ZDHC Wastewater and Sludge Laboratory Sampling and Analysis Plan

ZDHC

Version 3.0

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Definition terms

Visit the ZDHC Glossary to search for explanations on terminology used across this document and the ZDHC Foundation.

Sampler	An individual appointed by a ZDHC Approved Wastewater Laboratory to perform wastewater sampling activities.
ZDHC Signatory Approved Solution Provider	An organisation that the ZDHC has approved to deliver a specific service in alignment with the relevant ZDHC guidelines or documents. To achieve this status, the organisation must undergo an approval process in the Solution Provider Platform. If the application is successful, the organisation signs a contract and is then authorised to provide the designated service in accordance with the applicable ZDHC requirements. The approval validity is subject to periodic review and renewal.

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Introduction

This ZDHC Wastewater and Sludge Laboratory Sampling and Analysis Plan (hereafter referred to

as "SAP") is one of the key documents supporting the implementation of the ZDHC Wastewater and Sludge Guidelines V2. It does so by standardising procedures for laboratories to conduct sampling and analysis across the ZDHC Wastewater and Sludge Guidelines (which encompasses the textile and leather industries), ZDHC MMCF Guidelines V3.0, ZDHC Dissolved Pulp Guidelines V1.0 and ZDHC Recycled Polyester Guidelines V1.0.

The SAP provides a comprehensive framework for laboratories, including clear instructions for samplers and laboratory technicians to carry out wastewater and sludge sampling, analysis and reporting. The document includes key details such as the required types of containers, sampling procedures and information that must be included in the test report. Only ZDHC Approved Laboratories can perform testing and report results in the ZDHC Gateway. Only ZDHC qualified samplers may collect wastewater and sludge samples.

For reference, although the whole document should be read, each section of the SAP indicates the most relevant user involved in that specific content. Where no specific user is mentioned, all individuals responsible for that activity are considered equally concerned.

Please note that this does not exempt any involved parties from reading the entire document.

Data use

Wastewater and sludge data helps to promote the implementation of sustainable chemistry and best practices in the industry. It can be used for critical decision making and strategic direction for manufacturing facilities. The laboratory must maintain the integrity of test data generated under the ZDHC Roadmap to Zero Programme.

Revision history

In the spirit of continuous improvement, the SAP will be reviewed and revised as needed to incorporate learnings and opportunities identified during the practical application and implementation of these procedures. This version has gone through significant changes, listed below.

Table 1: Revision History of the ZDHC Wastewater and Sludge Laboratory Sampling and Analysis Plan

Version Number	Changes	Publication Date
Version 2	• The definition of pretreatment was included. • As mentioned in sampling specific to wastewater, adjusted content in wastewater collection. • As mentioned in sampling specific to the sludge adjusted content in sludge collection. • As mentioned in the standard test methods requirement for sludge adjusted content	October 2025
Version 2.1	List changes • Added single grab sampling for homogenisation tanks. • Adjusted content in the standard test method requirements for wastewater and sludge. • Added test report requirements. • Added appendices D and E.	November 2022
Version 2.0	• Added information for the parameters added with ZDHC MRSL V2.0. • Restructured content to avoid repeating information. • Added information from the sludge task team. • Added information from the ZDHC Wastewater Council. • Added information from the MMCF task team. • Added information from the Leather task team. • Changed from the EPA MDL and RL terminology to the ISO LOD and LOQ terminologies. • Change guidance for collecting liquid sludge samples.	November 2021
Version 1.3	• Added sampling and analysis of conventional parameters specific to the ZDHC Leather Wastewater Guidelines Addendum. This includes: faecal coliform, chloride, sulfate and total dissolved solids. • Added reference to, and content from, the ZDHC Wastewater and Sludge Sampling Procedure/Training. • Added guidance for composite wastewater sampling at facilities that do not have continuous production cycles. • Emphasised that the ZDHC specified methods for colour and chemical oxygen demand (COD) must be used without exception. • Changed sample storage temperature from	January 2021

	<4°C to the ISO recommended temperature of 2° to 8°C.	
Version 1.2	• Added sampling and analysis of parameters specific to the ZDHC MMCF Wastewater Interim Guidelines.	April 2020
Version 1.1	• Added sampling and analysis procedures specific to sludge. • Improved sampling and analysis procedures for wastewater. • Ensured full alignment with ZDHC Wastewater Guidelines Version 1.1 requirements. Including, but not limited to, standard methods for analysis. • Changed the expected sample temperature and applied it to all relevant sections throughout the wastewater section of the document. • Changed the recommended holding time for halogenated solvents. • Adjusted the target value for multi-point calibration. • Adjusted the calibration check for total coliform. • Updated and clarified the reporting and deliverable requirements in section 3.3 to align with the latest developments within ZDHC.	June 2019
Version 1.0	• Initial publication of the ZDHC Wastewater Laboratory Sampling and Analysis Plan.	June 2019

Related work

This document is part of a set of guidelines and solutions provided by ZDHC. All stakeholders (manufacturing facilities, brands/retailers, and laboratories) are expected to follow the most current guidance documents and practical tools listed below:

- ZDHC Wastewater and Sludge Guidelines
- ZDHC Root Cause Analysis and Corrective Action Plan Template
- ZDHC List of ZDHC Approved Laboratories for ZDHC Wastewater and Sludge Guidelines Testing
- ZDHC MMCF Guidelines

- ZDHC MMCF Guidelines Industry Standard Implementation Approach
- ZDHC Root Cause Analysis and Corrective Action Plan Template for MMCF Facilities
- ZDHC Dissolved Pulp Guidelines
- ZDHC Dissolved Pulp Guidelines Industry Standard Implementation Approach
- ZDHC Recycled Polyester Guidelines
- ZDHC Recycled Polyester Guidelines Industry Standard Implementation Approach
- ZDHC Manufacturing Restricted Substances List (ZDHC MRSL)

Other reference materials to be considered in conjunction with this document, not pertaining to ZDHC are:

- USEPA 833-B-89-100: POTW Sludge Sampling and Analysis Guidance Document, United States Environmental Protection Agency 1989.
- ISO 5667- 13: Guidance on Sampling Sludge.
- ISO 5667- 10: Guidance on Sampling of Wastewater

Sample collection (mainly for samplers)

ZDHC has developed a sampling procedure and sampler training accessible through the ZDHC Academy. Its purpose is to promote consistent sampling and collection of field data. Samplers must complete the training and successfully pass a test at the end. The academy will assign a sampler accreditation number.

All samplers that collect ZDHC samples must have the ZDHC accreditation sampler ID number. A reference checklist of materials needed for sampling can be found in Appendix D.

The use of the On-Site App is mandatory for sampling activities.

Definition of pretreatment

Pretreatment is a process where a facility reduces, alters or eliminates pollutants in wastewater discharge prior to release of the water to the CETP. Some of the pretreatment examples include, but are not limited to, pH adjustment, filtration, biological treatment of the wastewater and other physical/chemical processes. The pretreatment process may or may not generate sludge.

The processes below, when installed as a standalone system, cannot be considered as pretreatment:

- Grit removal with the mesh size >6mm
- Equalisation tank/homogenisation tank (if no chemicals are added in the equalisation tank/homogenisation)

The processes below, when installed as a standalone system, to be considered as pretreatment:

- Grit removal with the mesh size <6mm
- Equalisation tank/homogenisation tank (if chemicals are added in the equalisation tank/homogenisation)

Sample locations

The Wastewater and Sludge Guidelines (WSG) provides the sampling matrix, shown here in Tables 2, 2a and 2b. The WSG acknowledges the difference between facilities with direct, indirect, zero liquid discharge and those with an 'average total industrial wastewater generated, that is equal to or more than 15 m^3/day'. The WSG apply to industrial wastewater. Where a supplier combines their industrial wastewater with domestic wastewater, the resulting combined wastewater is classified as industrial wastewater, to which the guidelines apply. Refer to the Wastewater and Sludge Guidelines for more information.

Table 2- Testing Requirements for Suppliers that Generate on Average, Equal To, or More than 15m^3 of Industrial Wastewater per Day

Test Parameters and Sample Locations/ Discharge Type	**ZDHC MRSL (a) (d)** Sample untreated wastewater and test MRSL parameters	**ZDHC Heavy Metals (d)** Sample discharged wastewater/ effluent	**ZDHC Conventional Parameters and Anions (d)** sample discharged wastewater/ effluent	**ZDHC Sludge (d)** Sample sludge and test as per the major sludge disposal pathway
Direct	Yes	Yes	Yes	Yes
Indirect with pretreatment (with sludge)	Yes	Yes	No	Yes
Indirect with pretreatment (without sludge)	Yes	Yes	No	No
Indirect without pretreatment	Yes (c)	Yes (b)	No	No
Zero liquid discharge (ZLD)	Yes	No	No	Yes

a= Excluding ZDHC Heavy Metals
b= For the facilities with indirect without pretreatment discharge type ZDHC MRSL substances as well as ZDHC heavy metals (arsenic, cadmium, chromium (VI), lead, mercury) as explained in the above table should be sampled and tested only at untreated wastewater as there is no pretreatment involved.
c= Composite sampling is a must.
d= RCA/CAP must in case of any detection.

Table 2a - MMCF Facilities Specific Wastewater Testing

Test Parameters and Sample Locations/ Discharge Type	**ZDHC Heavy Metals (d)** sample discharged wastewater/effluent	**ZDHC Conventional Parameters and Anions (d)** sample discharged wastewater/effluent	**ZDHC Sludge (d)** Sample sludge and test as per the major sludge disposal pathway
Direct	Yes	Yes	Yes

Indirect with pretreatment (with sludge)	Yes	No	Yes
Indirect with pretreatment (without sludge)	Yes	No	No
Indirect without pretreatment	Yes (b)	No	No
Zero liquid discharge (ZLD)	No	No	Yes

b= For the facilities with indirect, without pretreatment discharge type ZDHC heavy metals (copper, lead, nickel, zinc, total chromium) should be sampled and tested only at untreated wastewater as there is no pretreatment involved.
d= RCA/CAP must in case of any detection.

(Wastewater testing requirements for MMCF facilities do not depend on the volume of wastewater generated. Even if generated wastewater is less than 15m^3 per day, all parameters mentioned in the MMCF Guidelines apply to a MMCF facility.)

Table 2b: Testing Requirements for Suppliers that Generate less than 15m^3 of Industrial Wastewater per Day

Test Parameters and Sample Locations/ Discharge Type	**ZDHC MRSL (a)** Sample untreated wastewater and test MRSL parameters	**ZDHC Heavy Metals** Sample discharged wastewater/ effluent	**ZDHC Conventional Parameters and Anions** Sample discharged wastewater/effluent	**ZDHC Sludge** Sample and test as per the major sludge disposal pathway
Direct	No	Yes	Yes	No
Indirect with pretreatment (with sludge)	No	No	No	No
Indirect with pretreatment (without sludge)	No	No	No	No

Indirect without pretreatment	No	No	No	No
Zero Liquid Discharge (ZLD)	No	No	No	No

a= Excluding ZDHC heavy metals

Sampling specific to wastewater

ZDHC wastewater parameters

The wastewater parameters for testing are listed in the ZDHC Wastewater and Sludge Guidelines (for textiles, leather), ZDHC Man-Made Cellulosic Fibres Guidelines, ZDHC Dissolved Pulp Guidelines and ZDHC Recycled Polyester Guidelines.

Wastewater sample collection (mainly for samplers)

1. Samples shall only be taken by trained, qualified and approved samplers. Laboratories shall nominate their samplers to undertake the training using the Solution Provider Platform.

2. Wastewater samples shall be collected as composite samples following ISO 5667 - 10 guidelines: "Guidance on Sampling of Wastewater".

3. Sampling using calibrated and refrigerated autosamplers is preferred. To ensure representative samples, composite sampling must be performed for no less than six (6) hours, or a length of time that represents the entire production process cycle (which could be more or less than six (6) hours), with no more than one (1) hour between discrete samples. Each discrete sample must be of equal volume. The composite sample container must be cooled during sampling.*

* Longer composite sampling may be required to satisfy regulatory requirements. More information on temperature measurements can be found in Table 3 in the Wastewater and Sludge Guidelines.

4. Supervision of Automatic Samplers: When an automatic sampler is used (e.g., set up by the laboratory technician to collect multiple aliquots over a defined period), the

sampling process must always be supervised. Lack of supervision may result in insufficient control over the sampling conditions (e.g., dilution of wastewater with clean water, discontinuous discharges, or other irregularities).
Therefore, the technician is required to remain on site for the entire duration of the sampling activity to ensure proper oversight and reliability of the sampling process.

5. If necessary, samplers from a ZDHC Approved Solution Provider can collect discrete samples by hand for no less than six (6) hours, with no more than one (1) hour between discrete samples. Each discrete sample shall be of equal volume and combined to produce one composite sample. The composite sample container must be cooled during sampling.

6. All of the sample containers will be filled from the same bulk composite sample. A minimum sample volume of 20 litres is required in order to fill all of the sample collection containers. Regarding the volume needed for the analysis, each laboratory shall ensure that the volume of water shipped will allow for duplicate tests and quality control. The sampler shall co-ordinate the exact volume for testing with their laboratory.

7. Samples must be taken during a time representing continuous and normal production and continuous and normal wastewater treatment. Collect wastewater samples in a way that represents the entire production cycle. As part of a document control purpose, facility management needs to confirm the fact that sample collection has been done during normal factory operation. The form could be found in the template under Appendix D.

NOTE: The ZDHC Approved Wastewater Laboratory will request a 10 day (including weekends, but excluding public holidays) production window from the facility. Upon receiving this request, the facility is required to specify the specific days within that window during which production activities will occur at its peak. This timeframe will then be designated as the sampling window. During the sampling window, the laboratory reserves the right to perform sampling on any production day through the sampler from a ZDHC Approved Wastewater Laboratory, without specifying the exact date in advance. This process follows a semi-announced sampling approach, ensuring both operational feasibility and the integrity of the sampling process are maintained. The facility will be notified that a sampler from a ZDHC Approved Wastewater Laboratory may visit at any point during the declared production days, and should be prepared to accommodate and extend the fullest co-operation to the sampler for sampling purposes.

8. Start the production cycle wastewater sampling after a time that compensates for the lag time in the effluent treatment system (ETP). For example, if the plant starts

production at 8:00 AM and the lag time in the ETP is 3 hours, then ETP wastewater discharge sampling will start at 11:00 AM.

9. In situations where there is a homogenisation tank in which the average holding time is greater than 12 hrs, a single sample collected from such a tank can be treated as a composite sample. However, the volume of wastewater in a homogenisation tank must represent the entire production process/cycle.

The holding time (or HRT) is calculated as:
Holding time (HRT) = Volume of the tank (m^3) / flow rate (m^3/hr)

Flow rate can be calculated as:
Flow rate (m^3/hr) = Daily flowrate (m^3/hr) / 24

10. Samples must not be taken if the wastewater is diluted.

11. For a direct discharge facility, the following parameters must be measured every hour during the composite sampling: pH, temperature difference or temperature absolute, persistent foam, wastewater flow rate, total chlorine and dissolved oxygen. The hourly measurements will be reported with the field data report form, and the average will be reported with the ZDHC Gateway Electronic Data Reporting (EDR) template.

12. Wastewater flow rate (m^3/day) must be collected and reported with the laboratory test results using the EDR template. Collect flow rate data seven times over a six hour period to get the hourly average. Then, multiply by 24 (hours), or the length of time the plant operated daily, to get m^3/day.

13. Appendix D provides access to the required ZDHC Wastewater Sampling Field Data Form and Representative Sample Declaration.

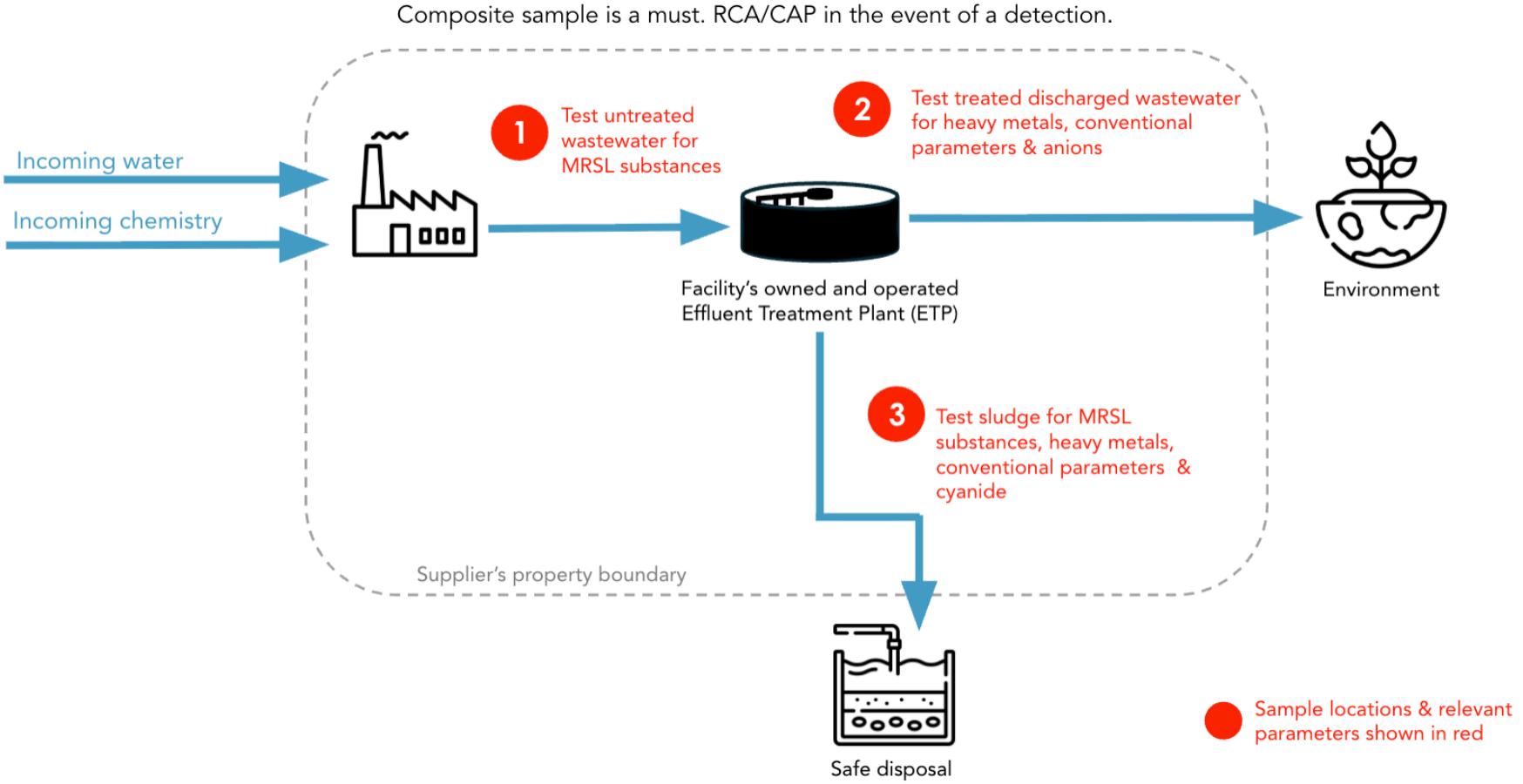
14. If a supplier has multiple discharge locations (discharged wastewater/effluent) for industrial wastewater, samples must be taken and analysed for each discharge location.

15. Samples from multiple sample locations/addresses must be tested separately and not blended together. Each facility with a separate address needs an individual AID on the ZDHC Gateway to report their wastewater and sludge data and generate a ClearStream Report individually.

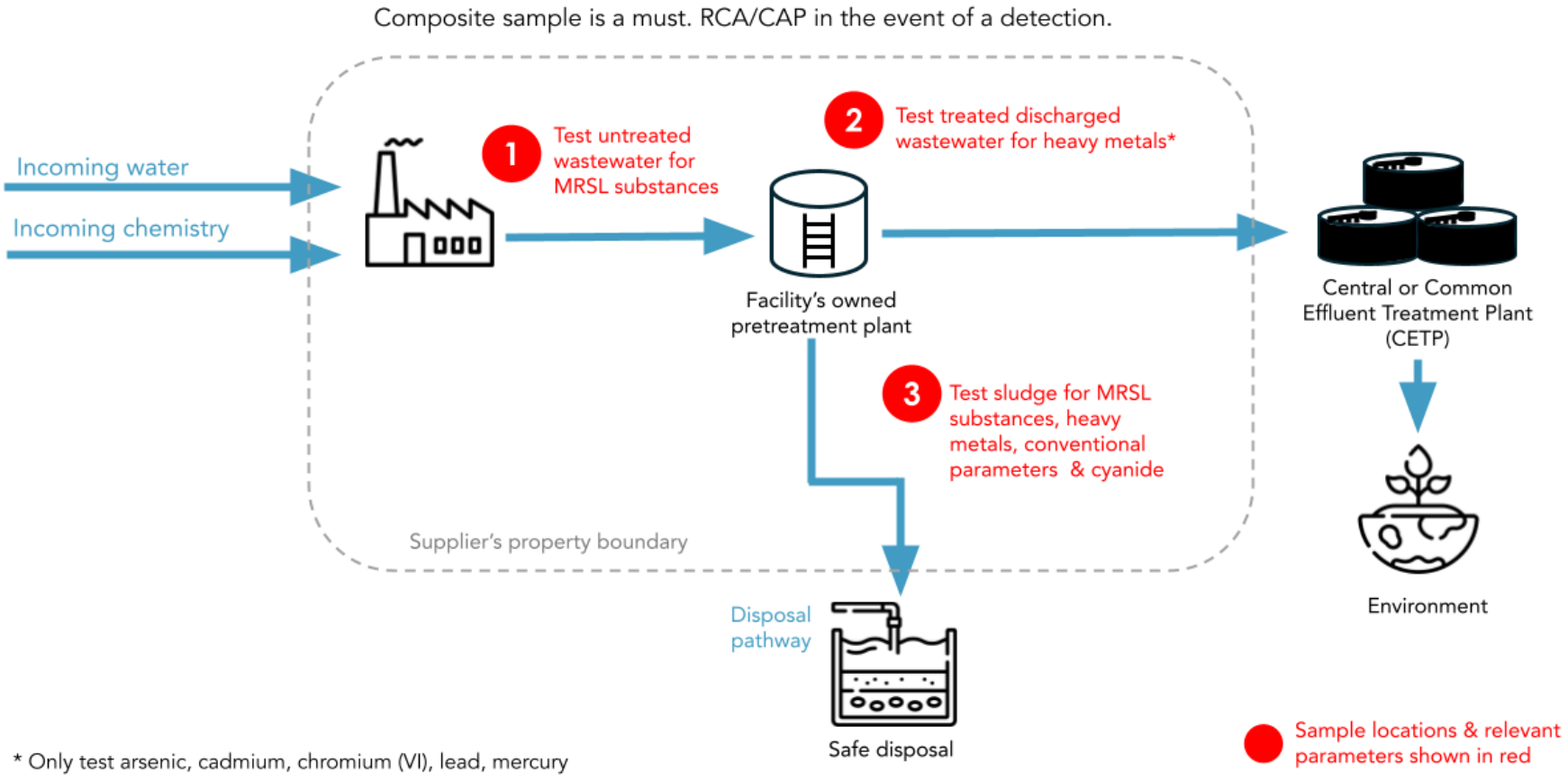
16. Suppliers must meet ZDHC's definition of zero liquid discharge (ZLD) treatment system.

17. Specific requirements for sampling ZDHC MRSL, conventional parameters, anions and metals (refer to Wastewater and Sludge Guidelines).
    - Sampling of untreated wastewater:
        - Sampling shall occur at the equalisation or homogenisation tank, where the wastewater generated across the facility is collected and homogenised or equalised.
    - Sampling of discharged wastewater (effluent):
        - Sampling shall occur at a point closest to the location where the industrial wastewater leaves the property boundaries of the supplier.

Direct Discharge

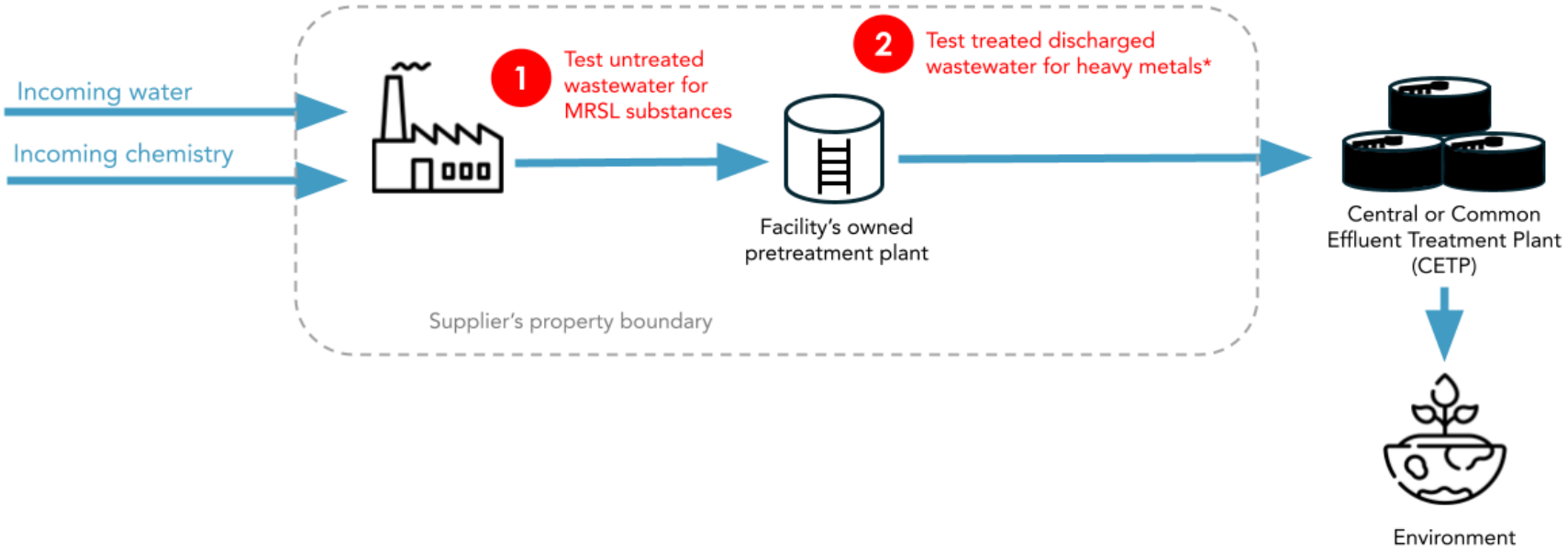


Indirect Discharge with Pretreatment (with sludge)



Indirect Discharge with Pretreatment (without sludge)

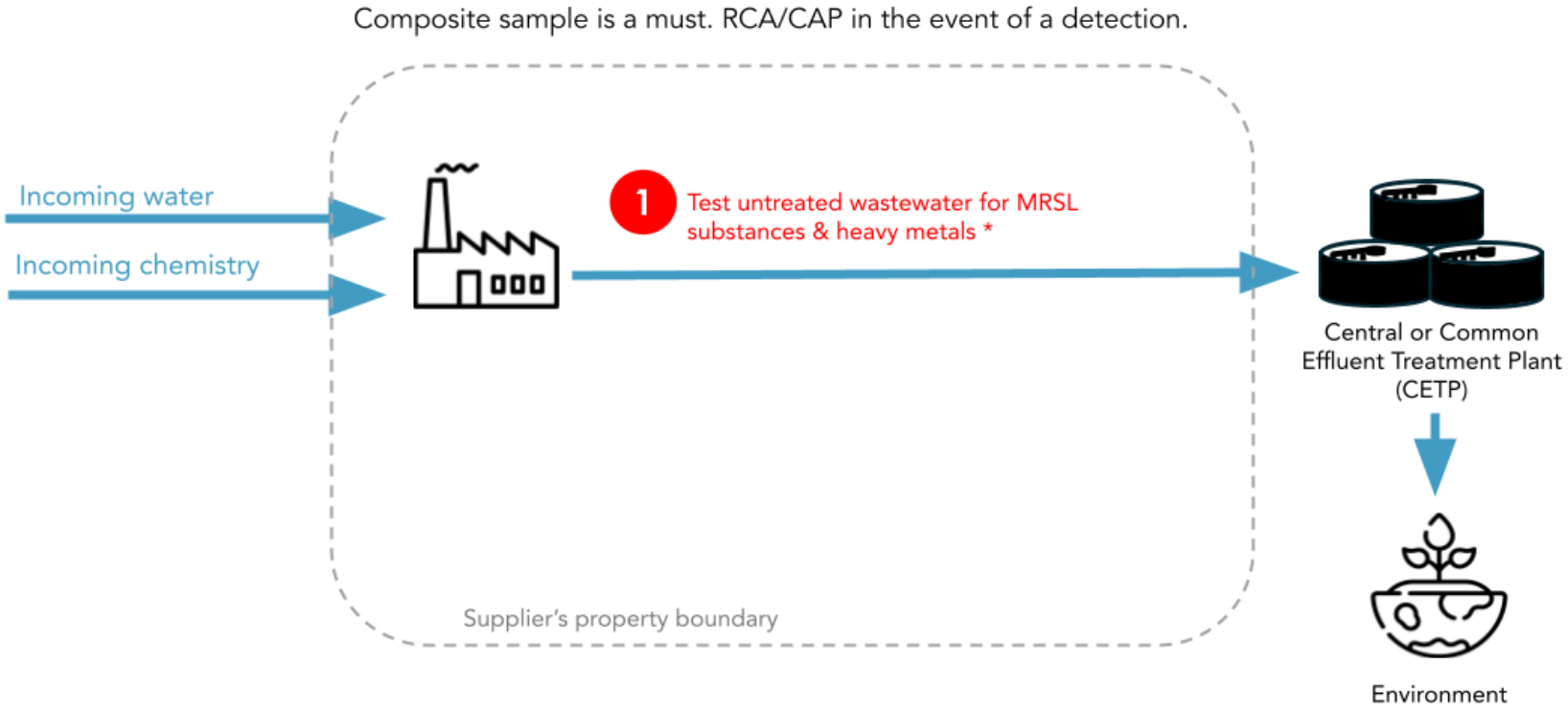
Composite sample is a must. RCA/CAP in the event of a detection.



* Only test arsenic, cadmium, chromium (VI), lead, mercury

Sample locations & relevant parameters shown in red

Indirect Discharge without Pretreatment

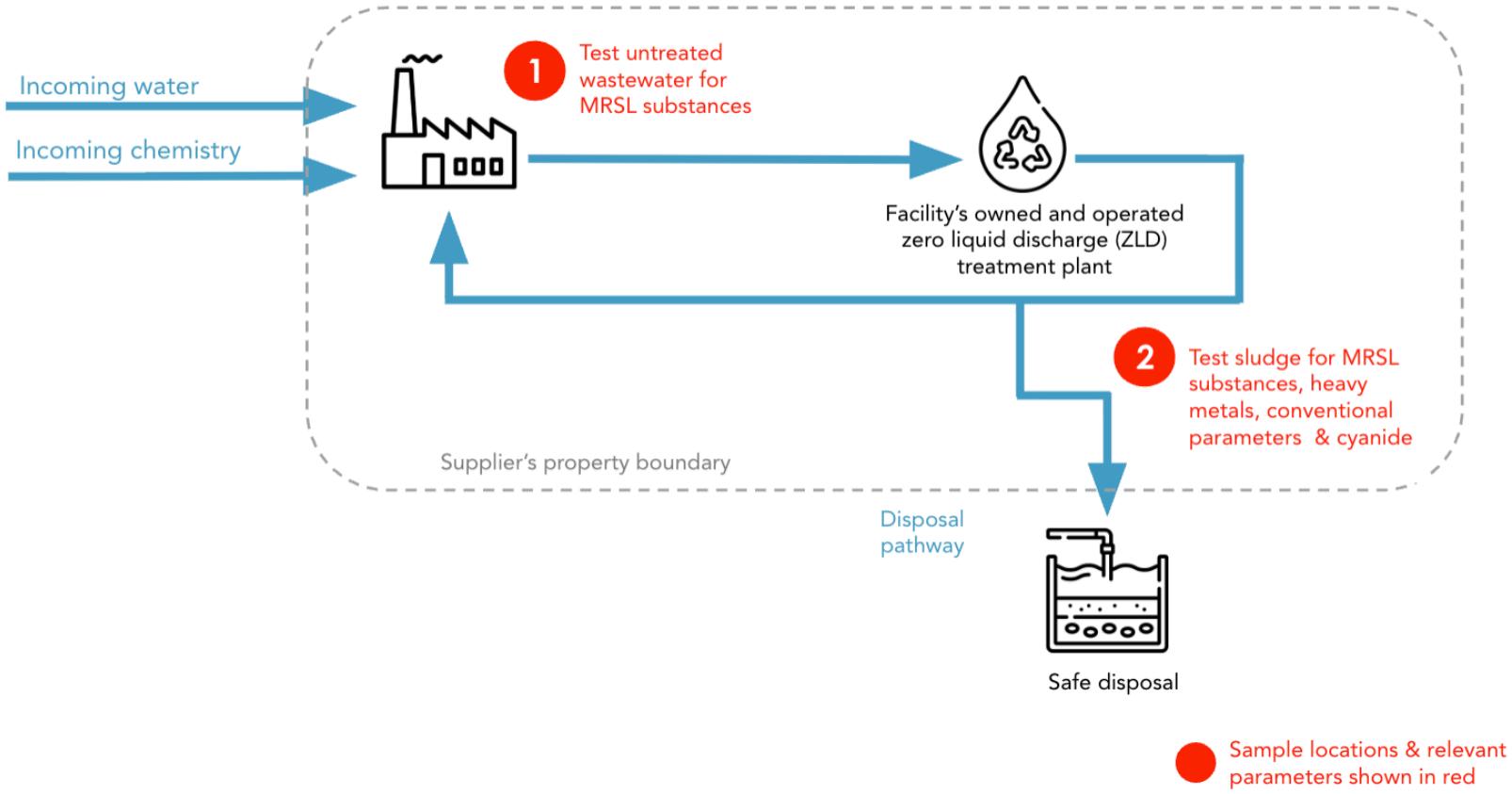


* Only test arsenic, cadmium, chromium (VI), lead, mercury

Sample locations & relevant parameters shown in red

Zero liquid discharge (ZLD)

Composite sample is a must. RCA/CAP in the event of a detection.



- Testing for persistent foam:
  - Foam is a naturally occurring phenomenon in aeration basins in which biological wastewater treatment occurs. Samplers should include photographs of the foam they witnessed in the final lab report, along with the time and date of taking such photos. The foam colour should be similar to the liquid in the aeration basin, should dissipate quickly and should be contained within the aeration basin. If the foam is higher than 45 centimetres (by visual estimation) then it could result in permanent foam being discharged onto the surface of receiving waters and should be noted. For direct discharge facilities, samplers should check for persistent foam on the surface of receiving waters at the point of discharge and the presence or absence of foam should be noted. This should be checked at the same location used for sampling the temperature difference. This test is to be done on-site by the sampler and should be checked at the same location used for ΔT* sample checks. In case the receiving body is not accessible or risky to access for the sampler, a visual estimation of the foam in the aeration basin should be done. If the foam is higher than 45 centimetres in height (by visual estimation), then it could result in permanent foam being discharged onto the surface of receiving waters and should be reported as 'fail' for the foam parameter.

* ΔT is the difference between the temperature of the receiving body and the effluent discharged by the facility.

- Testing for temperature difference:
  - Testing for temperature difference is only applicable to direct discharge facilities. Take the temperature of the discharged wastewater and the receiving body. Subtract the temperature of the receiving body from the temperature of the discharge to give the delta temperature difference, which can be a positive or a negative value. The Wastewater and Sludge Guidelines discharge limits only refer to a positive value, which produces an overall increase in the temperature of the receiving body of water.
- Testing for temperature absolute:
  - Testing for temperature absolute is only applicable to direct discharge facilities and is to be tested only if the following situations apply:
    - The receiving body may be several kilometres away from the point of discharge and the facility is discharging the effluent into the receiving body through a pipeline.
    - Accessing the location of the receiving body to measure its temperature can be risky in terms of injury to the sampler or damage to equipment.
    - The effluent is discharged directly into the ground.

- In all such cases where access to the receiving body is not possible or unsafe, the laboratory should report "temperature absolute" at the location at which discharged wastewater (effluent) exits a facility's boundary and is released into an external environment or designated channel.

Wastewater sample containers and preservatives (mainly for samplers)

1. Table 3 presents standard sample collection containers and preservatives for the wastewater parameters specified in the Wastewater and Sludge Guidelines.

2. The appropriate sample collection container and preservative can vary depending on the standard test method used. Therefore, verify the proper container and preservative with the test method used at the laboratory.

3. A temperature indicator bottle should be included with each shipping container. The temperature indicator bottle will be clearly labelled. If the sample shipping time is short, such as across town, then collect a sub-sample from one of the sample containers and measure and report that as the temperature received at the lab.

4a. Field blanks shall be collected for the following parameters:
- Total phosphorus
- Mercury
- Halogenated solvents / Volatile Organic Compounds (VOC)
- Perfluorinated and polyfluorinated chemicals (PFCs)

4b. The field blanks shall use the same containers as the samples and will be filled with reagent grade laboratory water (ASTM D1193 or ISO 3696 water grade 1).

Table 3: Wastewater Sample Containers and Preservatives

Wastewater Parameter	**Sample Container and Minimum Size**	**Standard Preservative** (verify with lab method to be used)
Shipping temperature of indicator bottle for all test parameters	calibrated thermometer to +/- 1C°	room temperature water
ZDHC Conventional including Anions		

Ammonia-ntrogen	P,G,FP 500-ml	H2SO4 < pH 2 keep cool - between 2˚C and 8˚C
AOX	P,G,FP 500-ml	HNO3 pH 1-2, keep cool - between 2˚C and 8˚C
Biochemical Oxygen Demand 5-days concentration (BOD5)	P,G,FP 1,000-ml	keep cool - between 2˚C and 8˚C
Chemical Oxygen Demand (COD)	P,G,FP 100-ml	H2SO4 < pH 2 keep cool - between 2˚C and 8˚ C
Chloride	P,G,FP 100-ml	keep cool - between 2˚C and 8˚C
Colour [m-1] (436nm; 525nm; 620nm)	P,G,FP 500-ml	keep cool - between 2˚C and 8˚C
Cyanide	P, FP 1,000-ml	NaOH > pH12 0.1 ml of 10% sodium thiosulfate keep cool - between 2˚C and 8˚C
Dissolved Oxygen (DO)	NA	measured in the field
E.coli	P,G clean, sterile, non-reactive, 125-ml	0.1 ml of 10% sodium thiosulfate keep in the dark and cool - between 2˚C and 8˚C
Oil and grease	glass, wide mouth PTFE lined lid 1,000-ml	HCl or H2SO4 < pH 2 keep cool - between 2˚C and 8˚C
Persistent foam	NA	measured in the field
pH	NA	measured in the Field
Phenol	P,G PTFE lined lid 500-ml	H2SO4 < pH 2 keep cool - between 2˚C and 8˚C
Temperature difference	NA	measured in the field
Temperature absolute	NA	measured in the field
Total chlorine	NA	measured in the field
Total Dissolved Solids (TDS)	P,G 200-ml	keep cool - between 2˚C and 8˚C
Total nitrogen	P,G,FP 100-ml	H2SO4 < pH 2 keep cool - between 2˚C and 8˚C

Total phosphorus	P,G,FP 100-ml	H2SO4 < pH 2 keep cool - between 2˚C and 8˚C
Total Suspended Solids (TSS)	P,G 200-ml	keep cool - between 2˚C and 8˚C
Sulfate	P,G,FP 100-ml	keep cool - between 2˚C and 8˚C
Sulfide	P, FP 100-ml	4 drops 2N zinc acetate NaOH > pH 9 keep cool - between 2˚C and 8˚C
Sulfite	P,G,FP 100-ml	1-ml 2.5% EDTA keep cool - between 2˚C and 8˚C
ZDHC Heavy Metals		
Chromium (VI)	G acid washed 40-ml Brown Glass VOA vial	0.45µm filter in field, add buffer * to pH 9.0-9.5 keep cool - between 2˚C and 8˚C
Heavy metals (antimony, arsenic, barium, cadmium, chromium-total, cobalt, copper, lead, nickel, selenium, silver, tin, zinc)	P, G, FP acid washed 250-ml	HNO3 < pH 2 keep cool - between 2˚C and 8˚C
Mercury	P, G, FP acid washed 500-ml	HNO3 < pH 2 keep cool - between 2˚C and 8˚C
ZDHC MRSL		
Alkylphenol (AP) and alkylphenol ethoxylates (APEOs): including all isomers	G 1,000-ml FP lined lid	keep cool - between 2˚C and 8˚C
Anti-microbials & biocides	G 1,000-ml FP lined lid	keep cool - between 2˚C and 8˚C
Chlorinated paraffins	G 1,000-ml FP lined lid	keep cool - between 2˚C and 8˚C
Chlorobenzenes and chlorotoluenes	G 1,000-ml FP lined lid	keep cool - between 2˚C and 8˚C
Chlorophenols	G 1,000-ml FP lined lid	keep cool - between 2˚C and 8˚C
DMFa	G 1,000-ml FP lined lid	keep cool - between 2˚C and 8˚C
Restricted aromatic amines (cleavable from azo-colourants)	G 1,000-ml FP lined lid	keep cool - between 2˚C and 8˚C

Dyes – Carcinogenic or equivalent concern	G 1,000-ml FP lined lid	keep cool - between 2˚C and 8˚C
Dyes – Disperse (sensitising)	G 1,000-ml FP lined lid	keep cool - between 2˚C and 8˚C
Dyes – Navy blue colourant	G 1,000-ml FP lined lid	keep cool - between 2˚C and 8˚C
Flame retardants	G 1,000-ml FP lined lid	keep cool - between 2˚C and 8˚C
Glycols /glycol ethers	G 1,000-ml FP lined lid	keep cool - between 2˚C and 8˚C
Halogenated solvents	three x 40-ml amber VOA vial no headspace	HCl < pH 2 keep cool - between 2˚C and 8˚C
Organotin compounds	G 1,000-ml acid washed FP lined lid	keep cool - between 2˚C and 8˚C
Other/miscellaneous chemicals	G 1,000-ml FP lined lid	keep cool - between 2˚C and 8˚C
Perfluorinated and polyfluorinated chemicals (PFCs)	P 1,000-ml no FP lined lid	keep cool - between 2˚C and 8˚C
Phthalates – including all other esters of ortho-phthalic acid	G 1,000-ml FP lined lid for each parameter (2 needed)	keep cool - between 2˚C and 8˚C
Polycyclic Aromatic Hydrocarbons (PAHs)	G 1,000-ml FP lined lid for each parameter (2 needed)	keep cool - between 2˚C and 8˚C
UV absorbers	G 100-ml FP lined lid	keep cool - between 2˚C and 8˚C
Volatile Organic Compounds (VOC)	three x 40-ml amber VOA vial no headspace	HCl or H2SO4 or HNO3 < pH 2 keep cool - between 2˚C and 8˚C

(P= plastic, G= amber glass, FP= fluoropolymer)

* Buffer = EPA Method 218.6. Dissolve 33g of ammonium sulphate in 75-ml of ASTM D1103 Type 1 or ISO 3696 Type 1 water, add 6.5-ml of sp.gr.ammonium hydroxide. Dilute to 100-ml with ASTM D1103 Type-1 or ISO 3696 Type 1 water.

Table 3a: Wastewater Sample Containers and Preservatives for MMCF facilities

Wastewater Parameter	Sample Container and Minimum Size	Standard Preservative (verify with lab method to be used)
Shipping temperature of indicator bottle for all test parameters	calibrated thermometer to +/- 1C˚	room temperature water

ZDHC Conventional including Anions		
Ammonia-nitrogen	P,G,FP 500-ml	H2SO4 < pH 2 keep cool - between 2˚C and 8˚C
AOX	P,G,FP 500-ml	HNO3 pH 1-2, keep cool - between 2˚C and 8˚C
Biochemical Oxygen Demand 5-days concentration (BOD5)	P,G,FP 1,000-ml	keep cool - between 2˚C and 8˚C
Chemical Oxygen Demand (COD)	P,G,FP 100-ml	H2SO4 < pH 2 keep cool - between 2˚C and 8˚ C
Colour [m-1] (436nm; 525nm; 620nm)	P,G,FP 500-ml	keep cool - between 2˚C and 8˚C
Oil and grease	Glass, wide mouth PTFE lined lid 1,000-ml	HCl or H2SO4 < pH 2 keep cool - between 2˚C and 8˚C
Persistent foam	NA	Measured in the field
pH	NA	Measured in the Field
Phenol	P,G PTFE lined lid 500-ml	H2SO4 < pH 2 keep cool - between 2˚C and 8˚C
Temperature difference	NA	Measured in the field
Temperature absolute	NA	Measured in the field
Total nitrogen	P,G,FP 100-ml	H2SO4 < pH 2 keep cool - between 2˚C and 8˚C
Total phosphorus	P,G,FP 100-ml	H2SO4 < pH 2 keep cool - between 2˚C and 8˚C
Total Suspended Solids (TSS)	P,G 200-ml	keep cool - between 2˚C and 8˚C
Sulfate	P,G,FP 100-ml	keep cool - between 2˚C and 8˚C
Sulfide	P, FP 100-ml	4 drops 2N zinc acetate NaOH > pH 9 keep cool - between 2˚C and 8˚C
ZDHC Heavy Metals		
Heavy metals (chromium-total, copper, lead, nickel, zinc)	P, G, FP acid washed 250-ml	HNO3 < pH 2 keep cool - between 2˚C and 8˚C

(P= plastic, G= amber glass, FP= fluoropolymer)4

* Buffer = EPA Method 218.6. Dissolve 33g of ammonium sulphate in 75-ml of ASTM D1103 Type 1 or ISO 3696 Type 1 water, add 6.5-ml of sp.gr.ammonium hydroxide. Dilute to 100-ml with ASTM D1103 Type-1 or ISO 3696 Type 1 water.

Laboratory required reporting limits and standard methods for analysis and testing wastewater (mainly for laboratory managers)

The required test methods and reporting limits are presented in the ZDHC Wastewater and Sludge Guidelines (for textiles, leather) and ZDHC MMCF Guidelines (for MMCF).

Standard test methods for requirements for wastewater (mainly for laboratory managers)

1. The approved laboratory analytical methods for each wastewater parameter are based on requirements in the European Union, the United States of America, India and China and are presented in the ZDHC Wastewater and Sludge Guidelines and MMCF, Dissolved Pulp and Recycled Polyester Guidelines. The analytical methods were selected in collaboration with the ZDHC Wastewater Council.

2. An"equivalent" analytical method can be used, such as those required by the regulatory agency in the region where the wet processing/manufacturing occurs. Equivalent methods must be pre-approved by ZDHC and/or the ZDHC Wastewater Council. The pre-approval process is conducted so that data from one lab can be compared directly and equivalently to any other lab in the ZDHC Roadmap to Zero Programme.

3. Methods specified by ZDHC for colour and Chemical Oxygen Demand (COD) must be used without exception.

4. For some parameters, validated cuvette methods can be used alternatively as indicated in the Wastewater and Sludge Guidelines.

Sampling specific to sludge

ZDHC sludge parameters

Sludge parameters are listed in the ZDHC Wastewater and Sludge Guidelines and MMCF Guidelines.

Sludge sample locations (mainly for samplers)

1. Refer to the chapter Wastewater and Sludge Guidelines in the Suppliers Roadmap to Zero to understand the main purpose of testing sludge and how to classify the sludge disposal pathways.

2. The suppliers must identify to qualified sampling personnel the locations where sludge is generated and stored at the supplier facility. The qualified sampling personnel will determine the proper sampling locations as per the guidance provided to them.

3. Collected sludge samples must be representative of the chosen ZDHC disposal pathways.

4. It may be necessary to collect various types of sludge samples throughout a supplier's ETP. Sludge types can include:
    a. Precipitation/flocculation/coagulation to remove organics
    b. Anaerobically digested secondary treated sludge
    c. Aerobically digested secondary treated sludge
    d. Primary treated sludge
    e. Biomass, secondary treated and tertiary treated sludge
    f. Mechanically dewatered sludge
    g. Dried sludge
    h. Sludge generated from evaporation processes
    i. Ash from the incineration of any type of waste at an on-site incinerator
    j. Residue collected from the grit removal if the filter mesh size is < 6mm

Sludge sample collection (mainly for samplers)

- Samples shall only be taken by trained and qualified samplers. Laboratories can nominate samplers to train using the Solution Provider Platform.

- Composite sludge samples are one sludge type taken over a period of time. The composite sludge sample can be taken from a hopper, roll-off or sludge storage areas. It must represent the facility's sludge production and storage cycle for the entire volume of that sludge type.

- At facilities with on-site incineration, the sampler will collect and separately test two samples:
    - Residual incineration ash
    - Composite sludge

- Sludge samples shall be collected as composite samples following the USEPA 833-B- 89- 100.[1] The ISO 5667- 13 "Guidance on Sampling Sludge" document also provides reference sludge sampling guidance and more detailed information on sampling devices.

- ISO 5667-13 "Guidance on Sampling Sludge" describes multiple sample collection devices.

- The most appropriate way of sampling in any situation depends on several factors:
    - Safe access to the sampling point by personnel.
    - The practicality of installing and maintaining automatic equipment (if appropriate).

- In general, automatic sampling devices, which are widely used for wastewater streams, do not work well for sludge streams because of the solids content and viscosity of sludges.[1] Due to this, manual composite sampling is required.

- For collecting solid and semi-solid sludge samples:
    - When sampling heaps of air-dried sludge lifted from drying beds or stockpiles of sludge cake, it is important to obtain portions of sludge from throughout the mass; not just from the surface layer.
    - For de-watered cakes, dried sludge powder or compost product, combine equal amounts collected at various locations/depths for each grab sample. This will obtain a more representative composite sample.

- To produce a sample from multiple sample locations (e.g. two or more de-watering units), combine the grab samples from each location (equal amounts or weighted, based on flow or solids flux data) in a plastic or stainless-steel bucket and thoroughly mix the sample (with a scoop or spoon). Then transfer it to sample containers.

- When sampling drying beds, divide each bed into quarters. From the centre of each quarter, collect a single core sample through the entire depth of the sludge using a coring device. Usually, a small amount of sand will be collected. Avoid large amounts of sand. Combine and thoroughly mix in a plastic or stainless-steel bucket and transfer to sample containers.

- A sample shall be collected as one composite sample of that sludge type. The composite sample shall represent the entire volume of that sludge type. A sampling grid pattern may be needed, and core samples may be required to obtain a representative sample. Collect samples in a manner that represents the entire sludge volume.

9. Collecting liquid sludge with low solids content:

•Liquid sludges with low viscosity (of lower solids content) may not have sufficient dry matter to facilitate laboratory testing of all the wastewater parameters. Therefore, at the lab's discretion, these samples will be collected using wastewater containers and preservatives, analysed like a wastewater sample and reported as a sludge sample on a weight/volume basis (mg/L). The laboratory analyst can be consulted about the quantities of sludge required at the lab.

10. Collecting liquid sludge with a high solids content

- All laboratory sample containers must be filled from the same bulk composite sample. A minimum sample volume of six (6) litres is needed to fill all the containers. The laboratory may require extra volume for quality assurance samples.

- Collect samples in a manner that represents the entire flow at the sampling point, over the entire sampling period.[1]

- These procedures should be followed when sampling from a tap:[1]
    - Allow sufficient time following pump start-up to clear the line of stagnant sludge.
    - Allow sludge to flow from the tap for several seconds prior to sampling. This will flush out stagnant sludge and solids accumulated in the tap.

- To prevent solids separation in the sample, use glass stirring rods or stainless-steel

spoons to mix the sample before splitting or transferring any portion of it to another container.

- With sludge processing trains, samples from taps on the discharge side of sludge pumps are well mixed since flow at this point in the system is turbulent with minimal solids separation within the flow stream.

- If a sample is drawn from a tap on a pipe containing sludge that is distant from the sludge pumps, the average flow velocity through the pipe should be greater than 2 feet per second (fps). Average velocities of less than 2 fps (0.6 m/s) result in solids separation and settling and affect sample solids content, depending on the location of the tap (top, side or bottom of the pipe).

- Given a choice, a tap on the side of the pipe is preferable. In addition, the tap should be a large size to encourage draw from the entire cross-section of flow when fully open without clogging the flow.

- If the sludge solids tend to separate into different fractions, mix the samples adequately to obtain a representative sample. If they do not mix, collect separate samples. Some pollutant parameters are predominantly associated with the solid fraction, while others are associated with the liquid phase.

11. Sampling equipment must be made of materials that will not contaminate or react with the sludge and must have adequate capacity to avoid oxidation if the bottle is not completely filled. The best material choices are glass and stainless steel because they are relatively inert.[1]

12. If available, sludge flux (weight/time) and/or sludge flow data (volume/time) must be collected and reported with the laboratory sample test report PDF. Typically, the sludge dewatering equipment is not operated 24/7 and will run 2 or 3 cycles per day, depending on equipment sizing and sludge volume produced. Monitoring flow rates will not represent the daily operations and flows/generation of sludge. In such cases, sludge flux and flow data are not necessary to collect or record.

13. The facility shall provide all necessary assistance to the sampler, including but not limited to:

- Access to all relevant areas.
- Provision of information, e.g. flow rates, facility layout, flow path processes, etc.
- Relocation of parts of solid sludge piles where needed to access deeper layers.
- Provision of safety gear and warnings on any specific hazards present.

Sludge sample containers and preservatives (mainly for samplers)

1. Table 4 presents standard sample collection containers and preservatives for sludge samples generated from a wastewater effluent treatment plant. There is a separate Table 4a for sample collection containers and preservatives for sludge samples generated from MMCF facilities.

2. The appropriate sample collection container and preservative can vary depending on the analytical procedure used. Therefore, verify the proper container and preservative with the analytical method used at the laboratory.

3. Caution should be exercised as containers can become pressurised due to gas production in wastewater sludges and explosive situations can occur. Care should be taken, particularly when glass containers are used, to prevent a build-up of gas pressure and to minimise the dispersion of fragments if an explosion occurs.[2]

4. A temperature indicator bottle shall be included with each shipping container to measure the temperature of samples at their time of arrival at the laboratory. The temperature indicator bottle will be clearly labelled.

5. When collecting samples, fill the container to 4/5 full to enable expansion of samples and provide room for gases that may be produced.[1]

6. For solid sludge samples (cake, powder, ash), adding a chemical preservative is generally not useful since the preservative does not usually penetrate the sludge matrix. Preservation is achieved by keeping the temperature between 2°C and 8°C.

Table 4: Sludge Sample Containers and Preservatives

Sludge Parameter	Sample Container Minimum Size	Standard Preservative (verify with lab analytical method to be used)
ZDHC Conventional including Anions		
% Solids	125-ml P,G	keep cool - between 2˚C and 8˚C
Cyanide	P, G 1,000-ml wide mouth	NaOH > 12 pH, keep cool between 2˚C and

	PTFE lined lid	8˚C approx 2-ml 10N NaOH
Fecal coliform	P,G clean, sterile, non-reactive, 125-ml	0.1 ml of 10% sodium thiosulfate keep in the dark and cool - between 2˚C and 8˚C
Paint filter test	250-ml P,G	keep cool - between 2˚C and 8˚C
ZDHC Total Metals		
Chromium (VI)	P, G acid washed 300-ml wide mouth	keep cool between 2˚C and 8˚C
Mercury	P, G acid washed 500-ml wide mouth	HNO3 < pH 2 keep cool between 2˚C and 8˚C
Total metals / heavy metals (antimony, Arsenic, barium, cadmium, chromium-total, cobalt, copper, lead, nickel, selenium, silver, tin, zinc)	P, G acid washed 1,000-ml wide mouth	HNO3 < pH 2, keep cool between 2˚C and 8˚C
ZDHC MRSL		
Alkylphenol (AP) and alkylphenol ethoxylates (APEOs): including all isomers	Six G 1,000-ml PTFE lined lid wide mouth	0.008% Na2S2O3 V/W keep cool between 2˚C and 8˚C
Chlorotoluenes		
Polycyclic Aromatic Hydrocarbons (PAHs)		
Flame retardants		
Perfluorinated and polyfluorinated chemicals (PFCs)		
Phthalates – including all other esters of ortho-phthalic acid		

(P= plastic, G= amber glass, FP= fluoropolymer)

Table 4a: Sludge Sample Containers and Preservatives for MMCF Facilities

Sludge Parameter	Sample Container Minimum Size	Standard Preservative (verify with lab analytical method to be used)
ZDHC Conventional including Anions		

% Solids	125-ml P,G	keep cool - between 2˚C and 8˚C
Faecal coliform	P,G clean, sterile, non-reactive, 125-ml	0.1 ml of 10% sodium thiosulfate keep in the dark and cool - between 2˚C and 8˚C
Paint filter test	250-ml P,G	keep cool - between 2˚C and 8˚C
ZDHC Total Metals		
Total metals / heavy Metals (chromium-total, copper, lead, nickel, zinc)	P, G acid washed 1,000-ml wide mouth	HNO3 < pH 2, keep cool between 2˚C and 8˚C

(P= plastic, G= amber glass, FP= fluoropolymer)

Standard test methods requirements for sludge (mainly for laboratory managers)

1. The approved laboratory analytical methods for each sludge parameter are based on requirements in the European Union, the United States of America, India and China and are presented in the ZDHC Wastewater and Sludge Guidelines. The analytical methods were selected in collaboration with the ZDHC Wastewater Council.

2. An equivalent analytical method can be used, such as those required by the regulatory agency in the region where the wet processing/manufacturing occurs. Equivalent methods must be pre-approved by ZDHC.

3. All test results will be reported on a dry weight basis for sludge that is tested as solids. For sludge that is tested as liquid, the results shall be reported as mg/l as (mg/l = mg/kg).*

* Explanation for exception (liquid sludge): Any detection of % solids which is up to or less than 5% is an indicator that there is a high moisture in the sludge. In such cases, the sampling of sludge is to be considered as liquid sludge.

4. The required reporting limits were established with consideration of achieving these levels with good laboratory practices.

5. Laboratories will test all sludge samples for certain organic ZDHC MRSL compounds outlined in the sludge testing requirements. Total metals, conventional and anion sludge parameters will be tested based on the major sludge disposal pathway opted by the supplier. Only if the total metals threshold values are exceeded, the laboratory will conduct leachate testing of the sludge for the metals that exceed the total metals threshold. The limits for conventional sludge parameters, organic MRSL compounds, leachate metals and

the total metals thresholds are shown in the Wastewater and Sludge Guidelines.

Example methodology of applying the total metals threshold value for arsenic:

Step 1: Conduct total metals analysis.
Step 2: Determine if the total metals results are greater than 10mg/kg for arsenic on a dry weight basis.
Step 3: If greater than 10mg/kg, conduct a Toxicity Characteristic Leaching Procedure (TCLP) test.
Step 4: Analyse the TCLP extract for total arsenic.
Step 5: Compare TCLP arsenic results with the ZDHC TCLP limits.

Sample holding time (mainly for laboratory managers)

1. In general, minimising the time between sample collection and analysis will provide more reliable and representative analytical data.

2. Test results will be reported if the sample exceeds the maximum holding time. Any test results for samples that exceeded the maximum holding time must be flagged with the following data qualifier: "Maximum holding time exceeded" in the ZDHC Gateway Electronic Data Reporting (EDR) template.

3. ZDHC encourages the use of local labs to meet short holding time requirements.

Sample holding time specific to wastewater

Table 5 presents the recommended and maximum holding times for each of the wastewater testing parameters.

Table 5: Wastewater Sample Holding Times

Wastewater Parameter	Recommended Holding Time	Maximum Holding Time
ZDHC Conventional including Anions		
Ammonia-nitrogen	7-days	28-days
AOX	-	6-months

Biochemical Oxygen Demand 5-days concentration (BOD5)	6-hours	48-hours
Chemical Oxygen Demand (COD)	7-days	28-days
Chloride	-	28-days
Colour [m-1] (436nm; 525nm; 620nm)	-	48-hours
Cyanide	24-hours	14-days
Dissolved Oxygen (DO)	measure in the field	15 - min
E.coli	6-hours	24-hours
Oil and grease	-	28-days
Persistent foam	measure in the field	-
pH	measure in the field	6-hours
Phenol	24-hours	28-days
Temperature difference	measure in the field	15 -min
Temperature absolute	measure in the field	15 -min
Total chlorine	measure in the field	15 - min
Total Dissolved Solids (TDS)	24-hours	7-days
Total nitrogen	-	28-days
Total phosphorus	-	28-days
Total Suspended Solids (TSS)	24-hours	7-days
Sulfate	-	28-days
Sulfide	-	7-days
Sulfite	-	48-hours
ZDHC Heavy Metals		
Chromium (VI)	24-hours	28-days

Heavy metals (antimony, arsenic, barium, cadmium, chromium-total, cobalt, copper, lead, nickel, selenium, silver, tin, zinc)	28-days	6-months
Mercury	-	28-days
ZDHC MRSL		
Alkylphenol (AP) and Alkylphenol Ethoxylates (APEOs): Including All Isomers	-	Extraction: 7-days from collection Analysis: 40-days from extraction
Anti-microbials & biocides	-	
Chlorobenzenes and chlorotoluenes	-	
Chlorinated paraffins	-	
Chlorophenols	-	
DMFa	-	
Restricted Aromatic Amines (Cleavable from Azo-colourants)	-	
Dyes – Carcinogenic or equivalent concern	-	
Dyes – Disperse (sensitising)	-	
Dyes – Navy blue colourant	-	
Flame retardants	-	
Glycols /glycol ethers	-	
Organotin compounds	24-hours	
Other/miscellaneous chemicals	-	

Perfluorinated and polyfluorinated chemicals (PFCs)	-	
Phthalates – including all other esters of ortho-phthalic acid	-	
Polycyclic Aromatic Hydrocarbons (PAHs)	-	
UV absorbers	-	
Halogenated solvents	7-days	14-days
Volatile Organic Compounds (VOC)	7-days	14-days

Table 5a: Wastewater Sample Holding Times for MMCF Facilities

Wastewater Parameter	Recommended Holding Time	Maximum Holding Time
ZDHC Conventional including Anions		
Ammonia-nitrogen	7-days	28-days
AOX	-	6-months
Biochemical Oxygen Demand 5-days concentration (BOD5)	6-hours	48-hours
Chemical Oxygen Demand (COD)	7-days	28-days
Colour [m-1] (436nm; 525nm; 620nm)	-	48-hours
Oil and grease	-	28-days
Persistent foam	measure in the field	-
pH	measure in the field	6-hours
Phenol	24-hours	28-days
Temperature difference	measure in the field	15 -min
Temperature absolute	measure in the field	15 -min
Total nitrogen	-	28-days

Total phosphorus	-	28-days
Total Suspended Solids (TSS)	24-hours	7-days
Sulfate	-	28-days
Sulfide	-	7-days
ZDHC Heavy Metals		
Heavy metals (chromium-total, copper, lead, nickel, zinc)	28-days	6-months

Sample holding time specific to sludge

Table 6 presents the recommended and maximum holding times for each of the sludge testing parameters.

Table 6: Sludge Sample Holding Time

Sludge Parameter	Recommended Holding Time	Maximum Holding Time
ZDHC Conventional including Anions		
% Solids	2-days	7-days
Cyanide	24-hours	14-days
Faecal coliform	6-hours	24-hours*
Paint filter test	2-days	7-days
pH	15-min	24-hours
ZDHC Heavy Metals		
Chromium (VI)	24-hours	28-days
Heavy metals (antimony, arsenic, barium, cadmium, chromium-total, cobalt, copper, lead, nickel, selenium, silver, tin, zinc)	28-days	6-months
Mercury	-	28-days
ZDHC MRSL		

Alkylphenol (AP) and alkylphenol ethoxylates (APEOs): Including all isomers	-	Extraction: 7-days from collection Analysis: 40-days from extraction
Chlorotoluenes	-	
Polycyclic Aromatic Hydrocarbons (PAHs)	-	
Flame retardants	-	
Phthalates – including all other esters of ortho-phthalic acid	-	
Perfluorinated and polyfluorinated chemicals (PFCs)	-	

* Faecal Coliform has a 24-hour holding time. It is acceptable to collect a grab sample for faecal coliform and ship before the composite if that will satisfy the holding time.

Table 6a: Sludge Sample Holding Time for MMCF Facilities

Sludge Parameter	Recommended Holding Time	Maximum Holding Time
ZDHC Conventional including Anions		
% Solids	2-days	7-days
Faecal coliform	6-hours	24-hours*
Paint filter test	2-days	7-days
pH	15-min	24-hours
ZDHC Heavy Metals		
Heavy metals (chromium-total, copper, lead, nickel, zinc)	28-days	6-months

* Faecal Coliform has a 24-hour holding time. It is acceptable to collect a grab sample for faecal coliform and ship before the composite if that will satisfy the holding time.

Sample shipments (mainly for samplers)

Use a 24-hour (overnight) delivery service for samples shipped to a laboratory. If 24-hour delivery is not available, consider using a ZDHC Approved Wastewater Laboratory to conduct

a short hold time analysis.

1. To avoid shipping delays and compromising sample holding times, contact the shipping company before sample collection. The shipping company can help determine the appropriate customs arrangements that must be made.

2. Appendix B: Shipping and customs forms provides sample shipping forms that may help to avoid delays in customs.

3. Samples must be shipped in cool boxes with sufficient insulation and artificial refrigerant ("blue ice"), or ice contained in double zip-lock bags, to maintain a sample temperature of between 2°C and 8°C for the entire duration of transportation. Shipping containers that leak fluid, such as melting ice water, will likely be returned to the shipper.

4. The sample cool boxes must be sealed with custody tape, signed and dated by the sampling crew.

5. The sampler is responsible for handling, processing and custody of the samples. This includes taking samples to the nearest servicing airport, bus station or other carriers.

6. Containers should be held upright during shipment. Use bubble wrap around individual glass containers and other adequate packing materials to prevent movement during shipment, cushion from shock and reduce the risk of leakage.

7. The sampling team must maintain physical custody or use a custody seal tape on the sample cool boxes.

Sample receipt, handling and custody

1. The laboratory shall be available to receive sample shipments at any time the delivery service is operating, including weekends.

2. The sample temperature shall be measured and recorded immediately upon opening the shipping container and prior to unpacking the samples or removing the packing material.
   - The laboratory shall use the shipping container temperature indicator bottle reading as the sample temperature.
   - To determine the temperature, invert the bottle several times, remove the cap and insert a calibrated thermometer.
   - Allow a minimum of three (3) minutes, but no greater than five (5) minutes, prior to

taking the measurement. The thermometer used shall be calibrated and capable of measuring within an accuracy of ±1°C.

- If a temperature indicator bottle is not present in the shipping container, an alternative means of determining shipping container temperature can be used.

- Under no circumstances shall a thermometer or any other device be inserted into a sample bottle to determine shipping container temperature. However, a small aliquot of the sample removed from the container can measure temperature. This small aliquot must be discarded and not returned to the sample container.

- Other devices which can measure temperature, such as an infrared thermometer, may be used if calibrated to ±1°C.
- The desired sample temperature when received at the laboratory is between 2°C and 8°C. If the sample temperature exceeds 10°C when received at the laboratory, the laboratory shall contact the client and inform them of the temperature deviation. The client may decide not to perform testing on these samples.

3. Each sample shipment requires a chain of custody log maintained from the time of collection, during the entire analytical process and until sample disposal. A chain of custody document provides a record of sample transfer from person to person. This document helps protect the integrity of the sample by ensuring only authorised persons have custody of the sample. An example of a chain of custody form is presented in Appendix A: Example chain of custody form. The sampling team must maintain physical custody or use a custody seal tape on the sample cool boxes.

4. A document attesting that the sampler has been in the factory needs to be signed by both the sampler and the customer (with a stamp).

5. If the laboratory encounters problems with samples or related documentation (e.g. mixed media, sample pH, sample documentation and paperwork such as traffic report/ chain-of-custody), the laboratory shall immediately contact the sampler for a resolution.

6. For wastewater and liquid type sludge, pH for all aqueous/water sample containers received by the laboratory shall be measured and recorded at the laboratory to demonstrate that proper preservation was performed.
    - Measure pH using test strips, a hand-held electronic pen or pH meter. To prevent sample contamination, measure the pH on a small aliquot of the sample removed from the container. This small aliquot must be discarded

and not returned to the sample container.

- Under no circumstances shall a strip or any device be inserted into a sample bottle for the purpose of determining pH.

Sample storage (mainly for laboratory managers)

All samples will be stored under custody at 2˚C - 8˚C in the laboratory (unless otherwise specified by the standard test method).

1. The samples may be disposed of 60 days after the laboratory submits the final ZDHC Gateway Electronic Data Report (EDR).

Laboratory quality systems (mainly for laboratory managers)

The minimum quality assurance measures for organic chemical analysis of wastewater and sludge are presented in Table 7.

Table 7: Minimum Quality Assurance Measures for Organic Chemical Analysis

Measure	Description	Target value	Frequency	Points to be checked (examples)	
A.Routine					
Method blank	covering sample preparation and measurement	< LOD	1 per batch	cleanliness of laboratory glassware and equipment	
Calibration check	an independently sourced/ prepared standard	± 20 %	1 per batch	check instrument condition/ drift, clean and recalibrate, stability of standards	
Internal standard	for GC methods. substance with physico-chemical properties similar to	50 – 150%	every sample	Correction of injection error. Method specific: surrogate as	choice of internal standard or surrogate or a

	the analyte			alternative	combination the reof is method/ analyte dependent
Surrogate	substance which has similar physico-chemical properties to the analyte	method specific	every sample	Check sample preparation procedure and internal standard correction. Method specific: internal standard as alternative	
Duplicate	duplicate undergoing complete process	< 35 % RPD	1 per batch and every 20 samples	reproducibility of method	
Matrix spike Matrix Spike Duplicate (MS/MSD)	spike into sample matrix (duplicate) undergoing complete process	±20% recovery and < 35% RPD	1 per batch	parameter recovery with sample matrix influence and reproducibility	
B. For Method Validation					
Multi-point calibration	min. 5-point calibration excl. origin	$r^2 \leq 0.990$	validation	linearity, working range	
Recovery (LCS)	extract the standard through the test procedure without matrix	method and analyte specific	validation	recovery without influence of matrix	
Repeatability (matrix spike replicate)	matrix spike replicates under repeatability conditions	method and analyte specific	validation	robustness, recovery with matrix influence, UoM, general fitness for use	
Limit of Detection	ISO/TS 13530:2009 4.4.3 4.4.7	less than WWG RL Ideal: ≤ ½ WWG RL	validation	LOD	

Note:

The general suitability of the analytical approach is demonstrated via method validation. Due to this demonstration, initial method validation is deemed to sufficiently cover most sample types and a comparatively leaner quality control (QC) programme is applied.

- Specific, problematic sample matrices, which are not covered by the validation, require additional QC measures.
- In case of conflict, quality assurance measures specified by the applied standard, the accreditation body or the local authority shall prevail.

Quality systems specific to wastewater

The quality assurance testing in Table 8 is required for each ZDHC wastewater parameter. This data may be requested and used by ZDHC to assess data quality and validate the analytical results.

Table 8: Required Quality Assurance Testing for Wastewater

DQI	Field Blank	Method Blank	Calibration Check*	Lab Duplicate	ISTD and Surrogate	Matrix Spike & Matrix Spike Duplicate
Frequency	1 per batch	1 per batch	1 per batch	1 in 20	every sample	1 in 20
DQO	< LOD	< LOD	+/- 20%	+/- 35%	method specific	20% recovery 35% RPD
ZDHC Conventional including Anions						
Ammonia-nitrogen	-	X	X	X	-	X
AOX	-	X	X	X	-	X
Biochemical Oxygen Demand 5-days concentration (BOD5)	-	X	X	-	-	-
Chemical Oxygen Demand (COD)	-	X	X	X	-	X
Chloride	-	X	X	X	-	-

Colour [m-1] (436nm; 525; 620nm)	-	X	X	-	-	-
Cyanide	-	X	X	X	-	X
Dissolved Oxygen (DO)	-	-	X	field test	-	-
E.coli	-	X	-	X	-	-
Oil and grease	-	X	X	X	-	-
Persistent foam	-	-	-	-	-	-
pH	-	-	X	-	-	-
Phenol	-	X	X	X	-	X
Total chlorine	-	-	X	field test	-	-
Temperature difference	-	-	-	-	-	-
Temperature absolute	-	-	-	-	-	-
Total Dissolved Solids (TDS)	-	-	X***	X	-	-
Total nitrogen	-	X	X	X	-	X
Total phosphorus	X	X	X	X	-	X
Total Suspended Soldis (TSS)	-	-	X***	X	-	-
Sulfate	-	X	X	X	-	-
Sulfide	-	X	X	X	-	-
Sulfite	-	X	X	X	-	-
ZDHC Heavy Metals						
Chromium (VI)**	-	X	X	X	-	X

Heavy metals (antimony, arsenic, barium, cadmium, chromium-total, cobalt, copper, lead, nickel, selenium, silver, tin, zinc)	-	X	X	X	X	X
Mercury	X	X	X	X	X	X
ZDHC MRSL						
Alkylphenol (AP) and alkylphenol ethoxylates (APEOs): including all isomers	-	X	X	X	X	X
Anti-microbials & biocides	-	X	X	X	X	X
Chlorinated paraffins	-	X	X	X	X	X
Chlorobenzenes and chlorotoluenes	-	X	X	X	X	X
Chlorophenols	-	X	X	X	X	X
DMFa	-	X	X	X	X	X
Restricted aromatic amines (cleavable from azo-colourants)	-	X	X	X	X	X
Dyes – carcinogenic or equivalent concern	-	X	X	X	X	X
Dyes – disperse (sensitising)	-	X	X	X	X	X
Dyes – Navy blue colourant	-	X	X	X	X	X
Flame retardants	-	X	X	X	X	X
Glycols /glycol ethers	-	X	X	X	X	X

Halogenated solvents	X	X	X	X	X	X
Organotin compounds	-	X	X	X	X	X
Other/miscellaneous chemicals	-	X	X	X	X	X
Perfluorinated and polyfluorinated chemicals (PFCs)	X	X	X	X	X	X
Phthalates – including all other esters of ortho-phthalic acid	-	X	X	X	X	X
Polycyclic Aromatic Hydrocarbons (PAHs)	-	X	X	X	X	X
UV absorbers	-	X	-	X	-	X
Volatile Organic Compounds (VOC)	X	X	X	X	X	X

Table 8a: Required Quality Assurance Testing for Wastewater for MMCF Facilities

DQI	Field Blank	Method Blank	Calibration Check*	Lab Duplicate	ISTD and Surrogate	Matrix Spike &Matrix Spike Duplicate
Frequency	1 per batch	1 per batch	1 per batch	1 in 20	every sample	1 in 20
DQO	< LOD	< LOD	+/- 20%	+/- 35%	method specific	20% recovery 35% RPD
ZDHC Conventional including Anions						
Ammonia-nitrogen	-	X	X	X	-	X
AOX	-	X	X	X	-	X

Biochemical Oxygen Demand 5-days concentration (BOD5)	-	X	X	-	-	-
Chemical Oxygen Demand (COD)	-	X	X	X	-	X
Colour [m-1] (436nm; 525; 620nm)	-	X	X	-	-	-
Oil and grease	-	X	X	X	-	-
Persistent foam	-	-	-	-	-	-
pH	-	-	X	-	-	-
Phenol	-	X	X	X	-	X
Temperature difference	-	-	-	-	-	-
Temperature absolute	-	-	-	-	-	-
Total nitrogen	-	X	X	X	-	X
Total phosphorus	X	X	X	X	-	X
Total Suspended Soldis (TSS)	-	-	X***	X	-	-
Sulfate	-	X	X	X	-	-
Sulfide	-	X	X	X	-	-
ZDHC Heavy Metals						
Heavy metals (chromium-total, copper, lead, nickel, zinc)	-	X	X	X	X	X

* Calibration check conducted with a second source standard.

Quality systems specific to sludge

The quality assurance testing in Table 9 is required for each ZDHC sludge parameter. This data will be reported with the sample results and used by ZDHC to assess data quality and validate the analytical data.

Table 9: Required Quality Assurance Testing for Sludge

DQI	Field Blank	Method Blank	Calibration Check*	Lab Duplicate	ISTD and Surrogate	Matrix Spike & Matrix Spike Duplicate
Frequency	1 per batch	1 per batch	1 per batch	1 in 20	every sample	1 in 20
DQO	< LOD	< LOD	+/- 20%	+/- 35%	method specific	20% recovery 35% RPD
ZDHC Conventional including Anions						
% Solids	-	-	X***	X	-	-
Cyanide	-	X	X	X	-	X
Faecal coliform	-	-	-	X	-	-
Paint filter test	-	-	-	X	-	-
pH	-	-	X	X	-	-
ZDHC Heavy Metals						
Chromium (VI)	-	X	X	X	-	X
Heavy metals (antimony, arsenic, barium, cadmium, chromium-total, cobalt, copper, lead, nickel, selenium, silver, tin, zinc)	-	X	X	X	X	X

Mercury	X	X	X	X	X	X
ZDHC MRSL						
Alkylphenol (AP) and alkylphenol ethoxylates (APEOs): including all isomers	-	X	X	X	X	X
Chlorotoluenes	-	X	X	X	X	X
Polycyclic Aromatic Hydrocarbons (PAHs)	-	X	X	X	X	X
Flame retardants	-	X	X	X	X	X
Phthalates – including all other esters of ortho-phthalic acid	-	X	X	X	X	X
Perfluorinated and polyfluorinated chemicals (PFCs)	-	X	X	X	X	X

Table 9a: Required Quality Assurance Testing for Sludge for MMCF Facilities

DQI	Field Blank	Method Blank	Calibration Check*	Lab Duplicate	ISTD and Surrogate	Matrix Spike & Matrix Spike Duplicate
Frequency	1 per batch	1 per batch	1 per batch	1 in 20	every sample	1 in 20
DQO	< LOD	< LOD	+/- 20%	+/- 35%	method specific	20% recovery 35% RPD
ZDHC Conventional including Anions						

% Solids	-	-	X***	X	-	-
Faecal coliform	-	-	-	X	-	-
Paint filter test	-	-	-	X	-	-
pH	-	-	X	X	-	-
ZDHC Heavy Metals						
Heavy metals (chromium-total, copper, lead, nickel, zinc)	-	X	X	X	X	X

* Calibration check conducted with a second source standard.
*** Check balance with reference weight.

Test report requirements (mainly for laboratory managers)

Reports shall be in line with the requirements of ISO 17025 and test reports shall be uploaded to the Gateway. At a minimum, they should include:

1. Factory name
2. Factory address
3. On-Site App project number (Include the project number assigned in the ZDHC On-Site App)
4. Sampling method (e.g., grab, composite,...)
5. Type of discharge (e.g., direct, indirect with or without pretreatment, with or without sludge, ZLD)
6. ZDHC sampler name and ZDHC certificate number
7. Sampling start date and time (including the time of each aliquot, if applicable)
8. Sampling end date and time (including the time of each aliquot, if applicable)
9. Date on which the sample was received by the laboratory
10. Testing period (indicate clearly the period during which each parameter or group of parameters was tested)
11. Sludge flux and/or sludge flow data, if available
12. Temperature of the sample upon receipt at the laboratory

13. Temperature deviations (indicate if the sample temperature upon receipt exceeds 8°C)
14. Holding time compliance (clearly note whether the holding time was evaluated and respected)
15. Subcontracted parameters (clearly identify any tests performed by a subcontracted laboratory)
16. Photos with relative time and date
17. Remarks (if necessary)
18. Test method reporting requirements:

Only test methods approved through the ZDHC Solution Provider Platform must be included in the report. Depending on the nature of the approved method, the following rules apply:

- **If a standardised method has been approved**, include the method as-is, with full reference to its official designation (e.g., ISO, EPA).
- If an **in-house method has been approved**, clearly reference the internal procedure, including the method identification or version number.
- If the **approved method is a standardised method adapted for a different matrix**, you must:
    - Reference the original standard method
    - Indicate that it was "**applied with reference to**" the standard, or state "**modified according to [standard], adapted for [matrix type]**"
    - Do not label this as a true in-house method unless it significantly deviates from the original.

Only parameters relevant to the ZDHC Wastewater and Sludge Guidelines, based on the applicability and type of discharge, should be included in the report uploaded in the ZDHC Gateway.

IF THE UNCERTAINTY IS REPORTED:

If the laboratory includes uncertainty values for test results, these should be calculated and presented following recognised standards (e.g., ISO/IEC 17025). Uncertainty for the parameters must be calculated.

It is the responsibility of the laboratory to apply the predefined decision rule (as per ISO17025), considering the measurement uncertainty and relevant acceptance criteria, to determine the conformity.

Minimum required reporting limits (mainly for laboratory managers)

The Level of Detection (LOD) for each parameter must first be determined. The LOD is established using ISO/TS 13530:2009 4.4.3. The procedure requires a complete, specific and well-defined analytical method. All analytical method sample processing steps must be included when determining the LOD. The LOD for an analytical procedure may vary as a function of sample type.

The Level of Quantification (LOQ) will be established using ISO/TS 13530:2009 4.4.7.

ZDHC requires laboratories to provide their LOD and LOQ on the Solution Provider Platform. To be a ZDHC Approved Wastewater Laboratory, the laboratory LOQ must be at or lower than the ZDHC Aspirational limit (conventional and metals) and at or lower than the ZDHC MRSL wastewater reporting limit. If the ZDHC minimum LOQ cannot be achieved, an alternate method must be approved by ZDHC. Alternatively, a sub-contracted laboratory can be used. The ZDHC minimum required LOQ's are presented in the ZDHC Wastewater and Sludge Guidelines. These were established with consideration of achieving these levels with good laboratory practices.

Detect and non-detect test results

In order to report a non-detected (ND) value, the ND must include a numerical value that represents the laboratory calculated LOQ for each parameter and where the laboratory calculated LOQ is at or lower than the lowest ZDHC limits (e.g. MRSL wastewater, MRSL sludge and wastewater Aspirations limits). Non-detection is not allowed for certain parameters including colour, pH and temperature.

Reporting requirements (mainly for laboratory managers)

Only ZDHC Approved Wastewater Laboratories can perform sampling, testing and report results into the ZDHC Gateway on behalf of the suppliers.

Reporting means submitting test data/results to the ZDHC Gateway Wastewater Module. The integrity of the test data in the Gateway must be at its highest possible standard. This will support the ZDHC Roadmap to Zero Programme in making scientific and data-driven decisions necessary to shape the future of our industry.

1. All ZDHC Accepted Laboratories are expected to follow the data reporting requirements specified in the ZDHC Gateway Electronic Data Reporting (EDR) System Guidelines for wastewater and sludge.

2. All test data must be reported using the EDR template generated in the ZDHC Gateway.

3. The use of reporting formats other than those approved in the EDR will not be accepted.

4. The following data qualifiers can be used when a numerical value is not entered for a parameter:
    - "Insufficient Sample" - not enough sample volume was provided.
    - "QA/QC" - sample analysis did not meet ZDHC data quality objectives

Data validation

ZDHC routinely requests laboratory quality assurance/quality control information from randomly selected ZDHC Approved Laboratories. The following information may be requested for data review:

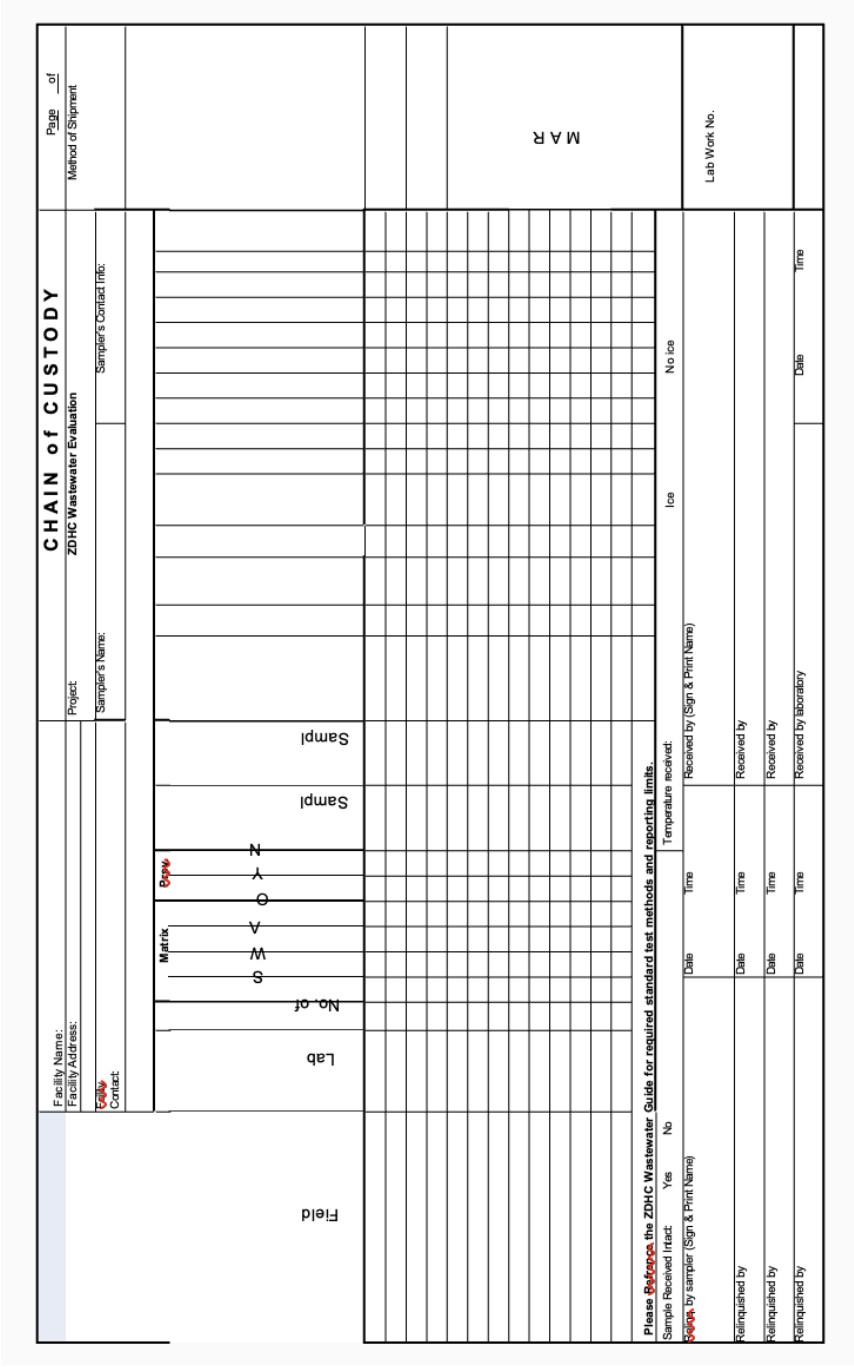
- Calibration curves
- Method blank instrument reports
- Instrument calibration check sample reports
- Laboratory control sample instrument reports
- Laboratory duplicate instrument reports
- Matrix spike and matrix spike duplicate instrument reports
- Sample custody documentation

The quality assurance and quality control results associated with the ZDHC sample will be assessed by ZDHC to validate the analytical data. Quality assurance results that fail to meet ZDHC guidelines may result in the rejection of sample results.

References

1. USEPA 833-B-89-100: POTW Sludge Sampling and Analysis Guidance Document, United States Environmental Protection Agency 1989.
2. ISO 5667- 13: Guidance on Sampling Sludge.

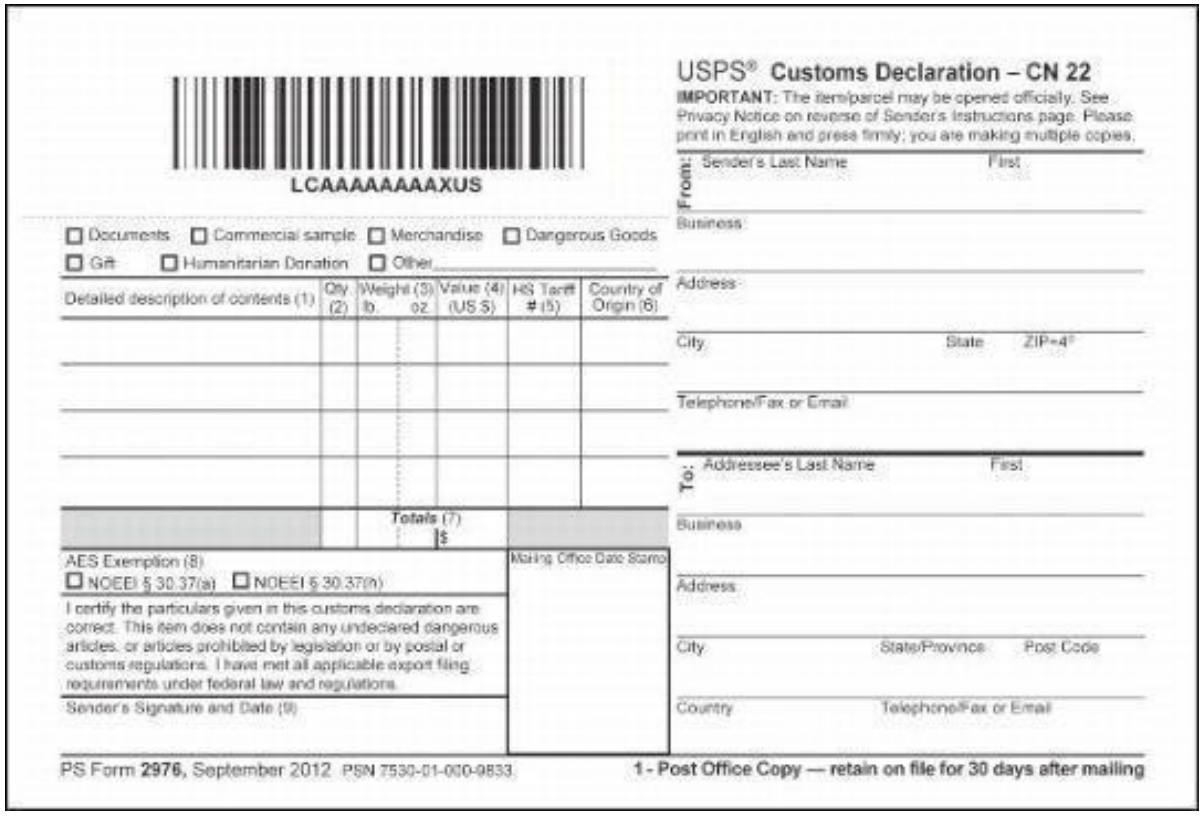
Appendix A: Example of chain of custody form



Appendix B: Examples of shipping and customs forms

The following are examples of forms to be completed and accompany the samples during international shipment, which will minimise the chance of shipping delays in customs:

1. Safety Data Sheet (SDS) for water samples. This illustrates that the wastewater samples are not some types of known hazardous material.
2. Customs declaration forms for various countries, such as the one presented below, can be found at this link.
3. Toxic Substance Control Act (TSCA) certification. This form certifies that a sample is not a material which is hazardous to ship by air freight.



LCAAAAAAAAXUS

USPS® **Customs Declaration – CN 22**

IMPORTANT: The item/parcel may be opened officially. See Privacy Notice on reverse of Sender's Instructions page. Please print in English and press firmly; you are making multiple copies.

☐ Documents ☐ Commercial sample ☐ Merchandise ☐ Dangerous Goods
☐ Gift ☐ Humanitarian Donation ☐ Other

Detailed description of contents (1)	Qty (2)	Weight (3) lb. oz.	Value (4) (US $)	HS Tariff # (5)	Country of Origin (6)
		Totals (7) $			

AES Exemption (8)
☐ NOEEI § 30.37(a) ☐ NOEEI § 30.37(h)

I certify the particulars given in this customs declaration are correct. This item does not contain any undeclared dangerous articles, or articles prohibited by legislation or by postal or customs regulations. I have met all applicable export filing requirements under federal law and regulations.

Sender's Signature and Date (9)

Mailing Office Date Stamp

From: Sender's Last Name First
Business
Address
City State ZIP+4®
Telephone/Fax or Email

To: Addressee's Last Name First
Business
Address
City State/Province Post Code
Country Telephone/Fax or Email

PS Form **2976**, September 2012 PSN 7530-01-000-9853 **1 - Post Office Copy — retain on file for 30 days after mailing**

TOXIC SUBSTANCE CONTROL ACT (TSCA) CERTIFICATION

Date: _____

(CHECK ONE SECTION ONLY)

POSITIVE CERTIFICATION:

_____ "I CERTIFY THAT ALL CHEMICAL SUBSTANCES IN THIS SHIPMENT COMPLY WITH ALL APPLICABLE RULES OR ORDERS UNDER TSCA AND THAT I AM NOT OFFERING A CHEMICAL SUBSTANCE FOR ENTRY IN VIOLATION OF TSCA OR ANY APPLICABLE RULE OR ORDER THEREUNDER."

- OR -

NEGATIVE CERTIFICATION:

_____ "I CERTIFY THAT ALL CHEMICALS IN THIS SHIPMENT ARE NOT SUBJECT TO TSCA."

COMPANY NAME: _____

COMPANY ADDRESS: _____

AUTHORIZED NAME: _____

AUTHORIZED SIGNATURE: _____

TITLE: _____

FEDERAL EXPRESS AWB#: _____

RETURN TO: _____

IF THE CERTIFIER IS UNSURE IF THEIR CHEMICAL SUBSTANCE IS SUBJECT TO TSCA COMPLIANCE, CONTACT THE ENVIRONMENTAL PROTECTION AGENCY, TSCA, ASSISTANCE OFFICE, WASHINGTON, D.C. (202) 544-1404 BETWEEN 8:30 AM AND 5:00 PM EST.

REVISED May 7, 1990

Toxic.392

Appendix C: Wastewater sample collection – field kit checklist

- Camera
- The following ZDHC sample collection forms:
    - Wastewater Sampling Request Form
    - Wastewater Sample Chain-of-Custody Form
    - Shipping and Customs Forms
    - Wastewater Sampling Field Data Form or MMCF Sampling Field Data Form
    - Lab Sample Container List with preservatives
- Auto sampler with refrigeration capabilities
- Ice or cold packs, or a portable freezer/fridge with a working range of 1-4˚C if manual sampling is necessary and acceptable
- Ice or cold packs for shipping the samples
- ISO 17025 traceable thermometer for measuring sample temperature
- pH measurement equipment and supplies
    - Meter with temperature compensation capabilities. pH / LF temperature compensation: pH temperature compensation for indication of the pH value at the current temperature, with LF measurement converted to the reference temperature of 20°C
    - Automatic Temperature Compensation (ATC) probe. This can be done manually if an ATC is not available.
    - High quality probe
    - Buffer solutions 4, 7, and 10
    - Laboratory control sample (LCS) or correlation testing sample with a known value
- Total chlorine measurement
    - For DPD test: hand-held meter, DPD reagent packets, distilled water for rinsing, GEL standard to check meter calibration
- Dissolved oxygen measurement
    - For meter and probe: hand-held meter, oxygen probe, membrane kit for probe

- Lab sample container kit with preservatives
- Cold storage container (like a cooler) for shipping samples
- Facility information that has been requested ahead of the sampling date:
    - Legal discharge permit
    - Sampling locations – the crew may need multiple sample collection kits

    - Drawing of the facility layout that includes identifying sample collection points
    - Point of contact for the owner of the analytical data – for example, a Bband that is paying for the testing.
- Safety equipment required by the lab, facility, brand, or others. This may include safety glasses, safety shoes, a high-visibility vest, a hard hat, or other items.
- ICE packs or ICE with zip-lock bags
    - Homogenisation container 20 litres