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 mono/di/tri Sodium Phosphate solution (generated during production of 4,7- dichloroquinoline) for manufacturing of Tri Sodium Phosphate crystals





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# <u>Procedure for grant of authorization by State Pollution Control Boards (SPCBs)/Pollution Control Committees (PCCs) for utilization of Hazardous waste</u>

- 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 Central Pollution Control Board (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.
  - f) Monitor closely the quantity of hazardous waste (spent mono, di & tri sodium phosphate solution) being sent by generators and the quantity being utilized by authorized facilities in production of tri sodium phosphate crystals.
- 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 at least 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 3<sup>rd</sup> party institutions or laboratories having EPA, 1986/NABL/IS017025 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) Rules, 2016 (HOWM Rules, 2016) to CPCB and also upload the same on SPCB/PCC website, periodically.
- 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 (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 EPA notified and OSHA 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 utilizer of spent mono/di/tri SP solution shall maintain daily records in National Hazardous Waste Tracking System (NHWTS).

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# 112.0 Utilization of spent mono/di/tri SP solution:

Type of HW	Type of HW Source of generation	
Spent mono/di/tri Sodium Phosphate (SP) solution (Category 28.1 of Schedule- I of HOWM Rules, 2016		

#### 112.1 Source of Waste:

Spent mono/di/tri SP solution generated during production of 4,7- dichloroquinoline (pharma/drug product) is categorized as hazardous waste at 28.1 of Schedule-I of HOWM Rules, 2016, which is required to be disposed in an authorized disposal facility in accordance with authorization condition, when not utilized as resource recovery.

Table 1 Typical Characteristics of Spent mono/di/tri SP solution:

Sr. No.	Parameters	Unit	Value
1	pH 25°C		9.12
2	Purity	%	>30.5
3	COD	mg/l	1028.1
4	TOC	mg/l	2.15
5	Cyanide	mg/l	5.12
6	Lead	mg/l	2.05
7	Nickel	mg/l	5.12
8	Copper	mg/l	3.19
9	Chromium	mg/l	7.52

Note: SPCBs/PCCs to check the characteristics of spent sodium phosphate solution 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 or may be referred to CPCB

#### 112.2 Utilization Process

The spent mono/di/tri SP solution (generated during production of 4,7- dichloroquinoline) received in tankers is transferred to the raw material storage cum settling tanks. The clear solution from storage tank is transferred for crystallization and fresh NaOH is added to raise the pH of spent solution up to range of 12-14. At this increased pH, mono and di sodium phosphate converts into tri sodium phosphate. The reactions that occurs are as follow:

$$NaH_2PO_4 + 2NaOH \rightarrow Na_3PO_4 + 2H_2O$$
  
 $Na_2HPO_4 + NaOH \rightarrow Na_3PO_4 + H_2O$ 

Also, the addition of NaOH results in an exothermic reaction which in turn accelerates the crystallization of tri sodium phosphate. The batch mass (i.e. the crystallized Tri Sodium Phosphate and the mother liquor) is then transferred to a centrifuge to filter out Tri Sodium Phosphate crystals. The mother liquor is stored in storage tanks and further reused in subsequent batches in the crystallization process to increase the recovery.

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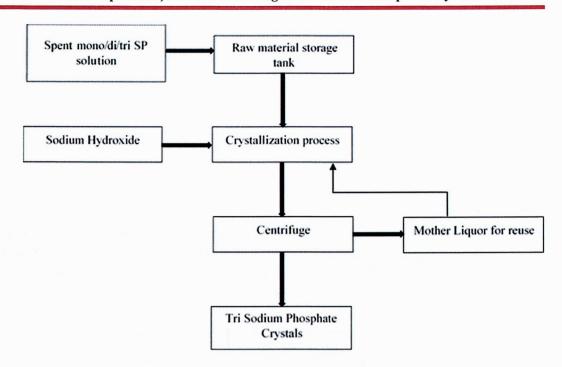


Figure-1.: Process flow diagram for utilization of spent mono/di/tri SP solution for manufacturing of tri sodium phosphate crystals

## 112.3 Standard operating procedure for utilization of spent mono/di/tri SP solution

This SoP is applicable only for utilization of spent mono/di/tri SP solution (generated during production of 4,7- dichloroquinoline) in manufacturing of tri sodium phosphate crystals.

- 1) The Spent mono/di/tri SP solution shall be procured only in SPCB/PCC authorized barrels/closed tanks mounted over vehicles fitted with requisite safe guards ensuring no spillage of the hazardous waste.
- 2) The Spent mono/di/tri SP solution shall be stored in SPCB authorised tanks (preferably non-reactive HDPE/FRP tank) dedicated storage tanks on corrosive resistant & impervious tiles flooring area within premises with caution sign.
  - Further, storage sheds/ area shall have proper slope and seepage collection pit to collect seepage / floor washing. The collected seepage / floor washing shall be channelized to Effluent Treatment Plant for further treatment. The bottom residue if any accumulated in storage tank, shall be filtered and transferred to Common hazardous waste TSDF.
- 3) The unit shall install storage tanks undercool, dry, well ventilated covered storage shed(s) within premises, as authorized by the concerned SPCB/PCC under HOWM Rules, 2016.
- 4) The unloading and transfer of Spent mono/di/tri SP solution shall be carried out through a dedicated mechanical transfer pump with fixed pipeline in a closed system. No manual handling shall be practiced.
- 5) Treatment and disposal of wastewater (if any):
  - Wastewater generated from floor-washings, spillages, storage tank washing, and discarded mother liquor shall be treated Physico-Chemically in an ETP to comply with surface water discharge standards or may be sent to CETP for final disposal as prescribed by SPCB/PCC.

In case of zero-discharge, the treated water from ETP may be managed as per conditions Waste Management-II Division, CPCB, Delhi 3 | Page

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stipulated by SPCB/PCC. Otherwise, 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.

- 6) The hazardous wastes namely, storage tank sludge, settling residues, etc. (if any) generated 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 the generation of the waste in accordance with the authorization issued by the concerned SPCB/PCC. Such storage area shall be covered with proper ventilation.
- 7) It shall be ensured that the Spent mono/di/tri SP solution is procured from the industries, which have valid authorization from the concerned SPCB/PCC as required under HOWM Rules, 2016.
- 8) Transportation of Spent mono/di/tri SP solution shall be carried out by sender (generator) or receiver (utilizer) only after obtaining authorization from the concerned SPCB/PCC under HOWM Rules, 2016. Requisite manifest documentation shall be followed as laid down under the said Rules.
- 9) The unit shall not sell the recovered product to any trader and concerned SPCB/PCC shall ensure the same. The tri sodium phosphate crystals manufactured from spent mono/di/tri SP solution shall be directly given to actual end users only. The names of end-users shall be reported to SPCBs/PCCs.
- 10) Prior to the utilization of Spent mono/di/tri SP solution, the unit shall obtain authorization for storage, utilization and disposal of Spent mono/di/tri SP solution from the concerned SPCB/PCC under HOWM Rules, 2016.
- 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) 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.
- 13) The unit shall provide suitable fire safety arrangements and flame proof electrical fittings.
- 14) 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.

#### 112.4 Product Usage/Utilization

- 1. Tri sodium phosphate crystals manufactured by utilizing Spent mono/di/tri SP solution (generated during production of 4,7- dichloroquinoline) permitted for industrial uses only such as production of detergent, cleaning agent, degreaser, buffering agent, pesticide, etc.
- 2. The end use usage of product (Tri sodium phosphate crystals) derived utilizing Spent mono/di/tri SP solution shall in no case be utilized as raw material for production of food

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& food processing, pharma & fertilizer sector.

3. The unit shall label the product as "This Tri sodium phosphate crystals has been manufactured by utilizing Spent mono/di/tri SP solution generated from pharma/drug sector".

## 112.5 Record/Returns Filing

- 1) The unit shall maintain a passbook issued by concern SPCB/PCC and maintain details of each procurement of spent mono/di/tri SP solution as mentioned below:
  - 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 mono/di/tri SP solution, 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 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.
- 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/PCC.
- 5) Each procurement, production and transportation details (along with quantity of hazardous waste generated & disposed) shall be maintained on the National Hazardous Waste Tracking System developed by CPCB. Unit shall use NHWTS to manage the manifest, enter daily records of quantity generated, disposed, etc.

#### 112.6 Standards

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

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PM <sub>10</sub>	10 mg/m <sup>3</sup> , TWA
Sodium Hydroxide	2 mg/m <sup>3</sup> , TWA
Formaldehyde	0.75 ppm, TWA
Phosphoric acid	1 mg/m <sup>3</sup> , TWA

TWA-Time-weighted average

- 2) 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.
- 3) Standard for wastewater discharge: Treated effluent (if any) 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

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(i) zero discharge as per consent or (ii) non-availability of common Effluent Treatment Plant (CETP), the unit shall achieve zero discharge by setting up adequate captive treatment facility.

## 112.7 Siting of Industry

Facilities for utilization of Spent mono/di/tri SP solution 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. This may not be applicable to the facility already engaged in the production of tri sodium phosphate crystals and have obtained CTE/CTO.

## 112.8 Size of Plant and Efficiency of Utilization

It is expected that 1 MT of tri sodium phosphate crystals shall be produced by utilizing approximately 3.8 MT of spent mono/di/tri SP solution (of purity around 30%)

Therefore, requisite facilities of adequate size of storage shed and other plant & machineries shall be installed accordingly.

## 112.9 Checklist of Minimal Requisite Facilities

Sr. No.	Particulars
1.	Storage tank for spent mono/di/tri SP solution with caution signs, proper covers, acid-proof lined floors and spillage collection system.
2.	Cool, dry, well-ventilated covered sheds for spent mono/di/tri SP solution storage tanks (above the ground), product storage, and process activities within premises and dedicated area for storage of mother liquor generated during utilization.
3.	Reaction Vessels / crystallizer & centrifuge.
4.	Closed system with feeder & other equipment for unloading of spent solution.
5.	Mechanical Pumps and Fixed Pipelines for transfer of spent mono/di/tri SP solution.
6.	Systems for removal of bottom sludge from storage tanks, filtering, storage, etc. and disposal through CHWTSDF.
7.	Fume ventilation systems for reaction vessel/crystallizer unit with suction hood, duct, ID fan, scrubber and a stack.

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