells (Figure 2), format 18650 (18mm diameter; 65mm height). This cell type is the most common in any e-bike battery packs. Four different technologies have been tested, with two different cathode chemistries, NCA and NMC. For the NCA chemistry, the chosen cells are the Sony VC7 [8] and the Sanyo Panasonic GA [9]; for the NMC chemistry the chosen cells are LG Chem MJ1 [10] and Sony NC1 [11]. The main characteristics of the cells have been reported from the correspondent datasheets and are listed in Table 1, together with the total number of samples received for testing.

A reference colour is assigned to each technology, the same colour will be used univocally throughout all the present report.

Table 1: main characteristics of the four different cell technologies tested in this project.

Cell type	A	B	C	D
Cell name	MJ1	VC7	GA	NC1
Manufacturer	LG Chem	Sony	Panasonic	Sony
Chemistry	NMC	NCA	NCA	NMC
Nominal capacity [mAh]	3500	3500	3300	2900
Nominal voltage [V]	3.635	3.6	3.6	3.6
Standard charge current [mA]	1700	1000	1475	1925
Maximum charge current [mA]	3400	-	-	3000
Standard discharge current [mA]	680	-	-	2750
Maximum discharge current [mA]	10000	-	-	8000
Maximum voltage [V]	4.2	4.2	4.2	4.2
Minimum voltage [V]	2.5	2.5	2.5	2.5
Current cut-off [mA]	50	100	67	100
Max Ch. Time [min]		330	270	180
Weight [g]	49	47.2	48	45.3
Samples tested [#]	132	132	132	127

3.2 Cell testing methodology: diagnostic vs. validation tests

The whole testing methodology has been based onto two sub-set of tests: diagnostic tests and validation tests:

- Diagnostic tests: they represent the core of the whole methodology. A representative sample of cells are subjected to several characterization tests with different duration to get all the important performance parameters. This parameters or indicators are then related to the measured SoH to find meaningful correlations.
- Validation tests: a wider sample of cells is tested in two different tests sites with two different purposes:
  - SoH estimation: At CSEM, the cells are subjected to the same fast characterisation tests (as in the diagnostic tests) to compute the indicators and estimate the SoH.
  - SoH measurement: At Libattion, the same cells are tested to measure the SoH and to validate the estimation precision.

Both the diagnostic tests and validation tests are the resulting combination of the same macroscopic testing phases (Figure 4):

1. Phase A: composed of subsequent fast characterization techniques performed at random cell's voltages (i.e. the voltage at which the cell has been received). This test includes resistance measurement, efficiency measurement and EIS measurement.
2. Phase B: composed of the solely SoH measurement. The cell is completely discharged and then a full charge-discharge cycle is applied to measure the actual capacity.
3. Phase C: composed of the same tests of Phase A, but at nominal voltage. The nominal voltage has been fixed to 3.6V for all the four cell technologies.

Practically, for each technology, a reduced number of samples (32 cells) has been randomly selected to make a "control group" to run the diagnostic tests. This control group performed all the three phases of tests, from A to C, as shown in Figure 5. Instead, the remaining cells – "experimental group"- (100 cells) performed a fast test, i.e. only the block A and block C. It should be noted that a voltage regulation phase was necessary to bridge phase A and C. This is done by applying an intermediate charging or discharging phase to bring the cell at nominal voltage (Figure 5).

The details on test duration at CSEM are given with the maximum testing time recorded per channel:

- Diagnostic tests - Phase A + phase B + phase C: duration of about 24h. One shift of 8 cells tested per day resulting in 16 working days (3.2 weeks) of tests with a total number of 128 tested cells.
- Validation tests @ CSEM: Phase A + voltage regulation phase + phase C: duration of about 8h. Two shifts per day resulting in 25 working days (5 weeks) with a total number of 395 tested cells.
- It should be noted that the testing duration was affected by the EIS measurements that can be only multiplexed on the testing equipment.
- Validation tests @ Libattion: Phase B: thanks to the large testing capacity, the results were obtained within a week after receival of cells.

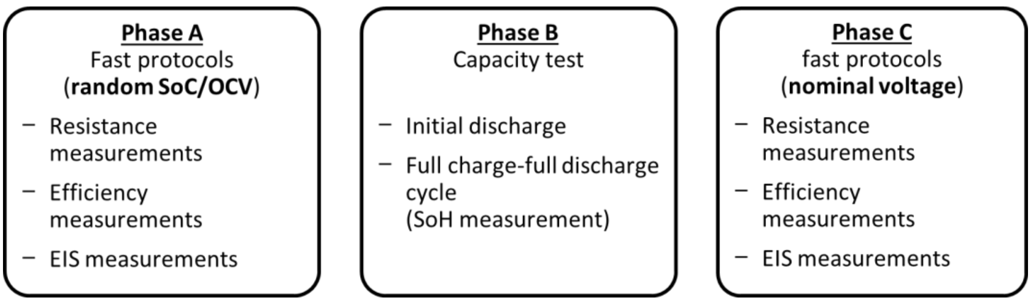


Figure 4 - Representation of the three phases that have been developed to build the testing protocols.

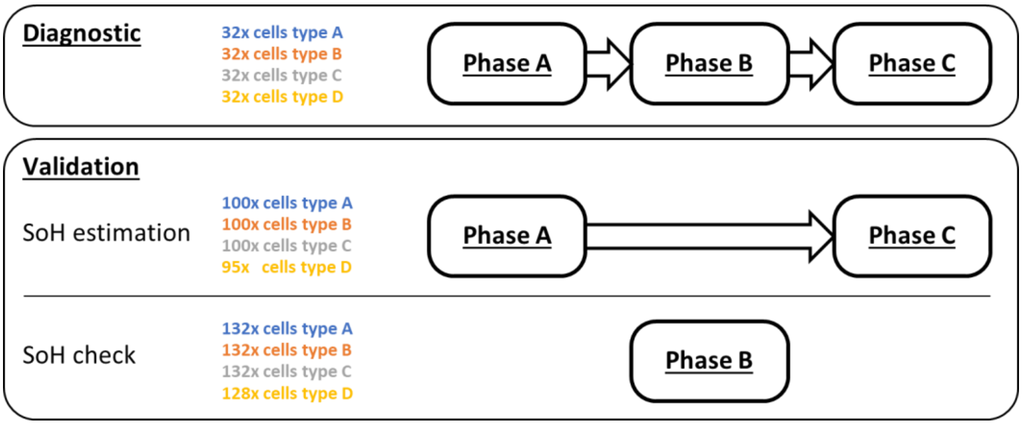


Figure 5 - Combination of the blocks given in Figure 5 to make the testing protocols for diagnostic and validation parts of testing methodology. The arrow entering Phase C implies a voltage regulation phase to reach nominal voltage.

3.3 Cell testing protocols in details

All the tests have been performed at controlled temperature of 20°C respecting two main constraints:

- Voltage limits: following the intervals given by the datasheets, minimum voltage 2.5V and maximum voltage 4.2V.
- Temperature limit of 45°C. This constraint has been fixed by CSEM in agreement with the testing guidelines provided by Libattion.

3.3.1 AC/DC resistance tests

The AC/DC resistance tests are used to measure the internal resistance of the cell.

The AC resistance test is an impedance measurement obtained by exciting the cells with an AC current at a fixed frequency of 1kHz. The Measured AC voltage signal shall have an amplitude of 10 mV, current excitation is derived consequently. The impedance is then derived by relating voltage and current signals. The results of the test are given as the average value of four repeated measurements. The 1kHz impedance value is a very well known in the battery domain, almost all battery manufacturers show this value in their datasheets. This protocol is developed as predefined function in the Biologic tester BT Lab® software. It can be seen also as a single point withing an EIS measurement (more details in section 3.3.3). In the following the resistance measured obtained by this test is depicted as RAC.

The DC resistance test is based on the standard IEC 61960 [12]. The test is performed both in charging and discharging conditions. After initial rest, the cell is discharged with a current rate of 0.2C for 10s and with a higher rate of 1C for 1s. After that the cell goes under rest for 30s. The resistance (RDC) is calculated as follows:

$$RDC = \frac{V_a - V_b}{I_a - I_b} \tag{1}$$

where V_a is the voltage measured at the end of 10s pulse at 0.2C; V_b is the voltage measured at the end of 1s pulse at 1C and I_a and I_b are respectively the current corresponding to 0.2C and 1C.

The same calculation is repeated for the charging phase. Depending on the initial condition of the cell if the voltage is too low or too high one of the two tests is skipped. In detail, the measurement in discharging phase is skipped when the voltage is lower than 3.4V and the measurement in charging phase is skipped when the voltage is higher than 4V.

3.3.2 Efficiency tests

The efficiency tests are performed with repeated symmetric cycles at reduced DoD. CSEM has worked in the past to prove the use of reduced cycles to measure the efficiency of lithium-io cells [13]. Symmetric charging/discharging phases have been used; the same efficiency test is repeated at different DoD windows. The energy efficiency is calculated as follow:

$$EE\ [\%] = \frac{Discharge\ capacity\ [mWh]}{Charge\ capacity\ [mWh]} \cdot 100 \tag{2}$$

3.3.3 EIS tests

The EIS test includes the measurement of the impedance values over a predefined range of signal excitations. EIS is a very powerful method mainly because it is a non-destructive technique to characterize the electrical properties of materials and their interfaces. In a linear system, the response to a sinusoidal input excitation at a given frequency is a sinusoidal output signal at the same frequency with different amplitude and phase. The impedance Z(t) at the given frequency is defined as follow:

$$Z(t) = \frac{E_t(t)}{I_t(t)} = \frac{E_0 \cdot \sin(\omega t)}{I_0 \cdot \sin(\omega t + \varphi)} \tag{3}$$

The time required for measuring an EIS spectrum depends on the number of points measured and on the selected frequency. More than one complete sinusoid is required for each impedance measurement (the number of complete sinusoids depends on the specific instrument and it is in the range of 1-5). On average, the measurement requires around 15 minutes.

3.3.4 Capacity test

This test includes full charge-discharge cycle to determine the residual capacity (i.e. SoH measurement). The specific protocol of this test has been agreed between CSEM and Libattion based on previous experience at Libattion on the chosen cell technologies. It is based on full charge (constant current-constant voltage) and discharge (constant current) of the cell.

The equations to calculate initial SoC and SoH are given in Table 2.

Table 2: variables calculated by the parameters listed in **Error! Reference source not found.** and their equations.

Name of variable	Equation
Initial State of Charge	$Initial\ SoC\ [\%] = \frac{Initial\ discharge\ capacity\ [mAh]}{Full\ discharge\ capacity\ [mAh]} \cdot 100$
State of Health	$SoH\ [\%] = \frac{Full\ discharge\ capacity\ [mAh]}{Nominal\ capacity\ [mAh]} \cdot 100$

3.4 Data exporting and indicators to estimate SoH

Given the large number of cells tested, the amount of experimental data generated was extremely large. Raw data files were produced by the battery tester Software for every single cell test. Specific python routines have been developed to analyse the raw data and extract the relevant variables for the project purposes. These extracted data have been directly organized in specific Office Excel™ spreadsheet to visualise the main variables and to control if tests show strange behaviour (i.e. voltage limits, temperature limits, etc.). In the next few sub-sections more details about the extracted and/or computed variables are presented.

The indicators to estimate SoH have been selected between all the available variables analysing the most suitable and reliable correlations. Three indicator types have been selected:

- Resistance-based indicators (R-based).
- Efficiency-based indicators (EE-based).
- EIS-based indicators (EIS-based).

4 Diagnostic tests result and discussion

As presented in Chapter 0, the whole SoH estimation process is based on the investigation of existing correlations between the measured indicators (i.e. the values resulting from the different testing procedures applied on the cells) and the measured SoH. This is done during the diagnostic tests, which account for phase A (fast procedures at random voltage), phase B (capacity measurements), phase C (fast procedures at nominal voltage). For time reasons, the correlations are investigated on a small "control group" of 32 cells for each technology. The found correlations will be then used on a "experimental group" of 100 cells for each technology to estimate the SoH which will be then validated from a standard SoH measurement by a third party (i.e. Libattion).

In some of the following sub-sections of the present Chapter, the case of the technology type B and the energy efficiency-based indicator will be used as an example. The objective is to present the rationale of the SoH estimation methodology, which is composed by the following parts:

- Preliminary check: this serves to evaluate the status of the cells (e.g. over-charged, over-discharged, etc.).
- SoH measurements: these serve to evaluate the aging status of the cells.
- Correlation assessment: this is the core of the procedure where the correlations are investigated.

4.1 Preliminary check of tested cells

The preliminary check of tested cells is done by analysing the initial voltage done at the beginning of the tests. The results are shown in Figure 6 and the averaged, maximum, and minimum voltage values are listed in Table 3 per each cell type. Most of the cells show an initial voltage between 3.4V and 4.2V, meaning that the battery packs are usually disposed at medium or high SoC. Type A and type B cells have similar averaged voltage, respectively 3.82V and 3.77V. Type C instead shows an averaged voltage of 4.12V, very close to the maximum value. This means that almost all the cells are fully charged. On the contrary, type D cells show the lowest averaged voltage (3.59V) due to a high number of cells fully discharged, with a voltage lower than 3.4V, one sample at 2.82V.

These preliminary observations allow us to anticipate that it is likely that the SoH estimation of type C and type D cells will be impacted respectively by the very high and low averaged voltage values measured. However, in the case of type C the values are uniform, while in the case of type D the values are more dispersed: so that, the impact on SoH estimation could be worst for type D cells.

For this reason, a filtering option will be super-imposed on the measured indicators to verify if the correlations improve when excluding those cells which show extreme values of initial voltages (see section 4.3 for more details).

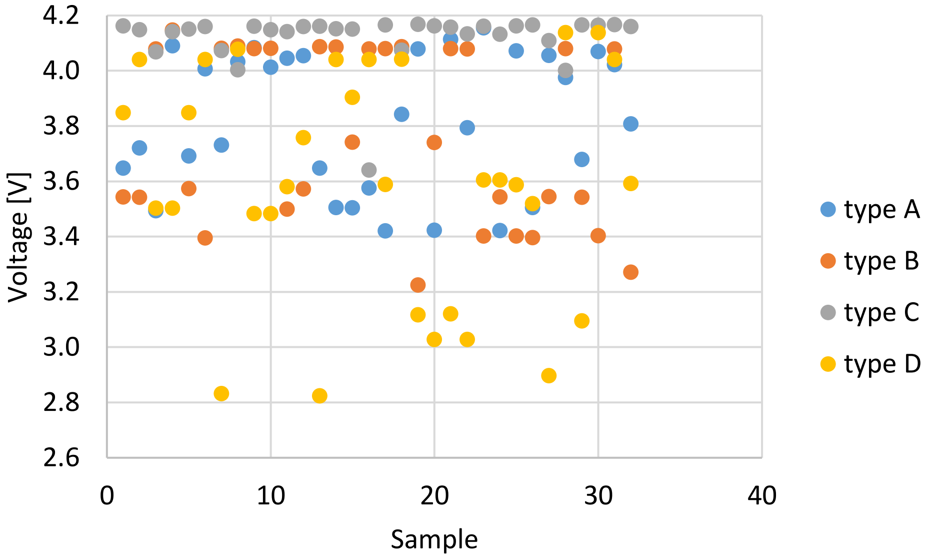


Figure 6 – Initial voltage of the cells under diagnostic test - control group – 32 sample for each technology: MJ1 (type A), VC7 (type B), GA (type C) and NC1 (type D).

Table 3: averaged, maximum and minimum voltage of the cells under diagnostic tests for each technology: MJ1 (type A), VC7 (type B), GA (type C) and NC1 (type D)

	Test type	Mean(V_{ini})	Max(V_{ini})	Min(V_{ini})
Type A (MJ1)	Diagnostic (32 samples)	3.82 V	4.16 V	3.42 V
Type B (VC7)	Diagnostic (32 samples)	3.77 V	4.15 V	3.23 V
Type C (GA)	Diagnostic (32 samples)	4.12 V	4.17 V	3.64 V
Type D (NC1)	Diagnostic (32 samples)	3.59 V	4.14 V	2.82 V

4.2 SoH measurement

The SoH values have been computed by using the expression given in Table 2 and they are shown in Figure 7. The averaged, maximum, and minimum SoH values of each cell technology are listed in Table 4. Looking at type B, C and D cells all the samples have SoH higher than 85%, except for one cell at 73% (type B). The averaged SoH is 91% for type B, 95% for type C and 90% for type D. On the contrary, type A cells have lower SoH values, mostly around 80-90% with an averaged value of 87%.

These preliminary observations allow us to conclude that the tested samples are surely aged cells but not second life cells. Usually second-life cells are defined for SoH < 80%, parameter which was relaxed to 85% for the present project. This is an advantage from the point of view of re-using cells with still a very high residual capacity, but it is a disadvantage from the point of view of studying the given population to find feasible correlations all over a wide range of possible SoH.

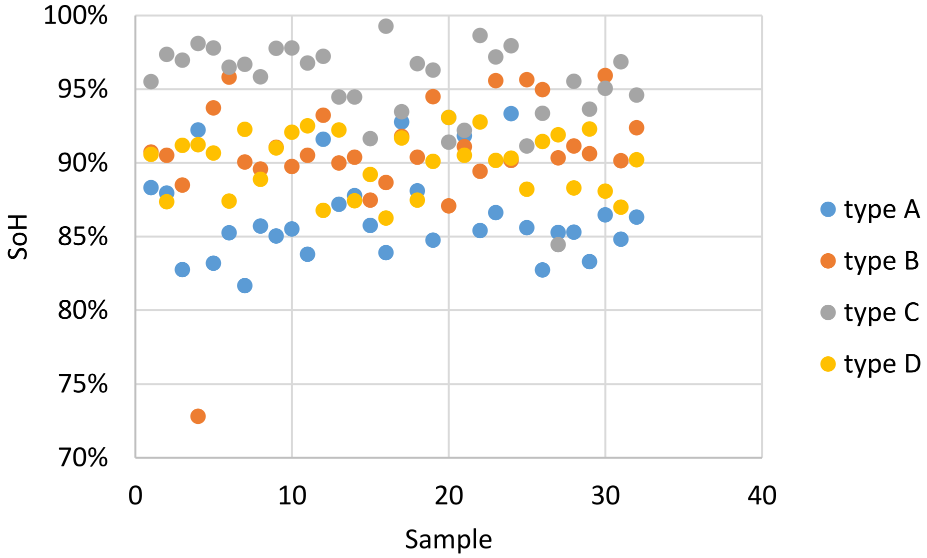


Figure 7 – Measured SoH under diagnostic test - control group – 32 sample for each technology:
MJ1 (type A), VC7 (type B), GA (type C) and NC1 (type D).

Table 4: averaged, maximum, and minimum SoH value of the cells under diagnostic tests for each technology: MJ1 (type A), VC7 (type B), GA (type C) and NC1 (type D)

	Test type	Mean(SoH)	Max(SoH)	Min (SoH)
Type A (MJ1)	Diagnostic (32 samples)	86.68%	93.35%	81.67%
Type B (VC7)	Diagnostic (32 samples)	90.76%	95.93%	72.82%
Type C (GA)	Diagnostic (32 samples)	95.41%	99.28%	84.46%
Type D (NC1)	Diagnostic (32 samples)	90.03%	93.08%	86.26%

4.3 Correlation assessment

As already stated at the beginning of present chapter, the correlation assessment will be detailed for a specific case: Chemistry type B – EE-based indicator.

For every single indicator and technology type, the correlation assessment has been repeated for three different conditions:

1. Random voltage: to assess the presence of any correlations without having to perform any initial SoC regulation on the cells. This means that the cells are tested as they have been received. This condition leads to a faster indicator assessment.
2. Nominal voltage: to assess the presence of any correlations when an initial SoC regulation is performed. This means that all cells are brought to the same SoC or voltage levels before starting the indicator assessment.
   This condition leads to a faster indicator assessment.
3. Random voltage filtered: to assess the presence of any correlations without having to perform any initial SoC regulation but discarding cells which show undesirable initial conditions.
   This condition leads to a timing compromise with respect to condition 1 and condition 2.

With respect to the latter, the filtering of cells has been based on the cell's initial voltage (Table 3). Thresholds have been set for OCV values correspondent to initial SoC > 95% and SoC < 20%: 4.15V and 3.52V respectively. Outside this SoC values, any Li-ion cells show a clear non-linear behaviour which impact both on resistance and efficiency. Exceptions at this rule have been set for Type C and Type D because of their specific average initial states (i.e. not having to discard all cells to respect the filtering conditions): (i) cell of type C have been found to be fully charged on average, therefore the maximum allowed voltage has been relaxed to 4.2V (SoC ≈ 100%); (ii) cell of type C have been found to be deeply discharged on average, therefore the minimum allowed voltage has been relaxed to 3.4V (SoC < 5%).

Table 5: filtering conditions on the cell's initial voltage for each technology:
MJ1 (type A), VC7 (type B), GA (type C) and NC1 (type D)

	Test type	Max(V_{ini})	Min(V_{ini})
Type A (MJ1)	Diagnostic (32 samples)	4.15 V	3.52 V
Type B (VC7)	Diagnostic (32 samples)	4.15 V	3.52 V
Type C (GA)	Diagnostic (32 samples)	4.20 V	3.52 V
Type D (NC1)	Diagnostic (32 samples)	4.15 V	3.40 V

4.3.1 Random voltage

Table 6 shows the results of the efficiency tests carried out at random voltage.

Table 6: Efficiency tests results at random voltage for cell type B (VC7)

Cell type	SoH	Charge capacity		Discharge capacity		EE-based indicator
B	[%]	[mAh]	[mWh]	[mAh]	[mWh]	[%]
B-1	90.75%	35.01	127.63	34.98	121.27	95.10%
B-2	90.52%	35.01	127.54	34.98	121.11	95.06%
B-3	88.51%	35.01	146.03	34.99	139.75	95.77%
B-4	72.82%	35.02	144.80	34.97	136.12	94.13%
B-5	93.75%	35.01	128.27	34.98	122.78	95.82%
B-6	95.83%	35.01	123.29	34.99	117.15	95.09%
B-7	90.06%	35.01	145.89	34.99	140.05	96.07%
B-8	89.60%	35.00	140.78	35.00	135.25	96.07%
B-9	91.06%	35.01	146.03	35.02	140.10	95.91%
B-10	89.76%	35.02	145.96	35.02	140.13	96.00%
B-11	90.52%	35.01	126.26	35.04	119.89	94.90%
B-12	93.24%	35.02	128.27	35.02	122.81	95.73%
B-13	90.00%	35.02	140.82	34.98	135.18	96.10%
B-14	90.40%	35.01	140.79	34.98	135.19	96.11%
B-15	87.49%	35.01	134.69	35.03	127.90	94.90%
B-16	88.69%	35.00	140.82	34.99	135.13	95.97%
B-17	91.81%	35.01	146.04	35.02	140.08	95.89%
B-18	90.39%	35.01	140.77	34.97	135.17	96.13%
B-19	94.51%	35.02	118.72	35.03	110.99	93.45%
B-20	87.09%	35.02	134.67	35.02	127.79	94.90%
B-21	91.11%	35.02	146.01	35.02	140.06	95.92%

Cell type B	SoH [%]	Charge capacity [mAh]	Charge capacity [mWh]	Discharge capacity [mAh]	Discharge capacity [mWh]	EE-based indicator [%]
B-22	89.44%	35.02	146.09	35.02	139.94	95.77%
B-23	95.58%	35.01	123.36	35.03	117.28	95.02%
B-24	90.17%	34.99	127.56	35.04	121.38	95.02%
B-25	95.66%	35.01	123.40	35.02	117.29	95.02%
B-26	94.97%	35.01	123.33	35.01	117.20	95.03%
B-27	90.35%	35.02	127.60	35.03	121.39	95.09%
B-28	91.16%	35.02	146.00	35.02	140.01	95.90%
B-29	90.63%	35.02	127.53	35.02	121.33	95.12%
B-30	95.93%	35.02	123.36	35.04	117.35	95.07%
B-31	90.15%	35.02	145.84	35.03	140.19	96.09%
B-32	92.39%	35.00	119.99	35.04	112.88	93.95%

The objective of the analysis is to look for the presence of any correlation between the SoH and the chosen indicator (i.e. energy efficiency EE in this case). Figure 8 shows the values measured on the 32 cells (control group) of technology B.

The correlation is measured with the following equation:

$$r = \sum_{i=1}^{n} \frac{\frac{(x_i - \bar{x})(y_i - \bar{y})}{n-1}}{s_x s_y} \tag{4}$$

If |*r*|> 0.5 the correlation exists. The closest to unit, the better it is.

if |*r*|< 0.5 the correlation does not exist.

Moreover, two conditions can be verified:

- *r* is positive: a positive correlation is found. This means that when SoH is larger than average, the indicators tends to be larger than average, and vice versa.
  This is an expected behaviour for the case of efficiency-related indicators.
- *r* is negative, a negative correlation is found. This means that when SoH is larger than average, the indicators tends to be smaller than average, and vice versa
  This is an expected behaviour for the case of resistance-related indicators.

The analysis can proceed only if two requirements are met contemporarily on the correlation factor *r*: (i) |*r*|> 0.5, (ii) the correlation shows the expected behaviour.

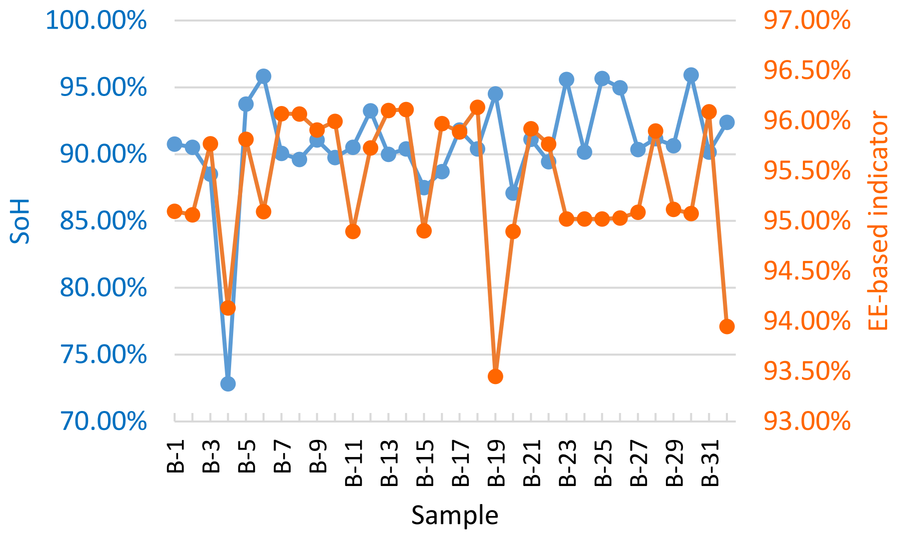


Figure 8 – assessed variables trends at random voltage for cell type B (VC7)

For the case of Figure 8, for instance, the correlation factor is r = 0.07, which is respecting requirement (ii) but not requirement (i). Therefore, the analysis process shall stop and the measured variable (i.e. energy efficiency measured at random voltage) cannot be used as indicator to estimate the SoH.

4.3.2 Nominal voltage

As done in previous section, the same analysis is repeated also for efficiency tests at nominal voltage (procedure presented in section 3.3.2). Table 7 shows the extracted measurements.

Table 7: Efficiency tests results at nominal voltage for cell type B (VC7)

Cell type	SoH	Charge capacity		Discharge capacity		EE-based indicator
B	[%]	[mAh]	[mWh]	[mAh]	[mWh]	[%]
B-1	90.75%	35.01	129.12	34.98	122.67	95.10%
B-2	90.52%	35.01	129.09	34.98	122.59	95.06%
B-3	88.51%	35.01	128.78	34.99	122.91	95.51%
B-4	72.82%	35.02	129.87	34.98	121.91	94.00%
B-5	93.75%	35.02	128.68	34.98	123.15	95.81%
B-6	95.83%	35.01	128.65	34.99	123.16	95.81%
B-7	90.06%	35.01	128.84	34.98	123.08	95.62%
B-8	89.60%	35.00	128.70	34.99	123.02	95.61%
B-9	91.06%	35.01	128.84	35.02	123.34	95.71%
B-10	89.76%	35.01	128.88	35.00	123.10	95.55%
B-11	90.52%	35.01	128.98	35.04	122.92	95.24%
B-12	93.24%	35.02	128.68	35.02	123.22	95.74%
B-13	90.00%	35.02	128.85	35.02	123.11	95.55%
B-14	90.40%	35.02	128.77	35.03	123.22	95.64%
B-15	87.49%	35.01	129.46	35.03	122.22	94.37%
B-16	88.69%	35.00	128.73	35.04	123.07	95.49%
B-17	91.81%	35.01	128.79	35.03	123.34	95.73%
B-18	90.39%	35.01	128.80	35.01	123.13	95.60%

Cell type	SoH	Charge capacity		Discharge capacity		EE-based indicator
B	[%]	[mAh]	[mWh]	[mAh]	[mWh]	[%]
B-19	94.51%	35.01	128.59	35.03	123.30	95.84%
B-20	87.09%	35.02	129.45	35.02	122.13	94.35%
B-21	91.11%	35.02	128.71	35.01	123.22	95.75%
B-22	89.44%	35.02	128.84	35.03	123.14	95.53%
B-23	95.58%	35.01	128.64	35.03	123.29	95.78%
B-24	90.17%	34.99	129.00	35.04	122.80	95.05%
B-25	95.66%	35.01	128.70	35.02	123.30	95.78%
B-26	94.97%	35.02	128.66	35.02	123.27	95.80%
B-27	90.35%	35.02	129.05	35.04	122.78	95.09%
B-28	91.16%	35.02	128.67	35.02	123.16	95.71%
B-29	90.63%	35.02	129.07	35.02	122.78	95.13%
B-30	95.93%	35.02	128.65	35.03	123.33	95.84%
B-31	90.15%	35.01	128.81	35.03	123.24	95.63%
B-32	92.39%	35.00	128.70	35.04	123.12	95.54%

In this case, the correlation factor is r = 0.78, which respect both requirements for the analysis to continue. Figure 9 also confirms the goodness of the correlation. However, it should always be remembered that this is coming at the detriment of the timing required to run the tests.
If compared with Figure 8, the two variables (i.e. SoH and energy efficiency) are showing clear symmetrical trends, resulting from an existing positive correlations. There exist few deviations to the general trends (e.g. sample B-1 and B-2) and this will have to be assessed in terms of impact onto the SoH estimation precision (see Chapter 5).

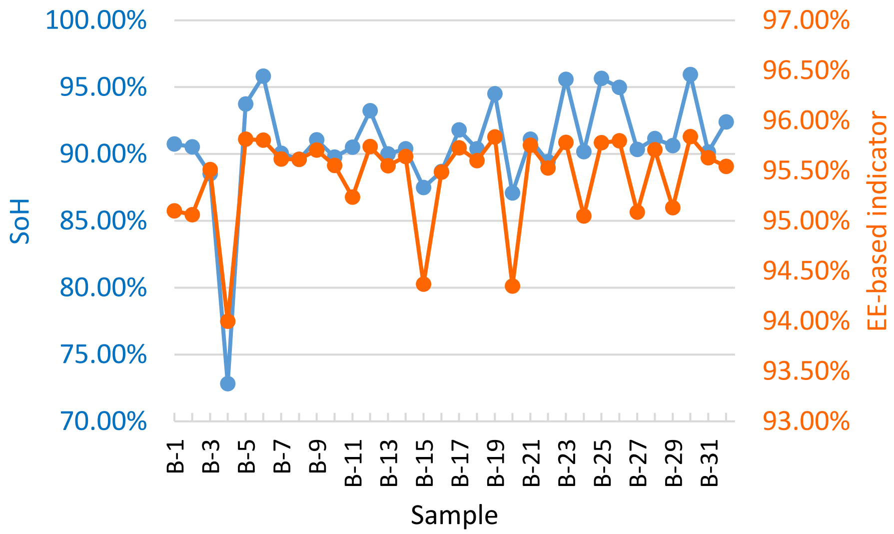


Figure 9 – assessed variables trends at nominal voltage for cell type B (VC7)

As said, in this case the analysis can proceed to find the best fit between the indicator and the SoH. A linear fit is chosen, and the parameters given by the fitting equation are stored for the SoH estimation.

4.3.3 Random voltage (filtered)

Finally, the last step is to assess the presence of any correlations without having to perform any initial SoC regulation but discarding cells which show undesirable initial conditions. As per Table 5 and for the case of technology B, this means that all cells which show initial voltage above 4.15V or below 3.52V, should be kept out from the analysis.

Table 8 shows how this is working in the case under analysis. 7 cells out of 32 (i.e. the 22% of the total) will not be used in the correlation analysis.

Table 8: Efficiency tests results at random voltage (filtered) for cell type B (VC7)

Cell type	SoH_C	Charge capacity		Discharge capacity		EE- based indicator	V_{ini}
B	[%]	[mAh]	[mWh]	[mAh]	[mWh]	[%]	[V]
B-1	90.75%	35.01	127.63	34.98	121.27	95.10%	3.54
B-2	90.52%	35.01	127.54	34.98	121.11	95.06%	3.54
B-3	88.51%	35.01	146.03	34.99	139.75	95.77%	4.08
B-4	72.82%	35.02	144.80	34.97	136.12	94.13%	4.15
B-5	93.75%	35.01	128.27	34.98	122.78	95.82%	3.57
~~B-6~~	~~95.83%~~	~~35.01~~	~~123.29~~	~~34.99~~	~~117.15~~	~~95.09%~~	~~3.40~~
B-7	90.06%	35.01	145.89	34.99	140.05	96.07%	4.08
B-8	89.60%	35.00	140.78	35.00	135.25	96.07%	4.09
B-9	91.06%	35.01	146.03	35.02	140.10	95.91%	4.08
B-10	89.76%	35.02	145.96	35.02	140.13	96.00%	4.08
~~B-11~~	~~90.52%~~	~~35.01~~	~~126.26~~	~~35.04~~	~~119.89~~	~~94.90%~~	~~3.50~~
B-12	93.24%	35.02	128.27	35.02	122.81	95.73%	3.57
B-13	90.00%	35.02	140.82	34.98	135.18	96.10%	4.09
B-14	90.40%	35.01	140.79	34.98	135.19	96.11%	4.09
B-15	87.49%	35.01	134.69	35.03	127.90	94.90%	3.74
B-16	88.69%	35.00	140.82	34.99	135.13	95.97%	4.08
B-17	91.81%	35.01	146.04	35.02	140.08	95.89%	4.08
B-18	90.39%	35.01	140.77	34.97	135.17	96.13%	4.09
B-19	94.51%	35.02	118.72	35.03	110.99	93.45%	3.23
B-20	87.09%	35.02	134.67	35.02	127.79	94.90%	3.74
B-21	91.11%	35.02	146.01	35.02	140.06	95.92%	4.08
B-22	89.44%	35.02	146.09	35.02	139.94	95.77%	4.08
~~B-23~~	~~95.58%~~	~~35.01~~	~~123.36~~	~~35.03~~	~~117.28~~	~~95.02%~~	~~3.40~~
B-24	90.17%	34.99	127.56	35.04	121.38	95.02%	3.54
~~B-25~~	~~95.66%~~	~~35.01~~	~~123.40~~	~~35.02~~	~~117.29~~	~~95.02%~~	~~3.40~~
~~B-26~~	~~94.97%~~	~~35.01~~	~~123.33~~	~~35.01~~	~~117.20~~	~~95.03%~~	~~3.40~~
B-27	90.35%	35.02	127.60	35.03	121.39	95.09%	3.54
B-28	91.16%	35.02	146.00	35.02	140.01	95.90%	4.08
B-29	90.63%	35.02	127.53	35.02	121.33	95.12%	3.54
~~B-30~~	~~95.93%~~	~~35.02~~	~~123.36~~	~~35.04~~	~~117.35~~	~~95.07%~~	~~3.40~~
B-31	90.15%	35.02	145.84	35.03	140.19	96.09%	4.08
~~B-32~~	~~92.39%~~	~~35.00~~	~~119.99~~	~~35.04~~	~~112.88~~	~~93.95%~~	~~3.27~~

Thanks to the filtering out of some of the cells, the correlation factor greatly improves to r = 0.62, which is worse than the nominal voltage case, but surely better than the random voltage case without any filtering. Most importantly, the correlation factor respects both requirements for the analysis to proceed.

As already stated, the result improvements come directly from the filtering out of some cells. Practically, the "discarded cells" can be interpreted in two ways, accordingly to the different objectives:

- On-hold cells: if the objective is to scrap the least number of cells as possible, the filtered-out cells shall be charge or discharged before including them into the correlation analysis (e.g. bringing them between the same voltage limits).
  However, it must be acknowledged that cells which are found in extreme voltage conditions, which were potentially stored in that conditions for long period, will most probably show high non-linear behaviour if compared to correctly treated cells. This will surely have an impact on the correlation analysis and ultimately on the SoH estimation precision.
- Scrapped cells: if the objective is to perform the fastest assessment as possible, one can think of applying this filtering triage to select which cells to maintain and which cell to scrap. The remaining cells can be then used to assess correlation (diagnosis phase), which should then be applied to cells which follow the same filtering rules.

4.4 Discussion

The full analysis explained in section 4.3 has been applied to all indicators (i.e the three typologies: R-based, EE-based and EIS-based indicators) and to all technologies. Two KPIs were analysed: the success rate (i.e., the percentage of found correlation over the total tested) and the average correlation factor. When a meaningful correlation has been found (i.e. $|r|> 0.5$ and sign of r as expected), the parameters of the best-fit linear functions have been used for SoH estimation in the validation phase.
Regardless from the specific assessment condition, Table 9 presents an aggregated analysis about the found correlations. The main findings are the following:

- Tests results on technology D did not return any good correlation between any of the tested indicators and the measured SoH. When the correlation factor was found to be $|r|> 0.5$, the correlation was not showing the correct behaviour.
- Technology C has been found to give the best correlation performances. These cells were found to be all at very similar conditions: SoH > 90% and V_{ini} > 4.1 (see Table 3 and Table 4). Most probably they were used in similar way along their lifetime.
- Correlations were found for all technologies with an average success rate of 65% and an average correlation factor of 0.73.
- No indicator typology is showing the best results for all the technologies:
  - The resistance-based indicators are showing the best success rate on average, 78%.
  - This means that R-based indicators might be the best one to be used if one would like to have the highest probability to find correlations in a new technology to be tested.
  - The EIS-based indicators are showing the best correlation factor on average, 0.77.
  - The EIS-based indicators might be the best one to be used if one would like to have the most accurate results in terms of SoH estimation, at the expense of the testing time.
  - The efficiency-based indicators are the only providing successful correlation rates in random test conditions for all the three technologies.
  - This means that the EE-based indicators might be the most robust to be used among the different chemistries if one would like to speed-up the testing procedures as much as possible.

It is therefore necessary to analyse the different indicators in the light of their performances in estimating the SOH.

Table 9: Analysis of the correlation found for the different tested technologies.

	Success rate [%]	|mean(*r*)|
Type A (MJ1)		
R-based indicators	67 %	0.61
EE-based indicators	67 %	**0.76**
EIS-based indicators	**78 %**	0.65
Weighted average	70 %	0.67
Type B (VC7)		
R-based indicators	**67 %**	0.74
EE-based indicators	50 %	0.75
EIS-based indicators	56 %	**0.79**
Weighted average	57 %	0.76
Type C (GA)		
R-based indicators	**100 %**	0.75
EE-based indicators	67 %	0.66
EIS-based indicators	33 %	**0.88**
Weighted average	67 %	0.76
Type D (NC1)	-	-
Average (all tech.)		
R-based indicators	**78 %**	0.70
EE-based indicators	61 %	0.72
EIS-based indicators	56 %	**0.77**
Weighted average (all indicators)	65 %	0.73

As regards of the three different assessment conditions (i.e. random voltage, nominal voltage, random voltage but with filtering option), the main findings were:

1. Random voltage tests were showing correlations only in the 25% of the cases (i.e. all indicators and all technologies considered). The average correlation factor was found to be $r = 0.66$.
2. Nominal voltage tests, as expected, were showing better results: correlations were found in the 68% of the cases with a higher average correlation factor $r = 0.76$.
3. The filtering option almost double the success rate in random voltage tests to 43%, also increasing the average correlation factor to $r = 0.73$.

Given the above findings, it is quite clear that tests at random voltage rarely provide a correlation and when a correlation is provided, it is never confirmed for all the tested technologies. For this reason, the other two testing conditions must be carefully evaluated. However, in the case of the nominal voltage condition, validation results are needed to understand if the better correlation factor are directly translated into much higher SoH estimation performances; only in this case the higher testing time required can be counterbalanced. If this is not the case, the practice of filtering-out some badly conditioned cells might provide a compromise alternative to the previous one.

5 Validation test results and discussion

As presented in Chapter 0, the validation of the SoH estimation process is run on “experimental groups” of 100 cells for each technology. The validation process is done in three steps:

1. SoH measurements: that is to apply phase B test onto the “experimental groups”. These tests have been carried out at Libattion.
2. SoH estimation: that is to estimate the SoH by applying the found correlations of **Error! Reference source not found.** over the indicators measured onto the “experimental groups”. These measurements have been carried out at CSEM.
3. Assess the SoH estimation error: that is to quantify the difference between point 1 and point 2.

As regards of point 2, it should be noted that some testing issues occurred at Libattion facilities, which reduced the testing capabilities only on the technology A.

5.1 Preliminary check of tested cells

As in the case of diagnostic tests, a preliminary check of the cells has been done on the initial voltage. The results of the measurements that have been performed at CSEM are shown in Figure 8. The averaged, maximum, and minimum voltage values observed are listed in Table 10 for each different cell technology. This also includes the samples that performed diagnostic tests with the aim of comparing the two groups of cells. In general, all the observation made in Section 4.1 are also applicable here: the control groups are well representing the whole population of cells per each different cell technology. Type A and Type B cells have a uniform distribution around the averaged value, with most of the voltage values higher than 3.2V. As regards type C, the cells have a uniform distribution at high voltage and in a very small interval: except for 3 samples, all the measurements are between 4.10V and 4.16V. Finally type D cells have a large dispersion of voltage values, in between 2.82V and 4.14V.

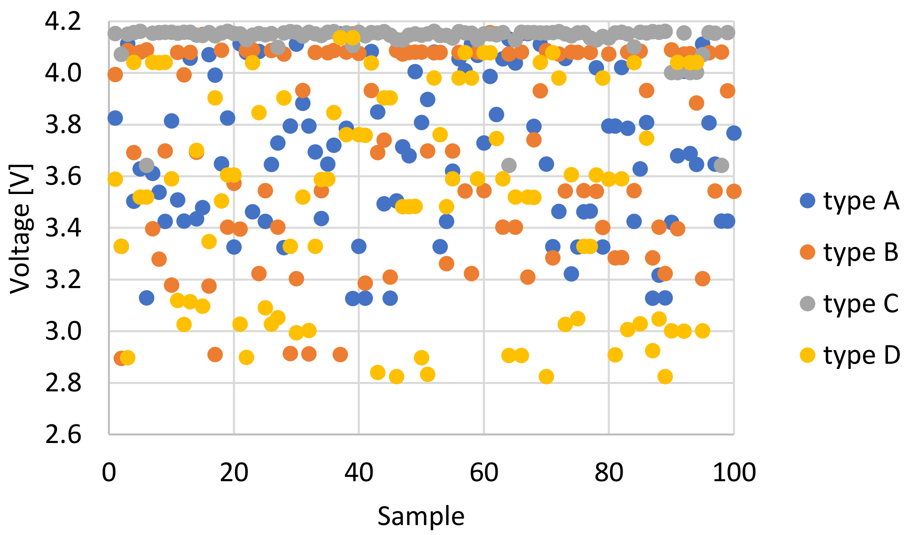


Figure 10 – Initial voltage of the cells under validation test - experimental group – 100 sample for each technology: MJ1 (type A), VC7 (type B), GA (type C) and 95 cells of NC1 (type D).

Table 10: averaged, maximum and minimum voltage of the cells under diagnostic tests and under validation test at CSEM for each technology: MJ1 (type A), VC7 (type B), GA (type C) and NC1 (type D)

	Test type	Mean(V_{ini})	Max(V_{ini})	Min(V_{ini})
Type A (MJ1)	Diagnostic (32 samples)	3.82	4.16	3.42
	Validation (100 samples)	3.70	4.15	3.13
Type B (VC7)	Diagnostic (32 samples)	3.77	4.15	3.23
	Validation (100 samples)	3.73	4.16	2.90
Type C (GA)	Diagnostic (32 samples)	4.12	4.17	3.64
	Validation (100 samples)	4.13	4.16	3.64
Type D (NC1)	Diagnostic (32 samples)	3.59	4.14	2.82
	Validation (95 samples)	3.50	4.14	2.82

5.2 SoH measurements

As anticipated, the SoH measurements on the “experimental groups” should be done by a third party. In the case of the BAT4SEI project, it has been carried out by Libattion.

In addition to that, the “control group”, which was originally tested by CSEM, has been also tested by Libattion. This allows CSEM to verify the presence of any difference between the obtain measurements at the two different testing facilities. Table 11 shows the obtained results. It should be noted that the above-mentioned issues on Type A has halved the control group for that technology.

Table 12 shows that there exist not-negligible differences (maximum difference are up to 4%) between the two testing facilities. In order to reduce the impact on the SoH estimation process, the average ΔSoH has been netted over the original measurements from Libattion. This allowed to derive the closest proxy of the hypothetical SoH measurements at CSEM premises. The following validation analyses are based on this assumption.

Table 11: SoH measurements obtained at CSEM and Libattion on the “control groups” of the tested technologies.

	Test type: Diagnostic	Mean(SoH)	Max(SoH)	Min (SoH)
Type A (MJ1)	CSEM (32 samples)	86.68%	93.35%	81.67%
	Libattion (16 samples)	90.03%	95.40%	82.91%
	CSEM (16 samples)	88.89%	93.35%	82.76%
Type B (VC7)	CSEM (32 samples)	90.76%	95.93%	72.82%
	Libattion (32 samples)	90.97%	94.91%	73.09%
Type C (GA)	CSEM (32 samples)	95.41%	99.28%	84.46%
	Libattion (32 samples)	96.66%	100.36%	83.73%
Type D (NC1)	CSEM (32 samples)	90.03%	93.08%	86.26%
	Libattion (32 samples)	92.22%	95.31%	88.07%

Table 12: SoH deltas between CSEM and Libattion from the “control groups” of the tested technologies.

	Test type	Mean(ΔSoH)	σ(ΔSoH)	Max(ΔSoH)	Min (ΔSoH)
Type A (MJ1)	Diagnostic (16 samples)	-1.13%	1.12%	-3.62%	1.08%
Type B (VC7)	Diagnostic (32 samples)	-0.21%	0.40%	-1.24%	1.20%
Type C (GA)	Diagnostic (32 samples)	-1.26%	0.38%	-2.11%	0.73%
Type D (NC1)	Diagnostic (32 samples)	-2.19%	0.83%	-4.08%	-0.75%

Figure 11 shows the corrected SoH values on the “experimental groups” (i.e. cells which undertake the phase B of validation tests at Libattion). The averaged, maximum and minimum SoH values of each cell technology are listed in Table 13 and compared with the “control groups” (i.e. cells which undertake the phase B of diagnostic tests at CSEM).

It can be concluded, that the chosen “control groups” (i.e. a randomly selected samples) are well representative of the population of the same technology. However, it is also confirmed by test on the validation samples that the tested cells are not second life cells for most of the cases. Usually, second-life cells are defined for SoH < 80%.

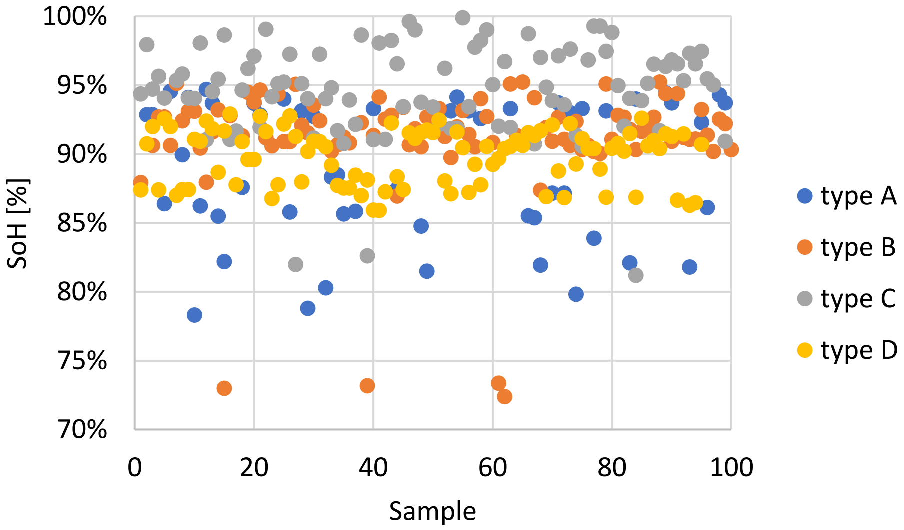


Figure 11 – Measured SoH under validation test - experimental group -: 54 cells of MJ1 (type A), 96 cells of VC7 (type B), 97 cells of GA (type C), and 95 cells of NC1 (type D).

Table 13: comparison of average, maximum and minimum SoH values between the cells under validation tests and cells under diagnostic tests.

	Test type	Mean(SoH)	Max(SoH)	Min (SoH)
Type A (MJ1)	Diagnostic (32 samples)	86.68%	93.35%	81.67%
	Validation (54 samples)	89.05%	94.70%	78.32%
Type B (VC7)	Diagnostic (32 samples)	90.76%	95.93%	72.82%
	Validation (96 samples)	91.15%	94.70%	72.39%
Type C (GA)	Diagnostic (32 samples)	95.41%	99.28%	84.46%
	Validation (97 samples)	94.74%	100.38%	81.20%

Type D (NC1)	Diagnostic (32 samples)	90.03%	93.08%	86.26%
	Validation (95 samples)	89.80%	92.91%	85.91%

5.3 SoH estimation results

As done in Chapter 0, the case of the technology type B and the EE-based indicator will be used as an example to detail the validation procedure.

As explained in Chapter 0, the “experimental group” sub-set has undertaken the phase A and phase C characterisation tests at CSEM. Thanks to these tests, the energy efficiency has been measured for all cells, including the 96 samples of type B.

Based on the correlations found and presented in section 4.3, the best-fit functions are applied on the measured efficiency to estimate the SoH of the “experimental group”. Table 14 shows the obtained results for the case of type B. It should be noted that the column random voltage-based measurement is kept empty because no correlation was found for that specific case (see section 4.3.1).

The SoH estimation is then compared with SoH measurements to assess the SoH estimation error for every single sample. Finally, the whole estimation performance for every single indicator is evaluated in term of mean error, its standard deviation, its maximum and minimum value.

Table 14: SoH estimation results based on EE-based indicator for cell type B (VC7)

		Random		Nominal		Random (filtered)		
Cell type B	SoH measured [%]	SoH **estimation** **[%]**	SoH Error **[%]**	SoH **estimation** **[%]**	SoH Error **[%]**	SoH **estimation** **[%]**	SoH Error **[%]**	Vini [V]
B-101	87.93%			90.14%	2.21%	88.66%	0.73%	3.99
B-102	Not tested							2.90
B-103	90.64%			92.43%	1.78%	91.60%	0.95%	4.09
B-104	92.70%			90.78%	1.92%	89.62%	3.08%	3.69
B-105	92.70%			93.68%	0.98%	90.99%	1.71%	4.08
B-106	90.64%			92.66%	2.01%	91.36%	0.71%	4.09
B-107	95.13%			91.74%	3.39%			3.40
B-108	92.42%			90.66%	1.75%			3.28
B-109	93.13%			91.83%	1.30%	90.23%	2.90%	3.70
[…]								
B-195	93.24%			90.72%	2.53%			3.20
B-196	91.39%			93.48%	2.10%	91.60%	0.21%	4.08
B-197	90.19%			87.57%	2.62%	86.44%	3.74%	3.54
B-198	92.53%			93.03%	0.50%	90.36%	2.17%	4.08
B-199	92.22%			92.61%	0.39%	90.68%	1.53%	3.93
B-200	90.33%			87.56%	2.77%	86.48%	3.85%	3.54
Results:	*Mean(E)*				2.29%		1.66%	
	σ(E)				1.91%		1.61%	
	Min(E)				-10.46%		-10.24%	
	Max(E)				5.54%		3.85%	

		Random		Nominal		Random (filtered)		
Cell type B	SoH measured **[%]**	SoH **estimation** **[%]**	SoH Error **[%]**	SoH **estimation** **[%]**	SoH Error **[%]**	SoH **estimation** **[%]**	SoH Error **[%]**	Vini [V]
	Refuse rate				0.00%		30.21%	

For the case of technology type B and the EE-based indicator, it is interesting to notice that the measurements of energy efficiency at random voltage (filtered) are providing better SoH estimation result if compared to the ones at nominal voltage. An improvement of 0.5% on the average error and 0.30% on the standard deviation have been found. This is most probably due to the high impact of badly conditioned cells (as anticipated in section 4.3.3). However, in the case of the random voltage (filtered) measurements one should account for a 30% refuse rate, which means that 29 cells over the 96 tested shall be scrapped or put on-hold.

5.4 Discussion

The full estimation process applied in the previous section has been applied to all indicators (i.e. R-based, EE-based and EIS-based) and to all technologies. As in the case of diagnostic tests, type D cells did not give any relevant correlation. So that, the following remarks are only related to technology A, B and C.

Table 15 presents an aggregated analysis about the same estimation results. Within the same typology of indicator, Three KPIs were analysed: the time required to successfully carry out the test, the average estimation error, and its standard deviation. The main findings are the following:

1. Tests at random voltage require 2 minutes (R-based), 14 minutes (EE-based), or 40 minutes (EIS-based). The SoH estimation error is approx. 2.4% with standard deviation of 1.8%. However, section 4.4 discussed that the correlation is found only in the 25% of the cases; therefore, the above encouraging results must be associated to very fortunate cases, for instance the technology C were all cells were at high voltage, so not really in random condition.
2. Tests at nominal voltage increase the testing time of a factor of almost 3 and do not bring advantage in terms of SoH estimation precision, which decrease of 21%. This is certainly due to the high impact of badly conditioned cells (i.e., cells stored at high/low voltage for a long time) which make a relevant non-linear impact also when tested at nominal voltage. It should be also noted that tests at nominal voltage with filtered cells have not be analysed. However, it makes sense to expect an improvement in the SoH estimation in both the average and standard deviation values.
3. Tests at random voltage with a filtering triage improve the SoH estimation of almost 20% and reduce the standard deviation of the same factor. As anticipate in section 4.3.3, there could be two option for the filtered cells:
    - they are scrapped: the testing times are the same of point 1, but the scrapping rate can vary from a minimum of 30% for type B cells to a maximum of 54% in case of type A cells.
    - they are put “on-hold”: they will have to be brought within a predefined voltage window with an increase of testing time of about 35%.

However, also given the results at point 2, one might prefer to scrap the cells instead of regulating them. This allows to exclude all the non-linearities coming from bad cells. However, the impact of high scrap rates must be economically assessed.

Table 15: Analysis of the validation results on the different tested technologies

	Test time [min]			mean(E) [%]			σ(E) [%]		
	r	n	r(f)	r	n	r(f)	r	n	r(f)
Type A (MJ1)									
R-based indicators	1	45	14	3.28	3.56	2.76	1.83	2.07	1.59
EE-based indicators	-	59	29	-	3.19	2.19	-	1.90	1.49
EIS-based indicators	40	83	53	3.30	4.66	3.27	1.83	2.65	1.66
Type B (VC7)									
R-based indicators	2	47	10	2.15	2.14	0.89	2.68	2.36	0.56
EE-based indicators	-	69	15	-	2.10	1.66	-	1.89	1.61
EIS-based indicators	-	85	47	-	2.70	1.31	-	2.41	0.96
Type C (GA)									
R-based indicators	2	64	2	1.83	2.78	1.83	1.71	1.76	1.71
EE-based indicators	14	82	14	2.51	2.90	2.51	1.71	1.77	1.71
EIS-based indicators	40	102	40	1.33	2.22	1.33	1.28	1.65	1.28
Type D (NC1)	-	-	-	-	-	-	-	-	-
Average (all tech.)									
R-based indicators	2	52	8	2.42	2.83	1.83	2.08	2.06	1.29
EE-based indicators	14	70	19	2.51	2.73	2.12	1.71	1.85	1.60
EIS-based indicators	40	90	47	2.31	3.20	1.97	1.56	2.24	1.30
Weighted average (all indicators):	18	70	25	2.41	2.92	1.97	1.78	2.05	1.40

As regards of the best indicator to be used, the main findings were:

- Resistance-based indicators provide estimation error below 2.5% on average (the lowest SoH estimation error was below 1%). Moreover, R-based tests are very fast to be performed, below 5 minutes on average in random voltage testing conditions. However, both RAC and RDC do not show persistent performances through different technologies. For instance, RDCr and RACr were found correlated only for type B and C cells; however, RDCr shows similar performances among the two technologies with an error around 2%, while RACr shows error around 1% in one case and 3% error in the other case.
- EIS-based indicators show high estimation error. Combined with the high testing time required to perform EIS test this could be a showstopper. However, two points goes into favour of this measurement:
  - As regards of the estimation error, it should be noted that most of the correlations were found for technology A, which overall presents the highest estimation error among all indicators.
  - As regards of the testing time, it should be noted that EIS measurements can be fasten if a specific frequency region should be targeted. In the case of the BAT4SEL project, it is found that the most interesting EIS-based indicator is based on the high- and mid-frequency regions of the EIS spectrum. In such a case, if opportunely tuned, the test can be reduced to be below 10 minutes.
- Efficiency-based indicators show stable estimation errors around 2%. Moreover, as anticipated in section 4.4, the EE-based indicator at random voltage was among the few providing

correlations for all the 3 tested technologies. This is confirmed also by a satisfactory SoH estimation error always below the 2.5%. Given the found robustness and the moderate testing time (below 15 minutes on average), the EE-based indicators might represent the best compromise for a fast SoH estimation of used cells.

6 Conclusion

Project BAT4SEL investigated the opportunity to accurately estimate SoH of second-life cells (i.e. aged cells with SoH < 80%) by using fast testing protocols. The project objective was to give a statistical quantification of the trade-off between the SoH estimation precision (i.e. averaged error and std. deviation) and the required testing time to obtain the same estimation.

The whole statistical analysis was based on the test of a high number of cells, which have been provided by Libattion to CSEM. In total 532 cells were tested at CSEM and Libattion facilities, with more than 130 cells for each of the four selected technologies: the Sony VC7 (NCA), the Sanyo Panasonic GA (NCA), the LG Chem MJ1 (NMC) and the Sony NC1 (NMC).

The testing methodology has been based onto two different sub-set of tests: diagnostic tests and validation tests. In the first sub-set, a representative sample of cells have been subjected to characterization tests to find existing correlations between the extracted indicators and the measured SoH. In the second sub-set, a wider sample of cells has been tested with the same protocols and validated by comparing the SoH values measured at Libattion and the SoH values estimated at CSEM.

Several indicators have been tested for correlation. They belong to different typologies: resistance-based indicators, efficiency-based indicators, and EIS-based indicators. Computing these indicators from the associated testing protocols require different testing time and different testing machine capabilities.

The correlations assessment has been verified for three different testing conditions: (i) random voltage condition, meaning that the cells are tested as received without performing any preliminary regulation; (ii) nominal voltage condition, meaning that all the cells are brought to the same voltage threshold before starting any procedure; (iii) random voltage filtered, meaning that some cells are discarded based on a voltage range defined arbitrarily by experts.

Given the results presented in section 4.4 (diagnostic test results) and section 5.4 (validation test results), it is possible to conclude that:

1. *BAT4SEL project was about aged cells but not about second-life cells.* The measured SoHs of the tested technologies were not exactly the ones of second-life cells. An average SoH > 90% has been found among the 4x32 cells used for characterization. This fact does not restrict the applicability of the testing methodology.
2. *All tested indicators bring a significant reduction on the testing time of 70%*, with respect to the 5 hours standard discharge-charge-discharge cycle. In the case of tests at random voltage with filtering option, resistance-based indicators requires 8 minutes (-97%), EE-based indicators requires 19 minutes (-93%), while EIS-based indicators requires 45 minutes (-85%).
3. *SoH validation measurements has shown biases*. The SoH measurements performed at CSEM and at Libattion were giving different values even though applied on the same cells. Therefore, an average ΔSoH=1.25% has been netted in the estimation procedure as explained in section 5.2. This might have impacted on the results.
4. *The characterization process takes 1 month per technology*. This includes:
    1. The time required to characterize a chosen technology through testing protocols. It should be noted that the mapping of any technology is a living process: it is suggested to include new cells into the correlation process from time-to-time.
    2. The time required to extract, elaborate, and analyse the raw data to extract the meaningful correlations, as don in this project.

    It should be noted that the above time does not include all the work necessary to develop and industrialize an automated procedure to speed up the SoH estimation in real application.

5. *The testing equipment might influence the results.* Uncertainties are reduced by:
   - The precision and accuracy of the battery tester. The results obtained in this project are based on measurements performed with a state-of-the-art battery tester. Industrial equipment should be calibrated such that the unavoidable impact on the estimation error is minimized.
   - The temperature at which tests are carried out. Some measurements (e.g. EIS measurements) are temperature sensitive. It is suggested to actively control the temperature by running tests in controlled environment (i.e. thermostatic chambers).
6. *Not all the technologies tested brought to positive results*. Technology D (NC1, NMC from Sony) did not show any correlations with respect of any of the tested indicator.
7. *Tests at random voltage condition rarely show correlations*. Only in 25% of the cases the correlation was found. Many of them were for technology C, which was showing 95% of the tested cells with V_{in} > 4.1V(so that not in random condition).
8. *A pre-triage process is a good practice*. Few highlights about that:
   3. Excluding “bad cells”, which are outside a predefined voltage band, increases the probability of finding correlations of about 50%.
   4. It is better to scrap “bad cells” rather than regulating them. This was confirmed by tests at nominal voltage, which were showing higher SoH estimation error. However, the scrap rate has been found to be around 40%, therefore it can have a clear economic impact.
   5. In case one would prefer to keep triaged cells, the testing time will increase of around 35%.
9. *There is not the best indicator for all*. One should choose according to his/her needs:
   6. As regards of accuracy, both resistance-based and EIS-based measurements provided an average estimation error around 1.2% with standard deviation of the same around 1.2%.
   7. As regards of robustness, the only indicator showing consistent correlations among all the tested technologies was the energy efficiency measurement.
   8. As regards of testing time, the fastest protocols are the resistance-based ones taking, which require only few minutes.
   9. As regards of machine applicability, resistance-based and efficiency-based measurements are the most suited.

7 Outlook and next steps

Project BAT4SEL confirmed the validity of the accelerated testing approach developed by CSEM on a statistically representative numbers of various Li-ion cells that are commonly used by Libattion.

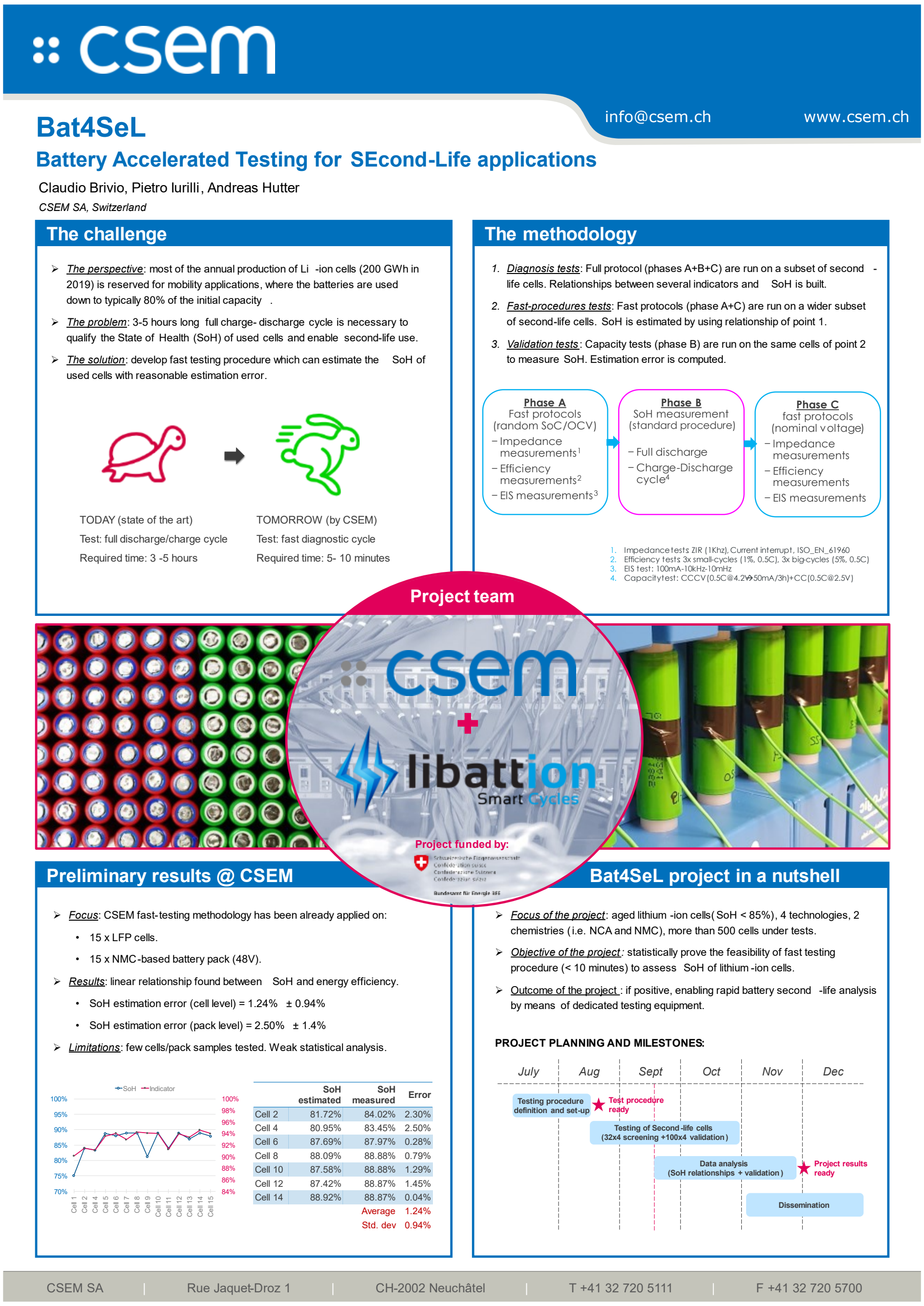
Libattion as a potential end user of the investigated approaches has identified the following main routes for the exploitation of the obtained know-how and resutls from the BAT4SEL project:

1. Implementing the BAT4SEL estimation algorithm into existing testing machine at Libattion.
2. Verify the applicability of the fast-testing approach at pack level. Making sorting at pack level rather than on cell level will simplifying the handling burden.
3. Implementing the fast-testing protocol directly into BMSs to track battery pack status, optimise lifetime, and plan replacment in a continuous manner and during the first use of the battery. Two options for this exploitation route have been identified:
    - Develop new BMS dedicated for second-life application with advantage in term of diagnostic capabilities, balancing current and manufacturing.

8 Publications

The BAT4SEL project methodology has been presented in September 2020 at the 25th International Congress for Battery Recycling ICBR 2020, Salzburg, Austria.

The project has been presented in form of a Poster presentation, which is shown hereafter:



Positive feedback was gathered where interests was raised from the following stakeholders: a trans-national regulation authority which aims at regulating the aftermarket of used battery packs, a foreigner national authority, a foreigner company active in the second-life business, a national start-up aiming at revolutionising the handling of used battery pack from electric vehicle.

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