

**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**



cpcb

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Central Pollution Control Board
(Ministry of Environment, Forest & Climate Change, Government of India)
Parivesh Bhawan, East Arjun Nagar,
Shahdara, Delhi – 110032

Shafiq

Procedure for grant of authorisation by SPCBs/PCCs for utilization of Hazardous Waste

- (i) While granting authorisation for utilization of hazardous wastes, SPCBs/PCCs shall ensure the following:
- a. The waste (intended for utilization) belongs to similar source of generation as specified in SoPs.
 - b. The utilization process is similar to the process of utilization described in SoPs.
 - c. End-use / product produced from the waste shall be same as specified in SoPs.
 - d. Authorisation be granted only after verification of utilization process and minimum requisite facilities as given in SoPs.
 - e. Issuance of passbooks (similar to the passbooks issued for recycling of used oil, waste oil, non-ferrous scrap, etc.) for maintaining records of receipt of hazardous wastes for utilization.
- (ii) After issuance of authorization, SPCB shall verify the utilization process, checklist and SOPs on quarterly basis for initial 02 years; followed by random checks in the subsequent period for at least once a year.
- In-case of lack of requisite infrastructures with the SPCB/PCC, 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.
- (iii) SPCB shall provide half yearly updated list of units permitted under Rule 9 of HOWM Rule, 2016 to CPCB and also upload the same on SPCB website, periodically. Such updated list shall be sent to CPCB half yearly by July and January respectively.
- (iv) 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.
- (v) In case 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 thereof.
- (vi) The source and work zone standards suggested in the SoPs 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.

D. Kapil

43.0 Utilization of Spent Aluminum 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 Hazardous and Other Waste (Management and Transboundary Movement) Rules, 2016	During the production of CPC Green and 2, 4, 6-Trimethyl Benzoyl Chloride	As a supplementary resource to manufacture liquid Aluminum Hydroxide Chloride/ Poly Aluminum Chloride for further use in ETP (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 manufacturing 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

S. 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

Dr. P. K. Singh

Standard Operating Procedure and Checklist of Minimal Requisite Facilities - Utilization of Spent Aluminium Chloride generated during manufacturing of CPC Green and 2, 4, 6-Trimethyl Benzoyl Chloride

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

S. 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 utilisation process shall involve following steps:

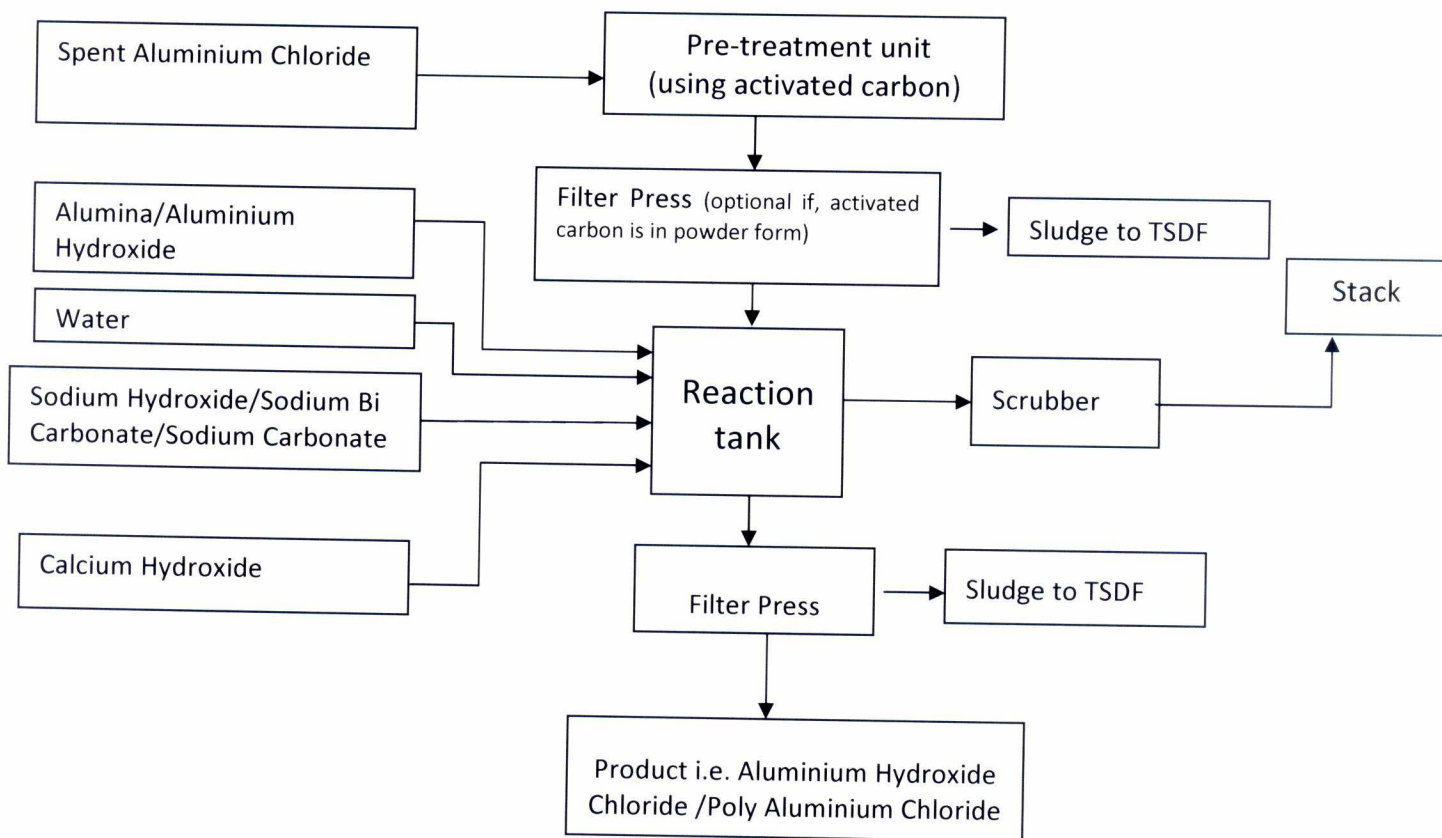
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- (a) In case of utilisation of Spent Aluminium Chloride generated from 2, 4, 6-Trimethyl Benzoyl Chloride, the spent solution is transferred into the pre-treatment unit where the Spent Aluminium Chloride is treated with activated carbon to remove 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 vessel in stage wise manner. The solution is kept in agitation to complete the 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 send to the TSDF for final disposal.

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

Process Flow Diagram of manufacturing of Aluminum Hydroxide Chloride / Poly Aluminum Chloride



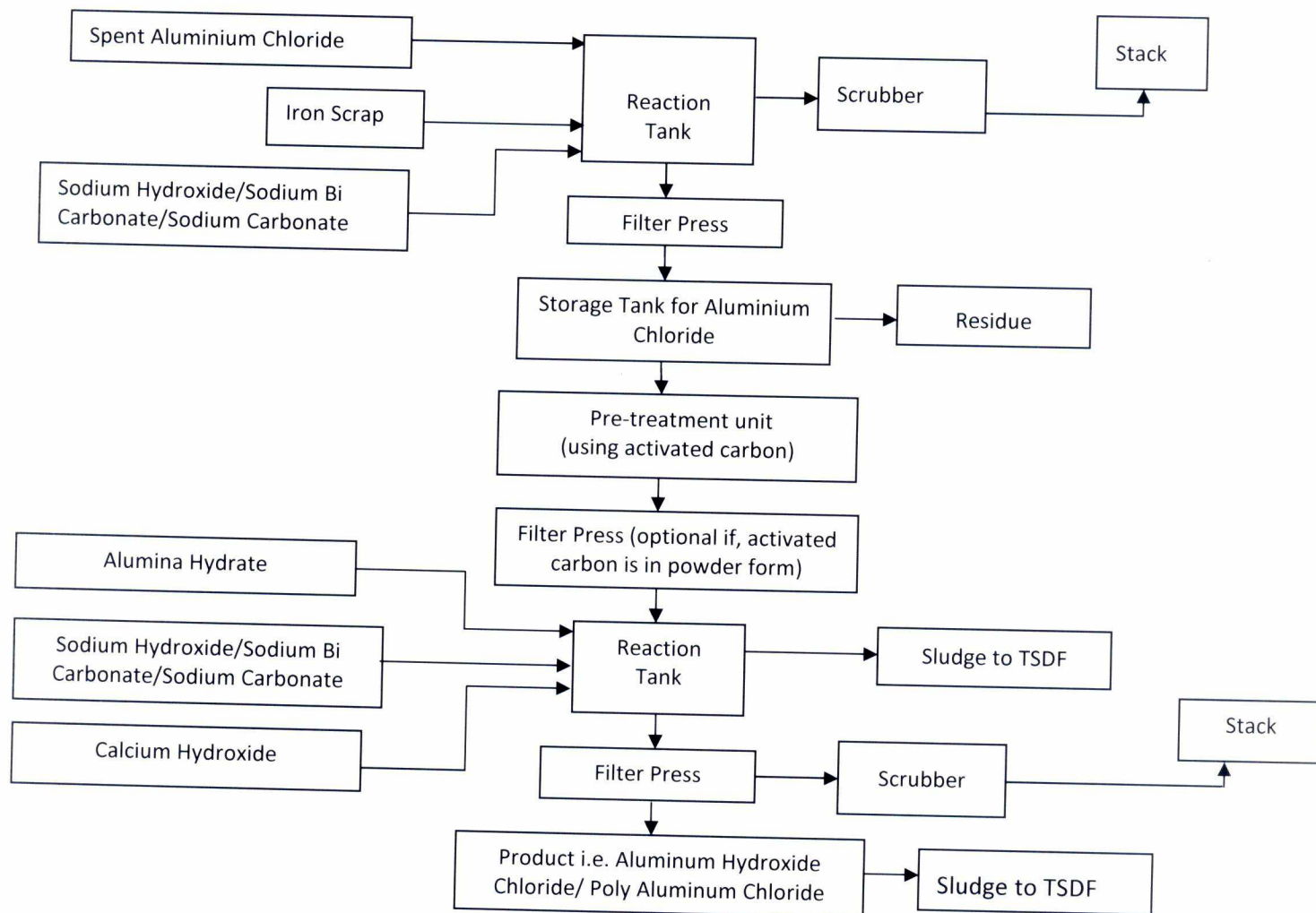
- (b) In case of utilisation of Spent Aluminium Chloride generated from CPC green, the spent solution is firstly charged into the reaction vessel and the other raw materials i.e iron scrap and sodium hydroxide /sodium bi-carbonate / sodium carbonate are added into reaction vessel. The solution is kept 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

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Standard Operating Procedure and Checklist of Minimal Requisite Facilities - Utilization of Spent Aluminium Chloride generated during manufacturing of CPC Green and 2, 4, 6-Trimethyl Benzoyl Chloride

the filter press is further treated with activated carbon and transferred to 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 vessel in stage wise manner. The solution is kept in agitation to complete the 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 send to the TSDF for final disposal.

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



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43.2 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 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 (i.e. Aluminium Hydroxide Chloride /Poly Aluminum Chloride) manufactured by utilizing aforesaid hazardous waste) as “This Aluminum hydroxide/Poly Aluminum Chloride has been manufactured by utilizing Spent Aluminium Chloride and not to be used in Water Treatment Plant”.

43.4 Standard Operating Procedure (SoP) for utilization

This 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 Aluminum 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 Aluminum Chloride into a 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 authorised by the concerned State Pollution Control Board/Pollution Control Committee under the Hazardous and Other Wastes (Management & Transboundary Movement) Rules, 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 Aluminum Chloride shall be transfer through mechanized system into the pre-treatment unit to remove color and organic impurities by treating the Spent Aluminum Chloride with Activated Carbon.

In case of the Spent Aluminum Chloride generated from CPC green manufacturing process, the pre-treatment with Activated Carbon shall be carried after removal of Copper from the Spent Aluminum Chloride by treating the same with iron scrap and sodium

hydroxide in the mechanized agitated reaction vessel. The same should be 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 agitator 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 hydroxide is received as the final product from the filter press.
- (9) Acid fume/vapor 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 solution. 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/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 procured 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 Aluminum Chloride and residues generated during utilisation 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 & Transboundary Movement) Rules, 2016 and in compliance with other provisions of said rules.
- (18) The residue generated from filter press during Copper removal shall be sent to SPCB/PCC authorised recycler/utiliser 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 with 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 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.
- (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.

43.5 Records/Return 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, date wise utilization of the same, quantity of Aluminium Hydroxide Chloride/Poly Aluminium Chloride manufactured, 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 utilised, 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) Rules, 2016, to concerned SPCB.
- (4) 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.

43.6 Standards

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

PM10	-	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 period of time, and is applied to irritants and other materials that have immediate effects
(Reference: Occupational Safety and Health Standards 1910:1000);

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

PM	-	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.

43.7 Sitting of Industry

Facilities for processing of Spent Aluminium Chloride shall preferably be located in a notified industrial area or industrial park/estate/cluster.



43.8 Size of Plant & Efficiency of utilisation

About 23030 Kgs of Spent Aluminium Chloride (generated from 2, 4, 6-Trimethyl Benzoyl Chloride) alongwith other material would be required to produce 25140 Kgs of Aluminum Hydroxide Chloride and 18260 Kgs of Spent Aluminium Chloride (generated from CPC Green) alongwith other material would required to produce 18800 Kgs of Aluminum Hydroxide Chloride. Therefore, requisite facilities of adequate size shall be installed accordingly as mentioned under para 43.10 below.

43.9 On-line detectors / Alarms / Analyzers

In case of continuous process operations, 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.

43.10 Checklist of Minimal Requisite Facilities

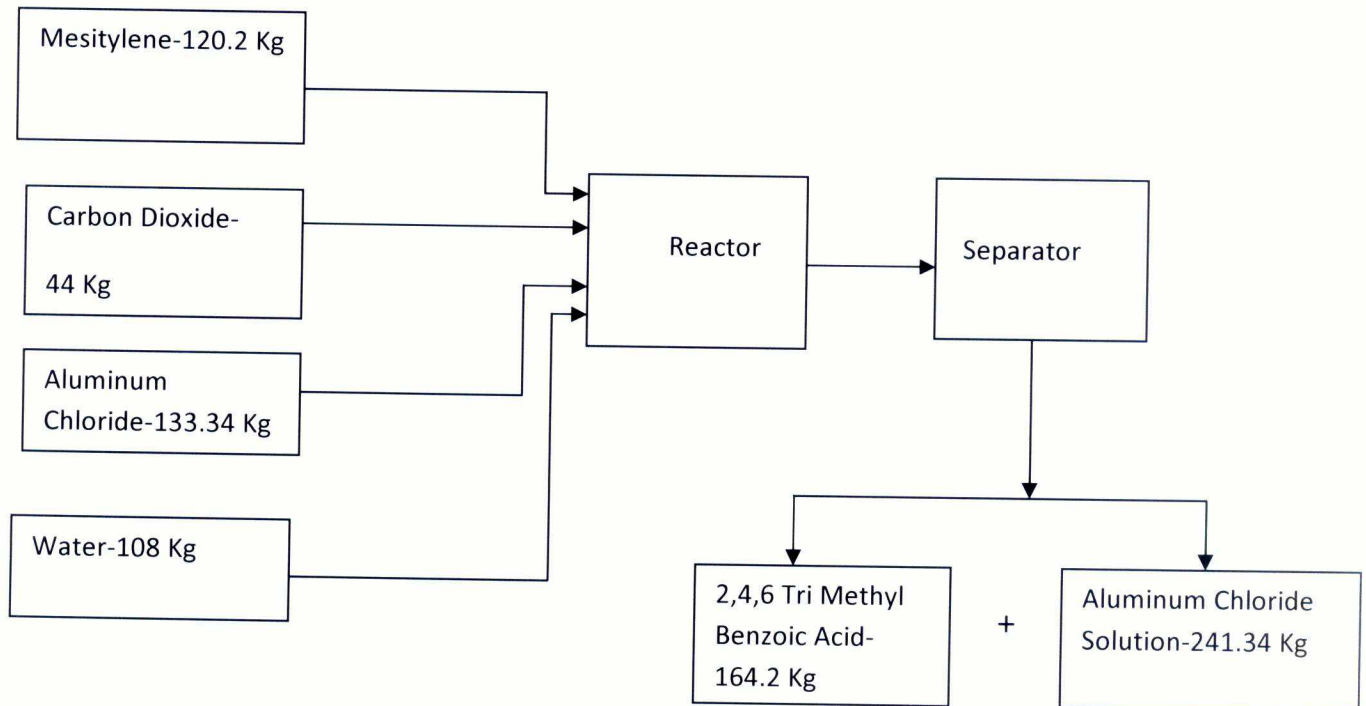
S. No.	Requisite Facilities
1.	Storage shed(s) for storage of Spent Aluminium Chloride in acid proof container only under cool, dry, well-ventilated covered storage shed(s) within premises.
2.	Separate Storage area for storage of liquid Aluminium Hydroxide Chloride /Poly Aluminium Chloride
3.	Storage Tanks 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 unloading 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

Standard Operating Procedure and Checklist of Minimal Requisite Facilities - Utilization of Spent Aluminium Chloride generated during manufacturing of CPC Green and 2, 4, 6-Trimethyl Benzoyl Chloride

	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 analysers shall be installed for PM and TOC in the stack emission and be connected to the concerned SPCB/PCC server.

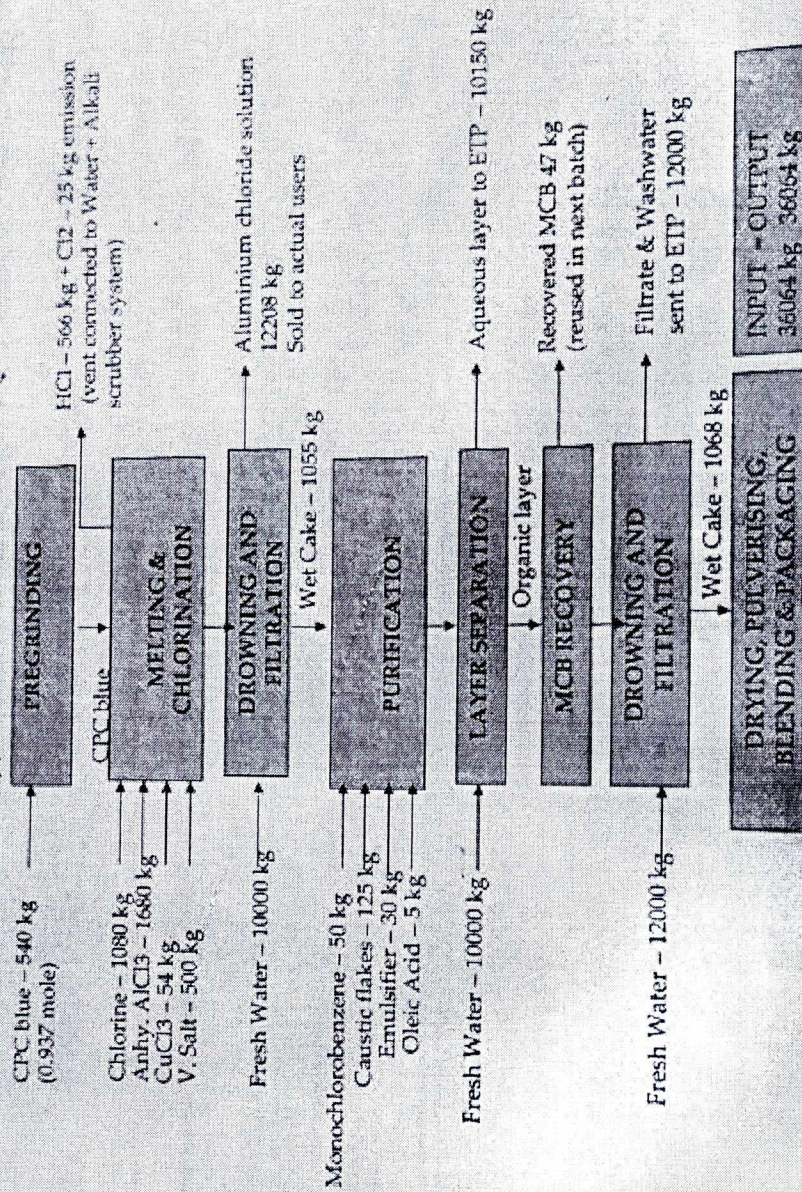
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Manufacturing process of 2,4,6 Tri Methyl Benzoic Acid



Manufacturing Process - CPC GREEN

Flowsheet (with Material Balance):



CPC GREEN - 1000 kg (0.886 mole)

AVDHOT PIGMENTS PVT. LTD., Ank