

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

**Recovery of Caffeine from Caffeine Liquor
(Containing Caffeine < 2.5 %)**



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**Central Pollution Control Board
(Ministry of Environment, Forest & Climate Change,
Government of India)
Parivesh Bhawan, East Arjun Nagar,
Shahdara, Delhi – 110032**

Recovery of Caffeine from Caffeine Liquor (Containing Caffeine < 2.5 %)

Procedure for grant of authorization by State Pollution Control Board (SPCBs)/Pollution Control Committee (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 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 same source of generation as specified in SoP.
 - b. The utilization shall be same 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, SPCBs/PCCs 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. The compliance reports shall be submitted to CPCB by July every 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/PCCs shall provide half yearly updated list of units permitted under Rule 9 of Hazardous & Other Wastes (Management & Transboundary Movement) [HOWM] Rules, 2016 to CPCB and also upload the same on SPCB/PCC 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/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 same 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 studies and developing SoPs thereof.
- 7) The source and work zone standards suggested in the SoP are based on E(P)A notified and OSHA/NAAQ standard, respectively. However, SPCBs/PCCs may impose more stringent standards based on the location or process specific conditions.



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80.0 Utilization of hazardous waste (H.W.):

Type of HW	Source of generation	Recovery/ Product
Caffeine Liquor (Containing Caffeine < 2.5 %) (Category: 28.1 Schedule-I of HOWM Rules – 2016)	Generated during manufacturing of Caffeine in Pharmaceutical industry.	Recovery of Caffeine

80.1 Source of Waste:

Sodium theophylline is reacted with di-methyl sulphate in alkaline condition, after the reaction is completed caffeine crude is generated which undergoes filtration followed by purification. Pure caffeine from purification stage is collected as product. The mother liquor from the said filtration is extracted with chloroform. The resultant extract is caffeine with chloroform which undergoes distillation to separate both components, where caffeine from the bottom of distillation is collected as product and the distillate (i.e. chloroform) is recycled back to the extraction column as a solvent. The other resultant raffinate (Caffeine Liquor Containing Caffeine < 2.5 %) is categorized as Hazardous waste listed at Category: 28.1, Schedule-I of HOWM Rules – 2016.

Table 1. Typical Characteristics of H.W.

Sr. No.	Parameter	Unit	Result
1.	Caffeine content	%	2.43
2.	Sodium sulphate	%	1.8
3.	Methanol	mg/Kg	114.4
4.	Chloroform	mg/Kg	7.33
5.	Arsenic	mg/Kg	BLQ
6.	Lead	mg/Kg	BLQ

80.2 Resource recovery Process

The caffeine recovery process involves extraction of Caffeine Liquor Containing Caffeine < 2.5 % with chloroform. The resultant extract is caffeine with chloroform which undergoes distillation to separate both components, caffeine as bottom product and chloroform as distillate, for recycling in extraction column as a solvent. After caffeine recovery the raffinate (caffeine content < 0.1 %) is treated in Multiple Effective Evaporator (MEE). The condensate from MEE is treated in Reverse Osmosis (RO) plant, permeate from RO plant is used in utility section for cooling tower and ancillary activities. Reject from RO, MEE concentrate is processed in Agitated Thin Film Dryer (ATFD) and salt is sent to CHWTSDF for disposal.

80.3 Product Usage / Utilization

1. The product Caffeine is generally used as an intermediate component in pharmaceutical industry and also utilized in beverage industries.
2. The Product i.e. Caffeine shall comply Bureau of Indian Standards (BIS) IS: 11911 – 1986 or any other notified standards as required for further respective utilization.

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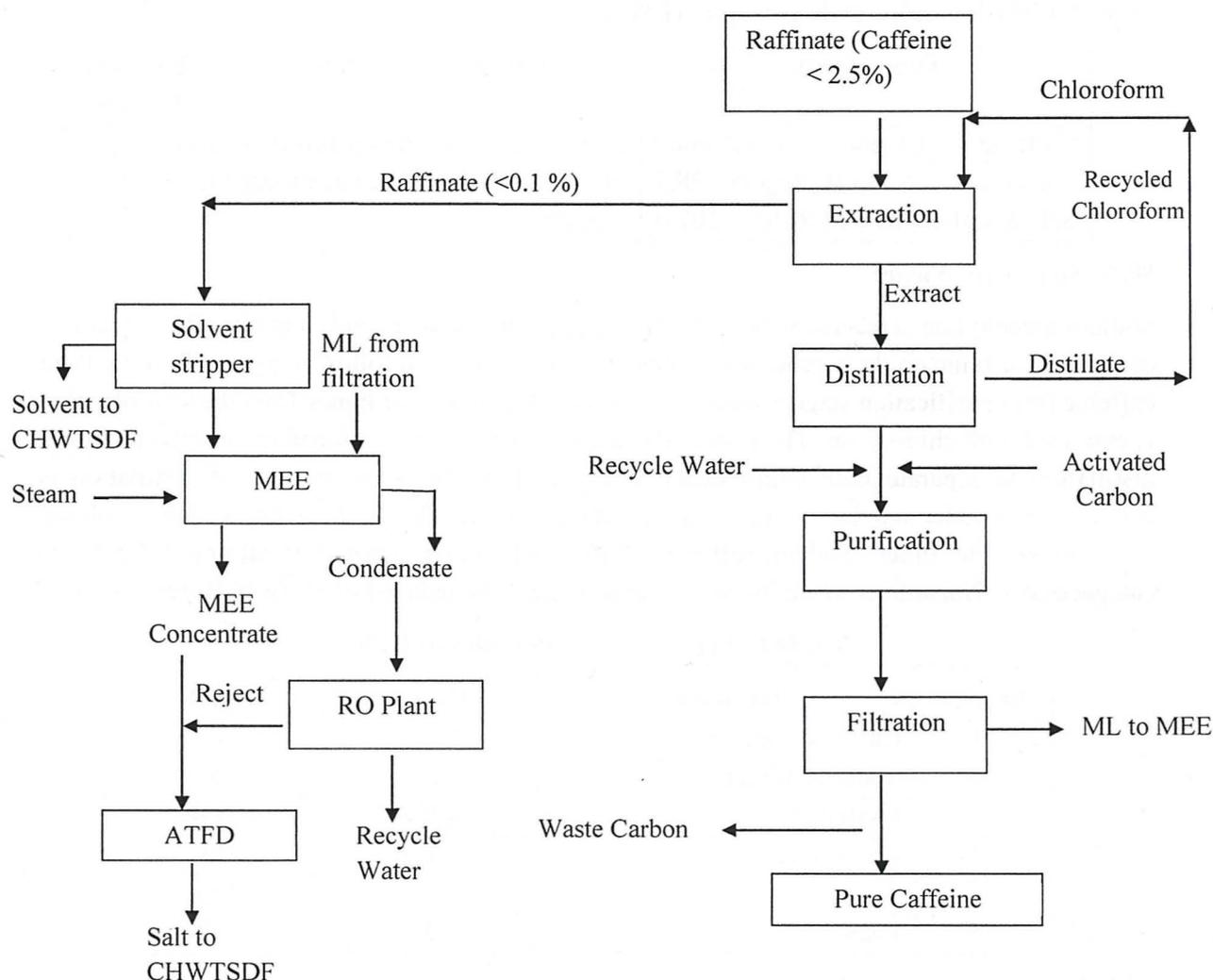


Figure: 1-Process flow diagram for utilization of hazardous waste.

80.4 Standard Operating Procedure for utilization of Caffeine Liquor:

This SoP is applicable only for Recovery of Caffeine from Caffeine Liquor (Containing Caffeine < 2.5 %).

- 1) Caffeine Liquor shall be procured only in SPCB/PCC authorized barrels/closed tanks mounted over vehicles fitted with requisite safeguards.
- 2) Caffeine Liquor shall be stored in dedicated storage tanks on acid proof brick lined area under covered storage shed within premises. Further, storage sheds 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.
- 3) Transfer of Caffeine Liquor from storage tank shall be carried out through dedicated mechanical transfer pump with fixed pipeline.

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- 4) Material transfer / handling in entire recovery process shall be done without manual interventions in closed system.
- 5) The unit shall provide separate storage tanks for storage of fresh chemicals and hazardous waste. The storage tanks should be at designated place with proper cover and with acid brick lining floors.
- 6) 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.
- 7) The unit shall provide Multiple Effective Evaporator (MEE) followed by RO plant and Agitated Thin Film Dryer for handling of raffinate generated during recovery process. The permeate generated from RO plant shall be reused in process or utility & scrubber make up to achieve ZLD.
- 8) Treatment and disposal of wastewater generated from floor-washings, spillages, equipment washing, scrubber bleed, during process (i.e. from MEE or from raffinate distillation) 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, the treated waste water from ETP may be managed as per conditions stipulated by the SPCB / PCC. 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.
- 9) The hazardous wastes generated (solvent generated from solvent stripper (installed before MEE) and salts generated from ATFD) 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.
- 10) The unit shall ensure that the Caffeine Liquor is procured from authorized industries as required under HOWM Rules, 2016.
- 11) Transportation of Caffeine Liquor shall be carried out by sender (generator) or receiver (utilizer) only after obtaining authorization from the concerned SPCB under HOWM Rules, 2016. Requisite manifest document shall be followed as laid down under the said Rules.
- 12) Prior to utilization of Caffeine Liquor, the unit shall obtain authorization for generation, storage and utilization of Caffeine Liquor from the concerned SPCB/PCC under HOWM Rules, 2016.
- 13) In case of environmental damages arising due to improper handling of hazardous wastes including accidental spillage during generation, storage, processing, transportation and

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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.

- 14) The unit shall provide suitable fire safety arrangements and flame proof electrical fittings.
- 15) 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.

80.5 Record>Returns Filing

- 1) The unit shall maintain a passbook issued by concern SPCB/PCC and maintain details of each procurement of Caffeine Liquor 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 Caffeine Liquor, 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.

80.6 Standards

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

PM ₁₀	5 mg/m ³ TWA
Chloroform	240 mg/m ³ Ceiling limit

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

**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) Monitoring of the above specified parameters for Work zone emission shall be carried out quarterly for first year followed by at least annually in the subsequent year of utilization.

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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 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 consent or non-availability of the common Effluent Treatment Plant (CETP), zero discharge shall be met.

80.7 Siting of Industry

Facilities for utilization of Caffeine Liquor shall 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.

80.8 Size of Plant and Efficiency of Utilisation

Approximately 9,550 Kg Caffeine liquor (caffeine content <2.4 %) may recover 220 Kg of Caffeine. Therefore, requisite facilities of adequate size of storage shed and other plant & machineries shall be installed accordingly.

80.9 Checklist of Minimal Requisite Facilities:

Sl. No	Particulars
1.	Dedicated storage tank for storage of Caffeine Liquor with acid proof brick lining and proper slope & seepage collection pit.
2.	Mechanical transfer pumps with fixed pipeline for transportation and handling of Caffeine Liquor.
3.	Suction system followed by Scrubber (APCD) to the tanks where raffinate after caffeine recovery is stored and also other potential spaces for fugitive emissions.
4.	Material transfer / handling in entire recovery process shall be done without manual interventions in closed system.
5.	Multiple Effective Evaporator (MEE) followed by RO plant and Agitated Thin Film Dryer for handling of raffinate generated.
6.	Extractor, Distillation unit, MEE, Solvent stripper, RO unit, ATFD.

