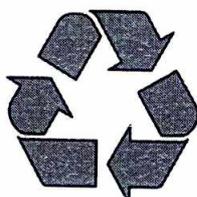


**Standard Operating Procedure and Checklist of Minimal Requisite Facilities
for utilization of hazardous waste under Rule 9 of the Hazardous and Other
Wastes (Management and Transboundary movement) Rules, 2016**

**Utilization of Spent Ammonium Chloride generated during production of
Hexamethyl Di Silazane for manufacturing of Ammonium Chloride**



June, 2018

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 authorisation 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) should have similar source of generation as specified in SoPs.
 - b. The utilization process should be 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 shall be granted only after verification of minimum requisite facilities installed and after verification of utilization process as given in SoPs.
 - e. Issuance of passbooks (similar to the passbooks issued for recycling of used oils, waste oil, non-ferrous scraps, etc.) for maintaining records of receipt of hazardous wastes for utilization.
- 2) After issuance of authorization, SPCB shall verify the utilization process, checklist and SOPs on quarterly basis for during the initial 02 years; followed by random checks in subsequent period for at least once in every 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) SPCB/PCCs 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 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 SoPs are based on the E(P)A notified and OSHA standards respectively, however, SPCB/PCC may impose more stringent standards based on the location or process specific conditions

45.0 Utilization of Spent Ammonium Chloride:

Type of HW	Source of generation	Recovery/Product
Spent Ammonium Chloride (Category no. A 10 of Schedule II of HOWM Rules, 2016)	During production of Hexamethyl Di Silazane (Pharma intermediate)	production of Ammonium Chloride

45.1 Source of Waste

The Spent Ammonium Chloride solution (25 %) is generated during the production of Hexamethyl Di Silazane in the Bayer's process which involves reaction between HCl acid,

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Liquified Ammonia and Hexamethyl Di Siloxane. Typical Characteristics of the waste is given below:

Specific gravity	1.071 gm/cm ³
NH ₄ Cl	25.15 %
Organic compound as Hexamethyl di Siloxane	Not Detected
Organic compound as Hexamethyl di Silazane	Not Detected

The aforesaid Spent Ammonium Chloride solution is categorised as Hazardous waste at S. No. A 10 of Schedule II of HOWM Rules, 2016 which are required to be disposed in authorized disposal facility in accordance with authorization condition, when not utilized as resource recovery.

45.2 Utilization Process

The Spent Ammonium Chloride solution (25%) is filled in a storage tank and allowed to settle for a day. The solution is pumped into neutralization tank where it is neutralized by adding Hydrochloric acid to get Ammonium Chloride. The solution is heated at a temperature of 110⁰ C, to evaporate water content and concentrate the solution. The concentrate is taken into crystalliser where hot solution is cooled from 110⁰ C to 45-50⁰ C for about 2 hrs. Solution from the crystalliser is taken to centrifuge where mother liquor is separated and collected. The collected mother liquor is re-circulated back to evaporation pan along with fresh Spent Ammonium Chloride solution. Wet cake from centrifuge containing 4-5 % moisture is dried in continuous rotatory drier by passing hot air at 250⁰ C.. The dried Ammonium Chloride is crushed, screened and packed as product. The flow diagram of utilization process of spent ammonium chloride is shown in Figure 1.

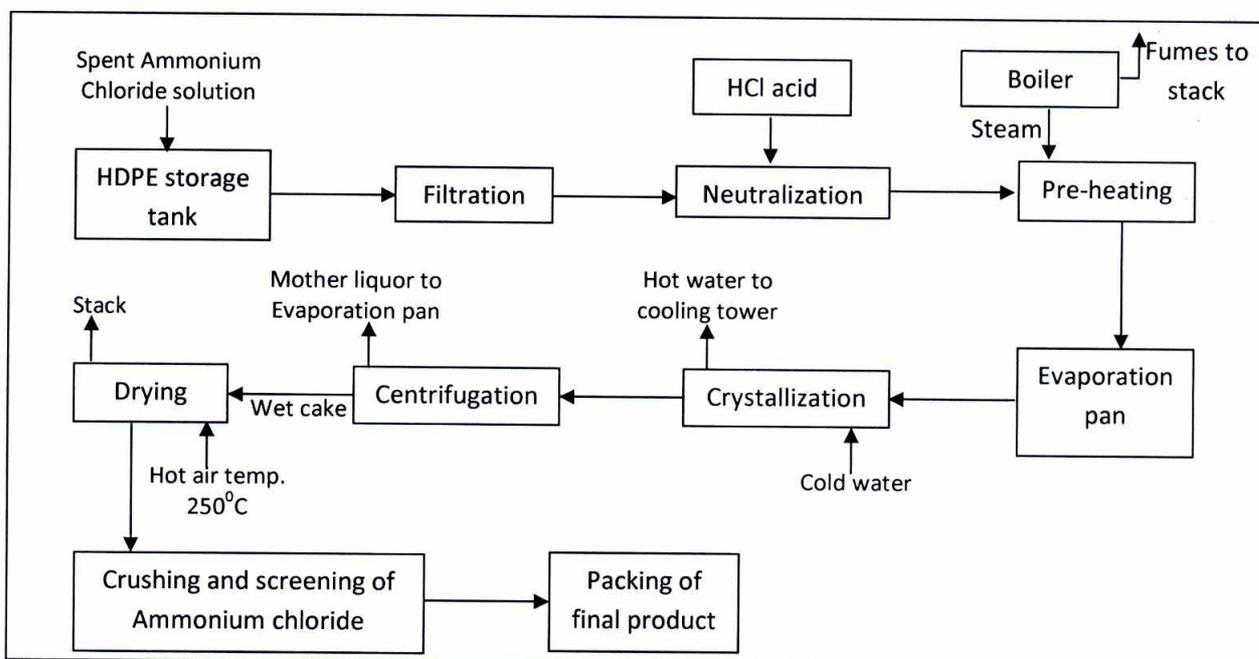


Figure 1: Flow diagram of Utilization of Spent Ammonium Chloride for manufacturing of Ammonium Chloride

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45.3 Product Usage / Utilization

The Ammonium Chloride will be utilised in Pharmaceutical sector, Galvanizing unit, Battery manufacturing and Fertilizer industry.

The product i.e. Ammonium Chloride shall comply with the Bureau of Indian Standard: IS 1113:1965 for use as fertilizer.

The unit shall label its product (i.e. Ammonium Chloride) manufactured by utilizing aforesaid Hazardous waste as "This Ammonium Chloride has been manufactured by utilizing Spent Ammonium Chloride, generated from Hexamethyl Di Silazane manufacturing process."

45.4 Standard Operating Procedure for utilization

This SoP is applicable only for the utilization of Spent Ammonium Chloride generated during manufacturing of Hexamethyl Di Silazane.

- 1) The Spent Ammonium Chloride 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 Spent Ammonium Chloride in to 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 Spent Ammonium Chloride may be 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.
- 4) There shall be no manual handling of the hazardous wastes (Spent Ammonium Chloride) chemical process pump shall be used for transfer of Spent Ammonium Chloride through pipelines to the reaction vessel.
- 5) The entire process 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 ETP or reaction tank, as the cases may be, through chemical process pump.
- 6) Vent of all storage tanks and evaporation pan shall be connected to scrubber for treatment using acidic medium.
- 7) 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.
- 8) The unit shall ensure that the said utilization process and its associated activities shall be demarcated separately within the unit.

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- 9) The wood/ briquette fired boiler shall be connected to stack of height 30 meters above the roof top or at height prescribed by SPCB/PCC, whichever is higher.
- 10) Spent Ammonium Chloride shall be transferred to all the process units like evaporator, centrifuge and crystallizer through process pumps. The evaporator be a closed system and shall have vent ducts connected to common scrubbing system followed by dispersion through stack. The evaporator shall be kept under covered shed with proper ventilation in the process area.
- 11) The mother liquor generated from the centrifuge shall be collected through process pumps for reuse in the initial stage of the utilization process i.e evaporator.
- 12) The centrifuged mass is transferred to the dryer through mechanised system, wherein the particulate emissions shall be controlled through scrubber system followed by dispersion through stack.
- 13) 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 MSDS. The safety precautions of the worker shall be in accordance with the Factory Act, 1948, as amended from time to time.
- 14) It shall be ensured that the aforesaid hazardous waste is procured from the industries who have valid authorization for the same from the concerned State Pollution Control Board as required under Hazardous and Other Wastes (Management and Transboundary Movement) Rules, 2016.
- 15) SPCBs/PCCs shall ensure synchronization of generation and utilization of Spent Ammonium Chloride and the same shall be reflect in respective authorization specifying name and quantity.
- 16) Transportation of Spent Ammonium Chloride shall be carried out by sender or receiver (utilizer) only after obtaining authorisation from the concerned SPCB under the Hazardous and Other Wastes (Management & Transboundary Movement) Rules, 2016.
- 17) Prior to utilization of Spent Ammonium Chloride, the unit shall obtain authorization for generation, storage, and utilization of spent Ammonium Chloride solution from the concerned State Pollution Control Board under the Hazardous and Other Wastes (Management and Transboundary Movement) Rules 2016.
- 18) Treatment and disposal of wastewater:
Waste water generated from floor-washings, spillages, reactor washing, scrubber bleed, boiler blow down, cooler blow down etc. shall be treated Physico-Chemically in an ETP and may be sent to CETP for final disposal or treated further in a captive facility to comply with surface water discharge standards.

In case of zero discharge condition by SPCB/PCC, the treated waste water from ETP may be evaporated in Forced Evaporators like MEE. The concentrated liquid from the

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evaporator shall be sent to spray dryer for conversion into dry powder which may be disposed as given in para 19 below.

- 19) 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.
- 20) The unit shall provide suitable fire safety arrangements and flame proof electrical fittings.
- 21) The hazardous wastes generated (namely the ETP sludge, scrubber, filter residue, effluent powder generated from Spray Dryer/forced evaporator, product spillages, etc.) shall be collected and temporarily stored in non reactive drums / bags under a dedicated hazardous waste storage area 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.

It shall be ensured that the highly soluble dry-powdered effluent from MEE-Spray Dryer should be stabilized or immobilized with suitable cementing material prior to secured landfilling in TSDF.

- 22) 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.

45.5 Record>Returns Filing

- 1) The unit shall maintain a passbook issued by concern SPCB wherein the following details of each procurement of Spent Ammonium 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 Spent Ammonium Chloride, quantity, date wise utilisation of the same, quantity of Ammonium 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/PCC.

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- 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.

45.6 Standards

- 1) Fugitive emission in the work zone shall comply with the following standards:

HCl	7.0 mg/m ³ Ceiling limit
Ammonia	35.0 mg/m ³ TWA*

*Reference: Occupational Safety and Health Standard 1910.1000
TWA*: time-weighted average
The permissible Exposure Limit is 8-hours 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.

- 2) Source emission monitoring from the stack attached to the evaporators and dryer shall comply with the following standards or as prescribed by the concerned SPCB/PCC, whichever is stringent;

HCl vapour & Mist	35.0 mg/Nm ³
Ammonia	75.0 mg/Nm ³
PM	50.0 mg/Nm ³
TOC	20.0 mg/Nm ³

- 3) The product Ammonium Chloride for using in the fertilizer industry, shall comply with the following specifications:

Parameters	Requirements
Moisture percent by weight, Max	2.0
Ammonical nitrogen by weight, Mix	25.0
Chlorides other than ammonical chloride (NaCl), percent by weight (on dry basis), Max	2.0
Hexamethyl Disiloxane	Not present
Hexamethyl Disilazane	Not present
Trimethyl Chlorosilane	Not present
Methyl Dichlorofomate	Not present
Toluene	Not present
TOC	Not present

- 4) Monitoring of the specified parameters for source emission shall be carried out quarterly for the first year followed by at least annually in the subsequent year of utilization. Fugitive emission for specified parameters shall be carried out quarterly. The monitoring shall be carried out by NABL accredited or ISO17025 /EPA approved laboratories and the results shall be submitted to the concerned SPCB/PCC on a quarterly basis.

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5) Standards for wastewater discharge: The treated waste water 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. In case of zero discharge or no discharge condition stipulated in the said Consent or non-availability of the Common Effluent Treatment (CETP), zero discharge shall be met.

45.7 Siting of Industry

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

45.8 Size of Plant & Efficiency of utilisation

Maximum 5.5 MT of Spent Ammonium Chloride would be required to produce 1 MT of Ammonium Chloride. Therefore, requisite facilities of adequate size of storage shed and other plants & machineries as given in para 45.9 below shall be installed accordingly.

45.9 On-line detectors / Alarms / Analysers

Online emission monitoring systems shall be installed in case of continuous process operations for HCl vapour and mist and ammonia mist.

45.10 Checklist of Minimal Requisite Facilities

Sl. No	Particulars
1	Storage tank(s) of adequate capacity to store spent ammonium chloride of at least two weeks requirement. Storage tank(s) 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 may be below the ground provided it has HDPE liner system beneath the tank and leachate collection system below HDPE liner.
2	Cool, dry well-ventilated covered storage shed(s) for Spent Ammonium Chloride storage tanks within premises.
3	Mechanized system for transfer of Spent Ammonium Chloride from tanker to storage tank and storage tank to the each unit operation.
4	The process units shall have proper ventilation (preferably with ventilation ducts above the process units connected to ID fan with exhaust above roof level)
5	Filter like drum filter
6	Evaporation pan
7	Crystalliser
8	Centrifuge
9	Dryer (of adequate size operate electrically or by fuel) as permitted by the concerned SPCB/PCC.

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10	Boiler operated electrically or by fuel as permitted by the concerned SPCB/PCC. Depending upon type of fuel, suitable air pollution control device(s) shall be installed with the boiler followed by stack of height as prescribed by the concerned SPCB/PCC.
11	Suction arrangement to suck vapours from suction hood system provided over evaporation pan for channelizing the fumes to the common vent.
12	Adequate Effluent treatment plant so as to comply with standards/conditions prescribed by the concerned SPCB/PCC. Forced Evaporator followed by Spray dryer (in case of zero discharge condition by SPCB/PCC)
13	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.
14	Dedicated hazardous waste storage area for temporary storage of hazardous waste generated during utilization process.

PCB