

**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 Hydrochloric acid [generated during manufacturing of
Trichloroethylene / Perchloroethylene (or) Chlorinated Paraffin Wax (CPW)] for
manufacturing of 7ADCA**



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Central Pollution Control Board
(Ministry of Environment, Forest & Climate Change, Government of India)
Parivesh Bhawan, East Arjun Nagar,
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Utilization of Spent Hydrochloric acid [generated during manufacturing of Trichloroethylene / Perchloroethylene (or) Chlorinated Paraffin Wax (CPW)] for manufacturing of 7ADCA

Procedure for grant of authorization by SPCBs/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 SoPs for 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. 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.
- 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 during subsequent period for atleast once a year.
- 3) In-case of lack of requisite infrastructures with the SPCBs/PCCs, they may engage 3rd party institutions or laboratories having EPA,1986/NABL/ISO17025 accreditation / recognition for monitoring and analysis of prescribed parameters in SoPs for verification purpose.
- 4) 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 half yearly basis i.e., by July and January respectively.
- 5) Authorization 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.
- 6) 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 thereof.
- 7) 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.

67.0 Utilization of Spent HCl:

Type of HW	Source of generation	Recovery/Product
Spent Hydrochloric acid (Schedule-II Class B 15 Inorganic acids of HOWM Rules, 2016)	Spent hydrochloric acid generated from manufacturing of trichloro/perchloroethylene (or) Chlorinated Paraffin Wax	Manufacturing of 7ADCA.

67.1 Source of Waste:

Spent Hydrochloric acid generated from manufacturing of Trichloro / Perchloroethylene (or) Chlorinated Paraffin wax falls under Class B 15 (Inorganic acids) Schedule-II of HOWM Rules – 2016.

Table 1. Typical Characteristics of Spent HCl (generated from manufacturing of Trichloro/ Perchloroethylene) given below:

Sr. no.	Parameter	Unit	Result
1	Appearance	-	Pale yellow
2	pH	-	Acidic
3	% HCl	%	31.78
4	COD	mg/kg	2431
5	TOC	mg/kg	661
6	Sulphate as SO ₄	%	0.00066
7	Iron as Fe	mg/kg	15.56

Table 2. Typical Characteristics of Spent HCl (generated from manufacturing of Chlorinated Paraffin wax) given below:

Sr.no.	Parameter	Unit	Result
1	Appearance	-	Pale yellow
2	pH	-	Acidic
3	% HCl	%	34.46
4	COD	mg/Kg	2110
5	TOC	mg/Kg	527
6	Sulphate as SO ₄	%	0.00078
7	Iron as Fe	mg/Kg	17.40

67.2 Utilization Process:

7-Amino Deacetoxy Cephalosporanic Acid (7ADCA) is manufactured from Penicillin-G. In this manufacturing process Bis-Silyl Urea (BSU) required in the ring expansion step. BSU is prepared by using HCl gas. HCl gas is prepared by utilizing Spent HCl.

BSU Manufacturing process: The HCl gas is generated by the reaction of Spent HCl (30-35%) with H₂SO₄ (98%), and Spent H₂SO₄ (65-70%) also generated during this step. HCl gas is then purged with Hexamethyldisiloxane (HMDO) and toluene mixture for the preparation of Tri Methyl Chlorosilane (TMCS). Further BSU is produced by addition of such as TMCS, Ammonia gas, caustic and other constituents. Thus BSU generated will utilized in the manufacturing of 7ADCA. Mother Liquor/effluent generated from the process further treated in the ETP provided.

Spent Sulphuric acid and (or), any other scrubbing liquor shall be disposed as prescribed by SPCB/PCC in accordance of HOWM Rules, 2016.

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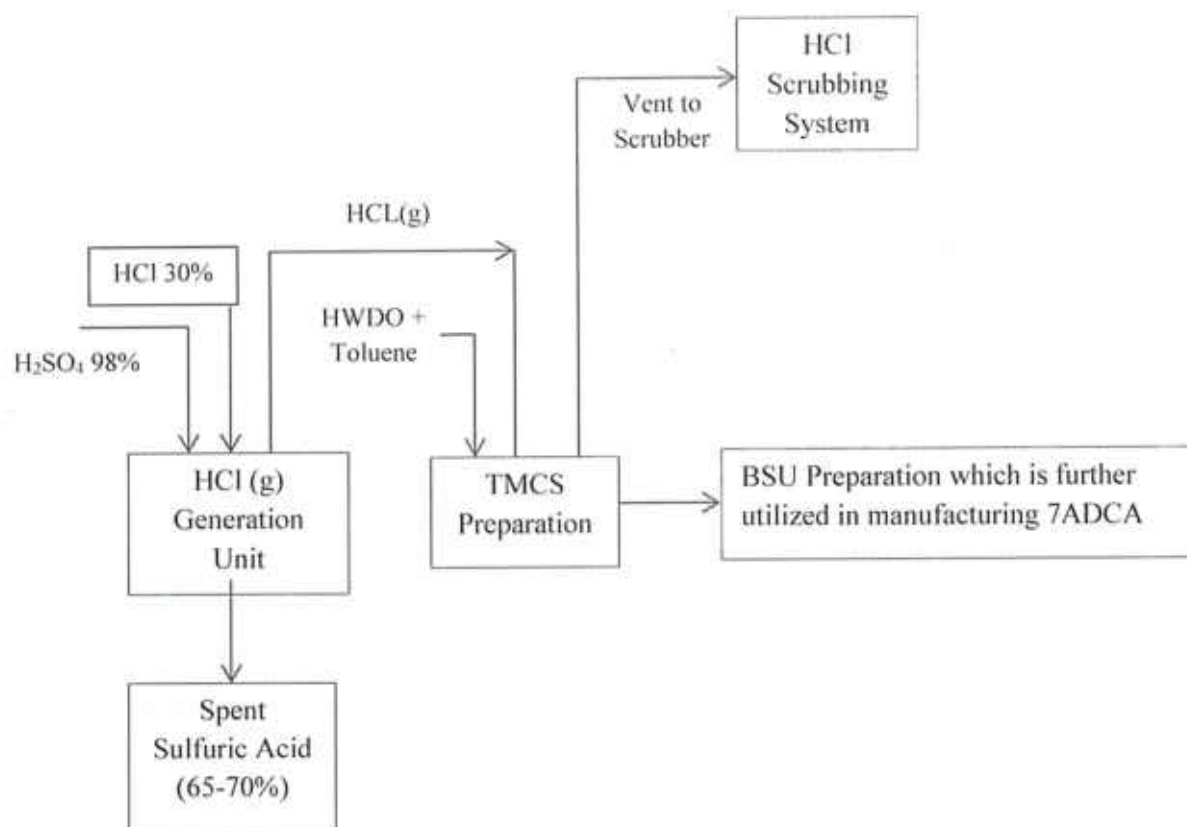


Figure: 1-Process flow diagram for utilization of hazardous waste

67.3 Product Usage / Utilization

7ADCA (7-Amino Deacetoxy Cephalosporanic acid) is an intermediate of Cefadroxil Active Pharma Ingredient (API). The product manufactured by utilizing Spent HCl shall comply as per Bureau of Indian Standards (BIS) or any other notified standards as required for further respective utilization.

67.4 Standard Operating Procedure for utilization

This SoP is applicable only for Utilization of Spent Hydrochloric acid [generated during Trichloroethylene / Perchloroethylene (or) Chlorinated Paraffin Wax (CPW)] for manufacturing of 7ADCA.

- 1) The Spent Hydrochloric acid shall be procured only in SPCB/PCC authorised barrels/closed tanks mounted over vehicles fitted with requisite safeguards ensuring no spillage of the same.
- 2) Spent Hydrochloric acid shall be stored in either HDPE or rubber lined steel tank and kept in acid proof brick lined dyke under shed. Unit shall provide slope and collection pit in storage area.
- 3) There shall be no manual handling of the Spent Hydrochloric acid. Spent Hydrochloric acid shall be unloaded from the closed tanker to the storage tank through pipelines using dedicated transfer pump. Spill containment arrangement shall be provided around the Spent Hydrochloric Acid storage tanks.

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- 4) Also the feeding of Spent Hydrochloric acid from storage tanks to the utilization reactors shall be done through pipelines using dedicated transfer pump.
- 5) The storage and handling of Spent Hydrochloric acid shall be done under a shed of proper vertical height and over imperviously lined flooring.
- 6) The unit shall install storage tanks 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.
Further, the storage area of Spent Hydrochloric acid 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, as the cases may be, through chemical process pump.
- 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) Adequate scrubbing system shall be provided to the reactors and storage tanks to treat the emissions.
- 9) The treated gases shall comply with emission norms and prior to dispersion into atmosphere through stack. The height of stack shall be a minimum of 6 m above the roof top or as prescribed by the concerned SPCB/PCC, whichever is higher.
- 10) Unit shall ensure the safety measures such as safety valves to appropriate equipment where, the high pressure process may involve.
- 11) 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.
- 12) Treatment and disposal of wastewater:
Wastewater generated from floor-washings, spillages, reactor washing, scrubber bleed including the wastewater from filtration shall be treated Physico-Chemically in an ETP or may be sent to CETP for final disposal or be 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 managed as per conditions stipulated by the SPCB/PCC.
- 13) 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.
- 14) The hazardous wastes generated (namely the Filter cake, other chemical sludge, other residues 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 MEE 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.
- 15) It shall be ensured that the Spent Hydrochloric Acid are procured from the industries, which have valid authorization from the concerned State Pollution Control Board as required under Hazardous and Other Wastes (Management and Transboundary Movement) Rules, 2016.

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- 16) Transportation of Spent Hydrochloric Acid shall be carried out by sender (generator) or receiver (utilizer) only after obtaining authorisation 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.
- 17) Prior to utilization of Spent Hydrochloric Acid, the unit shall obtain authorisation for generation, storage and utilization of Spent Hydrochloric Acid from the concerned State Pollution Control Board under Hazardous and Other Wastes (Management and Transboundary Movement) Rules, 2016.
- 18) 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.
- 19) The unit shall provide suitable fire safety arrangements and flame proof electrical fittings.
- 20) 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. The unit shall provide suitable fire safety arrangements and flame proof electrical fittings.

67.5 Record>Returns Filing

- 1) The unit shall maintain a passbook issued by concerned SPCB wherein the following details of each procurement of Spent Hydrochloric acid 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 Hydrochloric acid, date wise utilization of the same, hazardous waste generation and its disposal, etc. shall be maintained including analysis report of fugitive 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.
- 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.

67.6 Standards

Utilization of Spent Hydrochloric acid [generated during manufacturing of Trichloroethylene / Perchloroethylene (or) Chlorinated Paraffin Wax (CPW)] for manufacturing of 7ADCA

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

Particulate Matter	50 mg/Nm ³
NO _x	50 ppm
SO _x	100 ppm
HCl Mist	20 ppm
Ammonia (as NH ₃)	175 mg/Nm ³

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

PM ₁₀	5 mg/m ³ TWA* (PEL)
Cl ₂	3 mg/ m ³ TWA* (PEL)
HCl	7 mg/m ³ Ceiling Limit
Acid Mist (H ₂ SO ₄)	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.

3) Monitoring of the above specified parameters for source emission shall be carried out quarterly for 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 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 zero discharge or no discharge condition stipulated in the said consent or non-availability of the common Effluent Treatment Plant (CETP), zero discharge shall be met.

67.7 Siting of Industry

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

67.8 Size of Plant and Efficiency of Utilisation

1 MT of 7 ADCA is produced by using 4.874 MT of spent Hydrochloric Acid. Therefore requisite facilities of adequate size of storage shed and other plant & machineries shall be installed accordingly.

67.9 On-line Detectors / Alarms / Analyzers

In case of continuous process operations, online emission analyzers for PM, SO₂, NO_x in the stack shall be installed and the online data be connected to the server of the concerned SPCB/PCC

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67.10 Checklist of Minimal Requisite Facilities

Sr. No	Particles
1.	Storage tanks of adequate capacity to store Spent Hydrochloric Acid. Such storage tanks shall be placed above the ground and contained with low rise parapet/bund wall and acid proof floor with slope to collect spillages , if any, in to collection pit. Alternately, the 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 sheds for Spent Hydrochloric Acid storage tanks, product storage tanks and process activities within premises and dedicated hazardous storage area for temporary storage of hazardous waste generated during utilization process.
3.	Mechanized system for transfer of Spent Hydrochloric Acid from storage tanks to Lime Digester shall be installed. No manual handling shall be allowed in any case
4.	The process units shall have adequate scrubbing system consist of ventury scrubber, water scrubber and caustic scrubber at transfer point, storage tank to reactor, storage tanks and reaction vessels with stack of adequate height
5.	Spare vessel to transfer the reaction mass, if any, in case of leakage or damage to the Lime Digester.
7.	Pumps, pipes, feeders and other equipment for mechanical handling of Spent Hydrochloric Acid.
8.	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 Series LATS/80/2013-14.
9.	Adequate scrubbing system to the storage tanks and reactor.
10.	Standby pumps with online pH sensor for scrubbing media with hooter
