Standard Operating Procedure and Checklist of Minimal Requisite Facilities for utilization of hazardous waste under Rule-9 of the Hazardous and Other Wastes (Management and Transboundary Movement) Rules – 2016

Utilization of Black mass (generated by Lithium Ion Batteries dismantlers/recyclers or e-waste dismantlers/recyclers) for the recovery of Carbon/Graphite material and Metal compounds (Sulphates, Carbonates, Phosphates of Co, Mn, Ni, Li, Cu, Fe, Al & Na) by adopting Hydro-Metallurgy

CPCB

January, 2025

Central Pollution Control Board
(Ministry of Environment, Forest & Climate Change, Government of India)
Parivesh Bhawan, East Arjun Nagar,
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Procedure for grant of authorization by State Pollution Control Board (SPCBs)/Pollution Control Committee (PCCs) for utilization of Hazardous waste

1) While granting authorization for utilization of hazardous wastes, SPCBs/PCCs shall ensure that authorization is given only to those wastes for which Standard Operating Procedures (SoPs) for utilisation have been circulated by CPCB ensuring the following:

    a. The hazardous waste (intended for utilization) belongs to same source of generation as specified in SoP.

    b. The utilization shall be same as described in SoP.

    c. End-use/ product produced from the waste shall be same as specified in SoP.

    d. Authorization shall be granted only after verification of details and minimum requisite facilities as given in SoP.

    e. Issuance of passbooks (similar to passbooks issued for recycling of used oil, waste oil, non-ferrous scraps, etc.) for maintaining records of receipt of hazardous waste for utilization.

    f. Monitor closely the quantity of Black mass generated (generated by authorized lithium ion batteries dismantlers/recyclers or e-waste dismantlers/recyclers) and sold to utilizers.

2) After issuance of authorization, SPCBs/PCCs shall verify the compliance of checklist and SoP on quarterly basis for initial 1 year; followed by random checks during subsequent period for atleast once a year. The compliance reports may be submitted to CPCB.

3) In-case of lack of requisite infrastructures with the SPCBs/PCCs, they may engage 3rd party institutions or laboratories having EPA/NABL/ISO17025 accreditation/ recognition for monitoring and analysis of prescribed parameters in SoPs for verification purpose.

4) SPCBs/PCCs shall provide half yearly updated list of units permitted under Rule 9 of Hazardous & Other Wastes (Management & Transboundary Movement) [HOWM] Rules, 2016 to CPCB and also upload the same on SPCB/PCC website, periodically. Such updated list shall be sent to CPCB.

5) Authorization for utilisation shall not be given to the units located in the State/Union Territory where there is no Common TSDF, unless the unit ensures authorised captive disposal of the hazardous waste (if any generated during utilisation) or its complete utilisation or arrangement for transfer to authorised disposal facility.

6) In case of the utilization proposal is not same with respect to source of generation or utilization process or end-use as outlined in this SoP, the same may be referred to CPCB for clarification /conducting trial studies and developing SoPs thereof.

7) The source and work zone standards suggested in the SoP are based on E(P)A notified and OSHA/NAAQ standard, respectively. However, SPCBs/PCCs may impose more stringent standards based on the location or process specific conditions.

8) SPCBs/PCCs shall ensure that the utiliser of Black mass (generated from crushing and shredding of Lithium-ion batteries generated by batteries dismantlers/recyclers or e-waste dismantlers/recyclers) shall maintain daily records on National Hazardous Waste Tracking System (NHWTS). The manifest system should also be generated on NHWTS.

115.0 Utilization of hazardous waste (H.W.):

Type of HW	Source of generation	Recovery/ Product
Black mass (under the Category: A6/ A65/ A66 A68/ A72 of Schedule-II under HOWM Rules, 2016 accordingly as per the type of Li ion Battery)	Generated from dismantling, crushing and shredding of waste Lithium-Ion Batteries generated by Lithium Ion Batteries dismantlers/ recyclers or e-waste dismantlers/ recyclers.	Recovery of Carbon and Metal compounds (Sulphates, Carbonates, Phosphates of Co, Mn, Ni, Li, Cu, Fe, Al & Na) for downstream industrial use.

115.1 Source of Waste:

Black mass is hazardous material generated by authorised lithium ion batteries dismantling/recycling units or e-waste dismantling/recycling facilities involved in crushing and shredding of waste Lithium-Ion Batteries, after physically separating material like copper, plastics, Aluminum, steel, etc. It majorly comprising of the anode & cathode material; and categorized as Hazardous waste under A6/ A65/ A66 A68/ A72 of Schedule-II of HOWM Rules 2016 where constituents may vary depending on the type of Li ion battery from which Black mass is generated; which is required to be disposed in an authorized disposal facility in accordance with condition, when not utilized as resource recovery.

Table 1. Typical Characteristics of Black mass (BM from 2 types of Batteries studied during trial run)

S. NO.	TEST PARAMETERS	Unit	Results	
			Black mass from Battery type-1 (BM-1)	Black mass from Battery type-2 (BM-2)
1.	pH	-	8.90 - 9.02	6.0 - 8.26
2.	Moisture	%	2.27 - 3.4	6.4 - 8.1

3.	Total Organic Carbon	%	1.09 - 1.64	2.35
Leaching concentrations				
4.	Ammonia (as NH_3)	mg/L	3.68 - 3.77	1.38-1.53
5.	Cyanide (as CN-)	mg/L	7.15	5.96
6.	Sulphide (as S^{-2})	mg/L	BDL	BDL
7.	Phosphate (as PO_4)	mg/L	0.32 - 0.35	0.3
8.	Fluoride	mg/L	310.9 - 342	501.9 - 548
9.	Cobalt (as Co)	mg/L	259.6 - 285.3	3.48 - 3.79
10.	Iron (as Fe)	mg/L	0.462 - 0.508	11.69 - 12.81
11.	Nickel (as Ni)	mg/L	21.9 - 24.2	0.585 - 0.653
12.	Lead (as Pb)	mg/L	0.17	BDL
13.	Manganese (as Mn)	mg/L	277.1 - 302	0.602 - 0.661
14.	Zinc (as Zn)	mg/L	0.524	0.098
15.	Aluminium (as Al)	mg/L	98.4 - 105.7	61.8
16.	Cadmium (as Cd)	mg/L	BDL	BDL
17.	Copper (as Cu)	mg/L	234.4 - 259.8	52.68 - 58.6
18.	Antimony (as Sb)	mg/L	BDL	BDL
19.	Molybdenum (as Mo)	mg/L	BDL	BDL
20.	Mercury (as Hg)	mg/L	BDL	BDL
21.	Vanadium (as V)	mg/L	0.013	1.305
Total concentrations				
22.	Titanium (as Ti)	mg/L	BDL	BDL
23.	Lithium (as Li)	mg/L	38500 - 39400	25400

BDL: Below Detectable Limit (Sulphide <1.0 mg/L, Pb <0.1 mg/L, Cd <0.05mg/L, Sb<0.1mg/L, Mo<0.5mg/L, Hg<0.1mg/L, Ti<2.5mg/L)

ND: Not Detected

Note: It is expected that there may be large variation in metal constituents in Black Mass, depending on type of batteries used in production. SPCBs/PCCs need to check the characteristics of Black mass (by identifying type of waste Li ion battery used) prior to issuance of authorization, any significant deviation with respect to typical values mentioned in the table above may be examined with respect to the source and available process units or may be referred to CPCB.

115.2 Utilization process of Black mass at Production Stage:

Hydrometallurgical process is adopted to recover the metal compounds of Co, Mn, Ni, Li, Cu, Al & Carbon from the hazardous waste i.e., Black mass.

Black Mass alongwith water, Sulphuric acid and leaching reagent are fed to the leaching reactor. pH and temperature conditions are controlled during this process. The leachate is separated from the leached material via filtration, and the resulting solid residue (cake). The cake formed from the filtration of leached liquor is washed with water & collected as Graphite/carbon material. If the filtered cake contains traces of iron, then the same is re-leached followed by filtration to obtain graphite cake and Iron salt by precipitating the filtrate.

The filtered leach liquor is processed through a purification process through oxidising reagents and soda ash followed by filtration process. The obtained cake is leached for extraction of Copper and further stripped from the solution using sulfuric acid, yielding a copper sulfate solution. Subsequent evaporation results in the production of copper sulfate. Post-copper removal, the cake undergoes further purification/extraction processes to obtain Alumina (Aluminium Hydroxy Carbonate).

The filtrate generated from purification unit is directed to solvent extraction circuit to sequentially recover manganese, cobalt, nickel, and lithium by using organic extracting solvents.

The extracted manganese, Cobalt, Nickel and Lithium are stripped separately (solvent extraction with sulphuric acid followed by evaporation/ precipitation using soda ash to get respective manganese salts (Manganese sulfate/ Manganese carbonate, Cobalt salts (Cobalt sulfate/cobalt carbonate), Nickel salts (Nickel sulfate/ Nickel Carbonate), and Lithium salts (Lithium Sulfate/Lithium Carbonate).

The raffinate (the remaining filtrate after removal of metal compounds) from solvent extraction circuit contains mainly sodium sulphate solution which is further processed to produce Sodium sulphate through MEE route where Sodium sulphate crystals are recovered. The condensate water generated from MEE plant is recycled in the process for leaching or washings of process units etc.

Note: In liquid-liquid extraction, the extract is the layer with the solvent and the desired substance, while the raffinate is the remaining phase depleted of that substance.

A typical process flow diagram is given at figure given below:

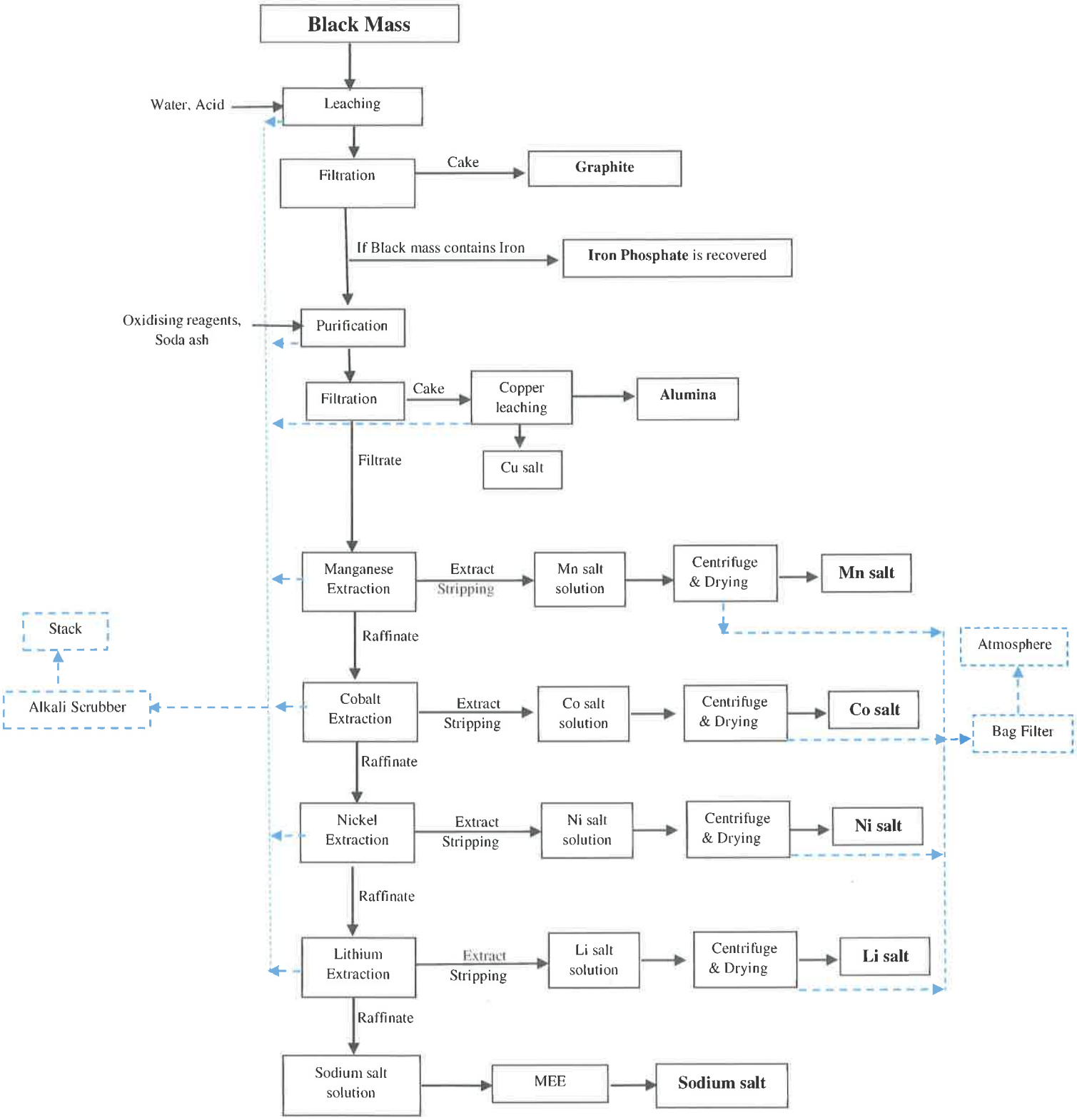

Figure: 1-Process flow diagram for utilization of Black mass

115.3 Standard Operating Procedure for utilization of Black mass:

This SoP is applicable only for utilization of Black mass (generated by lithium ion batteries dismantlers/recyclers or e-waste dismantlers/recyclers) for the recovery of carbon/graphite material and metal compounds (Sulphates, Carbonates, Phosphates of Co, Mn, Ni, Li, Cu, Fe, Al & Na) by adopting Hydro-Metallurgy. These SOPs shall become part of the comprehensive guidelines of CPCB on waste batteries recycling, if it involves captive utilization of black mass generated from lithium ion batteries recycling.

1) The utilizer (unit) shall procure the Black mass from the batteries dismantlers/ recyclers or e-waste dismantlers/ recyclers units complying with comprehensive guidelines on recycling of waste batteries issued by CPCB and authorised by SPCBs/PCCs, in leakproof HDPE bags in trucks registered with SPCB/PCC fitted with requisite safeguards ensuring no spillage.

2) There shall be a designated space for unloading/ keeping of HDPE bags of Black mass in storage sheds. The receiving waste shall be placed above the ground on a pallet, enclosed with low raise parapet wall to contain spillages, if any, alternatively, the bags may be placed above spill containment pallet.

3) The Black mass shall be stored in designated covered storage area (with caution sign) within premises on pallets placed on acid proof brick/tile lined floor area.

4) The unit shall provide separate storage tanks for other raw materials of utilization process i.e., solvents, reagent chemicals, intermediate raffinates, acids etc in dedicated storage area on acid proof brick/tile lined area within premises with caution sign.

5) The Storage and utilization area should have equipped with proper firefighting equipment and fire hydrant system to avoid the fire hazard especially in solvent storage area.

6) The Unit shall obtain necessary PESO license if required as per threshold quantum of solvents stored for the purpose of utilization process.

7) The entire utilization process area shall have leak-proof and acid proof tiles with adequate slope to collect spillages, if any, and shall be transferred to ETP or reaction tanks, as the case may be, through chemical process pump.

8) The unloading, storage, transfer and other handling of Black mass in entire utilization process shall be carried out through dedicated mechanical means minimizing manual intervention.

9) The unit shall ensure that the said utilization process and its associated activities shall be demarcated separately within premises.

10) The unit shall provide a suction hood connected to a bag filter system to control fugitive emissions during the charging of Black Mass (Particulate Matter) into the reactor.

11) The unit shall provide acid proof suction ducts connected to receive acidic fumes if any from the reactors (leaching and extraction units). These ducts may be connected to a common manifold duct, leading to an alkaline scrubbing system.

12) The unit shall provide bag filters to control metallic salt dust generated from the salt crushing process.

13) The unit shall provide fume extractions system followed by activated carbon filter to control VOC fumes in the solvent extraction unit area.

14) The treated gases / dust emissions from above mentioned pollution control systems shall comply with the emission standards and then only be released in the atmosphere through dedicated stacks. The stack height shall be a minimum of 30m from ground level or as prescribed by the concerned SPCB/PCC, whichever is higher.

15) Treatment and disposal of wastewater - Wastewater generated from the process, floor washing, spillage, reactor washing, scrubber bleed, condensate from MEE etc. may be reused in the process or treated physio chemically in an ETP to comply with wastewater discharge standards and may be sent to CETP for final disposal as prescribed by SPCB/PCC. In case of zero discharge, the treated waste water from ETP may be managed as per conditions stipulated by the SPCB/ PCC.

16) The treated effluent shall be discharged in accordance with the conditions stipulated in the Consent to Operate issued by concerned SPCB/PCC under the Water (Prevention and Control of Pollution) Act, 1974.

17) SPCBs/ PCCs shall ensure that recovered metal compounds containing traces of other heavy metals as impurities; may be permitted for end usage by industries only subject to meeting the criteria as supplementary material [as mentioned in section 115.4 (1)] and in no case shall be permitted for use in the food, pharma, animal feed, cosmetic, nutrient or fertilizer sector.

    Further, any fraction of recovered material not meeting the end usage quality requirements of downstream industries shall be treated as hazardous waste, which may be sent for co-processing in cement plants or disposed through TSDF.

18) The hazardous wastes (namely floor scrapping /sweepings, filter residue, ETP sludge etc.) generated shall be collected and temporarily stored in non-reactive drums/ bags category wise under a dedicated hazardous waste storage area having proper caution sign and be sent to authorized common TSDF or other authorized facility within 90 days from the generation of the waste in accordance with the authorization issued by the concerned SPCB/PCC. Such storage area shall have proper ventilation.

19) The unit shall ensure that the Black mass is procured from the industries, which have valid authorization from the concerned SPCB/PCC as required under HOWM Rules, 2016.

20) Transportation of Black mass shall be carried out by sender (generator) or receiver (utilizer) only after obtaining authorization from the concerned SPCB under Hazardous

and Other Wastes (Management and Transboundary Movement) Rules, 2016. Requisite manifest document shall be followed as laid down under the said Rules on national hazardous waste tracking system.

21) Prior to utilization of Black mass, the unit shall obtain authorization for collection, storage and utilization of Black mass from the concerned SPCB/ PCC under Hazardous and Other Wastes (Management and Transboundary Movement) Rules, 2016.

22) The unit shall maintain proper ventilation in the work zone and process areas. All personnel involved in the plant operation shall wear proper personal protective equipment (PPE) specific to the process operations involved and type of chemicals handled as per Material Safety Data Sheet (MSDS). The safety precautions of the worker shall be in accordance with the Factory Act, 1948, as amended from time to time.

23) In case of environmental damages arising due to improper handling of hazardous wastes including accidental spillage during generation, storage, processing, transportation and disposal, the occupier (sender or receiver, as the case may be) shall be liable to implement immediate response measures, environmental site assessment and remediation of contaminated soil / groundwater / sediment etc. as per the "Guidelines on Implementing Liabilities for Environmental Damages due to Handling & Disposal of Hazardous Wastes and Penalty" published by CPCB.

24) During the process of utilization and handling of hazardous waste the unit shall comply with requirement in accordance with the Public Liability Insurance Act, 1991 as amended, wherever applicable.

25) The unit shall provide suitable fire safety arrangements and flame proof electrical fittings.

115.4 Product Usage / Utilization

1) The recovered materials, i.e., graphite/carbon material and metal compounds (such as sulfates, carbonates, and phosphates of Co, Mn, Ni, Li, Cu, Fe, Al, and Na), shall be permitted for use in industries only under the following conditions:

   1.1 The recovered materials shall meet the quality criteria as supplementary material for downstream industrial usage with respect to purity including BIS quality specifications if any. Accordingly, the SPCBs/PCCs shall monitor the quality of recovered material required for domestic end users and the applicable standards such as BIS specifications.

   1.2 The end use of recovered materials i.e., Graphite/ Carbon material and metal compounds (Sulphates, Carbonates, Phosphates of Co, Mn, Ni, Li, Cu, Fe, Al & Na) shall not be utilized in the food, pharma, animal feed, cosmetic, nutrient or fertilizer sector in any form.

1.3 The recommended industrial uses of Recovered Products from the Black mass through hydro-metallurgical route are as follows:

S. No.	Name of products	End Use in industries for production of
1.	Graphite/Carbon material	Industrial Electrodes
2.	Aluminium carbonate Solid (Alumina)	Co-processing in Cement Plant
3.	Copper Salt	Pigments, paints, Varnish
4.	Manganese Salt	Pigments, Glaze, Ceramics
5.	Cobalt Salt	Pigments, electroplating, Catalyst, Ceramics, Paints, Pigments, purified cobalt salt
6.	Nickel Salt	Catalyst, Electroplating, Ceramics, purified nickel salt
7.	Lithium Salt	Glass, Ceramics, purified lithium salt
8.	Iron Salt	Paints, Pesticides
9.	Sodium Sulphate	Paper, Specialty Chemicals

2) Any fraction of recovered material not meeting the end usage quality requirements of downstream industries shall be treated as hazardous waste, which may be sent for re-processing or co-processing in cement plants or disposed through TSDF.

3) The unit shall label its products i.e. Recovered metal compounds/ Graphite prepared by utilizing aforesaid Black mass as "*This Carbon/[metal] salt has been prepared by utilizing Black mass (generated in crushing and shredding of Lithium-ion batteries)*".

115.5 Record/Returns Filing

1) The unit shall maintain a passbook issued by concern SPCB/PCC and maintain details of each procurement of Black mass as mentioned below:

- Address of the sender
- Date of dispatch
- Quantity procured
- Seal and signature of the sender
- Date of Receipt in the premises

Above records shall also be maintained on National Hazardous Waste Tracking System.

2) A log book with information on source and date of procurement of Black mass, date wise utilization of the same, hazardous waste generation and its disposal, etc. shall be maintained including analysis report of emission monitoring & effluent discharged, as applicable.

3) The unit shall maintain records on sales details of the recovered products (manufactured by utilizing Black mass).

4) The unit shall maintain record of hazardous waste generated, utilized and disposed as

per Form-3 & also file an annual return in Form-4 as per Rule 20 (1) and (2) of HOWM Rules, 2016, to concerned SPCB/PCC.

5) The unit shall submit quarterly and annual information on hazardous wastes consumed, its source, products generated or resources conserved (specifying the details like, type and quantity of resources conserved) to the concerned SPCB/PCC.

6) The unit shall use NHWTS to manage the manifest, enter daily records of quantity generated, disposed, etc.

115.6 Standards

1) Source emissions from the stack connected to reactors/ process unit shall comply with the following Emission standards or as prescribed by the concerned SPCB/PCC, whichever is stringent:

Particulate Matter	50 mg/Nm^3
Manganese as Mn	5 mg/Nm^3
Sulphuric acid mist	50 mg/Nm^3
Total Fluoride	25 mg/Nm^3
TOC	20 mg/Nm^3

2) Work zone emission in the work zone area shall comply with the following standards:

PM_{10}	5 mg/m^3 TWA* (PEL)
Sulphuric acid	1 mg/m^3 TWA* (PEL)
Hydrogen Fluoride	3 ppm TWA* (PEL)
Fluorides (as F)	2.5 mg/m^3 TWA* (PEL)
Manganese compounds (as Mn)	5 mg/m^3 #
Cobalt metal, dust, and fume (as Co)	0.1 mg/m^3 TWA* (PEL)
Copper Dusts and mists (as Cu)	1 mg/m^3 TWA* (PEL)
Nickel	1 mg/m^3 TWA* (PEL)

*PEL - Permissible Exposure Limit.

*Time-weighted average (TWA)- measured over a period of 8 hours of operation of process.

- A ceiling limit is one that may not be exceeded for any period of time, and is applied to irritants and other materials that have immediate effects.

3) Monitoring of the above specified parameters for Source emissions and Work zone emission shall be carried out quarterly for first year followed by at least annually in the subsequent year of utilization. The monitoring shall be carried out by ISO 17025 accredited or EPA, 1986 approved laboratories and the results shall be submitted to the concerned SPCB/PCC on a quarterly basis.

4) Standard for wastewater discharge: Treated effluent shall be discharged in accordance with the conditions stipulated in Consent to Operate issued by concerned SPCB/PCC under the Water (Prevention and Control of Pollution) Act, 1974. In case of (i) zero discharge as per consent or (ii) non-availability of the common Effluent Treatment Plant (CETP), the unit shall achieve zero discharge by setting up adequate captive treatment facility.

115.7 Siting of Industry

Facilities for utilization of Process residue shall be preferably located in a notified industrial area or industrial park/estate/cluster and in accordance with Consent to Establish issued by the concerned SPCB/PCC.

115.8 Size of Plant and Efficiency of Utilisation

During trial study, a total of 21.5 Tons of Black mass (14.9 Tons of BM-1 & 6.6 Kg of BM-2) was utilized. Accordingly, based on the available metals in the respective black mass samples the total metal recovery is 90% for cobalt; 93% for Nickel; 96% for Manganese; 83% for Lithium; 73% for Copper; 76% for Iron; 76% for Aluminum; and 97% for carbon shall be achieved. Therefore, based on the type of Black mass (generated from various types of Li ion batteries) requisite facilities of adequate size of storage shed and other plant & machineries shall be installed accordingly.

115.9 Online detectors/ Alarms/ Analyzers

In case of continuous process operations, online emission Analyzers for PM, HF, and TOC in the stack shall be installed and the online data be connected to the server of the concerned SPCB/ PCC.

115.10 Checklist of Minimal Requisite Facilities*:

Sl. No	Particulars
1.	Dedicated space for receiving black mas. Dedicated storage space under covered shed, for storage of Black mass bags or containers; raw chemicals, solvents. Pallets to store bags containing Black Mass. Containment of storage space with low raise wall/ bund.
2.	Acid and solvent storage tanks shall have proper ventilation, acid proof brick lining, proper slope and collection pit with caution sign under cool, dry, well-ventilated covered sheds shall have proper slope and seepage collection pit to collect seepage / floor washing.
3.	Storage and utilization area should have equipped with proper firefighting equipment including fire hydrant system.
4.	Sign boards indicating, storage, process, product and hazardous waste areas.
5.	Mechanised systems - for unloading, storage, transfer and other handling of Black mass in entire utilization process.
6.	Suction hood connected to a bag filter system (to control dust emission) while charging the Black Mass into the reactor.

7.	An alkaline scrubbing system connected with acid proof suction ducts to the reactors (leaching and extraction units) for control of acidic fumes. The scrubbing unit shall be connected to stack
8.	Bag dust collectors to the salt crushing processes connected to stack
9.	Fume extractions system followed by activated carbon filter in the solvent extraction unit area connected to a stack.
10.	Wastewater generated from the process, floor washing, spillage, reactor washing, scrubber bleed, condensate from MEE etc. may be reused in the process or treated physio chemically in an ETP to comply with wastewater discharge standards and may be sent to CETP for final disposal as prescribed by SPCB/PCC.
11.	Separate Leaching reactors for each stage of process
12.	Filtration units
13.	Extraction reactors (solvent / acid based)
14.	Stripping units
15.	Centrifuges
16.	Crystallization units.
17.	Evaporation/ drying units
18.	Crushing units
19.	MEE unit.
20.	Stack - to have sampling port, platform, access to the platform etc. as per the guidelines on methodologies for source emission monitoring published by CPCB under Laboratory Analysis Techniques LATS/80/2013-14. The air pollution control systems (fume scrubbers and bag dust collectors shall be connected to stack of height 30m or more as may be prescribed by SPCBs/PCCs.
21.	Online analyzers - for PM, HF, and TOC in the stack in case of continuous process operations.

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