Recycling of end of life battery packs for domestic raw material supply chains and enhanced circular economy

D2.2 Report on specification common and critical parameters for pilots' innovations (WP3, 4 and 5)

LEITAT – BEE – COMA – CESVI – FORD – CEA – ORANO – MTB - IND – IWKS – TOR

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Project details

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BATRAW Project

BATRAW main objective is to develop and demonstrate two innovative pilot systems for sustainable recycling and end of life management of EV batteries, consumer batteries, and battery scraps contributing to the generation of secondary streams of strategically important CRMs and battery RMs. The first pilot will deliver innovative technologies and processes for dismantling of battery packs achieving recovery of 95% of battery pack components and separating waste streams including cells and modules by semi-automated processes for recycling. BATRAW's second pilot will scale and demonstrate efficient pre-treatment and continuous hydrometallurgical recycling of battery cells and modules including innovative steps for C-graphite, Al and Cu separation from black mass and Mn extraction, achieving a recovery of the full range of battery RMs (Co, Ni, Mn, Li, C-graphite, Al and Cu) at selectivity of 90-98%. Innovations will be scaled and demonstrated in a pilot system with recycling capacity of 1 ton lithium-ion battery (LIB) packs dismantled per shift (8 hours) and treat 300 kg BM per day. BATRAW outcomes are of strategic importance within the prospects of the exponentially growing EU battery market and reducing EU import dependency of CRMs. The project will further promote the overall sustainability and circularity of battery products and raw materials by developing new procedures for battery repair and reuse, enabling faster diagnostics and conversion of EV packs into second life batteries, delivering eco-design guidelines for battery manufacturing, demonstrating blockchain platform for raw material tracking and supply chain transparency (Battery Passport) and delivering guidelines for safe transports and handling of battery waste. The project aims to maximize market uptake and impact through ambitious C&D&E plan including circular business models, innovations workshops, dissemination in EU platforms, policy briefs and other strategies to reach markets and stakeholders.

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BATRAW Consortium

PARTICIPANT NO	PARTICIPANT ORGANISATION NAME	COUNTRY
1	ACONDICIONAMIENTO TARRASENSE ASSOCIACION (LEITAT)	ES
2	INDUMETAL RECYCLING SA	ES
3	CENTRO DE EXPERIMENTACION Y SEGURIDAD VIAL MAPFRE SA	ES
4	BEEPLANET FACTORY SL	ES
5	COMANAI SL	ES
6	SIG DE RAEE Y PILAS SOCIEDAD LIMITADA	ES
7	COMMISSARIAT A L ENERGIE ATOMIQUE ET AUX ENERGIES ALTERNATIVES	FR
8	ORANO MINING	FR
9	RENAULT SAS	FR
10	MTB MANUFACTURING	FR
11	FRAUNHOFER GESELLSCHAFT ZUR FORDERUNG DER ANGEWANDTEN FORSCHUNG EV	DE
12	TECHNISCHE UNIVERSITAET BRAUNSCHWEIG	DE
13	ISLE UTILITIES BV	NL
14	MINESPIDER GERMANY GMBH	DE
15	FORD OTOMOTIV SANAYI ANONIM SIRKETI	TR
16	TORRECID SA	ES
17	CENTRE FOR EUROPEAN POLICY STUDIES	BE
18	POSCO	KR

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ABSTRACT

One of the challenges of a project of this type is to be able to establish a common methodology and working conditions in order to anticipate future problems in the project. Therefore, the objective of this deliverable is to outline the technical specifications and validation protocols for the pilots and the recovery procedures for the different raw materials. To achieve this objective, it will be necessary to layout the concept, workflows, and template for the information in order to be amassed and shared (on blockchain). This process will allow a faster information exchange between the partners.

The topics to be covered throughout this report will be the optimization of advanced procedures and devices for a fast characterization of EV packs in addition to setting up broaden secure electric deactivation strategies batteries and samples for the duration of BATRAW project. Another important point will be to construct and to validate a pilot line demonstrating semi-automatic dismantling of EV battery packs and modules both in a workstation and in a pilot and to check routes for valorising WEEE fractions from dismantling procedures. Finally, an alternative benchmark for leaching BM to achieve a greener process to replace inorganic acids will be studied. In a parallel way, benchmarking the use of liquid/liquid membranes as a potential alternative to current liquid/liquid extraction solvent extraction (SX); scale-up of electrochemical lithium recovery as a potential alternative to current liquid/liquid extraction. For this purpose, the specifications of the different processes mentioned above will be defined.

Revision History

The following table describes the mail changes done in the document since it was created:

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Version 2	21st December 2022	Update 1	B. Marín (LEITAT)
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Acronyms and abbreviations

ADR	Agreement concerning the International Carriage of Dangerous Goods by Road
AI	Artificial Intelligence
BMS	Battery Management System
BM	Black Mass
CAPEX	Capital Expenditure
CRM	Critical Raw Materials
DoA	Deed of Agreement
DES	Deep Eutectic Solvent
D	Deliverable
EJF	Electrochemical Junction Transfer
EoL	End of Life
EBRA	European Battery Recycling Association
EC	European Commision
EU	European Union
FMEA	Failure Mode and Effect Analysis
JRC	Joint Research Center
KPI	Key Performance Indicator
MSDS	Material Safety Data Sheet
MS	Milestone
M	Month
OPEX	Operational Expenditure
PIM	Polymeric Inclusion Membrane
RM	Raw Materials
RMIS	Raw Materials Information System
SoC	State of Charge
SoH	State of Health
SLM	Supported Liquid Membrane
T	Task
TRL	Technology Readiness Level
UN	United Nations
WEEE	Waste Electrical and Electronics Equipment
WP	Work Package

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Executive Summary

The report skeleton is built from a list of basic samples definition including BATRAW methodology (Chapter 2), then a list of specification related directly to BATRAW objectives for the first technical objectives (Chapter 3)

From DoA:

This report is related to T2.1. Disassembly, recycling processes, and validation specification [ORANO]LEITAT, CEA, TUB, IND, REC, CESV, COMA, REN, TOR M1-M6.

REN, CESVI, BEE, COMA, CEA, TUBS and LEITAT will carefully review the disassembly steps protocol from EV car to battery cells and complete a Failure Mode and Effect Analysis (FMEA) (D2.1).

CEA will define the dismantling program with support of REN and CESVI. COMA, BEE, LEITAT and TUBS will develop the technical specifications required for the disassembly of batteries to be done in WP3 at pilot level. Safety and automation specifications will be considered.

ORANO and IND will develop the technical specifications for each step of the recycling process in coordination with this task's partners (partners involved in WP3, 4 and 5). This includes the characteristics of the materials to be managed and treated at the beginning of each step of the process and delivered at the end of the process. The main parameters of the process to be monitored and the relevant analysis required to validate parameters of WP3, 4 and 5 will be defined (D2.2).

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LIST OF FIGURES

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1. Definition of the samples to be treated

Samples (EV battery packs/modules and BM from battery manufacturing scraps) will be delivered to all partners working on the optimisation of the different steps from the start of the project: Battery packs will be delivered to *WP2* and *WP3*, respectively, for second life applications, repair, and dismantling activities. Battery modules will be sent to *WP3* and *WP4* for dismantling and mechanical pre-treatment. Synthetic Black Mass will be prepared in *T5.1* starting from M3. This material will be used to start the leaching process optimisation before the BM is received from *WP4*. The BATRAW overall timing is illustrated in *Figure 1.* Samples of BM from battery manufacturing scraps will be delivered to *WP4* in M6 and samples of BM from domestic batteries will be delivered to *WP5* in M12. At M18 (*MS*2.2), battery packs and modules will be received by BEE and ORANO to prove the concept of each pilot line separately. The optimisation of the pilot line processes is starting at M3 (TRL4-5) and will continue till M36 (TRL6) where the optimised technologies are transferred to the pilots from M25 and not later than M36, corresponding to the achievements of the tasks at micro pilot level from *WP3*, 4 and 5. The pilot line activities will start from M18 (TRL6) and end by M40 (TRL7). As reported in *Figure 2* several steps are already available at TRL5- 6 prior to the project start. The integration of these technologies into the respective pilot lines will run in parallel to the optimisation of processes at lower TRL.

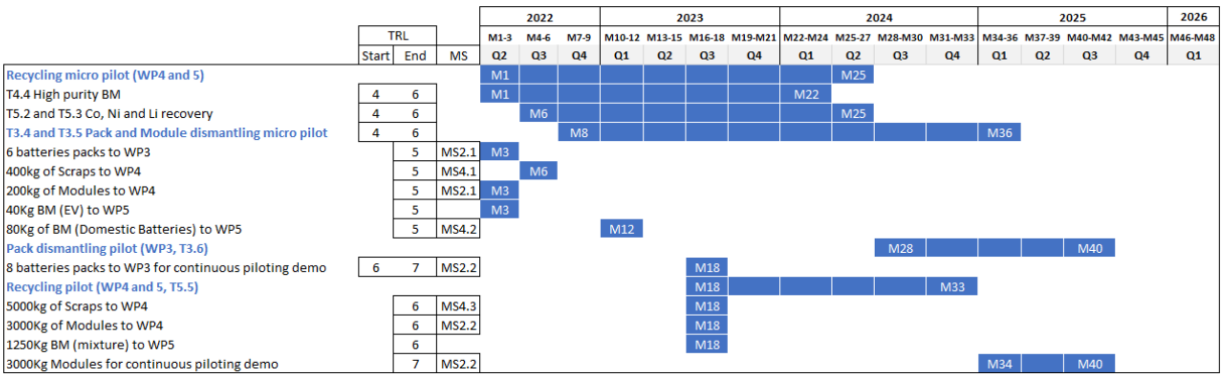

Figure 1.5 BATRAW methodology from micro pilot (TRL4-5) to pilot technology (TRL6) and system level (TRL7), including key milestones

FIGURE 1. BATRAW METHODOLOGY FROM MICRO PILOT (TRL(4-5)) TO PILOT TECHNOLOGY (TRL6) AND SYSTEM LEVEL (TRL7), INCLUDING KEY MS.

Collection and transport of samples

1. **EV battery packs and modules** – CESVI and BEE will deliver 7 battery packs and 180 modules, to the partners at micro pilot level, from the first months of the project (MS2.1, M3). CESVI, FORD and BEE will deliver 8 battery packs and 858 modules, to the partners at pilot level, at month (MS2.2, M18). It is estimated 15 packs (or 4,600tons) and 1038 modules (or 6,432tons) to be exchanged and treated along the project. The cell format will be essentially pouch and prismatic. These samples are currently in stock at partners CESVI, FORD and BEE. As mitigation plans if the samples are not available at the time of the project start then other consortium partners that have

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regular access to EV battery pack through their core activities (REC and REN) will provide the samples.

2. **Scraps from Li-ion battery manufacturing** – Wet and dry batteries, i.e., with or without electrolyte, coming from the European cell makers will be delivered to BATRAW via MTB. MTB will deliver 400kg of scraps to ORANO in WP4 at micro pilot level (M4.1, M6). An additional 5000kg will be delivered to ORANO at pilot level (M4.3, M20).

3. **BM from consumer batteries** – Lithium batteries present in WEEE, are mainly based on LCO and NMC chemistries, but batteries with cathode materials of different chemistries such as LFP, NCA and LMO can also be found. The BM coming from consumer batteries will be used to demonstrate the flexibility of the pilot (WP5) to recover Li, Ni, Mn, and Co independently of the purity of the BM. IND will deliver 80Kg of domestic battery BM to WP5 (M4.2, M12). IND will focus its activity to reach higher BM purity. The final quantity of BM to be treated at pilot level will be a mixture of BMs from the BATRAW WP4 mechanical pre-treatment of EV modules, scraps, and BM from domestic battery. The total quantity treated at pilot level is 4250kg BM at M20 (1250 kg) and M34 (3000 kg) to test respectively the hydrometallurgical pilot separately and in continuous operation with the pre-mechanical treatment.

4. **Synthetic BM** - Black Mass commercially available containing a mix of Li, Ni, Co, Mn. CEA will buy the synthetic BM (NMC622) and it will be distributed to LEITAT and ORANO to start activities in WP5 on the optimisation of processes from M3, before receiving real BM from WP4 .

5. **Synthetic liquors** – Synthetic liquors will be prepared by digestion in inorganic acids of the commercially available NMC622.

Working stations and scaling up of the technologies (TRL4-5 to TRL6) From M1 to M36, the optimisation of each innovation and its upscaling from micro pilot (TRL4-5) to technology demonstrated in relevant environment (TRL6) will be developed as following:

Technology	Scaling (from/to)	Partner (from/to)
Pack dismantled to module	1 pack dismantled each 3 hours/ 3 pack dismantled each 8 hours	LEITAT/BEE
Module dismantled to cell	1 module each 3 hours to 3 pack dismantled each 8 hours	TUBS/BEE
Pre-treatment in safe conditions and mechanical treatment, from module and/or cell to BM	From 30 kg/week to 300 kg/day	IWKS/ORANO
Cutting and cell component separation, specifically Al and BM separation	From 30 kg/week to 300 kg/day	MTB/ORANO
Electrolyte washing and C-graphite and Cu separation	From 100g/hours to 100Kg/day	CEA/ORANO
Leaching of the BM through inorganic acid, Mn and Li recovery.	From 5L/hours to 250L/hours	CEA/ORANO
Leaching of the BM through RTILs, DES and green solvent, Co and Ni separation and recovery through liquid-liquid membrane exchange and Li recovery through electrochemical process.	From 5L per batch/day to 250L/hours	LEITAT/ORANO
Li, Ni, and Co recovery through solvent extraction SX process (ORANO).	From 3L/hours to 250L/hours and improving efficiency from 90 to 95%	LEITAT/ORANO

FIGURE 2. LIST OF TECHNOLOGIES AND SCALE-UPS FROM M0 TO END OF PROJECT.

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Pilots: Disassembly, recycling, and hydrometallurgical processing (TRL6 to 7) The transport of batteries, as dangerous goods, will be evaluated and balanced to the transport of the full vehicle to the disassembly pilot location (Pilot 1 BEE, Spain) where the battery pack will be disassembled up to the module or the cell level. In case the performance electrical, mechanical, and thermal performances are satisfied, the disassembly of the pack will be stopped at module level. These modules can then be repaired, reintegrated in a new pack, or used in second life applications. In case the module is not fit for reuse, it will be explored if the disassembly to the battery cell level shows potential improvement of the recycling efficiency and BM purity. Modules and battery cells will be sent to the continuous pilot recycling line (Pilot 2, ORANO, France). Cathode material for lithium-ion manufacturing WP6 partners will receive C-graphite (*WP4)*, Li(OH).H2O, NiSO4.6H2O, and CoSO4.7H2O (WP5) for anode manufacturing and the proper synthesis of NMC622 and LNMO, respectively, as commodity product and Co-free material. LNMO will be produced at 25 kg batch level by TOR. POSCO will produce advance NMC9½½ at 2 kg/batch. The impact of the purity of the precursor from WP5 will be assessed and compared with commercial baseline and from their behaviour in full battery cell. The partners active in WP6 will prepare material (powder, ink, electrodes) and characterise them at all steps. REN will join the effort to participate to the assessment of the devices. The ranking of the materials produced, and their system associated, will be benchmarked following safety, cost, energy, power, and life cycle. Activities will start from M3 using commercial precursor starting with the definition, synthesis and assessment of a baseline material and system to enable comparison with BATRAW product.

Methodology to improve the sharing and assessment of relevant information (From M1) During all phases of the project, from WP1 up to WP7, MINE will evaluate which information is required from all project participants in their respective upstream and downstream supply or processing stages to carry out efficient recycling and assess the environmental and social sustainability of suppliers and components along supply chain, also considering the individual needs and limitations of the participants regarding confidentiality and transparency. Finally, MINE will evaluate or develop data models, templates and workflows and identify best practices and incentives regarding the sharing of information in an efficient and scalable way to facilitate the recycling of EV batteries and to improve the ecological and social sustainability of suppliers and components along the respective supply chains. This includes the generation of Battery Passports and Product Passports as well as the associated QR codes. MINE will assess whether data generated is of use to the EC RMIS (EC Raw Materials Information System). The JRC will be invited to join the BATRAW External Advisory Board if funding is granted.

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1.1. Battery pack and module from WP2 to WP2, 3, 4 and 5

BEE, Ford and CESVIMAP will receive several batteries coming from EVs, and they will obtain the necessary information about the battery parameters that may be useful for the consortium, such as number of modules, weights, dimensions, capacity, and battery chemistry. *Table 1* lists all the data collected from the batteries used for the project omitting all confidential information that cannot be added.

In addition, available information on other models currently on the market whose batteries could be used later in the process have been studied. The information has been provided by CESVIMAP thanks to its studies of these models, prior to the start of the project.

Finally, *Table 2* lists a few general parameters with respect to the modules and packs received.

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TABLE 1. TYPE OF BATTERY PACK AVAILABLE IN BATRAW PROJECT.

					Pack		Module			Cell
Type	Co	Partner	Origen	Destination	Nº available	kg	Nº available	Nº per pack	kg	Format
1	ES	BEE	Production waste	WP3 CEA	2	300	46	46	4	Pouch
2	ES	CESVI	EoL	WP3 CEA	40	300	816	48	4	Pouch
3	ES	CESVI	EoL	WP3 CEA	2	350	72	18	13	Pouch
4	ES	CESVI	EoL	WP3 CEA & LEITAT	2	243	16	8	28	Pouch, Prismatic
5	TR	FORD	Production waste	WP2 BEE (D2.6)	2	492	20	10	37	Pouch
6*	TR	FORD	Production waste	WP2 BEE (D2.6)	4	135	4	1	135	Pouch
7*	TR	FORD	Production waste	WP2 BEE (D2.6)	8	18	8	1	18	Pouch
8	ES	BEE	Production waste	WP2 BEE (D2.7)	1	350	2	18	13	Prismatic

*Models 6 and 7 are composed of 1 module packs. Both are going to be consider as isolated modules.

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Table 2. General parameters for BATRAW pack and module.

		Module level		Pack level	
Specification	Unit	Min	Max	Min	Max
Dimension					
Length	mm	303	540	1570	2300
Width	mm	200	375	1000	1500
High	mm	35	120	265	300
Weight	Kg	3	135	135	500
Electrical					
Energy	kWh	0.1	10	5	80
Nom. Volt.	Vn	7	25	360	360
Storage					
Temperature	ºC	20	30	20	30
Humidity	%	-	<85	-	<85

1.2. Scraps from Li-ion battery manufacturing from WP4 to WP5

Wet and dry batteries, i.e., with or without electrolyte, coming from the European cell makers will be delivered to BATRAW via MTB. MTB will deliver 400kg of scraps to ORANO in WP4 at micro pilot level (M4.1, M6). An additional 5000kg will be delivered to ORANO at pilot level (M4.3, M20).

1.3. Black Mass from WP4 to WP5

Consumer batteries vs. industrial batteries – Batteries are available in different sizes and shapes. According to their size, EBRA (European Battery Recycling Association) classifies the batteries into two groups: The small ones (powering torch, mobile phones, laptops…) are called 'portable' or 'consumer' batteries. The larger ones (powering professional tools, cars, etc.), specifically design for industrial applications are called 'industrial batteries'. This last category includes the batteries from hybrid or full electric cars, therefore EV batteries.

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BM from consumer batteries – Lithium batteries present in WEEE, are mainly based on LCO and NMC chemistries, but batteries with cathode materials of different chemistries such as LFP, NCA and LMO can also be found.

The BM coming from consumer batteries will have to lead with the impossibility of classifying the batteries according to their chemistry with total certainty. This is due to different reasons: The mix of batteries to be managed is very complex since it is a very heterogeneous mixture of different battery types and chemistries coming from different manufacturers and applications. In addition, the mix of batteries is contaminated with foreign materials such as wire, plastics, cardboards, other residues and so on.

The recycling process starts with a sorting step, which consists of removing the foreign materials and afterwards sorting the different batteries by chemistry. In order to assure an accurate sorting, one of the following two requirements would be needed: (1) technology able to identify different cathode materials of the batteries and sort them automatically, (2) labels on the battery indicating the chemistry.

Nowadays, there is a lack of technology for the identification and classification of batteries by their chemistry. Additionally, there is no label on the batteries that indicates the cathode material. This is the reason why the sorting is made manually and based on workers' knowledge gained thanks to their long experience in battery management.

During the manual sorting, batteries are separated attending the application they are coming from. For example, up to now batteries from laptops were considered Cobalt rich chemistry (high content in cobalt). However, this assumption is changing because the content of cobalt is decreasing and being replaced by LMO. Similarly occurs with batteries coming from other applications. In other words, batteries are separated according to different indicators (size, shape, color, voltage, the application they come from…), by which the battery chemistry is assumed, but errors may occur and the BM that is expected to be from a specific chemistry may contain material of other chemistries.

This BM will be used to demonstrate the flexibility of the pilot (WP5) to recover Li, Ni, Mn, and Co independently of the chemistry of the BM. IND will deliver 80Kg of domestic battery BM to WP5 (M4.2, M12). IND will focus its activity to reach higher BM purity. The final quantity of BM to be treated at pilot level will be a mixture of BMs from the BATRAW WP4 mechanical pre-treatment of EV modules, scraps, and BM from domestic battery. The total quantity treated at pilot level is 4250kg BM at M20 (1250 kg) and M34 (3000 kg) to test respectively the hydrometallurgical pilot separately and in continuous operation with the pre-mechanical treatment.

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BM form industrial batteries - This is the BM that comes from EVs batteries. As in the case of domestic batteries, the composition of the different components varies depending on the chemistry of the battery. For this group of batteries, the most common chemistries are NMC and LMO, however, there are also batteries whose chemistries are LFP, NCA.

The simplest way to classify batteries, and therefore the composition of their BMs, would be a classification according to the chemistry present in the battery. In order to do this effectively, prior identification of the battery chemistry by the disassembly team will be necessary.

Given that it will not be possible to obtain real BM until sometime after the start of the project and to be able to start working on the recovery processes of the different elements present in the batteries, it will be necessary to establish a synthetic BM as similar as possible to that which is expected to be extracted from the EV batteries. To do this, it is first necessary to know what composition is expected to be obtained from the real BM.

Actual BM – The composition of the actual BM will depend on the chemistry of the battery, the composition of the manufactured cathode (active material, binder, conductive carbon) and the anode used for each cathode type.

Different compositions are proposed depending on which chemistry is present in the battery. LMO, LCO, NCA, LFP, LNO, LTO and different NMC chemistries BMs compositions will be analysed. First, a summary of chemical composition (wt.%) of cathode and anode active material will be listed in *Tables 3 and 4.*

Later, expected composition of the actual BM from industrial batteries, including the electrodes, the battery cover and the electronic parts of the system will be displayed in *Table 5*. A division has been made according to the active material of the anode as the composition of the electrodes may vary in addition to the other components.

The sum of the information extracted from these tables will allow an accurate estimation of the composition of the real BM, however, all the information displayed in the tables will be subject to change when analysing the values from the actual BM. To complement these estimated values, it will be necessary to perform an ICP analysis of some of the BM obtained from the different batteries disassembled in the first stage of the project. These analyses will allow to know how adjusted the estimation has been and to readjust the discordant values.

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TABLE 3. EXPECTED COMPOSITION OF CATHODE ACTIVE MATERIAL FOR THE ACTUAL BM FROM INDUSTRIAL BATTERIES.

Cathode chemistry	Formula	Chemical composition (wt%)								
		Li	Ni	Mn	Co	Al	Ti	Fe	P	O
LCO	$LiCoO_2$	7.09	-	-	60.21	-	-	-	-	32.69
LNO	$LiNiO_2$	7.11	60.12	-	-	-	-	-	-	32.77
LMO	$LiMn_2O_4$	7.39	-	58.52	-	-	-	-	-	34.09
LTO	$LiTiO_2$	8.00	-	-	-	-	55.14	-	-	36.86
LFP	$LiFePO_4$	4.40	-	-	-	-	-	35.40	19.63	40.57
NCA	$LiNi_{0.8}Co_{0.15}Al_{0.05}O_2$	7.22	48.87	9.20	-	1.40	-	-	-	33.30
NMC532	$LiNi_{0.5}Mn_{0.3}Co_{0.2}O_2$	7.19	30.39	17.07	12.21	-	-	-	-	33.14
NCM622	$LiNi_{0.6}Mn_{0.2}Co_{0.2}O_2$	7.17	36.33	11.34	12.16	-	-	-	-	33.01
NMC811	$LiNi_{0.8}Mn_{0.1}Co_{0.1}O_2$	7.13	48.27	5.65	6.06	-	-	-	-	32.89

TABLE 4. EXPECTED COMPOSITION OF ANODE ACTIVE MATERIAL FOR THE ACTUAL BM FROM INDUSTRIAL BATTERIES.

Anode chemistry	Formula	Chemical composition (wt%)				
		Li	Ti	C	Si	O
Graphite	C	-	-	100	-	-
LTO	$Li_4Mn_5O_{12}$	6.04	52.15	-	-	41.81
LTO/Si (10%)	$Li_4Mn_5O_{12}$-Si	5.44	46.93	-	10.00	37.63

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Table 5. Expected composition of the actual BM from industrial batteries.

Battery	Type 1	Type 2
Anode	Graphite	LTO
Material composition (mass %)		
Cathode active material	22 - 25	27 - 29
Anode active material	15 - 17	18 - 20
Other elements present in the battery (mass %)		
Carbon	2 – 3	4 - 5
Binder	3 – 4	4 – 5
Cooper parts	13 – 14	2 - 3
Aluminum parts	12 – 13	13 - 14
Aluminum casting	9 – 10	8 - 9
Electrolyte solvent	12 - 14	13 -14
Plastics	4 - 5	3 - 4
Steel	0.1	0.1
Thermal insulator	1 - 2	1 - 2
Electronic parts	0.3 – 0.4	0.2 – 0.3

Synthetic BM – For the synthetic BM NMC622 will be used as baseline.

Black Mass commercially available containing a mix of Li, Ni, Co, Mn, CEA will buy the synthetic BM to be distributed to LEITAT and ORANO to start activities in WP5 on the optimisation of processes from M3, before producing real BM from WP4. CEA will buy and send NMC622 from TARGRAY to LEITAT(2Kg) and ORANO (5Kg) to start the first trials.

LEITAT and CEA performed an ICP analysis on the commercial NMC622(TARGRAY) that will be used as baseline.

For the first part of the study, pure NMC622 will be used as BM from which the synthetic liquors will be obtained. Once it has been verified that the extraction of the different raw materials is possible, the possibility of conducting a second part of the study adding other reagents to the commercial NMC622 will be considered. Some of the elements that would be interesting to add are lithium salts present in the electrolyte and Cu and Al sources to simulate all those components that have been dissolved in the BM and could not be removed in the previous stages of the process.

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TABLE 6. COMMERCIAL BM(TARGRAY NMC622) COMPOSITION.

	TARGRAY	CEA	LEITAT
Element	Chemical composition (w/w %)	Chemical composition (w/w %)	Chemical composition (w/w %)
Li	7-8	7.8	7.1
Ni	35.5-36.5	40.3	37
Mn	11.0-11.5	12.4	11
Co	11.5-12.5	12.6	12
Cu	< 0.005	0	<0.01
Fe	< 0.01	0	<0.1
Na	< 0.03	0.02	<0.1
Al	-	0.07	0.19
K	-	0	-

2. Pilot 1: semi-automatic battery pack disassembly

2.1. Specification for rapid characterization of EV

A quick characterization tool will be developed to determine packs SoC and SoH. The test method will be robustly design for different electrochemical technologies. At the same time procedures will be designed flexibles in order to adapt to battery pack models, operations and access inside.

The characterization of batteries is key to knowing their internal state. The assessment of SoH is normally carried out by means of a capacity measurement. However, the testing times and procedures to achieve this are excessive for the implementation of this method at a large scale.

In this context, a fast characterization procedure is sought in this project, so that SOH can be estimated from alternative parameters. The precision of the new method should be enough to allow the sorting of batteries between second-life use or recycling. The main challenge of any characterization procedure is the high voltage of battery packs, which requires specific equipment and increases handling risks. Moreover, access to the electric parts of the battery is required to perform any test, which can be difficult in electric vehicles.

Finally, in the method design, it will also be considered the available equipment and the necessary tools to proceed in a secure, accurate and rapid way.

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2.2. Specification for semi-automated and intelligent robotic processes (AI) for battery pack and module dismantling

2.2.1. KPI for semi-automated and intelligent robotic processes (AI) for battery pack dismantling at working station level.

LEITAT will develop the working station for semi-automated dismantling of the battery packs. A human-robot collaborative solution will be developed where the robot will assist the human operator in dismantling procedures.

Relevant process parameters and KPIs are listed in *Table 7.*

TABLE 7. KPI MICROPILOT SEMI AUTOMATIZATION OF BATTERY PACK DISASSEMBLY.

Parameters	Units	Initial KPI	Final KPI
Number of disassembled batteries per shift	Battery pack	1	2
Cycle time per battery	Hours	8	4
Battery detection	%	-	90
Correct unscrewing of screws by robot	%	-	90
Correct cable cut by robot	%	-	90
Automation Percentage of Operations over Cycle Ops.	%	0	50
Automation Time Percentage over Cycle Time	%	0	50
Percentage of Dangerous Human Operation over total	%	-	50
Reduction of needed workers	%	-	50
Nº of oral interactions between worker and robot over total interactions	% Oral interactions	-	50

2.2.2. KPI for semi-automated and intelligent robotic processes (AI) for battery pack dismantling at working station level

TUBS will develop the working station for semi-automated dismantling of the battery modules after the pack has been dismantled. A collaborative robot will be developed which trained using various learning methods. functionally integrated handling tools will be designed and developed for combined gripping and cutting/ joining operations.

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Relevant process parameters and KPIs are listed in *Table 8.*

TABLE 8.KPI MICROPILOT SEMI AUTOMATIZATION OF BATTERY MODULE DISASSEMBLY.

Parameters	Units	Initial KPI	Final KPI
Number of disassembled modules per shift	Module	10	13
Cycle time per module	Hours	0.8	0.6
Module detection	%	-	90
Correct unscrewing of screws by robot	%	-	90
Correct cable cut by robot	%	-	90
Automation Percentage of Operations over Cycle Ops.	%	0	50
Automation Time Percentage over Cycle Time	%	0	50
Percentage of Dangerous Human Operation over total	%	-	50
Reduction of needed workers	%	-	50
Nº of oral interactions between worker and robot over total interactions	% Oral interactions	-	30

2.2.3. KPI for semi-automated and intelligent robotic processes (AI) for battery pack and module dismantling at Pilot level

BEE and COMA with the support of TUBS and LEITAT will validate the pilot line and each of its working stations. Validation criteria will be based on inner distribution, pack dismantling capacity, error rate, safeness and security of each action, traceability of battery pack state and complete process time and similar. Corresponding validation report including techno-economic KPIs is *D3.8.*

Relevant process parameters and KPIs of the pilot level are listed in *Table 9*:

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TABLE 9.KPI TECHNOLOGY FOR PILOT SEMI AUTOMATIZATION OF BATTERY DISASSEMBLY.

Parameters	Units	Final KPI
Improvement in timing over a full manual process	%	30
Production capacity per shift	Battery pack	3
Batteries to repair	%	5
Batteries to reuse	%	5
Batteries to recycle	%	70
Waste mass	%	20
Operators per shift	Number of operators	2-3
Battery models	Number of different models	8
2nd life modules	%	20
Packs disassembled	Packs/day	3

2.3. Specification to recover battery pack components for recycling (modules, cells, WEEE, plastics)

BEE will evaluate ways for valorising waste fractions from dismantling process, including waste electronic valorisation e.g., WEEE like BMS and junction box. A procedure will be elaborated to evaluate final quality of the materials and their suitability to be part of second life products.

A complete circular approach will be carried out so that maximum percentage of EV battery pack components are reused. If they are damaged or unusable for this purpose, then WEEE or similar treatment inhouse or through a recycling partners will be made. This will be reported in *D3.2.*

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3. Pilot 2: pre-treatment and hydrometallurgical recycling - technical objectives

3.1. Specification for cell deactivation process in inert fluids

CEA will optimise a process for EV battery deactivation in an inert fluid, combined with a cutting step (confidential process (background) between CEA and ORANO). This process will be developed in accordance with all safety and security aspects and will be implemented on CEA site for validation on several types of cells (prismatic, pouch or cylindric cells) by performing pilot tests. CEA will provide safety recommendations to MTB and ORANO for the pilot demonstrator design for safe deactivation (*T5.4*). IND will test different deactivation technologies for lithium batteries coming from WEEE. The information regarding this topic will be displayed at *D4.1 Report on deactivation process and battery cells opening.*

TABLE 10.KPIS FOR CELL DEACTIVATION PROCESS IN INERT FLUIDS.

Parameters	Units	KPI
Number of modules treated per day	modules	Minimum 8
time to discharge 1 module to SOC = 0	Hours	< 4
Loss of Active Mass (NMC)	%	<1
Number of cycles allowed with inert liquid	cycles	Minimum 50
Versatility of the process	Types of cells	Prismatic and pouch

3.2. Specification for mechanical pre-treatment process for EV battery packs

ORANO with the support of MTB will develop the cutting process to open and to reduce the size of the battery modules and cells to sizes compatible with recycling processes. Different cutting solutions will be evaluated for different types of modules composed of prismatic, pouch or cylindric cells and compared in terms of quality, safety, and operational costs. Supported by ORANO, MTB will adapt cutting tools to further increase the recycling yield. MTB, IWK and IND will participate as experts in sorting solutions giving advice about the best practices to obtain good quality fractions for the subsequent actions. At the end of this task the most relevant tools will be assessed and tested to validate them with real data. The corresponding report will be delivered by MTB (*D4.2*).

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TABLE 11. KPIS FOR MECHANICAL PRE-TREATMENT PROCESS FOR EV BATTERIE PACKS.

Parameters	Units	KPI
Number of modules treated per day	modules	Maximum 50
Lead time to treat 1 module	Hours	< 4
Loss of Active Mass (NMC)	%	<1
Number of cycles allowed with inert liquid	cycles	Minimum 50
Active mass (NMC) purity	%	> 90
Cu and Al purity	%	> 95
Plastic recovery	%	> 90
Versatility of the process	Types of cells	Prismatic and pouch

3.3. Specification for pre-treatment of domestic batteries

IWKS and IND will develop a sorting system to separate the different fractions (plastics, Fe, Al, and Cu) and to produce the BM rich in metal content (Li, Ni, Mn, Co). IWKS will analyse the output fractions using element analytics (ICP-OES, XRF) and SEM/EDX mappings for the localization of impurities both in the black mass and on the plastic and metal fractions. As task leader, IWKS will check and evaluate the developed parameters to optimize the mechanical treatment step. These data will be considered as inputs to design the scale-up system and reported in *D4.4.*

3.4. Specification for a flexible and green hydrometallurgical process

LEITAT will benchmark RTILs and DES to extract selectively target metals from BM aiming to reach a greener process replacing current inorganic acids. Selected recovery routes will be tested at laboratory scale and the chemical composition of the resulting solutions versus the composition of the Black Mass starting material will be analysed. This method will be carried out in parallel and will compete with acid leaching, to see if it is a valuable option to implement in the process. CEA and ORANO will define a baseline for the NMC622 leaching conditions and LEITAT DES leaching.

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3.4.1. Inorganic Leaching

CEA and ORANO will define a baseline for acid leaching conditions for NMC622. As the conditions of this process are confidential between CEA and ORANO, LEITAT will propose two possibilities in case the above-mentioned process cannot be shared for the realisation of acid leaching.

For the inorganic leaching, mineral acid solutions like H_2SO_4 and HCl will be tested to achieve total dissolution of the NMC. Operational conditions are detailed in the *Table 12.*

TABLE 12. PROPOSED OPERATIONAL CONDITIONS FOR ACID LEACHING.

Acid	T (ºC)	Concentration	Liquid-solid ratio	Time (min)	Efficiency (%)	Liquor composition (g/L)
H_2SO_4	70	2 M	>33.3 g / L	90	Li: >99 Mn: >98 Ni, Co: >95	Li: 2-3 Ni: 11-12.5 Mn: 3-4 Co: 3.5-4.5
HCl	70	4 M	20 g / L	30	Li, Ni, Mn, Co: >95	Li: 3.5-4 Ni: 17.5-18.5 Mn: 5.5-6 Co: 5.5-6.5

3.4.2. DES Leaching

For the green leaching process, different Deep Eutectic Solvent (DES) mixtures will be tested in the NMC leaching process. Metal extraction efficiency will be compared in every mixture tested.

Operational conditions and results of the first trials are detailed in the *Table 13.* Reagents, temperature, and time have been omitted for confidentiality reasons.

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TABLE 13.DES LEACHING OPERATIONAL CONDITIONS.

Mixture number	T (°C)	Time (h)	Efficiency (%)	Liquor composition (mg/kg)
1	Very high	Long	Li: 60-70 Ni: <1 Mn, Co: 40-50	Li: 800-1100 Ni: <70 Mn: 850-1100 Co: 900-1200
2	Very high	Long	Li, Ni, Mn, Co: >85	Li: 1100-1400 Ni: 5900-6100 Mn: 1800-2000 Co: 1900-2100
3	High	Medium	Li, Ni, Mn, Co: 85-95	Li: 1100-1500 Ni: 5900-6800 Mn: 1800-2200 Co: 1900-2300
4	Medium	Long	Li, Ni, Mn, Co: 80-95	Li: 2100-2900 Ni: 10900-13300 Mn: 3400-4200 Co: 3500-4500

TABLE 14. KPI DES LEACHING PROCESS FOR CRM RECOVERY.

Parameters	Units	Micro pilot KPI	Pilot KPI	Beyond BATRAW
Leaching efficiency of the BM by DES	%	60	90	95
Purity of final products obtained after DES leaching	%	50	70	90
DES leaching solution produced per hour	L/h	2	20	100

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3.5. Specification for Mn extraction step in the HM process

The objective of this process step is to combine the cathode active material leaching step with manganese separation.

Under certain operating conditions, the dissolved manganese during leaching reacts with the metal oxides to form manganese dioxide and bring nickel, cobalt, and lithium into solution. This mechanism has been studied by the CEA and a publication has been made on this subject. (Billy, E.; Joulié, M.; Laucournet, R.; Boulineau, A.; De Vito, E.; Meyer, D. Dissolution mechanisms of LiNi1/3Mn1/3Co1/3O2 positive electrode material from lithium-ion batteries in acid solution. ACS Applied Materials & Interfaces **2018**, Emmanuel Billy_Trophées innovation recyclage– Catégorie académique- FEDEREC – 2019))

From a process point of view for the pilot, the manganese dioxide can then be easily filtered. However, the initial amount of dissolved Mn^{2+} is not sufficient to dissolve all the solid active material present in the reactor. It has been observed that the addition of $MnSO_4$ allows the dissolution reaction to continue. Subsequently, $MnSO_4$ will be used as a reducing agent to complete the dissolution of the active materials.

This process step has the advantage of separating Manganese upstream of the process, thus avoiding a liquid-liquid solvent extraction step (expensive process in terms of CAPEX and OPEX) later in the process. This process also allows to have a lower consumption of reducing agent compared to the classical black mass leaching processes realized today at industrial scale.

Within the framework of the project, the validation of the process step will first be carried out at CEA and the scaling up of the technology will take place on the ORANO site.

The first tests are performed on commercial NMC622 Targray materials. Later in the project, tests will be performed on real materials. In the case of the real black mass, it is expected that the amount of MnSO4 consumed for the complete dissolution of the NMC material will be lower than that used in the case of a commercial cathode material. This since the aluminum and copper residues in the collectors could themselves act as a reductant.

<u>Operating conditions:</u>

- Leaching with H_2SO_4.
- Addition of $MnSO_4$ to complete the dissolution of the metal oxides. The quantity of $MnSO_4$ to be added may vary depending on the stoichiometry and type of cathode materials to be dissolved.

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TABLE 15. KPIS FOR THE MN EXTRACTION IN THE HM PROCESS.

Parameters	Units	KPI
Leaching efficiency (Li)	%	> 98-99%
Leaching efficiency (Ni)	%	> 98-99%
Leaching efficiency (Co)	%	> 98-99%
[Mn] in the leachate	mg/L	< 100mg/L

Critical parameters for the process step:

- Temperature
- pH
- S/L ratio
- Dosage of $MnSO_4$

3.6. Specification for Co and Ni separation

Nowadays there are many methods available for metal recovery, but solvent extraction has been the most extended for industrial applications. Due to the need of large quantities of organic extractants, it is a very expensive process that needs to be improved. During this project, LEITAT will work on the separation and recovery of Co and Ni by the application of membrane technology, which is a cleaner technology that avoids great solvent consumption with lower energy requirements.

Polymer inclusion membranes (PIMs) are a very versatile type of polymer-based liquid membrane with an improved stability than supported liquid membranes (SLMs). PIMs are composed by a base polymer, an organic extractant and a plasticizer. The polymer provides the mechanical strength to the membrane, the extractant is an ion-exchanger or complexation agent which bonds to the specie of interest and is responsible of its transportation trough the membrane, and the plasticizer increases flexibility and elasticity of the membrane. The driving force is the gradient of concentration of the specie of interest between two aqueous solutions separated by the membrane: aqueous solution containing the specie and a stripping solution.

The work plan for membrane development will be as explained in the following tasks:

1. Selection of the extractants

The extraction capacity of Co and Ni by solvent extraction with different organic extractants (see *Table 16*) will be studied to find an efficient carrier for Co extraction and another efficient for Ni extraction. Liquid-liquid solvent extraction experiments

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will be carried out at different pHs to find the optimum conditions. The extractants which present higher capacity of extraction for Co and Ni will be selected for membrane development.

TABLE 16. ORGANIC EXTRACTANTS PROPOSED FOR LIQUID-LIQUID SOLVENT EXTRACTION EXPERIMENTS.

Extractant	Metal extraction	pH range
Aliquat 336	Co	2.5 – 5.5
Cyanex 272	Ni, Co	4.0 - 6.0
DEHPA	Mn, Co, Ni, Cu	2.0 – 4.2
Versatic acid	Ni	5.0 - 7.0
TOA	Co	< 6.0
LIX-84-I	Cu, Ni	4 – 8
LIX-84-ICNS	Ni, Co	8.7
LIX-984-N	Cu, Ni	7.3

TABLE 17. KPIS FOR NI AND CO LIQUID-LIQUID EXTRACTION IN HM PROCESS.

Parameters	Units	KPI
Extraction and stripping efficiency (Ni)	%	> 98-99%
Extraction and stripping efficiency (Co)	%	> 98-99%
Co and Ni salts		Battery grade specification

2. <u>Definition of stripping solution</u>

Once the extractant is selected, an aqueous stripping solution will be defined, containing a stripping agent with an optimal affinity for Co and Ni able to dissolve the metal and remove it from the membrane.

3. <u>Development of flat sheet polymer inclusion membranes</u>

Flat sheet PIMs containing the selected extractant will be developed containing cellulose triacetate (CTA) as base polymer and 2-Nitrophenyl octyl ether (2-NPOE) as plasticizer. These membranes will be prepared by drop-casting method and the optimal thickness will be studied. Alternatively, the use of polyvinylidene fluoride as base polymer will be also studied and optimized.

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4. Development of hollow fibre polymer inclusion membranes

To increase the membrane's active area, PIMs will be developed by dip-coating a porous PVDF hollow fibre membrane into a solution containing the extractant, the base polymer, the plasticizer defined before. The optimal conditions of the method will need to be further studied.

3.7. Specification for electrochemical Li recovery (ELR) in hydrometallurgical process

The objective of this final process will be to recover the maximum amount of Li present in the leachate using electrochemical techniques. For this purpose, it is intended to use a host structure such as the spinel LMO which, by means of an oxidation change in the manganese present in its structure, allows the insertion and disinsertion of the Li+ cations.

The major advantage of the electrochemical Li recovery process is the selectivity of the system towards Li ions. The presence of impurities remaining from previous processes such as Cu, Al, Mn, Co, or Ni does not present a problem since their size is too large to go inside the host structure. However, the existence of other impurities such as Na or K ions may present a problem for Li recovery. For this reason, a study of the selectivity of the LMO electrode for these impurities will be carried out.

LEITAT will optimise the upscaling of the most efficient lithium recovery from the BM dissolved from T5.1, from batch to batch to a continuous flow process. Lithium ion is pumped through electrode and released in neutral water, where the lithium will be recovered as a salt.

There are certain factors that can determine the electrochemical recovery of lithium such as the concentration of organic carbonates, the concentration of Li in the leachate, the pH of the leachate and the current applied in the electrochemical process.

Organic carbonates: These species are present in the electrolyte, and if they are not removed beforehand, they can fatally affect the electrochemical process. Organics carbonates can be adsorbed to the electrode surface and block charge transfer. Organic carbonates should be eliminated or at least reduced.

[Li+] concentration: The feasibility of the process can be improved by increasing the concentration (e.g. evaporating the solvent) and/or increasing the volume of the treatment cell.

pH: The most common industrial way to dissolve BM is to digest it in strong inorganic acids such as HCl or H_2SO_4, resulting in the formation of an acid leachate. At low pH there is no evidence of the intercalation process and only hydrogen evolution can be spotted. If the pH of the leachate is raised well-defined curves can be appreciated. In

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addition, pH rising can lead to the precipitation of organic carbonates and their extraction.

Applied current: The applied current can be related to the amount of Li released from the LMO structure, so ideally it should be as high as possible in order to recover as much Li as possible. However, if the value is too high, a decreased cation transfer yield is certainly observed due to a mechanical degradation of Electrochemical Junction Transfer (EJF) and to water oxidation. This reaction causes gas bubbles that could peel the matrix coating.

Factors relevant to the Li recovery process are listed in *Table 18*:

TABLE 18. SPECIFICATIONS FOR LI ELR.

Parameters	Units	Value Min	Value Max
Organic carbonates (PC, EC, DMC)	% v/v	0	5
[Li+]	mol · L-1	0.3	2
pH	-	5	9
Applied current	mA · cm-2	4	11

Once Li is released in neutral water, where the lithium will be recovered as a salt. The resulting salt will be recrystallized to form $LiOH.H_2O$ to be delivered to WP6. The optimization's process will consider the impact of the impurities, pH and initial [Li^+] concentration on the recovery efficiency and reported in *D5.3.*

KPIs for the ELR step are displayed in *Table 19*.

TABLE 19. KPIS FOR ELR IN THE HM PROCESS.

Parameters	Units	Baseline	KPI
Li recovery efficiency	%	90	95
Amount of treated liquor	L/h	3	250
Li salts	-	-	Battery grade specification

ORANO together with LEITAT will evaluate the performance of the ELR according to its process and integration feasibility.

Critical parameters for the pilot feasibility:

- Purity and recovery rate achieved.
- Equipment and materials required.
- Waste generated and energy consumption.
- Time consumed.
- Upscaling.

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3.8. Specification for chemical precursors for cathode and ceramic pigments materials synthesis

The following chapter is listing the specification for the purity grade and other parameter to be considered for ORANO pilot output.

3.8.1. Battery grade specifications for raw materials for synthesis of LMNO and ceramic pigments

The required purity of different precursors to be used for LMNO and ceramic pigments manufacturing are given bellow. It must be taken into consideration that indicated values are the first approximation and could vary in function of results during the development of the project.

On the other hand, it should be noted that the grade of hydration is not a sensitive parameter because all water is going to be eliminated during dry or high temperature synthesis processes.

Other impurities not included in next tables should be tested to know their influence in performance of final synthetized materials.

Lithium Hydroxide

Purity of $LiOH.H_2O$ should be 99 % with a content on LiOH of 56,5 wt% minimum. In terms of impurities, *Table 20* displays the limits on average on commercial material based on specifications:

TABLE 20. LITHIUM HYDROXYDE IMPURITIES SPECIFICATIONS.

CO_2	Na	K	Fe	Ca	Cl	Magnetic particles
< 0.35 %	<0.003 %	<0.003 %	<0.0008 %	<0.005 %	<0.005 %	<50 ppb

Any other deviation in impurities needs to be tested.

Manganese oxide

Purity of MnO_2 should be 92-95%. In terms of impurities, *Table 21* displays the limits in ppm on commercial material bases on specifications:

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TABLE 21. MANGANESE OXIDE IMPURITIES SPECIFICATIONS.

Fe	Ti	Al2O3	Na	K	Ca, Mg	Cu, Co, Cr, Ni, V, PB, Mo, Sn, As
30	10	300	2250	400	550	6

Manganese sulphate

The minimum metal content needed is 31.8wt% (Mn). In terms of impurities, *Table 22* displays the ideal limits in ppm on average on commercial material based on specifications:

TABLE 22. MANGANESE SULPHATE IMPURITIES SPECIFICATIONS.

Co	Fe	Ni	Cu	Na	Ca	Mg	Cl
100	100	5	100	3	12	12	5
Zn	Pb	Cr	Cd	Al	Si	K	
10	5	5	5	5	12	12	

Any other deviation in impurities needs to be tested. Nickel impurity is not limiting since Nickel is present in LMNO. For the manganese sulphate it is important to be selenium free.

TABLE 23. AVERAGE VALUES FOUND IN COMMERCIAL MANGANESE SULPHATE BATTERY GRADE.

Pb	As	Cd	Ca	Mg	Na	Zn	Cu
10	10	10	50	50	50	10	10

Average ppm in Mn sulphate. (Li ion manufacturing in Australia)

Nickel sulphate

Reference composition is $NiSO_4.6H_2O$ with a metal content 22.0-25 wt.% nickel (Ni) min. In terms of impurities, *Table 24* displays the ideal limits in ppm on average on commercial material based on their specifications:

TABLE 24. NICKEL SULPHATES IMPURITIES SPECIFICATIONS.

Co	Fe	Ni	Cu	Na	Ca	Mg	Cl
100	100	100	3	3	20	10	10
Zn	Pb	Cr	Cd	Al	Si	K	
10	3	5	1	1	10	10	

Any other deviation in impurities must be tested. Manganese is not limiting since manganese is present in LMNO. *Table 25* shows values found in commercial nickel sulphate battery grade depending on supplier.

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TABLE 25. COMMERCIAL NICKEL SULPHATE BATTERY GRADE SPECIFICATIONS DEPENDING ON SUPPLIERS.

Element	ppm		
	Europe	China	China
Co	2	10	30
Pb	1	2	6
As	1	1	1
Cd	1	2	2
Ca	1	7	5
Mg	1	20	20
Na	1	30	30
Zn	1	2	10
Cu	1	2	10

Average ppm in Mn sulphate. (Li ion manufacturing in Australia)

Cobalt Sulphate

For cobalt sulphate, as Co, wt % ≥ 20%

In terms of impurities, *Table 26* displays the ideal limits in ppm:

TABLE 26. COBALT SULPHATE SPECIFICATIONS.

Ni	Fe	Mn	Cu	Na	Ca	Mg
5	5	10	100	3	12	12
Zn	Pb	Cr	Cd	Al	Si	K
10	3	5	5	5	12	5

In the case of ceramic pigments, mainly oxides are used. The obtained precursors either oxides or the sulphates calcined into oxides should have following purity:

TABLE 27. OXIDES FOR CERAMIC PIGMENTS SYNTHESIS SPECIFICATIONS.

Oxide	Purity		d90 (micron)		Humidity
	Minimum	Optimum	Minimum	Optimum	
Cr_2O_3	> 95%	> 98%	8	2	< 1 %
Co_3O_4	> 95%	> 98%	8	4	< 1 %
Fe_2O_3	> 95%	> 98%	25	2	< 1 %
Mn_3O_4	> 95%	> 98%	6	2	< 1 %
NiO	> 95%	> 98%	16	5	< 1 %

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Other type of oxide precursors within above chemistries could be also considered.

4. Safety

REN, CESVI, BEE, COMA, CEA, TUBS and LEITAT will carefully review the disassembly steps protocol from EV car to battery cells and complete a Failure Mode and Effect Analysis (FMEA) (D2.1). The FMEA is a device used for the evaluation of capability disasters in a technique through figuring out their impact and severity inside the technique. It is a record that has been produced from the different partners of the BATRAW venture from special fields to count on viable issues and following mitigation plans regarding the automatization of the dismantling technique. The variety of perspectives within the partners has contributed to generate a huge record with quite a few cross-slicing knowledge, as a result being capable of cowl all the areas of the technique. However, the FMEA is a lively tool and, since improvement of the defined technique is not yet fully defined, an initial version of the device has been done in order to offer enough information to every partner to start developing and designing the entire dismantling process and differentiating among steps. This is a technique device so disasters and outcomes are centred on it and how they could fail.

5. Transport and storage

5.1. TRANSPORT

For transportation, the carrier should be informed about the danger level of the load, loading, and unloading locations in order to arrange the transport route. Lithium batteries transport always (waste or not) needs to be done under the *ADR (Agreement concerning the International Carriage of Dangerous Goods by Road)* regulation.

The ADR comprises regulations for road transport regarding packaging, load securing, classification, and labelling of dangerous goods. This means that required documents for hazardous goods transportation are ADR and Multimodal Dangerous Goods Form. These are mandatory documents necessary for customs clearance, land, and sea transportation.

Today, all EU members are signatories to the ADR. The ADR became effective through implementation in the respective national law). When lithium batteries reach their EoL they become hazardous waste, therefore the EU waste directive applies. Moreover, EU regulation is now under development, and it is foreseen to be implemented by 2023.

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5.2. STORAGE

Batteries should be stored in UN coded packaging with MSDS forms. The most important issue in storage is that the batteries are not stackable.

In Spain the Law 7/2022, of April 8, on waste and contaminated soil for a circular economy (Article 21) specifies the obligations of the initial producer or other possessor related to the storage, mixing, packaging and labelling of waste.

It is specified that containers or packaging containing hazardous waste must be clearly and visibly labelled, legible and indelible, at least in the official Spanish language of the State. In addition, it is also specified all the data and codification that the label must contain. However, regarding the storage, it is not clearly specified how the waste must be packaged, the text is ambiguous where it is only said that hazardous waste must be contained in a closed container, but no further guidelines are given regarding the types of containers, insulating materials that can be used to cover the waste, etc.

Within Indumetal Group, lithium batteries are always stored in drums covered with vermiculite (insulating material) and placed inside a concrete bunker. This procedure is part of a good internal practice to minimize the risks associated with lithium's reactivity, but it is not a mandatory procedure according to any national nor European law.

Lithium storage in Indumetal Recycling

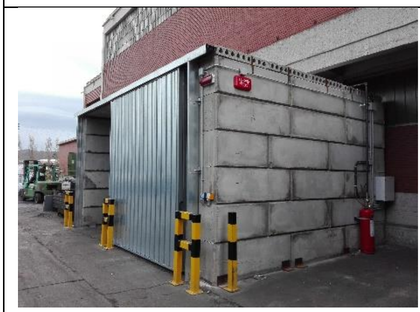

FIGURE 3.CONCRETE BUNKER

FIGURE 4. DRUMS INSIDE THE BUNKER

FIGURE 5. BATTERIES COVERED WITH VERMICULITE.

TABLE 28.BATRAW FIRST BATTERY DELIVERIES.

From	To	Shipping Date	Arrival Date	Cost euros/ton
Ford Turkey	BEE Spain	14.09.2022	30.09.2022	3880
BEE Spain	CEA Spain	07.10.2022	14.10.2022	1770

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6. Responsibility

As a general principle the responsibility it transferred from the partner delivering the pack or module to the partner receiving the pack or module. The responsible oversees the reuse or recycling of the pack and module.

7. Conclusion

A general outline has been successfully established for all the procedures that are going to be carried out in the project. All specifications were considered, starting from the arrival of the batteries, until the characteristics of the recovered materials (*e.g.,* purity, form) needed to close the cycle (*i.e.,* reincorporation into the battery chain production). This overview will allow a better understanding of the whole process, as well as the possibility to readapt technologies or procedures.

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8. Annexes

8.1. BATRAW objectives and KPI

Obj. nº	Type	KPI nº	Description	Baseline at month M0	Expected final Value	Expected Date	Task	Deliverable/milestone
1	Technical	1	Pilot 1: semi-automatic battery pack disassembly	Process is mostly manual limited by worker number and formation	1 ton/8 hours	M40	3.6	D3.7, D3.8
		1.1	Tool for rapid characterization of EV	> 1 hour	SoH in < 30 minutes	M17	2.4, 3.3	D2.6, D3.4, D3.9
		1.2	To develop and demonstrate semi-automated and intelligent robotic processes (AI) for battery pack and module dismantling	300kg/day (or 1 pack)	1 ton/8 hours	M36	3.4, 3.6	MS5, MS6, D3.5, D3.6
		1.3	To recover battery pack components for recycling	-	95% in weight	M15	3.7	D3.2
		1.4	To demonstrate technologies and processes at pilot line	300kg/day (or 1 pack)	1 ton/8 hours	M40	3.6	D3.7, D3.8
2	Technical	2	Pilot 2: pre-treatment and hydrometallurgical recycling	30Kg/week	300kg BM/day	M40	5.5	D5.5
		2.1	To upscale and demonstrate cell deactivation process in inert fluids	Module short circuit and Shredding	50 KWh pack deactivation	M18	4.1	D4.1
		2.2	To demonstrate mechanical pre-treatment process for EV battery packs achieving recovery of Al, Cu, and C and high-purity black mass	Pyrometallurgy to slag	High-purity black mass (>92% Co, Ni, Mn, Li)	M22	4.4	D4.4
		2.3	To upscale pre-treatment of domestic batteries and ensure separation of different fractions and to produce BM rich in Li, Ni, Mn, Co	Cryogenization of the full battery	BM separation for non-EV and domestic battery	M26	4.5	D4.6
		2.4	To upscale and demonstrate a flexible and green hydrometallurgical process	Inorganic acid used for leaching	Green solvents, efficiency > 95% and purity > 99%	M12	5.1	MS12
		2.5	To upscale and demonstrate innovative Mn extraction step	100g/h	250L/h	M20	5.1	MS10
		2.6	To upscale and demonstrate electrochemical Li recovery	3L/h	250L/h	M24	5.2	D5.3
		2.7	To demonstrate continuous recycling process in a scalable pilot line	30Kg/week	300kg BM/day	M40	5.5	D5.5
3	General	3	To deliver battery grade NMC811, MMC9½½ and LMNO cathodes	< 50€/kWh (battery grade cathode)	< 30€/kWh	M48	6.2	D6.3
		4	To build a prototype of second life battery from BATRAW sample	-	2nd life prototype	M17	2.4	D2.6
		5	To develop and deliver eco-design guidelines on pack manufacturing supporting better repair and dismantling of large battery packs	-	Guideline (eco-design pack construction)	M48	7.3	D7.3
		6	To develop and deliver guidelines and best practices for safe handling and transport of end-of-life battery waste	-	Guideline (pack handling and transport)	M24	2.3	D2.4
		7	To develop and demonstrate a blockchain platform for RM, product, and supply chain tracking	-	Battery Passport	M48	7.1	D7.1
		8	To feed the EC's Raw Materials Information System (RMIS) by the IMS; to contribute to policies and standardisation	-	2-3 policy briefs and associated dossier	M48	8.4	D8.4
		9	To fully substantiate an exploitation and business plan that takes a broad EU-wide focus, including Circular Business models	-	Exploitation and business plan	M45	7.7	D7.8
		10	To quantify environmental impacts and benefits of BATRAW results by LCA study	-	Environmental and social assessment report	M42	7.3	D7.4

FIGURE 6. DESCRIPTION OF BATRAW OBJECTIVES AND KPIS.

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