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Sodium-Ion and sodium Metal BAtteries for efficient and sustainable
next-generation energy storage

GRANT AGREEMENT No. 963542

SIMBA – Deliverable Report

<< D6.3 – Develop a safe storage, transport and opening protocol for the solid-state batteries at end of life >>

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Publishable summary

SIMBA aims to develop a highly cost-effective, safe, all-solid-state sodium-ion battery for large-scale stationary applications. Although in many ways sodium-ion batteries (SIBs) are similar to lithium-ion batteries (LIBs), there are still several persistent scientific and technical challenges to be addressed in understanding impact of storage conditions upon safety and transport.

Since LIBs were first commercialized in 1991, numerous regulations and directives have been issued to ensure their safety during storage, transport, and disposal. Although SIBs and LIBs are similar, they still have different properties, such as SIBs can be fully discharged. This deliverable is dedicated to evaluating the control measures during storage, transport and opening to minimize the risks. Firstly, we discussed the effect of low-temperature storage and explored the possibility of achieving a zero stage of charge. The transport at zero volts, and zero state of charge is explored, and the implications in safety compared to lithium ion batteries. Finally, the opening procedures of end-of-life baseline cells (BLC) and solid-state batteries (SSB) were demonstrated. The sorted battery components will be used to further reclaim and reuse in task T6.2.

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1   Purpose of the document

This document corresponds to D6.3 - Develop a safe storage, transport and opening protocol for the solid-state batteries at end of life. The relative regulations and control measures will be discussed in this deliverable report as a guideline.

1.1 Document structure

The document includes an introduction section, one section focused on the main results and discussion and a final paragraph on conclusion and remarks. The main results and discussion include the control measures for storage, transport and opening protocol for the A7 size baseline cells and solid-state batteries at end of life.

We received three single layer pouch cells, which contained a polymer membrane electrolyte, however there was liquid electrolyte still used in the electrodes to enable the ionic transport. Therefore we have included some work relating the base-line cell (BLC) which investigated the storage conditions at different states of charge, and with a short circuit at room temperature which was already included in deliverable 5.1. Due to the number of cells received a full experimental window was not able to be performed, however initial discharge and discussion with respect to the literature is included.

1.2 Deviations from original description in the Grant Agreement Annex 1 Part A

1.2.1   Descriptions of work related to deliverable in GA Annex 1 – Part A

1.2.2   Time deviations from original planning in GA Annex 1 – Part A

According to GA Annex 1-Part A, D6.3 was due in M24. However, this deliverable was shift to M38 due to delay of A7 sized solid-state pouch cells.

1.2.3   Content deviations from original plan in GA Annex 1 – Part A

N/A

2   Introduction

D6.3 is associated with T6.1 and T6.2 of WP6. T6.1 addresses; ST6.1.1 Storage, Transport Safety Base Line Cell (BLC) recycling Cell type 1 ST 6.1.2 discharge and stabilisation ST 6.1.3 tear down and opening The cells will be shredded with an isolated shredder for safe and efficient opening. This will be compared to dismantling techniques developed at UBham. The shredded fraction will be further refined and the trapping and elimination of the solvent/byproducts will be performed, including any HF that is produced.

 6.2 focuses on reclamation and reuse of materials from end-of-life baseline cell (D4.1)[4] and solid-state batteries (D4.7)[5] manufactured by WMG. The main goal for this report is to develop a safe storage, transport and opening protocol for the solid-state batteries before recycling.

Firstly, low temperatures storage and zero state of charge were evaluated in solid-state batteries storage. Then the relative regulations for transport were introduced and the packaging of end-of-life solid-state batteries were displayed. Finally, the safe opening protocol was developed for baseline cells and solid-state batteries.

3   Storage, transport and opening for BLC and SSB

3.1 Risk

In the past 30 years, the safety of LIBs has been greatly improved through improvements in materials, structure designs, system management, etc.[1] However, numerous fires and explosions have been reported around the world.[2] SIB is an alkali metal-ion battery which can be manufactured on the same lines as LIB, and has similar materials. The main difference is that aluminium can be utilized as the current collector on both the positive and negative electrodes. From previous published work on layered oxide vs hard carbon it has been shown that similar thermal runaway to LIB is possible, as there is a fuel and an oxidant.[3] However, in these cases when the layered oxides, is over charged it can break down and produce oxygen. Less is known about the Prussian white and hard carbon systems. Thermal runaway (TR) occurs typically at higher state of charges, as there is more energy which can dissipate quickly. TR can occur from an internal or external short circuit, overheating or overcharge. In each case the SEI layer that protects the lithiated or sodiated anode, starts to dissolve, and then when fully dissolved the sodiated carbon reacts with the electrolyte to reform the SEI. As the cell heats up, the electrolyte can ignite (fuel and oxidant) and the chemical reactions propagate, this is known as a thermal runaway event. In the case of layered oxide systems, the oxygen can fuel the fire. For Prussian white or blue / hard carbon systems, a similar effect is observed upon the hard carbon in terms of the SEI and SEI stability. However, the Prussian blue has cyanide bonds rather than oxygen, and the material is fully desodiated upon charge with a high voltage overpotential at 100% SOC, meaning that the materials is very difficult to overcharge, in addition, no oxygen is produced upon overcharge. Previous reported work shows that the Prussian white starts to break down at around 200 °C where the sodium content in the cathode is Na>0 and 300 °C at Na=0 in different atmospheres.[4] The main gases are HCN, CO_2, H_2O, at Na-1.5 whereas at Na=0 HCN and $(CN)_2$ was observed at 300°C. (Figure 1). Li et al[5] showed that thermal runaway however does still occur in these cells, and the energy evolution as measured from DSC was not insignificant, at 539 Jg^{-1} for charged cathode with electrolyte. That is higher than the 200 Jg^{-1} reported for LFP in a similar experiment.[6] A reaction mechanism was proposed which consists of electrolyte decomposition at ~ 200 °C followed by HCN release and then cyanide and electrolyte exothermic reaction which occur ~ 300 °C. However further clarification of this is required for this specific system.

The literature suggests that thermal runaway will occur with the Prussian white and hard carbon cells, and this can be initiated by generation of localised heating through crush, nail penetration or hotbox – as long as there is enough heat generation through ohmic heating of the cell during a potential short circuit. It also suggests that cyanide gases are formed. Thermal runaway and cell failures through nail

penetration could not be tested in the laboratory due to health and safety concerns and risks around cyanide gas formation.

It should however be noted that due to the low energy density in these cells, it is unlikely that a short circuit would result in this level of heat build-up. In addition, when considering the polymer electrolyte in the solid-state battery, the membrane does not contain the carbonates which propagate the thermal runaway reaction. Therefore more studies are required to ascertain the actual thermal runaway implications. The received SSB cell was a partial solid-state battery with liquid electrolyte in the electrodes.
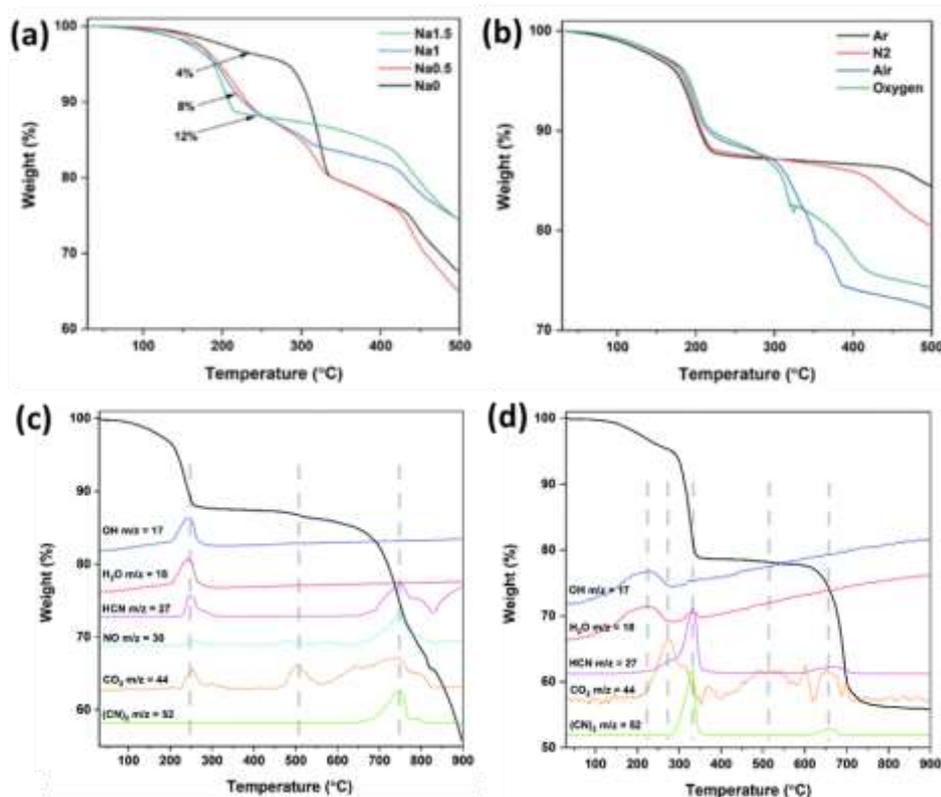

Figure 1 (a) TGA curves recorded with a heating rate of 5 °C min^{-1} in N$_2$. (b) TGA curve of Na1.5 under different atmospheres at a heating rate of 5° min^{-1}. (c) gas evolution of Na 1.5 and (d) Na0 under Argon at 10 °C min^{-1}.[4]

3.2 Storage

The storage conditions are investigated to ensure safety and also retention of state of charge. Safety is mainly related to potential gassing in storage due to unwanted side reactions and subsequent potential venting, whereas the retention of the state of charge is related to the internal chemical reactions, and potential SEI instabilities on the hard carbon over time, which also could result in gas being produced. Previous work illustrated in OCV stability showed that the OCV stayed stable for up to 1 month (LO/HC) and 2 months (PW/HC), and then the OCV decreased rapidly for the cell discharged to zero volts, the lower and higher SOC saw higher changes of OCV than partially charged (Figure 2). This we believe is due to the SEI dissolving into the electrolyte over time. Therefore, suggestions for storage are at low temperature, and partial state of charge in liquid electrolyte cells. A partial state of charge will retain the SEI for longer, and if the cell is to be used or reused then this needs to be considered. For polymer and solid state cells, it is expected that storage is possible at zero state of

charge as there is no requirement for a non-soluble interface, however we have not been able to test this hypothesis.
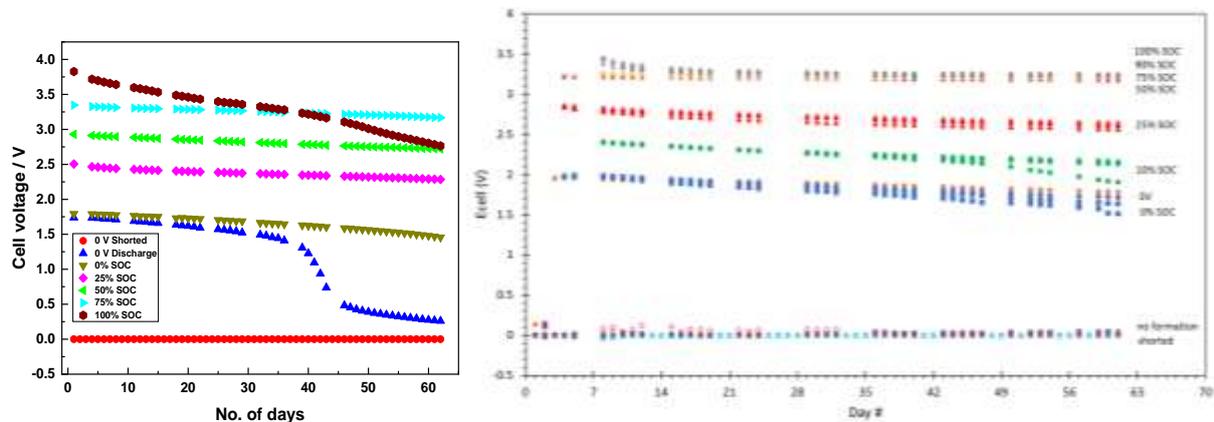

Figure 2 Summary of change in OCV for (a) LO/HC and (b) PW/HC liquid electrolyte cells (deliverable 5.1) stored at 25 °C

3.2.1 Lower storage temperature

Firstly, temperature affects reaction kinetics. Normally the rate of reaction is halved for every 10 °C decrease in temperature. Liu et al. reported that the charge transfer resistance, which relates to reaction kinetics, increasing 10-fold from 25 to 5 °C.[7] Lower temperature will slow the interphases dissolving into the electrolyte, reducing any self discharge.

The polymer membrane used in SSB was soaked in ethylene carbonate (EC)/diethylene carbonate (DEC)/Fluoroethylene carbonate (FEC) to ensure good contact between membrane and electrodes.[6] However, EC crystallizes on the electrode surfaces when stored at low temperature due to its high melting point, ~ 35 °C (Figure 3a). The electrochemical impedance spectroscopy (EIS) results in Figure 2b also displays SSB has larger charge transfer resistance (522 Ω) than that of BLC (0.1 Ω) after store at low temperature. Therefore although the storage at lower temperatures will reduce any self-discharge or voltage drop, due to poor movement of the sodium-ions in the electrolyte. The cell must return to room temperature for subsequent reuse of the cell to ensure that the electrolyte is liquid again. Further voltage stability improvements are expected from the polymer electrolyte system only, where the ions are more immobilised at higher temperatures.
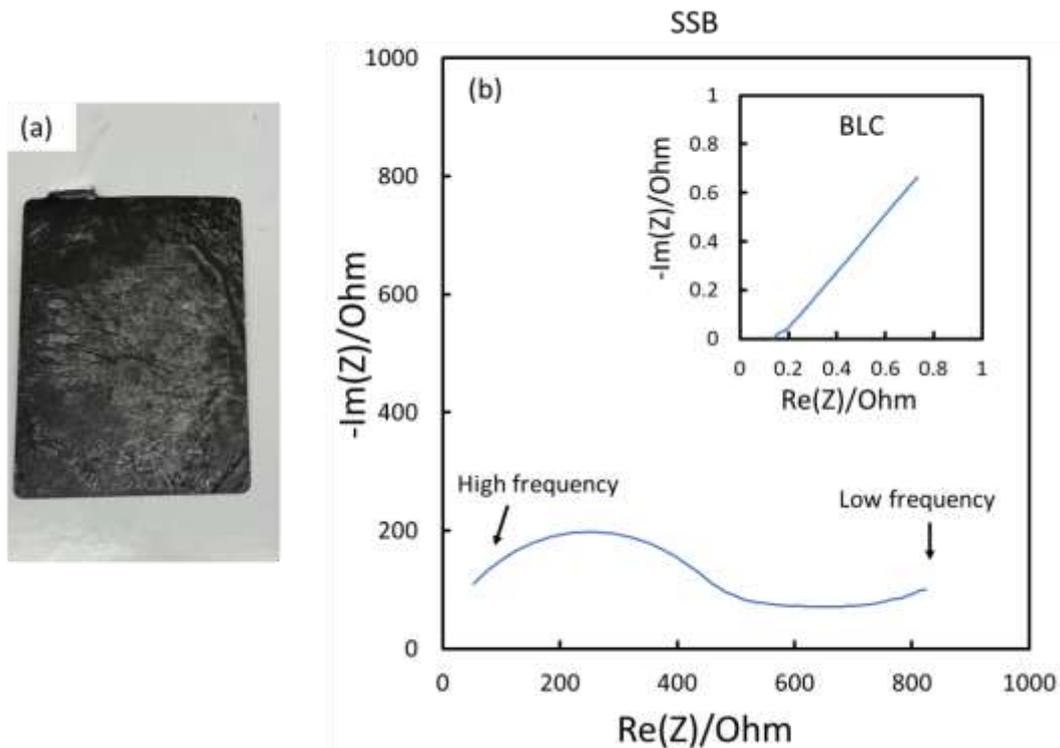
Figure 3 (a) PW electrode from end-of-life SSB and (b) Nyquist plot of end-of-life of SSB and BLC after storage at low temperature.

3.2.2   zero state of charge or zero volts

Previous work as shown in Figure 2 has investigated zero volts or zero state of charge (Deliverable 5.1). For safety reasons, storage with less energy is preferred, particularly if subsequent recycling is performed. In the cycling protocols for the BLC and SSB the voltage window is between 1.3 and 3.8 V,.[4,5] Figure 4a exhibits the capacity profile of BLC at end-of-life with discharge to 0V at 0.1 C (100 mA) and hold at 0 V until current drop to 0.01C (10 mA). It exhibits end-of-life cells retaining 270 mAh capacity, over 25% reversible capacity of BLC (1 Ah). After discharge, the open circuit voltage rebounds to 1.5 V open circuit voltage, as is expected from the anode and cathode thermodynamics. In the subsequent discharge, it only presents 12.5 mAh capacity and voltage rebounds to 1.4 V. Through two fully discharge cycles, most of the energy in the end-of-life cell is released, leaving less than 1% of the capacity, thereby reducing the risk of subsequent processing.
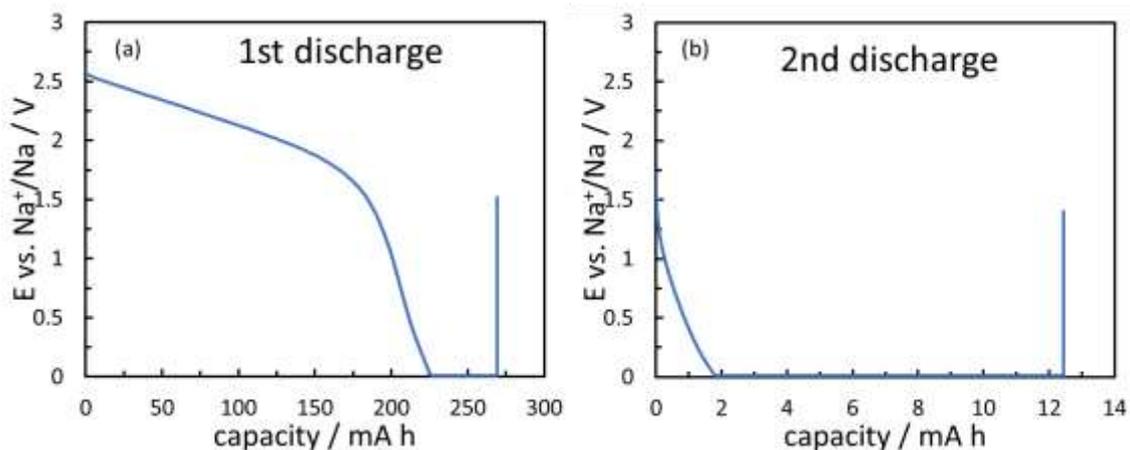

Figure 4 (a) first discharge profile and (b) second discharge profile of BLC at end-of-life.

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The same discharge procedure as BLC were also applied for the SSB at end-of-life (Figure 5). However, even the first discharge process only releases less than 10^{-4} mAh capacity..
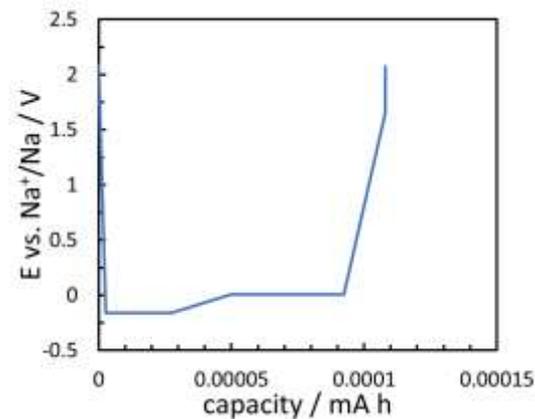

Figure 5 Discharge profile of end-of-life SSB.

For both the BLC and the SSB discharging to zero volts before processing takes the sodium from the anode and inserts it into the cathode. Therefore, in terms of recycling, this is preferred because the sodium will be mainly retained in the cathode fraction. However, the high overpotentials exhibited at end of life, indicate that significant degradation has occurred in the cells, this could be from an SEI build up, poor adhesion to current collectors, or degradation of the electrolyte and active materials. In all cases the energy levels are very low, suggesting that very little will happen during the opening stages.

Furthermore, the storage location of end-of-life lithium-ion batteries also needs comply with Annex XII of Regulation (EU) 2023/1542. The relative requirements are:
1. Treatment and any storage, including temporary storage, at treatment facilities, including recycling facilities, shall take place in sites with impermeable surfaces and suitable weatherproof covering or in suitable containers.
2. Waste batteries in treatment facilities, including recycling facilities, shall be stored in such a way that they are not mixed with waste from conductive or combustible materials.
3. Special precautions and safety measures shall be in place for the treatment of waste lithium-based batteries during handling, sorting and storage. Such measures shall include protection from exposure to:
   (a) Excessive heat, such as high temperatures, fire or direct sunlight;
   (b) Water, such as precipitation and flooding;
   (c) Any crushing or physical damage.

Waste lithium-based batteries shall be stored in their normally installed orientation, that is, never inverted, and in well-ventilated areas and they shall be covered with a high voltage rubber isolation. Storage facilities for waste lithium-based batteries shall be marked with a warning sign.

From our understanding it is likely that sodium-ion batteries will also need to comply with the same regulations. There is some evidence, as presented above, that sodium-ion will be less susceptible to safety concerns during storage due to their lower energy densities.

3.3 Transport
Different from storage, transportation only temporarily stores end-of-life batteries, and physical damage is more likely to occur due to the movement of the batteries. Currently there are the following regulations for battery transportation:

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1. United Nation Recommendation of the Transport of Dangerous Good, 21st revised Ed. 2019
2. Agreement concerning the International Carriage of Dangerous Goods by Road (ADR), ECE/TRANS/326, 2023
3. European Agreement concerning the International Carriage of Dangerous Goods by Inland Waterways (ADN), ECE/TRANS/325, 2023
4. Regulation concerning International Transport of Dangerous Goods by Rail (RID), OTIF, 2023
5. UN Committee of shipment hazardous waste regulation according to the provision ST/SG/AC.10/C.3/2019/71

The detailed requirements were discussed in D6.1.[7] This deliverable will describe detailed measures to cut down the risks during transportation.

As performed in ST6.1.1 Storage, Transport Safety **Recupyl** has identified and reported the main organization at world level in-charge of shipment regulation:

International organizations relevant to shipment of batteries	International Organization Europe connection	International Organization out of Europe
United Nation Recommendation of the Transport of Dangerous Good , 13th revised Ed. 2003		ASEAN countries: based on ADR but completed with a Framework for Facilitation of Goods transit
European Agreement International Carriage of Dangerous Goods by Road (ADR) ECE/TRANS/175, 2005	For ADR: UNECE Inland Committee WP.15, **Geneva**	MERCOSUR Countries: Agreement based on ADR-RID
European Agreement International Carriage of Dangerous Goods by Inland Waterway (ADN) ECE/TRANS/172, 2005	For ADN: UNECE Joint Meeting of Expert Regulation, **Geneva**	ANDEAN countries : legislation in preparation based on ADR
Regulation concerning International Transport of Dangerous Goods by Rail (RID) OTIF	For RID: OTIF Safety Committee, **Bern**	

Implementation must be managed by State members under the European Directive 96/49/EC.

In the segment of shipment, one section is dedicated to Damaged or Defective Lithium Cells and Batteries.

1- New or used lithium cells and batteries (not waste cells and batteries):
Defective or damaged but poses no additional risk in transport (See SP 188, PI 903 or SP 310), Defective or damaged and poses additional risk in transport (*Need packaging criteria and examples of what is defective and damage*)

2- Waste lithium cells and batteries: Defective or damaged but poses no additional danger in transport (See SP 188, PI 903, SP 310 or ADR requirements) Defective or damaged and poses additional risk in transport (*Need packaging criteria and examples of what is defective and damage*)

Identification	Class 1	Class 2	Class 3	Class 4	Class 5	Class 6	Class 7	Class 8	Class 9
	Explosive materials	Gases	Flammable liquids	Flammable solids	Peroxides activating fire	Toxic materials	Radio elements	Corrosives products	Other dangerous products
Labels									

Class 4 = Battery emit flammable gas in contact with water
High energy storage batteries Containing sodium

UN 3292 Batteries containing Sodium
(Actually only for Na batteries (Zebra))

Class 9 = Miscellaneous dangerous substances
UN 3090 Lithium batteries
UN3091 Lithium batteries contained in equipment or batteries packed with equipment
Lithium, lithium ions, lithium alloys based batteries

Different labels are used for identification of risk, and they are classified as explosive, gases, flammables, oxidizing, toxic, radioactive, etc.

Why batteries are classified as hazardous:
- Identification of the risk

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- Fitting with the existing list and classification
- By the electrolytes (corrosives class 8)
- By the reactive metals like Li (class 9)
- By the solvent or organic electrolytes (class 4).

What needed as batteries are classified as hazardous:
After Classification and Identification of the risk:

- Adapted and approval packaging
- Adapted procedures,
- Transport operation requirements, documents, signals, training, company management

The details of actions for this requirement are:

- Documentation (chap. 5.4)
- Labelling/Marking – package & transport unit (chap.5.2/5.3)
- Loading storage (part 7)
- Driver training (ADR part 8)
- Safety adviser (RID ADR 1.8.3)
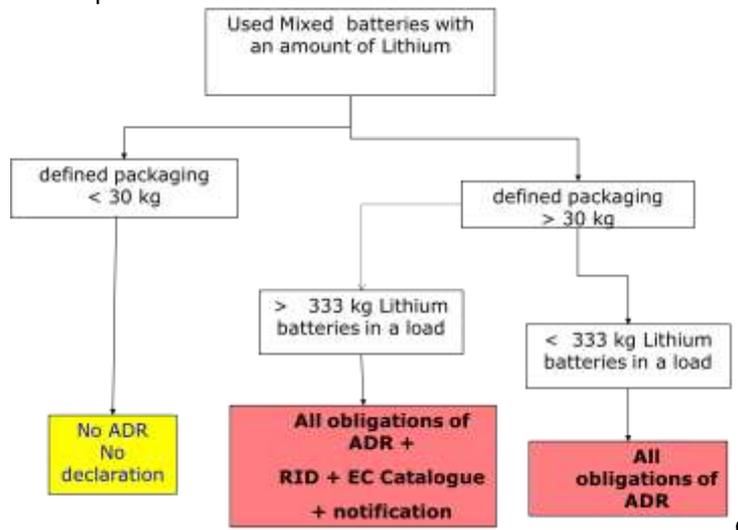- Safety obligations (consignor, loader carrier, Chap.1.4 RID/ADR).

ADR regulation and packaging of mixed batteries from primary collection was amended and final text was adopted in the FINAL INSTRUCTION: ECE-TRANS-WP15-AC21:

P903b	PACKING INSTRUCTION	P903b
This instruction applies to used cells and batteries of UN Nos. 3090 and 3091.		

Used lithium cells and batteries, with a gross mass of not more **than 500 g** collected for disposal, together with other used non-lithium batteries or alone, may be carried, without being individually protected, under the following conditions:
(1)   In 1H2 drums 4H2 boxes conforming to the packing group II performance level for solids or
(2) In 1A2 drums 4A boxes fitted with a PE bag and conforming to the packing group II performance level for solids. The PE bag shall :
have an impact resistance of at least 480 grams in both parallel and perpendicular planes with respect to the length of the bag,
have a minimum of 500 microns of thickness with electrical resistivity of more than 10 M ohms and low water adsorption rate over 24 hours at 25℃ lower than 0.01% the PE bag may be used once only.
(3)   In collecting trays with a gross mass of less than 30 kg made from non-conducting material meeting the general conditions of 4.1.1.1, 4.1.1.2 and 4.1.1.5 to 4.1.1.8.

Additional requirement:
The empty space in the packaging shall be filled with cushioning material. The cushioning may be dispensed when the package is entirely fitted with a plastic bag and the bag is closed.] Hermetically sealed packaging shall be fitted with a venting device according to 4.1.1.8. The venting device shall be so designed that an overpressure caused by gases does not exceed 10 kPa.

Example of adopted package is reported below:

Actual plastic bag (before fedding) Around 20 cm above the top of drums

Actual plastic bag (feeding step)

Actual plastic bag (after closing)

For household batteries the protocol of classification is:

c

To ensure complete safety considerations for the prototype cells, extensive packing and isolation protocols were followed. Non-combustible, non-electric conductivity and thermal insulation materials will be filled in the package and surround end-of-life batteries (Figure 5). Furthermore, the end-of-life batteries can be discharged to zero state of charge to minimize the retained energy.

As stated previously, in our current work, we have noted that the discharge to zero state of charge and the storage has more of an effect on recoverable energy due to the SEI instability, rather than any specific safety implications. Further work is required to further understand the stability for transport. If discharged to zero volts, and then the negative and positive terminals are connected (short circuited) little occurs. Anecdotally, some gas has occurred in cells which were not dried in the correct manner due to the likely reduction of water to produce hydrogen and oxygen. However, little occurs in cells that are correctly processed in dry atmospheres and not exposed to air before electrolyte filling. If the terminals are connected, there is no energy contained in the cell and this can be considered a bag of chemicals rather than an electrochemical cell. As patented by Faradion (US11159027B2) granted in the US.

One point to note is that Zero state of charge is suggested here rather than partial for storage, as it is assumed that the transport will be less time than the storage, and if transported at low temperatures

the effect of the SEI dissolution will be limited. A partial state of charge will give greater stability to the OCV, compared to zero state of charge.

The transporter must consider the temperature for transport, and the length of transport to storage. One point to note as stated in 3.2, if stored at room temperature or higher, the SEI may dissolve, and self-discharge and voltage drop occur. However, if transported at low temperatures this will be limited.

For solid state cells it is expected that zero state of charge for storage and transport will be preferable, due to the difference in SEI stability.
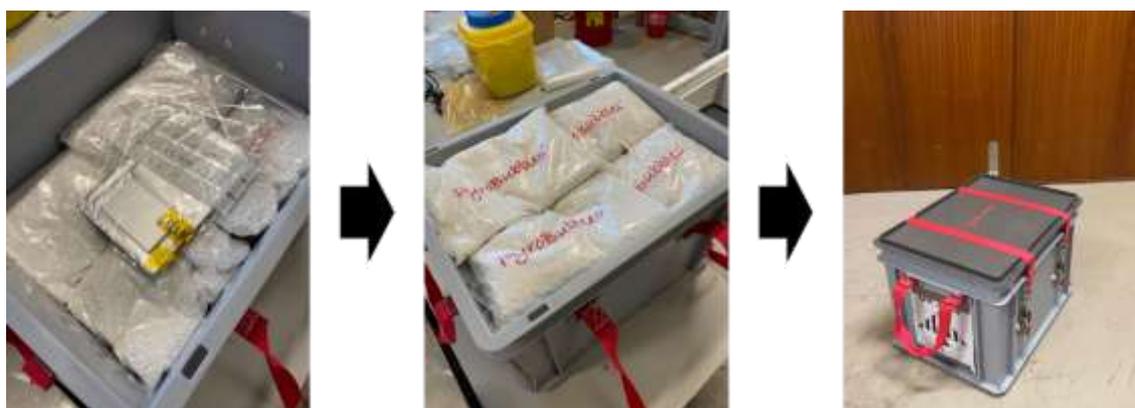
Figure 6 Package of end-of-life SSB

3.4 Opening

Compared with storage and transport, the opening process not only faces the risk of thermal runaway, due to short circuiting the cell, through puncture. There is also a risk of exposure with toxic and irritating chemicals, such as HF from the electrolyte degradation and CN from the cathode degradation, which are both hazardous. The main risks and control measures during the opening procedure are listed in Table 1. This section is dedicated to raising a protocol for safe opening of end-of-life cells by using the BLC and SSB developed in this project.

Table 1 Risks and control measures during the opening of end-of-life cells

Risk	Control measure
Internal short circuit	Fully discharged before opening
Release of toxic gas [D4.1]	Operate in a fume hood
Contamination of toxic and irritant chemicals	Wear personal protective equipment
Na deposited on negative/positive electrodes	Soak in isopropanol (IPA) over night

The procedure for opening BLC is listed below and displayed in Figure 6:
1. Place the battery flat in an insulated tray.
2. Carefully make a hole to release the gas.
3. Cut edge of the pouch cell.
4. Cut tabs.
5. Sorting positive, negative electrodes and separator.
6. Soak electrodes and separator in IPA separately overnight.
7. Dry in vacuum oven at 60 °C overnight
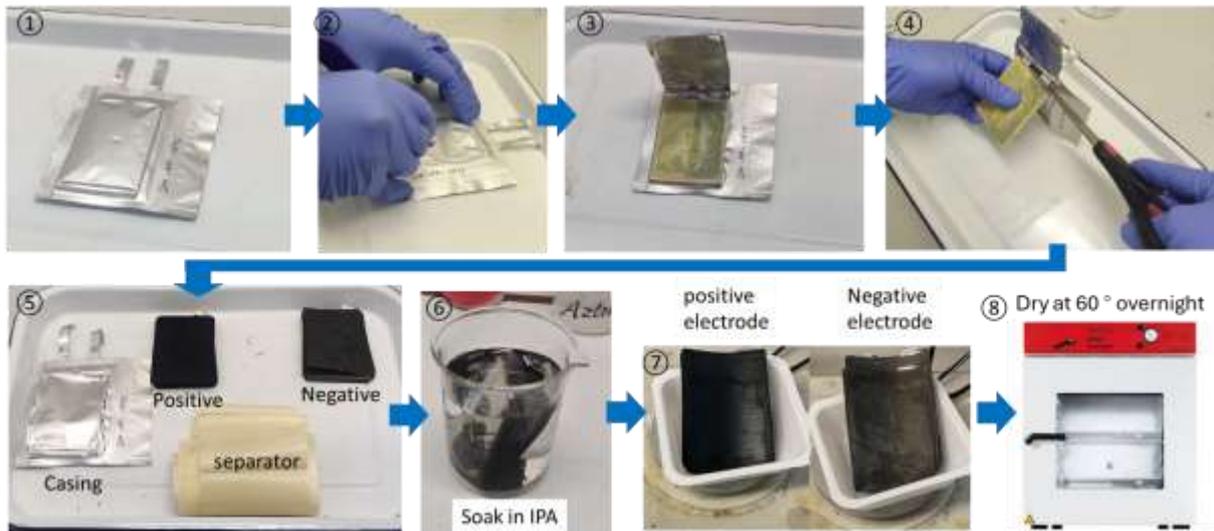8. Collect from oven and ready for direct recycling.

Figure 7 Opening process of end-of-life BLC.

The opening procedure of SSB is modified from BLC due to following differences:
- Less gas generated during cycling.
- The electrode and separator of SSB were stuck together due to the crystallization of EC during low temperature storage.

Figure 7 shows the procedure which are:
1. Cut edge of the pouch cell.
2. Cut tabs.
3. Soak electrodes and separator in IPA overnight.
4. Sorting positive, negative electrodes and separator.
5. Dry in vacuum oven at 60 °C overnight
6. Collect from oven and ready for direct recycling.
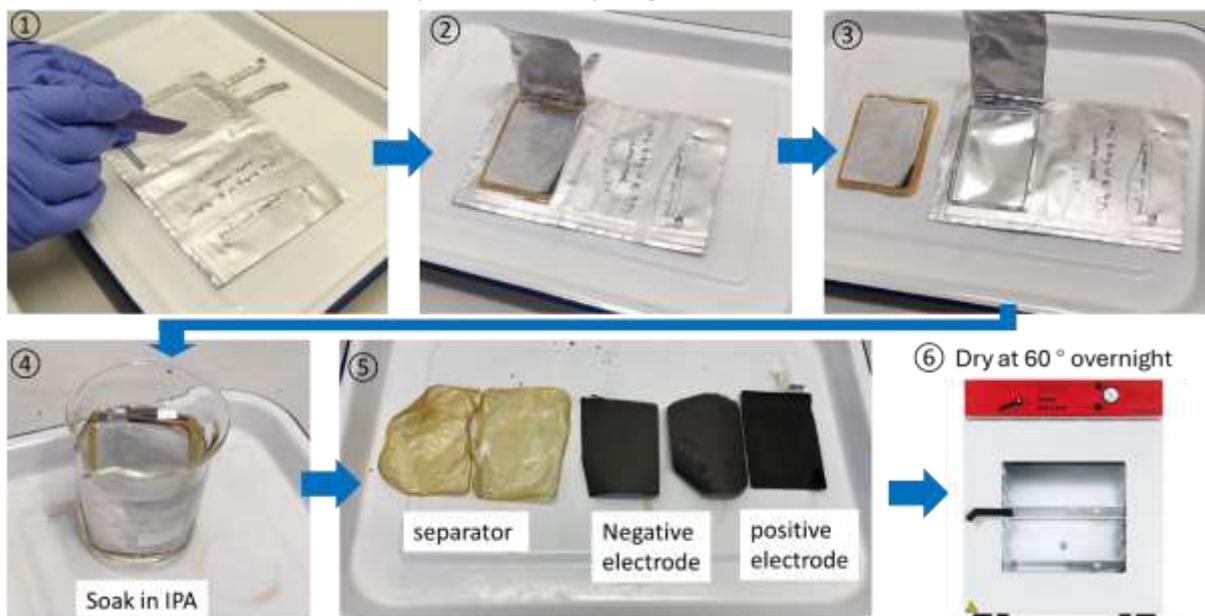
Figure 8 Opening process of end-of-life SSB.

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4   Conclusions and Recommendations

In this report, we discussed the risks and control measures during storage, transport, and opening end-of-life cells. Storage end-of-life SSB at low temperatures can effectively reduce calendar ageing and slow down the SEI dissolution processes. Zero state of charge or zero volts can also reduce storage and post processing risk. However, it should be noted that self-discharge and SEI dissolution can occur at room temperature and higher, potentially producing gas (although not observed in this work). It is recommended to store at zero state of charge and 10 °C. Then we introduced LIB regulations for transport and the packaging of end-of-life cells to minimize the risk, it is likely that NIB offers less risk than LIB due to the aluminium current collectors and potential to store at lower SOC or zero volts.

Finally, two opening processes of end-of-life BLC and SSB were displayed. The safety measures of storage, transport and opening were investigated in single sided A7 size end-of-life pouch cells. The end-of-life cells we received were prototype batteries (BLC ~1 Ah, SSB ~0.1 Ah), there is a certain gap with commercial batteries. Further study is suggested to using a upscaled commercial SIBs to evaluate the measures we developed in this report.

5   Risk Register

Risk No.	What is the risk	Probability of risk occurrence[1]	Effect of risk[2]	Solutions to overcome the risk
1	Insufficient solid-state batteries were received.	1	2	Basic electrochemistry performance was tested.

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[1] Probability risk will occur: 1 = high, 2 = medium, 3 = Low

[2] Effect when risk occurs: 1 = high, 2 = medium, 3 = Low

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7   Appendix A- Acknowledgement

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Project partners:

#	Partner	Partner Full Name
1	TUDa	TECHNISCHE UNIVERSITAT DARMSTADT
2	UU	UPPSALA UNIVERSITET
3	UBham	THE UNIVERSITY OF BIRMINGHAM
4	WMG	THE UNIVERSITY OF WARWICK
5	KIT	KARLSRUHER INSTITUT FUER TECHNOLOGIE
6	CEA	COMMISSARIAT A L ENERGIE ATOMIQUE ET AUX ENERGIES ALTERNATIVES
7	IFE	INSTITUTT FOR ENERGITEKNIKK
8	SAS	USTAV ANORGANICKEJ CHEMIE SLOVENSKA AKADEMIA VIED (Institute of Inorganic Chemistry, Slovak Academy of Sciences)
9	FHG	FRAUNHOFER GESELLSCHAFT ZUR FOERDERUNG DER ANGEWANDTEN FORSCHUNG E.V.
10	JM	JOHNSON MATTHEY PLC
11	Elkem	ELKEM AS
12	YUN	YUNASKO-UKRAINE LLC
13	SAFT	SAFT
14	Altris	ALTRIS AB
15	Recupyl	TES RECUPYL SAS
16	UNR	UNIRESEARCH BV

Appendix D – Disclaimer/Acknowledgement