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 Spent Aluminium Chloride generated during manufacturing of CPC Green and 2, 4, 6- Trimethyl Benzoyl Chloride
(Revised)

September, 2020

Central Pollution Control Board

(Ministry of Environment, Forest & Climate Change, Government of India)

Parivesh Bhawan, East Arjun Nagar,

Shahdara, Delhi – 110032

Procedure for grant of authorization by SPCBs/PCCs for utilization of Hazardous waste

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

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

   b. The utilization shall be similar to as described in SoP.

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

   d. Authorisation 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 ETP Sludge for utilization.

2) After issuance of authorization, SPCB shall verify the compliance of checklist and SoP on quarterly basis for initial 2 years; followed by random checks in the subsequent period for atleast once a year.

   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.

3) SPCBs shall provide half yearly updated list of units permitted under Rule 9 of Hazardous & Other Wastes (Management & Transboundary Movement) Rules, 2016 (HOWM Rules, 2016) to CPCB and also upload the same on SPCB website, periodically. Such updated list shall be sent to CPCB on a half yearly basis i.e., by July and January respectively.

4) Authorisation for utilisation shall not be given to the units located in the State/UT where there is no Common TSDF, unless the unit ensures authorised captive disposal of the hazardous waste (generated during utilisation) or its complete utilisation or arrangement of sharing with any other authorised disposal facility.

5) In case of the utilization proposal is not similar 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 utilization studies and developing SoPs.

6) The source and work zone standards suggested in the SoP are based on the E(P)A notified and OSHA standard respectively, however, SPCB/PCC may impose more stringent standards based on the location or process specific conditions.

43.0 Utilization of Spent Aluminium Chloride:

Type of HW	Source of generation	Recovery/Product
Spent Aluminium Chloride- Category Serial No.- 26.3 of Schedule I and C2 of Schedule II of HOWM Rules, 2016.	During the production of CPC Green and 2, 4, 6-Trimethyl Benzoyl Chloride	As a supplementary resource to manufacture liquid Aluminium Hydroxide Chloride/Poly Aluminium Chloride for further use in ETP and STP (as coagulant) and paper industry (as sizing material)

43.1 Source of Waste

The Spent Aluminium Chloride is generated during manufacturing process of CPC Green and 2, 4, 6-Trimethyl Benzoyl Chloride. The details on the manufacture process of the above two sources is given at Appendix A. The typical characteristics of the waste are given below.

Characteristics of Spent Aluminium Chloride Solution Generated from CPC Green manufacturing Industries

Sl. No.	Parameters	Unit	Result
1.	Moisture	%	78.09
2.	Aluminium Chloride as $AlCl_3$	%	18.8
3.	HCL	%	0.13
4.	V-salt (as Sodium Chloride)	%	1.38
5.	Calcium Chloride	%	<0.1
6.	Cuppers Chloride as $CuCL_2$	%	1.40
7.	TOC	mg/kg	16.75
Heavy Metals			
8.	Mercury as Hg	mg/kg	<0.1
9.	Arsenic as As	mg/kg	<0.1
10.	Cadmium as Cd	mg/kg	0.48
11.	Lead as Pb	mg/kg	0.62
12.	Iron as Fe	mg/kg	7.2
13.	Zinc as Zn	mg/kg	0.84
14.	Copper as Cu	mg/kg	923
15.	Chromium as Cr	mg/kg	0.56
16.	Manganese as Mn	mg/kg	2.14
Organic Compound			
17.	CPC-Blue	mg/kg	6.6
18.	CPC-Green	mg/kg	9.8
19.	Ferric Chloride	mg/kg	0.08

Characteristics of Spent Aluminium Chloride Solution Generated from 2, 4, 6-Trimethyl Benzoyl Chloride Manufacturing Industries

Sl. No.	Parameters	Unit	Result
1.	Moisture	%	72.30
2.	Aluminium Chloride as $AlCl_3$	%	25.20
3.	HCL	%	1.294
4.	V-salt (as Sodium Chloride)	%	1.1
5.	Calcium Chloride	%	<0.1
6.	TOC	mg/kg	66.50
Heavy Metals			
7.	Mercury as Hg	mg/kg	<0.1
8.	Arsenic as As	mg/kg	<0.1
9.	Cadmium as Cd	mg/kg	0.13
10.	Lead as Pb	mg/kg	0.44
11.	Iron as Fe	mg/kg	5.16
12.	Zinc as Zn	mg/kg	0.78

13.	Copper as Cu	mg/kg	1.92
14.	Chromium as Cr	mg/kg	0.46
15.	Manganese as Mn	mg/kg	0.94
Organic Compound			
16.	Acetyl Chloride	mg/kg	<0.1
17.	Mesitylene	mg/kg	8.0
18.	2, 4, 6-Trimethyl Benzoyl Chloride	mg/kg	10.90

43.2 Utilization Process

The utilization process shall involve following steps:

a) In case of utilization of Spent Aluminium Chloride generated from 2, 4, 6-Trimethyl Benzoyl Chloride, spent solution is transferred into the pre-treatment unit where the Spent Aluminium Chloride is treated with activated carbon to remove colour and organic impurities. The treated Spent Aluminium Chloride is then charged into the reaction vessel and the other raw materials i.e. alumina/aluminium hydroxide, sodium hydroxide /sodium bi-carbonate/ sodium carbonate and calcium hydroxide (all in solid form) are added into reaction. The reaction process is completed in 04-05 hours. After completion of process, the reaction mass is filtered through filter press. The product i.e. Aluminium hydroxide is obtained as liquid. The solid cake obtained from filter press is sent to the TSDF for final disposal.

The fumes from the reaction vessel are treated through venture scrubber and the scrubbed bleed generated from venture scrubber is further utilized in process for maintaining pH.

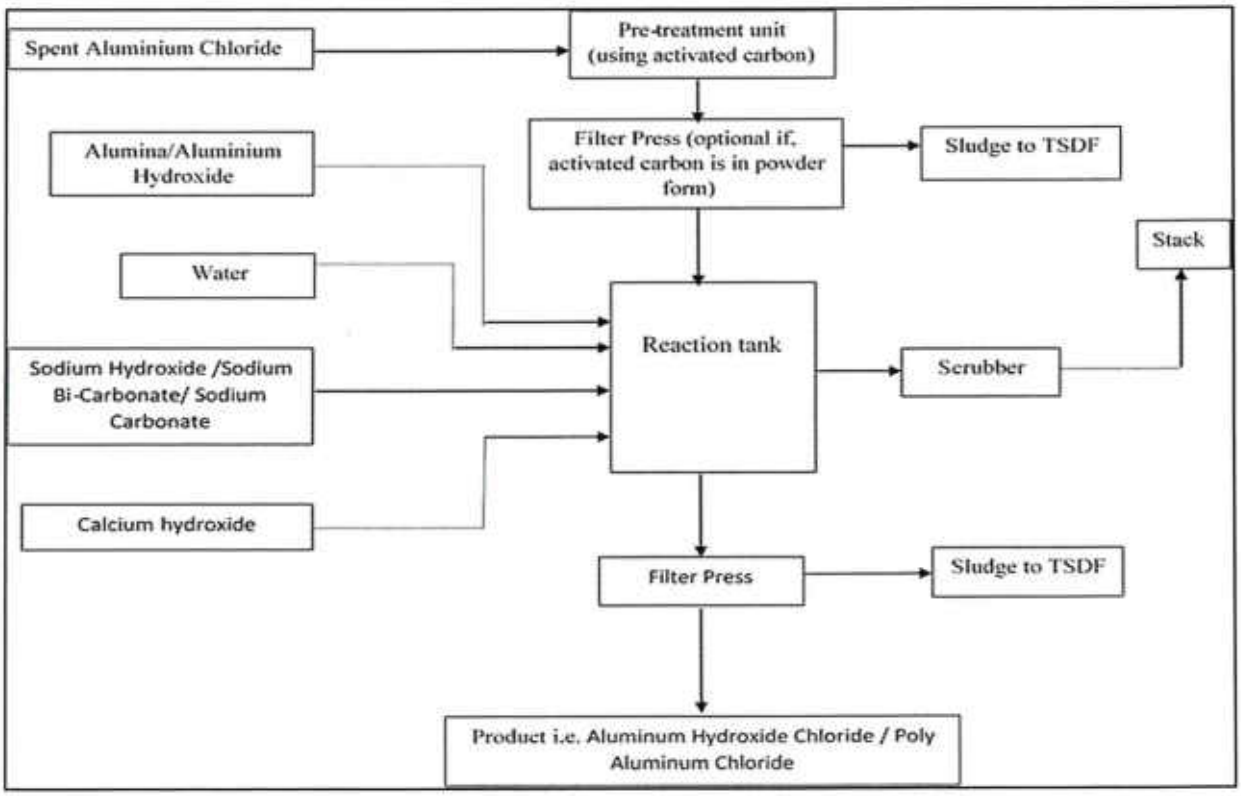

Figure: 1-*Process flow diagram for utilization of Spent Aluminium Chloride generated from 2, 4, 6-Trimethyl Benzoyl Chloride.*

b) In case of utilization of Spent Aluminium Chloride generated from CPC green, the spent solution is firstly charged into the reaction vessel and the other raw materials i.e. iron scrap and sodium hydroxide /sodium bi-carbonate/ sodium carbonate are added into reaction vessel. The solution is kept in agitation to complete the reaction and the reacted mass is filtered through filter press. The filtered precipitate is dried and taken out. The filtrate solution from the filter press is further treated with activated carbon and transferred to the reaction vessel and the other raw material i.e. alumina/aluminium hydroxide, sodium hydroxide /sodium bi-carbonate /sodium carbonate and calcium hydroxide (all in solid form) are added into reaction vessel in stage wise manner. The solution is kept in agitation to completion of process, the reaction mass is filtered through filter press. The product i.e. Aluminium hydroxide is obtained as liquid. The solid cake obtained from filter press is sent to the TSDF for final disposal.

The fumes from the reaction vessel (s) are treated through venture scrubber and the scrubbed bleed generated from venture scrubber is further utilized in process for maintain pH.

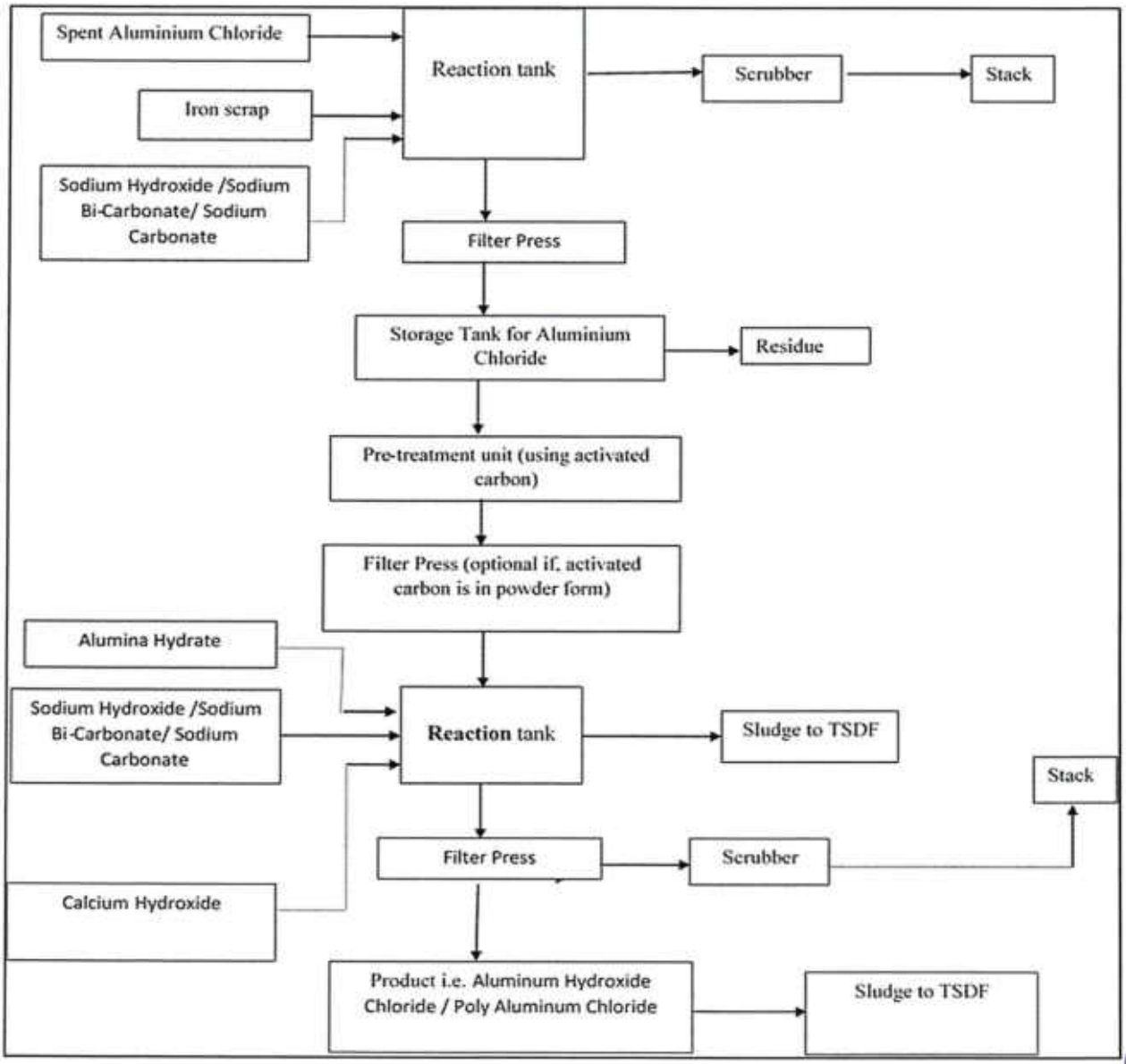

Figure: 2-*Process flow diagram for utilization of Spent Aluminium Chloride generated from CPC green.*

43.3 Product Usage / Utilization

The liquid Aluminium Hydroxide Chloride/Poly Aluminium Chloride will be utilized only in the Effluent Treatment Plant as coagulant and Pulp and paper industry as sizing material and in no case shall be used in Water Treatment Plant.

*The Aluminium Hydroxide Chloride (AHC) manufactured by utilizing spent Aluminium Chloride (generated from manufacturing of CPC Green and 2,4,6 Trimethyl Benzoyl chloride) shall be used as a coagulant in Sewage Treatment Plants (STPs) as per Para 43.4. In no case, Aluminium Hydroxide Chloride (AHC) shall be utilized for drinking water purification or water treatment plants. In no case, this treated sewage water shall be used in agricultural fields.

The product i.e. Aluminium Hydroxide Chloride /Poly Aluminium Chloride shall comply with the Bureau of Indian Standards: IS 15573:2018 for TOC i.e. 80 ppm (in liquid form)

The unit shall label its product (Aluminium Hydroxide Chloride/Poly Aluminium Chloride) manufactured by utilizing aforesaid hazardous waste) as "This Aluminium hydroxide/Poly Aluminium Chloride has been manufactured by utilizing Spent Aluminium Chloride and not to be used in Water Treatment Plant".

43.4 Utilization Process and Standard Operating Procedure for utilization of Aluminium Hydroxide Chloride as a coagulant in STPs:

Characteristics of Aluminium Hydroxide Chloride (AHC) generated from CPC green and 2, 4, 6 Trimethyl Benzoyl Chloride:

Sr No	Parameters	Unit	Results of AHC from CPC Green	Results of AHC generated 2,4, 6 Trimethyl Benzoyl Chloride
1	Moisture	%	73.71	67.80
2	Aluminium Hydroxide Chloride	%	15	20.88
3	HCl	%	< 0.01	< 0.01
4	V-Salt (as sodium chloride)	%	6.30	6.2
5	Calcium Chloride	%	4.90	5.1
6	Cuppers Chloride as $CuCl_2$	%	0.0087	-
7	TOC	mg/Kg	8.75	58
8	Mercury as Hg	mg/Kg	<0.1	< 0.1
9	Arsenic as As	mg/Kg	< 0.1	< 0.1
10	Cadmium as Cd	mg/Kg	0.16	0.05
11	Lead as Pb	mg/Kg	0.28	0.16
12	Iron as Fe	mg/Kg	5.8	0.54
13	Zinc as Zn	mg/Kg	0.36	0.20
14	Copper as Cu	mg/Kg	15	0.32
15	Chromium as Cr	mg/Kg	0.12	0.11
16	Manganese as Mn	mg/Kg	0.56	0.28
17	Organic Compounds	mg/Kg	CPC Blue: <5	Acetyl Chloride: < 0.1
			CPC Green: <5	Mestilylene: 2.0
			Ferric Chloride: 0.020	2,4, 6 Trimethyl Benzoyl Chloride: 2.8

Utilization Process of AHC as a coagulant in STP: The Aluminium Hydroxide Chloride (AHC) manufactured by utilizing spent Aluminium Chloride (generated from manufacturing of CPC Green and 2,4,6 Trimethyl Benzoyl chloride) as a coagulant in Sewage Treatment Plants (STPs). In coagulation and flocculation process, by using coagulant dissolved solids is transformed into insoluble solids. The dosing of coagulant is done in Flash Mixer/Equalisation tank. The effluent goes to screen chamber, grit chamber, Flash Mixer, primary clarifier/settling tank, secondary treatment (aeration tank/ anaerobic treatment), secondary clarifier/settling tank and treated effluent storage tank.

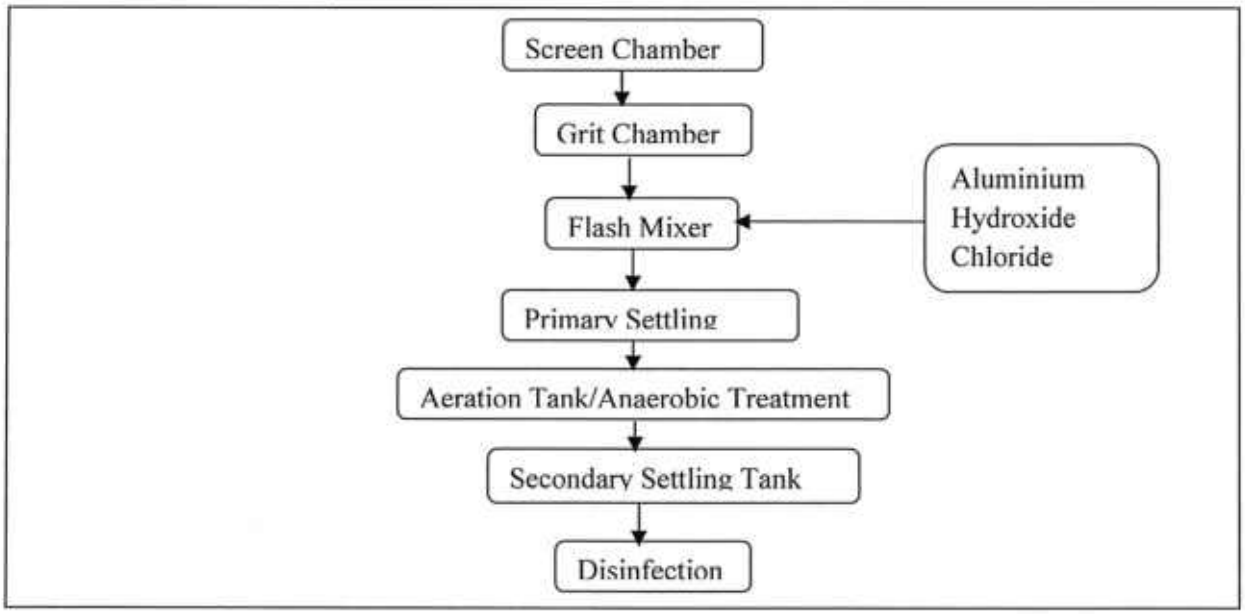

Figure: 3-*Process flow diagram for utilization of Aluminium Hydroxide Chloride as a coagulant in STP.*

Standard Operating Procedure for utilization of AHC as a coagulant in STP:

1) The AHC shall be transported in SPCB/PCC authorized tankers mounted on vehicles fitted with requisite safeguards ensuring no spillage of the same.
2) There shall be a designed space for unloading of AHC into the storage tank. The receiving storage tank shall be placed above the ground and contained with low raise parapet/bund wall with slope to collect spillages, if any, into collection pit. Alternatively, storage tanks for AHC may be kept below the ground provided it has HDPE liner system beneath the tank and leachate collection system below HDPE liner. In the event of leachate detection in the leachate collection system, corrective measures shall be taken immediately.
3) The unit shall install storage tank under cool, dry, well ventilated covered storage shed(s) within premises, as authorized by the concerned SPCB/ PCC under Hazardous and Other Wastes (Management & Transboundary Movement) Rules, 2016, so as to eliminate rain water intrusion.

   Further, the storage area shall have leak-proof floor tiles with adequate slope to collect spillage, if any, into a collection pit. The spillage from collection pit shall be transferred to reaction tanker or ETP, as the cases may be, through chemical process pump.
4) There shall be no manual handling of the AHC. Acid Proof pump shall be used for transfer of AHC through pipelines to equalization tank/flash mixer.

5) The vent of AHC storage tanks shall be connected to scrubber for treatment using alkaline medium.
6) The unit shall provide separate storage tanks for storage of chemicals and the storage tanks should be at designated place with proper cover and with acid brick lining floors.
7) The unit shall ensure that the said utilization process and its associated activities shall be demarcated separately within the unit.

43.5 Standard Operating Procedure for utilization

The SoP is applicable only for the utilization of Spent Aluminium Chloride generated during manufacturing of CPC Green and 2, 4, 6-Trimethyl Benzoyl Chloride to produce Aluminium Hydroxide Chloride/Poly Aluminium Chloride (in liquid form only).

1) The Spent Aluminium Chloride shall be transported in acid proof tankers mounted on vehicles fitted with requisite safeguards ensuring no spillage, as authorized by SPCB/PCC.

2) There should be a designated space for unloading of Spent Aluminium Chloride into an acid proof storage tank. The receiving storage tank shall be placed above the ground and contained with low raise parapet/bund wall & concrete/acid proof floor with slope to collect spillages, if any into collection pit.

3) The storage tank shall be kept under cool, dry, well-ventilated covered storage shed(s) within the premises, as authorized by the concerned State Pollution Control Board/Pollution Control Committee under the Hazardous and Other Wastes (Management and Transboundary Movement) Rule, 2016, so as to eliminate rain water intrusion.

4) There shall be no manual handling of the Spent Aluminium Chloride and Poly Aluminium Chloride. Acid proof pump shall be used for transfer of Spent Aluminium Chloride through pipelines.

5) The Spent Aluminium Chloride shall be transfer through mechanized system into the pre-treatment unit to remove colour and organic impurities by treating the Spent Aluminium Chloride with Activated carbon.

   In case of the Spent Aluminium Chloride generated from CPC Green manufacturing process, the pre-treatment with Activated Carbon shall be carried after removal of Copper from the Spent Aluminium Chloride by treating the same with iron scrap and sodium hydroxide in the mechanized agitated reaction vessel. The same should added through closed conveyor system. The reacted mass shall be transferred to the filter press and precipitated solid mass from the filter press is dried and packed and kept in Storage area.

6) The activated carbon treated Aluminium Chloride solution shall be transferred to the closed reaction vessel where alumina/aluminium hydroxide, sodium hydroxide /sodium bi-carbonate / sodium carbonate and calcium hydroxide are also added step by step in powder form into the reactor through closed conveyor system. The reactor shall be of acid proof material.

7) The solution in the reactor shall be agitated with the help of mechanized agitated for 4-4.5 hours and then transferred to filter press through pump for filtration. The solid mass from the filter press shall be removed mechanically and packed and kept in storage area.

8) The filtrate i.e. Liquid Aluminium Chloride is received as the final product from the filter press

9) Acid fume/vapour is expected to be liberated from the reaction tanks where iron alumina/aluminium hydroxide, sodium hydroxide /sodium bi-carbonate / sodium carbonate and calcium hydroxide is added to Spent Aluminium Chloride. Thus, the said reaction tanks shall be connected with hood over it to suck acid fume/vapour. The hood shall be maintained under suction followed by treatment in scrubber using alkaline medium and attached to stack of minimum height of 06 m above the roof top or as prescribed by the concerned SPCB/PCC, whichever is higher.

10) The entire process area shall have leak-proof and acid proof floor tiles with adequate slope to collect spillages, if any, into a collection pit. The spillages from collection pit shall be transferred to reaction tank, as the cases may be, through acid proof pump.

11) The product i.e. Aluminium Hydroxide Chloride/ Poly Aluminium Chloride shall comply with the Bureau of Indian Standards: IS 15573:2018 for TOC i.e. 80 ppm (liquid form).

12) The product (i.e. Aluminium Hydroxide Chloride/ Poly Aluminium Chloride) manufactured by utilizing aforesaid hazardous waste shall only be used in ETP as coagulant and Pulp & Paper industry as sizing agent.

13) The unit shall label its product (i.e. Aluminium Hydroxide Chloride/ Poly Aluminium Chloride) manufactured by utilizing aforesaid hazardous waste) as "This Aluminium Hydroxide Chloride/ Poly Aluminium Chloride has been manufactured by utilizing spent Aluminium Chloride and not to be used in Water Treatment Plant".

14) 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) such as Chemical goggles, full-face shield, or a full face respirator, Impervious gloves of chemically resistant material (rubber or neoprene), Body suits, aprons, and/or coveralls of chemical resistant material and impervious boots of chemically resistant material.

15) Treatment and disposal of wastewater:

    The pollution potentials are emissions from reactors and residues from filter press and there is no generation of wastewater and thereof on discharge of wastewater.

16) It shall be ensured that Spent Aluminium Chloride is produced from the industries that have valid authorization for the same from the concerned SPCB/PCC as required under Hazardous and Other Wastes (Management and Transboundary Movement) Rules, 2016.

17) Transportation of Spent Aluminium Chloride and residues generated during utilization shall be carried out by the sender or receiver (utilizer/TSDF operator) as per the authorization issued by concerned SPCB/PCC under the Hazardous and Other Wastes (Management and Transboundary Movement) Rules, 2016 and in compliance with other provision of said rules.

18) The residue generated from filter press during Copper removal shall be sent to SPCB/PCC authorized recycler/utilizer or disposed in SPCB/PCC authorized common or captive disposal facility.

19) The other residue generated from (filter press), product spillages etc. shall be collected and temporarily stored at designated place and proper cover and concrete/acid proof brick lining floor and be sent to authorized common TSDF or other authorized facility within 90 days from generation of the waste in accordance with the authorization issued by the concerned SPCB/PCC. Such storage area shall be covered with proper ventilation.

20) In case of environmental damages arising due to improper handling of hazardous wastes including accidental spillage during generation, storage, processing, transportation and disposal, the unit shall be liable to implement response measures, environmental site assessment and remediation of contaminated soil/ground water/sediment etc. as per the "Guidelines on Implementing Liabilities for Environment Damages due to Handling & Disposal of Hazardous Wastes and Penalty" published by CPCB.

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

43.6 Record/Returns Filing

1) The unit shall maintain a passbook issued by concerned SPCB wherein the following details of each procurement of Spent Aluminium Chloride shall be entered:
   - Address of the sender
   - Date of dispatch
   - Quantity procured
   - Seal and signature of the sender
   - Date of receipt in the premises
2) A log book with information on source and date of procurement of each type of the said hazardous wastes, quantity, and date wise utilization of the same, quantity of Aluminium Hydroxide Chloride / Poly Aluminium Chloride manufactured, utilization, 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 record of hazardous waste utilized, hazardous waste generated and disposed as per Form 3 & shall file annual returns in Form 4 as per Rule 20 (1) and (2) of the Hazardous and Other Wastes (Management and Transboundary Movement) Rule 2016, to concerned SPCB.

43.7 Standards

1) Fugitive emissions in the work zone shall comply with the following:

PM_{10}	-	5 mg/m^3 TWA*
HCL	-	7 mg/m^3 Ceiling#

TWA - Time-weighted average The Permissible Exposure Limit is 8-hour TWA*

 # A Ceiling limit is one that may not be exceeded for any perioid of time, and is applied to irritants and other material that have immediate effects

2) Source Emission in Stack attached to the reaction tanks shall comply with the following:

PM_{10}	-	50 mg/Nm^3
HCL Vapour & Mist	-	35 mg/Nm^3
TOC	-	20 mg/Nm^3

3) Monitoring of specified parameters for source and fugitive emission shall be carried out by NABL accredited or ISO17025/EPA recognized laboratories and the results shall be submitted quarterly to the concerned SPCB/PCC.

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

43.8 Siting of Industry

(1) Facilities for processing of Spent Aluminium Chloride shall preferably be located in a notified industries area or industrial Park/estate/cluster.

(2) Facilities for utilization of AHC shall be located in a STP and in accordance with Consent to Establish issued by the concerned SPCB/PCC.

43.9 Efficiency of utilisation

About 23030 Kgs of Spent Aluminium Chloride (generated form 2, 4, 6-Trimethyl Benzoyl Chloride) alongwith other material would be required to produce 25140 Kgs of Aluminium Hydroxide Chloride and 18260 Kgs of Spent Aluminium Chloride (generated from CPC Green) alongwith other material would require to produce 18800 Kgs of Aluminium Chloride. Therefore, requisite facilities of adequate size shall be installed accordingly as mentioned under para 43.11 below.

43.10 On-line detectors / Alarms / Analysers

In case of continuous process operation, online analysers shall be installed for PM and TOC in the stack emission. The on-line data shall be connected to the concerned SPCB/PCC server.

43.11 Checklist of Minimal Requisite Facilities

Sl. No.	Requisite Facilities
1.	Storage shed(s) for storage of Spent Aluminium Chloride in acid proof container only under cool, dry, well-ventilated covered storage shed(s) within premises.
2.	Separate Storage area for storage of liquid Aluminium Hydroxide Chloride/ Poly Aluminium Chloride
3.	Storage Tank should be of such size/capacity that it can store two weeks requirements
4.	Collection pit for collection of the spillage from storage area, process area and uploading area.

5.	Closed Mechanized system for – - Mixing of Spent Aluminium Chloride with alumina/aluminium hydroxide, sodium hydroxide /sodium bi-carbonate / sodium carbonate and calcium hydroxide. - Transfer of alumina/aluminium hydroxide, sodium hydroxide /sodium bi-carbonate / sodium carbonate and calcium hydroxide into process reactor
6.	Chemical process pumps (acid proof) for transfer of Spent Aluminium Chloride and Poly Aluminium Chloride from tanker to storage tank and subsequently to process area
7.	Activated Carbon Pre-treatment unit Filter press (optional in case activated carbon used in powder form)
8.	Closed Process reactor (reaction vessel (s)) attached to scrubbing system followed by stack of minimum height of 06 m above the roof top or as prescribed by concerned SPCB/PCC, whichever is higher
9.	Filter Press
10.	Dedicated separate covered hazardous waste (filter press residue, activated carbon, etc.) storage area to store hazardous generated during utilization process.
11.	Stacks 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.
12.	Product shall be stored in cool, dry, well-ventilated covered storage shed within premises.
13.	Online analyzers shall be installed for PM and TOC in the stack emission and be connected to the concerned SPCB/PCC server.

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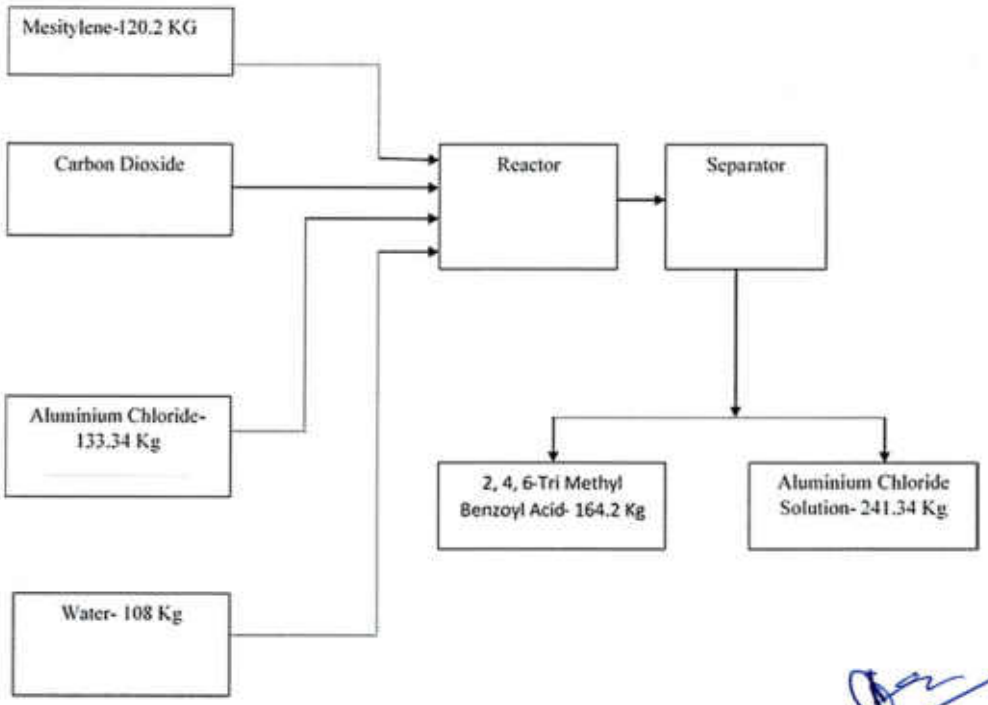
APPENDIX A

Manufacturing process of 2, 4, 6-Tri Methyl Benzoyl Acid



Manufacturing Process – CPC GREEN

(Flowsheet (with Material Balance):