

**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 Sulphuric Acid generated from dyes & dyes intermediate industries in production of another dyes and dye intermediate products**



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**Central Pollution Control Board**  
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## Utilization of Spent Sulphuric Acid generated from dyes & dye intermediate industries in production of another dyes and dye intermediate products

### 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 for maintaining records of receipt of spent acid 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 3<sup>rd</sup> 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.

### 53.0 Utilization of spent sulphuric acid:

Type of HW	Source of generation	Recovery/Product
Spent sulphuric acid (Category: 26.3 of Schedule I of HOWM Rules, 2016 and Inorganic Acids mentioned at S. No 15 in the foot note of Schedule II of HOWM Rules, 2016)	Generated from dyes & dyes intermediates industries	As a supplementary resource in manufacturing of dyes & dye intermediates

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### **53.1 Source of Waste**

The spent sulphuric acid is generated from manufacturing of dyes and dye intermediate industries and is categorized as Hazardous waste at S. No. 26.3 of Schedule I of HOWM Rules, 2016 and Inorganic Acids mentioned at S. No 15 in the foot note 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.

### **53.2 Utilization Process**

The spent sulphuric acid generated from various dyes and dye intermediate industries is used as raw material for production of another dyes and dye intermediate products (by replacing use of pure sulphuric acid). The production of dyes or dye intermediate products mainly includes unit operations like sulphonation, drowning, isolation, filtration, purification, neutralization, centrifugation, drying, pulverising, packing etc.

### **53.3 Product Usage / Utilization**

The spent sulphuric acid shall be utilised in manufacturing of dyes & dye intermediates except food processing industries.

The unit shall label its product (i.e. dye and dye intermediates) manufactured by utilizing aforesaid hazardous waste as "This product name has been manufactured by utilizing spent sulphuric acid, generated from dyes & dye intermediate industries".

### **53.4 Methodology for finalization of quality of spent sulphuric acid for utilisation in production of dye and dye intermediates**

- 1) MOEF&CC vide Office Memorandum No: SO 3518(E) dated 23/11/2016 notified the procedure to issue permission for the "Change in product mix without increase in pollution load". As per this notification, all SPCBs shall have to frame Technical Committee to implement the notification. All SPCBs have framed Technical Committee to comply the said notification.
- 2) It is envisaged that implementation of this SoP is done through this committee or other committee constituted for implementation of HOWM Rule, 2016, wherever scrutiny and assessment are required in this SoP. Accordingly, following shall be the responsibilities of Technical Committee while reviewing the application;
  - a) The technical committee shall decide the characteristics of spent sulphuric acid generated from manufacturing of dyes and dye intermediate industry for utilization in manufacturing of another dyes & dye Intermediate products.
  - b) The committee shall permit the quantity for utilization of spent acid for production of another dyes and dye intermediate products based on characteristics of spent acid, material balance and mass balance.
  - c) The source emission from the stack connected to reactors/ process stack and fugitive emission in the process/storage area shall comply with the standards prescribed in section 1.7 (i) and 1.7 (ii) of this SoP respectively. Standards prescribed in aforesaid section is not exhaustive these may vary depending upon characteristics of spent sulphuric acid and utilization process; however committee may take final decisions on these.
  - d) Hazardous waste generated during utilization of spent acid in production of various dyes and dye intermediate products shall be listed in the authorization issued by SPCBs along with its disposal mechanism.

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- e) Besides the minimal requisite facilities stipulated in this SOP at section 1.10, other requisite facilities for each of the utilization of spent sulphuric acid may vary depending on the unit operations of the respective utilization process and the same may be decided by the committee.
- f) The general conditions shall be complied as mentioned in the SoP.
- g) Based on the recommendation of the Technical Committee, SPCB shall grant authorization to unit under Rule 9 for permission to utilize spent acid for production of dyes and dyes intermediates.

### 53.5 Standard Operating Procedure for utilization

This SoP is applicable only for utilization of spent sulphuric acid generated during manufacturing of dyes and dyes intermediate industries in production of another dye and dye intermediate products.

- 1) The spent sulphuric acid 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 sulphuric acid into the storage tank. The receiving storage tank shall be placed above the ground and contained with low raise parapet/hund wall with slope to collect spillages, if any, into collection pit. Alternatively, storage tanks for spent sulphuric acid 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 hazardous wastes (spent sulphuric acid). Acid Proof pump shall be used for transfer of spent sulphuric acid through pipelines to the reaction vessel.
- 5) The vent of spent sulphuric acid 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.
- 8) Spent sulphuric acid shall be mixed with the reactants in closed vessel reactors. The reactor shall be kept under covered shed with proper ventilation in the process area.
- 9) Sulphuric acid mist and SO<sub>2</sub> are expected to be liberated from the reactors, where the spent sulphuric acid is added. Thus, the said reactors 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.

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- 10) 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.
- 11) The reactors, nutsche filters and centrifuges be a closed system and shall have vent ducts connected to common scrubbing system followed by dispersion through stack.
- 12) 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.
- 13) 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.

14) 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 and 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 evaporated in forced evaporators like MEE. The concentrated liquid from the evaporator shall be sent to spray dryer for conversion into dry powder which may be disposed as given in the para 15 below.

- 15) The hazardous wastes generated (namely the filter residue, ETP sludge, scrubber, effluent powder generated from spray dryer/forced evaporator, product spillages, damaged filter liners, 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.

- 16) Transportation of spent sulphuric acid 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 sulphuric acid, the unit shall obtain authorization for generation, storage, and utilization of spent sulphuric acid solution from the concerned State Pollution Control Board under the 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 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.

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- 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 the requirements in accordance with the Public Liability Insurance Act, 1991 as amended, wherever applicable.
- 21) SPCBs/PCCs shall ensure synchronization of generation and utilization of spent sulphuric acid and the same shall be reflected in respective authorization specifying name and quantity.
- 22) The treated effluent 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.

### 53.6 Record>Returns Filing

- 1) The unit shall maintain a passbook issued by concern SPCB wherein the following details of each procurement of spent sulphuric 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 sulphuric acid, quantity, date wise utilisation 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.

### 53.7 Standards

- 1) Source Emissions from the stack connected to Reactors /process stacks shall comply with the following standards or as prescribed by the concerned SPCB/PCC, whichever is stringent;

PM	: 50 mg/Nm <sup>3</sup>
H <sub>2</sub> SO <sub>4</sub> acid mist	: 50 mg/ Nm <sup>3</sup>
SO <sub>2</sub>	: 50 mg/ Nm <sup>3</sup>
TOC	: 20 mg / Nm <sup>3</sup>

- 2) Fugitive emission in the storage area shall comply with the following standards:

PM <sub>10</sub>	: 5 mg/m <sup>3</sup> TWA* (PEL)
Ammonia	: 35 mg/m <sup>3</sup> TWA* (PEL)
Hydrogen chloride	: 7 mg/m <sup>3</sup> TWA* (PEL)
Toluene	: 200 ppm
Sulphuric acid	: 1 mg/m <sup>3</sup> TWA* (PEL) 3 mg/m <sup>3</sup> TWA* (STEL)

\*PEL: Permissible Exposure Limit

\*time-weighted average (TWA): measured over a period of 8 hours of operation of process.

\*short term exposure limit (STEL): measured for 15 minutes duration of exposure



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- 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 NABL accredited or ISO17025 /EPA 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 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 Plant (CETP), zero discharge shall be met.

**53.8 Siting of Industry**

Facilities for utilization of spent sulphuric acid 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.

**53.9 On-line detectors / Alarms / Analysers**

In case of continuous process operations, online emission analysers for relevant parameters of the aforesaid emission parameters shall be installed following OCEMS guidelines of CPCB and such online data be connected to the server of the concerned SPCB/PCC.

**53.10 Checklist of Minimal Requisite Facilities**

Sl. No	Particulars
1	Storage tank(s) of adequate capacity to store spent sulphuric acid of at least two weeks requirement. Such storage tank(s) shall be placed above the ground and contained with low raise parapet/bund wall and acid proof floor with slope to collect spillages, if any, into collection pit. Alternately, the storage tank(s) 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 sulphuric acid storage tanks within premises.
3	Mechanized system for transfer of Spent Sulphuric Acid from tankers to storage tanks to reactor vessels.
4	The process shall have proper ventilation (preferably with ventilation ducts above the process units).
5	Reactors with suction hood connected via duct to scrubber and stack of adequate height as prescribed by concerned SPCB/PCC.
6	Nutsche Filters/Filter Press
7	Centrifuges
8	Adequate Effluent treatment plant so as to comply with standards/conditions prescribed by the concerned SPCB/PCC. Reverse Osmosis & Forced Evaporator (in case of zero discharge condition by SPCB/PCC)
9	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/20/2013-14.

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