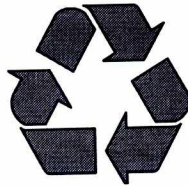


**Standard Operating Procedure and Checklist of Minimal Requisite Facilities
for utilization of hazardous waste under Rule 9 of the Hazardous and Other
Wastes (Management and Transboundary movement) Rules, 2016**

**Utilization of Spent Liquid Glauber Salt (generated during Para Base Vinyl
Sulphone manufacturing process) for production of Reactive Dye (Reactive
Orange 2 R)**



December, 2018

Central Pollution Control Board
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Shahdara, Delhi – 110032

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Utilization of Spent Liquid Glauber Salt (generated during Para Base Vinyl Sulphone manufacturing process) for production of Reactive Dye (Reactive Orange 2R)

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 Hazardous and Other Wastes (Management & Transboundary Movement) 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, thereof.
- 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

46.0 Utilization of Liquid Glauber Salt:

Type of HW	Source of generation	Recovery/Product
Spent Liquid Glauber Salt (Category no.26.1 of Schedule I of HOWM Rules, 2016)	Generated from the ethoxylation step of Para Base Vinyl Sulphone manufacturing process	As a supplementary resource in manufacturing of Reactive Dye (reactive orange 2 R).

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Utilization of Spent Liquid Glauber Salt (generated during Para Base Vinyl Sulphone manufacturing process) for production of Reactive Dye (Reactive Orange 2R)

46.1 Source of Waste

Spent Liquid Glauber Salt is generated during the ethoxylation step of Para Base Vinyl Sulphone (dye& dye-intermediate) manufacturing process. The aforesaid Spent Liquid Glauber Salt is categorised as Hazardous waste at S. No.26.1 of Schedule I 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.

Typical characteristics of the hazardous waste is given below:

Parameters	Results
pH	2.18
Lead as Pb	<0.02 mg/L
Chromium as Cr	0.024 mg/L
Copper as Cu	0.120 mg/L
Nickel as Ni	0.29 mg/L
Zinc as Zn	0.28 mg/L
Cadmium as Cd	<0.004 mg/L
COD	56027 mg/L
TOC	22310 mg/kg
Moisture	48.29 %

46.2 Utilization Process

Manufacturing process of Reactive Dye (Reactive Orange 2 R) involves charging of Vinyl sulphone into reaction vessel along with water to maintain the temperature between 0 to 5°C. Then hydrochloric acid/Sulphuric acid is added followed by sodium nitrite powder gradually till diazotization is completed. Excess nitrite is removed by adding sulfamic acid just before the coupling process. In the coupling process, sodium naphthionate is added to the diazotized vinyl sulphone. The reaction mixture is stirred and temperature is maintained between 0-5°C by adding ice. The crude dye liquid is transferred to the mixing vessel where Spent Liquid Glauber Salt is added. After proper mixing/blending, it is transferred to holding tank followed by spray dryer. The final product is then collected and packed. The process flow diagram of above utilization process is given in Fig. 1.

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Utilization of Spent Liquid Glauber Salt (generated during Para Base Vinyl Sulphone manufacturing process) for production of Reactive Dye (Reactive Orange 2R)

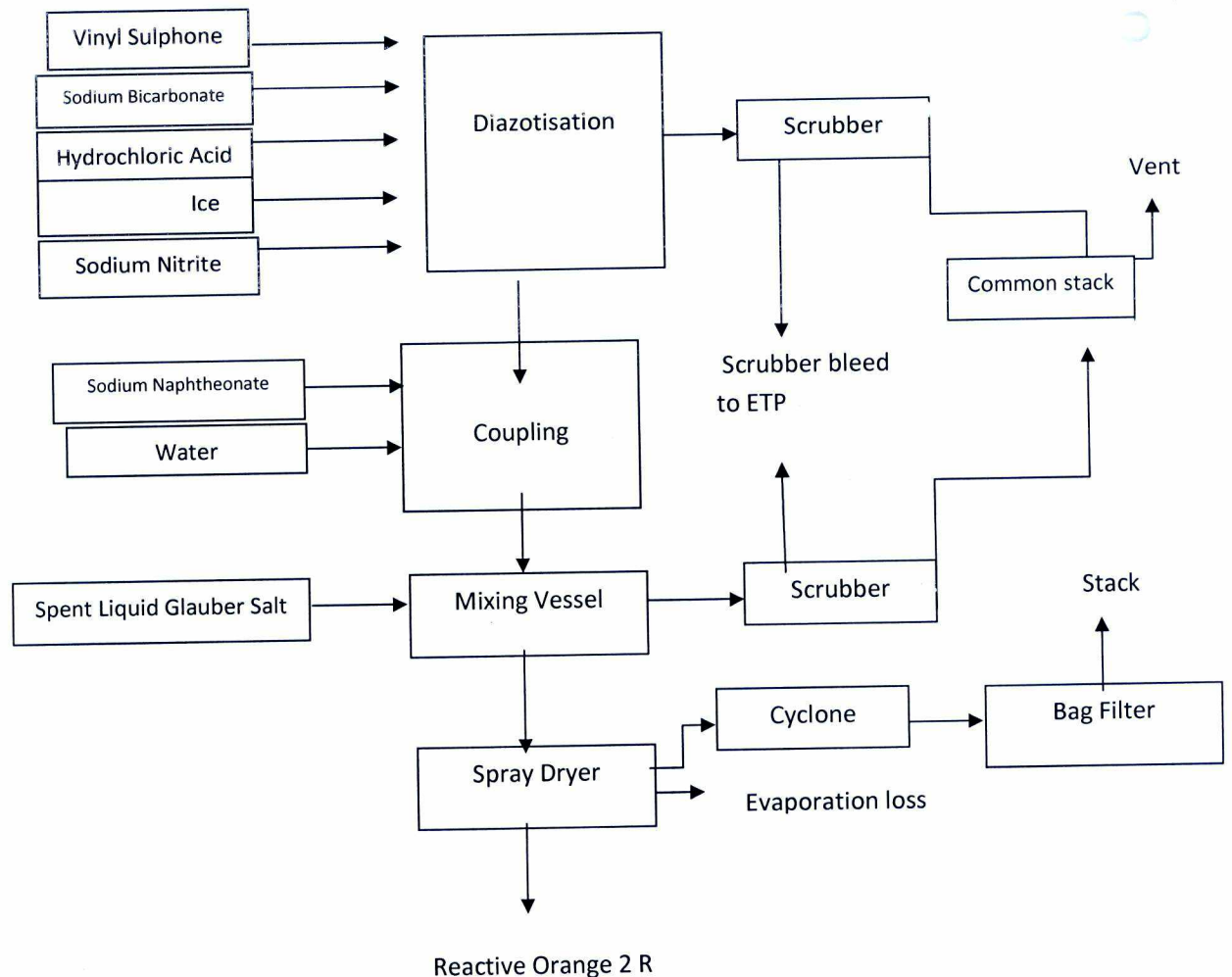


Fig. 1. Process flow diagram for production of Reactive Dye (Reactive Orange 2 R)

46.3 Product Usage / Utilization

Reactive dye is used for fabric dyeing in textile industry.

The unit shall label its product (i.e. Reactive dye) manufactured by utilizing aforesaid Hazardous waste as “This Reactive dye has been manufactured by utilizing spent liquid Glauber salt, generated from ethoxylation step of para base vinyl sulphone manufacturing process.”

46.4 Standard Operating Procedure for utilization

This SoP is applicable only for the utilization of Spent Liquid Glauber Salt generated during manufacturing of para base vinyl sulphone process for production of Reactive Dye (Reactive Orange 2 R).

- (1) Spent Liquid Glauber Salt shall be transported in SPCB/PCC authorized acid-proof tankers mounted on vehicles fitted with requisite safeguards ensuring no spillage of the same.

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Utilization of Spent Liquid Glauber Salt (generated during Para Base Vinyl Sulphone manufacturing process) for production of Reactive Dye (Reactive Orange 2R)

- (2) There should be a designated space for unloading of Spent Liquid Glauber Salt into a rubber lined storage tank. The receiving storage tank shall be placed above the ground and contained with low raise parapet/bund wall & acid proof floor with slope to collect spillages, if any, into collection pit.
Alternatively, storage tanks for Spent Liquid Glauber Salt 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 State Pollution Control Board/Pollution Control Committee 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 Liquid Glauber salt). Chemical process pump shall be used for transfer of Spent Liquid Glauber Salt through pipelines to the reaction vessel.
- (5) 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 ETP or reaction tanks, as the cases may be, through chemical process pump.
- (6) The unit shall provide separate storage tanks for the storage of chemicals and the storage tanks should be at designated place with proper cover and with acid brick lining floors.
- (7) Vinyl sulphone along with sodium bi carbonate and ice is charged in the diazotization reactor in the presence of sodium nitrite and hydrochloric acid maintaining the temperature up to 0-5°C after complete mixing, diazotized mass is sent for coupling where sodium naphtheonate and water is added and temperature is maintained up to 5°C.
- (8) Spent Liquid Glauber Salt shall be used in the mixing vessel after completion of reaction in reaction vessels (i.e. diazotization reactor and coupling) of the process. The mixer shall be transferred to dryer kept under covered shed with adequate safety gadgets provided to workers, as well as ensuring proper ventilation in the process area.
- (9) HCl is expected to be liberated from the diazotization reactor and mixing vessel. 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.
- (10) The treated acid fume/vapour shall comply with emission norms and shall be dispersed into atmosphere through stack of minimum height of 6 m above the roof top or as prescribed by the concerned SPCB/PCC, whichever is higher.
- (11) The spray dryer used during the utilization process shall be connected with cyclone followed by bag filter. The residue from bag filter is reused in the utilization process. The boiler attached with the spray dryer shall be operated electrically or by fuel 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.
- (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) such as Chemical goggles, full-face shield, or a full-face respirator,

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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.

- (13) Treatment and disposal of wastewater:

Following are the sources of wastewater from utilization process;

a) Waste water (generated from Floor washing/reactor wash/vehicle wash/spillages, etc.)

b) Scrubber Bleed Water (generated from Diazotization reactor and mixing vessel)

Waste water generated shall be treated Physico-Chemically in an ETP so as to comply with the prescribed inlet standards in case of CETP or be treated in captive ETP having adequate treatment facilities to comply with surface water discharge standards as stipulated in the Consent issued by the SPCBs/PCCs.

In case of zero discharge condition by SPCB/PCC, the treated waste water from ETP may be managed as per conditions stipulated by the SPCBs/PCCs

- (14) It shall be ensured that Spent Liquid Glauber Salt 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.
- (15) 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.
- (16) The hazardous waste i.e. residues generated (from Spray Dryer, 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. The residue from bag filter may be re used in the utilization process.
- (17) Transportation of Spent Liquid Glauber Salt and the 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.
- (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.
- (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.

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46.5 Record>Returns Filing

1) The unit shall maintain a passbook issued by concerned SPCB wherein the following details of each procurement of Spent Liquid Glauber Salt 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 Liquid Glauber salt, quantity, date wise utilisation of the same, quantity of reactive dye (reactive orange 2 R) 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.

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.

46.6 Standards

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

H ₂ SO ₄ Mist	1.0 mg/m ³ Ceiling limit
PM ₁₀	5.0 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 spray dryer, diazotization reactors and mixing vessel. shall comply with the following standards or as prescribed by the concerned SPCB/PCC, whichever is stringent;

a) Stack attached to Spray Dryer

Particulate matter	50.0 mg/Nm ³
HCl Vapour & mist	35.0 mg/Nm ³
Total heavy metals	0.5mg/Nm ³
TOC	20.0 mg/Nm ³

b) Stack attached to Diazotization reactor and mixing vessel

HCl Vapour & mist	35.0 mg/Nm ³
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SO ₂	0.5mg/Nm ³
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- 3) 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.

46.7 Siting of Industry

Facilities for utilization of Spent Liquid Glauber Salt 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.

46.8 Size of Plant & Efficiency of utilisation

Maximum 0.34 kg of Spent Liquid Glauber Salt would be required to produce 1 kg of reactive dye. Therefore, requisite facilities of adequate size of storage shed and other plants & machineries as given in para 46.10 below shall be installed accordingly.

46.9 On-line detectors / Alarms / Analysers

- a) Online emission monitoring systems shall be installed with data transmission to CPCB and SPCBs server in case of continuous process operations for HCl vapour and mist in stack attached to spray dryer.
- b) Online emission monitoring systems shall be installed with data transmission to CPCB and SPCBs server in case of continuous process operations for HCl vapour and mist in stack attached to diazotization reactors and mixing vessel.

46.10 Checklist of Minimal Requisite Facilities

S. No.	Requisite Facilities
1.	Storage tank(s) of adequate capacity to store Spent Liquid Glauber Salt of at least two weeks requirement. Such storage tank(s) shall be placed above the ground and contained with low raise parapet/bund wall & acid proof floor with slope to collect spillages, if any, into collection pit. Alternatively, 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 Liquid Glauber Salt storage tanks within premises.
3.	Mechanized system for transfer of Spent Liquid Glauber Salt from tankers to spray dryer
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.	Coupling Reactor

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6.	Diazotization Reactor
7.	Mixing Vessel
8.	Dryer (dryer of adequate size operated electrically or by fuel as permitted by the concerned SPCB/PCC.
9.	Suction arrangement to channelize emissions from diazotization reactor and mixing vessel, dryer, to the APCD and finally to the common stack of height as prescribed by the SPCBs/PCCs. Scrubber shall be installed to the reactor and vessel and cyclone followed by bag filter shall be install in case of dryer.
10.	Adequate Effluent treatment plant so as to comply with standards/conditions prescribed by the concerned SPCB/PCC.
11.	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.
12.	Dedicated hazardous waste storage area for temporary storage of hazardous waste generated during utilization process

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