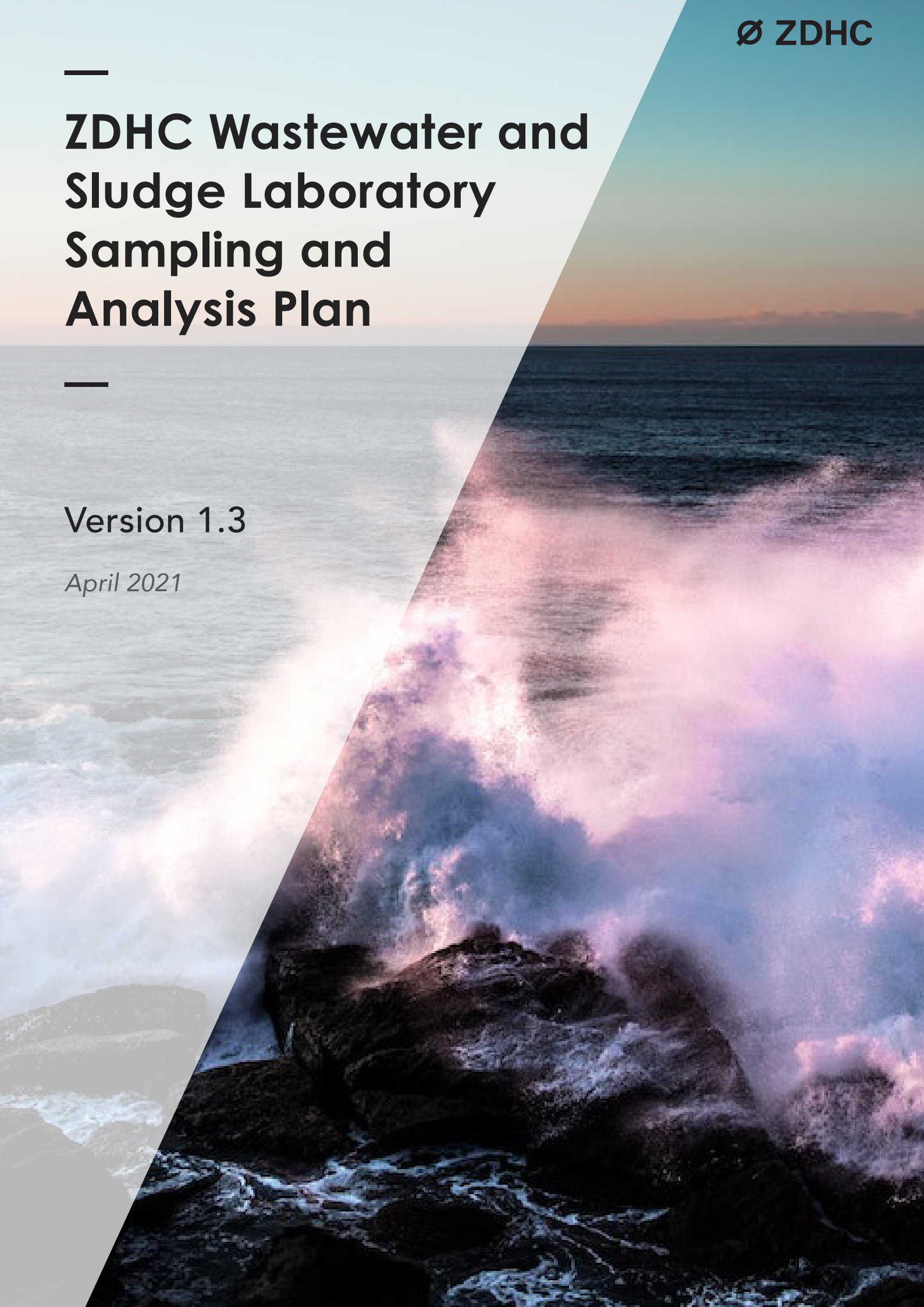
ZDHC

ZDHC Wastewater and Sludge Laboratory Sampling and Analysis Plan

Version 1.3

April 2021



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1.0.0 Revision History

In the spirit of continuous improvement, the ZDHC Wastewater and Sludge Laboratory Sampling and Analysis Plan (SAP) will be reviewed on a regular basis and revised as needed to incorporate learnings and opportunities identified during the practical application and implementation of these procedures. A historical record of updates to the SAP is noted in the figure below.

Figure 1: Revision History

Version Number	Changes	Time of Publication
Version 1.0	Initial publication of the ZDHC Wastewater Laboratory Sampling and Analysis Plan.	June 2019
Version 1.1	1. Added sampling and analysis procedures specific to sludge. 2. Improved sampling and analysis procedures for wastewater. • Ensured full alignment with ZDHC Wastewater Guidelines Version 1.1 requirements, including but not limited to standard method for analysis. • Changed the expected sample temperature and applied it to all relevant sections throughout the wastewater part of the document. • Changed the recommended holding time for halogenated solvents – see Figure 3. • Adjusted the target value for Multi-Point Calibration in Figure 4. • Adjusted Calibration Check for Total Coliform in Figure 5. • Updated and clarified the Reporting and Deliverable Requirements in Section 3.3.0 to align with latest development within ZDHC.	August 2019
Version 1.2	• Added sampling and analysis of parameters specific to the ZDHC MMCF Wastewater interim guidelines.	April 2020
Version 1.3	1. 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. 2. Added reference to and content from the ZDHC Wastewater and Sludge Sampling Procedure/Training. 3. Added guidance for composite wastewater sampling at facilities that do not have continuous production cycles. 4. Emphasised that the ZDHC specified methods for colour and Chemical Oxygen Demand must be used. There are no exceptions. 5. Changed sample storage temperature from < 4°C to the ISO recommended temperature of 2°C to 8°C.	January 2021
Version 1.3	Replaced Faecal Coliform with E.coli test.	April 2021

2.0.0 Introduction

The ZDHC Roadmap to Zero Programme takes a holistic approach to tackling the issue of hazardous chemicals in the global textile, apparel, footwear and leather industry. This holistic approach starts with the management of input chemistries, the management of day-to-day chemical use at production facilities (Suppliers), and the management of effluent generated and discharged by Suppliers.

With regards to effluent management, the ZDHC Wastewater Guidelines (always refer to the latest version, publicly available on the ZDHC website) are in place to harmonise wastewater parameters, limit values and test methods, as well as requirements for sampling, testing and reporting. This is an attempt to encourage and enable the industry, in particular brands, suppliers (manufacturing facilities) and testing laboratories, to work towards the same set of expectations.

This ZDHC Wastewater and Sludge Laboratory Sampling and Analysis Plan (SAP) document is one of the key elements to support implementation of the ZDHC Wastewater Guidelines. It does so by standardising procedures for laboratories to conduct sampling and analysis. In this document the ZDHC Wastewater Guidelines encompass ZDHC Sludge, Textiles,

Man-Made Cellulosic Fibres and Leather Wastewater Guidelines.

Purpose

This SAP provides a detailed framework for laboratories to perform testing to determine the concentration of parameters in wastewater and sludge.

The ZDHC Wastewater and Sludge Sampling Procedure/Training provides a detailed framework for the sampling process.

Data Uses

Wastewater and sludge test data helps to promote the implementation of sustainable chemistry and best practices in the industry.

The laboratory must be aware of the importance of maintaining the integrity of test data generated under the ZDHC Programme. The test data may be used to make major decisions regarding manufacturing facilities.

3.0.0 Wastewater

3.1.0 Sampling for Wastewater

3.1.1 Types of Samples and Sampling Points/Locations

Please refer to the ZDHC Wastewater Guidelines (WWG) for types of samples and sampling points/locations. The types of samples to be collected will depend on the testing option decided upon by the brand and supplier (manufacturing facility), but limited to the two testing options identified in the WWG. The WWG acknowledges the difference between facilities with a direct discharge, indirect discharge and on-site zero liquid discharge treatment plant.

3.1.2 Wastewater Sample Collection

1. Samples shall be taken by ZDHC trained and accredited samplers only. Please refer to the ZDHC Academy for training information.
2. Wastewater samples shall be collected as composite samples following ISO 5667 - 10 guidelines: “Water Quality Sampling Guidance for the preservation and handling of water samples”.
3. Sampling using calibrated and refrigerated auto samplers is preferred. To ensure representative samples, composite sampling must be performed 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. The composite sample container must be cooled during sampling.
4. If necessary, laboratory personnel 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.
5. All of the laboratory sample containers will be filled from the same bulk composite sample. A minimum sample volume of 20-litres is needed to fill all the containers. The laboratory may require extra volume for QA samples. The sampler shall coordinate extra volume requirements with the laboratory.
6. Samples must be taken during a time that represents continuous and normal production and continuous and normal wastewater treatment. Collect wastewater samples in a way that represents the entire production cycle. This may take a shorter or longer time than the specified 6-hour composite time.
7. 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.
8. Samples must not be taken if the wastewater is diluted, for example, by heavy rainfall.
9. Wastewater flow data (volume/time) must be collected and reported with the laboratory test results.

3.1.3 Wastewater Sample Containers and Preservatives

1. *Figure 2* presents standard sample collection containers and preservatives for the wastewater parameters specified in the ZDHC Wastewater 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 being used at the laboratory.

Figure 2: Wastewater Sample Containers and Preservatives

Wastewater Parameter	**Sample Container Minimum Size**	**Standard Preservative** (verify with lab method to be used)
Conventional		
Temperature	NA	Measured in the field
Total Suspended Solids (TSS)	P, G 200-ml	keep cool - between 2˚C and 8˚C
Total Dissolved Solids (TDS)	P,G 200-ml	keep cool - between 2˚C and 8˚C
COD	P, G, FP 100-ml	H_2SO_4 < pH 2 keep cool - between 2˚C and 8˚ C
Total-N	P, G, FP 100-ml	H_2SO_4 < pH 2 keep cool - between 2˚C and 8˚C
pH	NA	Measured in the Field
Colour [m-1] (436nm; 525; 620nm)	P, G, FP 500-ml	keep cool - between 2˚C and 8˚C
BOD-5 day	P, G, FP 1,000-ml	keep cool - between 2˚C and 8˚C
Ammonia-N	P, G, FP 500-ml	H_2SO_4 < pH 2 keep cool - between 2˚C and 8˚C
Total-P	P, G, FP 100-ml	H_2SO_4 < pH 2 keep cool - between 2˚C and 8˚C
AOX	P, G, FP 500-ml	HNO_3 pH 1-2, keep cool - between 2˚C and 8˚C
Oil and Grease	Glass, wide mouth PTFE lined lid 1,000-ml	HCl or H_2SO_4 < pH 2 keep cool - between 2˚C and 8˚C
Phenols	P, G PTFE lined lid 500-ml	H_2SO_4 < pH 2 keep cool - between 2˚C and 8˚C
Total Coliform [bacteria/100ml]	P, G clean, sterile, non-reactive, 125-ml	0.1 ml of 10% Sodium Thiosulfate keep cool - between 2˚C and 8˚C
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
Persistent foam	NA	Measured in the field
Cyanide	P, FP 1,000-ml	NaOH > pH12 0.1 ml of 10% Sodium Thiosulfate 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
Acute Aquatic Toxicity; Luminus Bacteria	G,FP 1,000-ml	keep cool - between 2˚C and 8˚C
Acute Aquatic Toxicity; Fish Egg		
Acute Aquatic Toxicity; Daphne		
Acute Aquatic Toxicity; Algae		
Total Hydrocarbon	Glass, wide mouth FP lined lid 1,000-ml	HCl or H_2SO_4 < pH 2 keep cool - between 2˚C and 8˚C
Carbon disulfide	Three x 40-ml amber VOA vial no head-space	HCl < pH 2 keep cool - between 2˚C and 8˚C
Chloride	P,G,FP 100-ml	keep cool - between 2˚C and 8˚C

Sulfate	P,G,FP 100-ml	keep cool - between 2˚C and 8˚C
Shipping temperature indicator bottle	calibrated thermometer to +/- 1C˚	room temperature water
Metals		
Antimony, Chromium-total	P, G, FP acid washed 250-ml	HNO_3 < pH 2 keep cool - between 2˚C and 8˚C
Cobalt, Copper, Nickel		
Silver, Zinc, Arsenic		
Cadmium, Lead		
Chromium-Hexavalent (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
Mercury	P, G, FP acid washed 500-ml	HNO_3 < pH 2 keep cool - between 2˚C and 8˚C
ZDHC MRSL V1.1		
AP and APEOs: Including All Isomers	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
Dyes – Azo (Forming Restricted Amines)	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
Flame Retardants	G 1,000-ml FP lined lid	keep cool - between 2˚C and 8˚C
Glycols	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 head-space	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
Perfluorinated and Polyfluorinated Chemicals (PFCs)	P 1,000-ml no FP lined lid	keep cool - between 2˚C and 8˚C
Otho-Phthalates – Including all ortho esters of phthalic acid	G 1,000-ml FP lined lid	keep cool - between 2˚C and 8˚C
Polycyclic Aromatic Hydrocarbons	G 1,000-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 H_2SO_4 or HNO_3 < pH 2 keep cool - between 2˚C and 8˚C

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

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

3. A temperature indicator bottle shall be included with each shipping container. The temperature indicator bottle will be clearly labelled.

4. Field blanks shall be collected for the following parameters:
   a. Total-P
   b. Coliform Bacteria
   c. Mercury
   d. Halogenated Solvents
   e. Volatile Organic Compounds (VOC)

5. The field blanks shall use the same containers as the samples and will be filled with ultra-pure laboratory grade water (ASTM D1193 or ISO 3696).

3.1.4 Wastewater Sample Custody

Each sample shipment requires a Chain of Custody log that is 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 to protect the integrity of the sample by ensuring only authorised persons have custody of the sample. An example Chain of Custody form is presented in *Appendix A*. The sampling team must maintain physical custody, or use custody seal tape on the sample cool boxes

3.1.5 Wastewater Sample Shipments

1. Use an a 24-hour (overnight) delivery service for samples that are shipped to a laboratory.

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

3. *Appendix B* provides sample shipping forms that may help to avoid delays in customs.

4. Samples will 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 duration of transportation. Shipping containers that leak fluid, such as melting ice water, will likely be returned to the shipper.

5. The sample cool boxes must be sealed with custody tape that is signed and dated by the sampling crew.

6. The sampler shall be responsible for all handling, processing, and custody of the samples, including taking samples to the nearest servicing airport, bus station, or other carrier.

3.2.0 Laboratory Analysis/Testing for Wastewater

3.2.1 Wastewater 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.
    a. The laboratory shall use the shipping container temperature indicator bottle reading as the sample temperature.
    b. To determine the temperature, invert the bottle several times, remove the cap, and insert a calibrated thermometer.
    c. Allow a minimum of three (3) minutes, but not 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.
    d. If a temperature indicator bottle is not present in the shipping container, an alternative means of determining shipping container temperature can be used.
    e. Under no circumstances shall a thermometer or any other device be inserted into a sample bottle for the purpose of determining shipping container temperature. However, a small aliquot of the sample removed from the container can be used for measuring temperature. This small aliquot must be discarded and not returned to the sample container.
    f. Other devices, such as an infrared thermometer, which can measure temperature may be used if they are calibrated to ±1°C.
    g. The desired sample temperature when it is received at the laboratory is between 2°C and 8°C.
        i. If the sample temperature is greater than 8°C and less than 10°C, the laboratory shall note the issue and provide an exception report with the sample test report.
        ii. 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. The 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.
    a. Measure pH using test strips, an electronic hand-held 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.
    b. Under no circumstances shall a strip or any device be inserted into a sample bottle for the purpose of determining pH.

4. 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.

3.2.2 Wastewater Sample Holding Time

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

2. *Figure 3* presents both the recommended and maximum holding times for each of the wastewater testing parameters.

3. If a sample exceeds the maximum holding time the test results will be reported. However, any test results that exceed the maximum holding time must be flagged with the following data qualifier: "Maximum holding time exceeded. Red flag in the ZDHC Gateway - Wastewater Module. Probable error in results due to the holding time."

Figure 3: Wastewater Sample Holding Times

Wastewater Parameter	Recommended Holding Time	Maximum Holding Time
Conventional		
Temperature	measure in the field	15 minutes
Total Suspended Solids (TSS)	24 hours	7 days
Total Dissolved Solids (TDS)	24-hours	7-days
COD	7 days	28 days
Total-N	-	28 days
pH	measure in the field	6 hours
Color [m-1] (436nm; 525nm; 620nm)	-	48 hours
BOD-5 day	6 hours	48 hours
Ammonia-N	7 days	28 days
Total-P	-	28 days
AOX	-	6 months
Oil and Grease	-	28 days
Phenols	24 hours	28 days
Total Coliform [bacteria/100ml]	6 hours	24 hours
E.coli	6 hours	24 hours
Persistent foam	measure in the field	-
Cyanide	24 hours	14 days
Sulfide	-	7 days
Sulfite	-	48 hours
Aquatic Toxicity; Luminus Bacteria, Fish egg, Daphne, or Algae	24 hours	48 hours
Total Hydrocarbon	-	28 days
Carbon disulfide	7 days	14 days
Chloride	-	28 days
Sulfate	-	28 days

Metals		
Trace Metals	28 days	6 months
Chromium-Hexavalent (VI)	24 hours	28 days
Mercury	-	28 days
ZDHC MRSL V1.1		
Alkylphenol (AP) and Alkylphenol Ethoxylates (APEOs): Including All Isomers	-	**Extraction:** 7-days from collection **Analysis:** 40-days from extraction
Chlorobenzenes and Chlorotoluenes	-	
Chlorophenols	-	
Dyes – Azo (Forming Restricted Amines)	-	
Dyes – Carcinogenic or Equivalent Concern	-	
Dyes – Disperse (Sensitising)	-	
Flame Retardants	-	
Glycols	-	
Halogenated Solvents	7 days	14 days
Organotin Compounds	24 hours	**Extraction:** 7-days from collection **Analysis:** 40-days from extraction
Perfluorinated and Polyfluorinated Chemicals (PFCs)	-	
Otho-Phthalates – Including all ortho esters of phthalic acid	-	
Polycyclic Aromatic Hydrocarbons (PAHs)	-	
Volatile Organic Compounds (VOC)	7 days	14 days

3.2.3 ZDHC Wastewater Parameters

The wastewater parameters, and the individual compounds for each parameter class, are listed in the ZDHC Wastewater Guidelines.

3.2.4 Standard Test Methods for Wastewater

1. The ZDHC approved standard test methods are presented in the ZDHC Wastewater Guidelines, *Appendix A Tables 1A-1B and Tables 2A-2N.*

2. The methods are based on requirements in the European Union, the United States of America, India, and China. Alternate methods, such as those required by the regulatory agency in the region where the wet processing/manufacturing occurs, may be used with prior review from ZDHC, with the exceptions of colour and chemical oxygen demand (COD).

3. The specified ZDHC methods for colour and Chemical Oxygen Demand must be used, there are no exceptions. For more information refer to ZDHC Wastewater Guidelines.

4. Testing for persistent foam.
    - Foam is a naturally occurring phenomena in aeration basins that enable biological wastewater treatment. To ensure accurate accounting of foam, sampling personnel are expected to take a digital photo of the foam they witness within the wastewater treatment system. They should include photographs of the foam they witnessed in the final laboratory report, along with the time and date of taking such photos.
    - The presence of foam is acceptable and meets the requirements of the Guidelines if its colour is similar to the liquid in the aeration basin, dissipating, no thicker than 45 centimetres by visual estimation, and is contained within the aeration basin.
    - If the foam is higher than 45 centimetres by visual estimation or is not contained within the aeration basin, then it does not meet the requirements of the Guidelines.

3.2.5 Required Reporting Limits for Wastewater

1. The minimum required reporting limits are presented in the ZDHC Wastewater Guidelines. These were established with consideration to achieving these levels with good laboratory practices. If the ZDHC reporting limits cannot be achieved, an alternate method or a sub-contract laboratory must be used.

2. The Method Detection Limit (MDL) for each parameter must be determined first and is used to establish the reporting limit. The MDL will be established using ISO/TS 13530:2009 4.4.3.

3. The Reporting Limit (RL) will be established using ISO/TS 13530:2009 4.4.7.

4. Non-detected test results will be reported using the calculated RL for each parameter and not the ZDHC discharge limit.

3.2.6 Quality Systems

1. The minimum quality assurance measures for organic chemical analysis of wastewater and sludge are presented in *Figure 4*.

Figure 4: Minimum Quality Assurance Measures for Organic Chemical Analysis

Measure	Description	Target value	Fre-quency	Points to be checked (examples)
A. Routine				
Method blank	Covering sample preparation and measurement	< MDL	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 re-calibrate, stability of standards
Internal Standard	For GC methods. Substance with physio-chemical properties similar to the analyte	50 – 150%	every sample	Correction of injection error. Method specific: surrogate as alternative
Surrogate	Substance which has similar physio-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 under-going complete process	< 35 % RPD	1 per batch and every 20 samples	Reproducibility of method
Matrix Spike Matrix Spike Duplicate	spike onto sample matrix (duplicate) undergoing complete process	± 20 % recov-ery and < 35 % RPD	1 per batch and every 20 samples	Parameter recovery from sample matrix and reproducibility

B. For method validation				
Multi-Point Calibration	Min. 5-point calibration excl. origin	$r^2 \geq 0.995$	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	MDL RL

Note:

- General suitability of the analytical approach is demonstrated via method validation.
- Matrix to be encountered for wastewater and sludge analysis is less diverse/concentrated than for process chemicals (ZDHC MRSL).
- Therefore, initial method validation is deemed to sufficiently cover most sample types and a comparatively leaner 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 local authority shall prevail.

2. The quality assurance testing in *Figure 5* is required for each of the ZDHC wastewater parameters. This data may be requested and used by ZDHC to assess data quality and validate the analytical results.

Figure 5 – Required Quality Assurance Testing for Wastewater

DQI	Field Blank	Method Blank	Calibration Check*	Lab duplicate	ISTD and Surrogate
Frequency	1 per Batch	1 per Batch	1 per Batch	1 in 20	Every Sample
DQO	< MDL	< MDL	+/- 20%	+/- 35%	Method Specific
Conventional					
Temperature	-	-	-	-	-
Total Suspended Solids (TSS)	-	-	X	X	-
Total Dissolved Solids (TDS)	-	-	X	X	-
COD	-	X	X	X	-
Total-N	-	X	X	X	-
pH	-	-	X	-	-
Colour [m-1] (436nm; 525; 620nm)	-	X	X	-	-
BOD-5 day	-	X	X	-	-
Ammonia-N	-	X	X	X	-
Total-P	X	X	X	X	-
AOX	-	X	X	X	-
Oil and Grease	-	X	X	X	-
Phenols	-	X	X	X	-
Total Coliform [bacteria/100ml]	X	X	-	X	-
E.coli	X	X	-	X	-
Persistent foam	-	-	-	-	-
Cyanide	-	X	X	X	-
Sulfide	-	X	X	X	-
Sulfite	-	X	X	X	-

Aquatic Toxicity; Luminus Bacteria, Fish egg, Daphne, or Algae	-	X	Reference Toxicant	X	-
Total Hydrocarbon	-	X	X	X	-
Carbon disulfide	X	X	X	X	X
Chloride	-	X	X	X	-
Sulfate	-	X	X	X	-
Metals					
Metals	-	X	X	X	X
Chromium-Hexavalent (VI)	-	X	X	X	X
Mercury	X	X	X	X	X
ZDHC MRSL V1.1					
Alkylphenol (AP) and Alkylphenol Ethoxylates (APEOs): Including All Isomers	-	X	X	X	X
Chlorobenzenes and Chlorotoluenes	-	X	X	X	X
Chlorophenols	-	X	X	X	X
Dyes – Azo (Forming Restricted Amines)	-	X	X	X	X
Dyes – Carcinogenic or equivalent Concern	-	X	X	X	X
Dyes – Disperse (Sensitising)	-	X	X	X	X
Flame Retardants	-	X	X	X	X
Glycols	-	X	X	X	X
Halogenated Solvents	X	X	X	X	X
Organotin Compounds	-	X	X	X	X
Perfluorinated and Polyfluorinated Chemicals (PFCs)	X	X	X	X	X
Otho-Phthalates – Including all ortho esters of phthalic acid	-	X	X	X	X
Polycyclic Aromatic Hydrocarbons (PAHs)	-	X	X	X	X
Volatile Organic Compounds (VOC)	X	X	X	X	X

* calibration check conducted with a second source standard

3.2.7 Wastewater Sample Storage

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

2. The samples may be disposed of 60 days after the final laboratory report is issued.

3.3.0 Reporting and Deliverable Requirements

Reporting[1] here means the submission of test data/results onto the ZDHC Gateway – Wastewater Module by ZDHC Accepted Laboratories on behalf of suppliers. It is vital that the integrity of the test data in the Gateway is at its highest possible standard. This will, among other things, support the ZDHC Roadmap to Zero Programme in making scientific and data driven decisions necessary to shape the future of our industry.

1. All test data must be reported using the ZDHC Gateway Electronic Data Reporting (**EDR**) System. The EDR guidelines and reporting template are publicly available on the ZDHC website.

2. The use of reporting formats other than those approved in the EDR will be deemed non-conformance. Such data is unacceptable and resubmission in the specified format will be required.

3. 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, available on the ZDHC website.

[1] For more information please read the latest version of the ZDHC Wastewater Guidelines and the ZDHC Gateway User Terms and Conditions document.

3.4.0 Data Validation

- ZDHC routinely requests laboratory QA/QC information from randomly selected ZDHC Accepted Laboratories. The following information may be requested for data review:
    a. Calibration Curves
    b. Method Blank Instrument Reports
    c. Instrument Calibration Check Reports
    d. Laboratory Control Sample Instrument Reports
    e. Laboratory Duplicate Instrument Reports
    f. Matrix Spike and Matrix Spike Duplicate Instrument Reports
    g. 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.

4.0.0 Sludge

4.1.0 Sampling for Sludge

4.1.1 Sludge Sampling Points/Locations

1. Please refer to the ZDHC Wastewater Guidelines (WWG) for the definition of sludge and the main purpose of testing sludge. Depending on the chosen testing option described in the WWG Section 9, sludge samples may be collected.

2. The suppliers must identify, to qualified sampling personnel, the points where sludge is generated and stored at the Supplier. The qualified sampling personnel will make a final determination as to the proper sampling locations.

3. Sludge samples will be collected to representative all of the sludges generated by the Supplier's ETP (Effluent Treatment Plant) that are disposed of or reused.

4. It may be necessary to collect various types of sludge samples throughout a given Supplier's ETP, including:

    a. Any Liquid Sludge Waste (not to be confused with wastewater) containing low or high amounts of solids.
    b. Anaerobically Digested Sludge
    c. Aerobically Digested Sludge
    d. De-watered Sludge Cake
    e. Primary Sludge
    f. Tertiary Sludge
    g. Biomass Sludge (Secondary Sludge)
    h. Compost Product
    i. Dried Sludge Solid Waste
    j. Incinerator Ash

4.1.2 Sludge Sample Collection

1. Samples shall be taken by ZDHC trained and accredited samplers only. Please refer to the ZDHC Academy for training information.

2. Samples must be taken during a time that represents continuous and normal production and continuous and normal wastewater treatment. Collect sludge samples in a way that represents the entire production cycle. This may take a shorter or longer time than the specified 6-hour composite time.

3. Start the production cycle sludge 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 sludge sampling will start at 11:00 AM.

4. Sludge samples shall be collected as composite samples following: USEPA 833-B-89-100 "POTW Sludge Sampling and Analysis Guidance Document". The ISO 5667- 13 "Guidance on Sampling Sludge" document also provides reference sludge sampling guidance and more detailed information on sampling devices.

5. The most appropriate way of sampling in any situation depends on several factors:
    a. safe access to the sampling point by personnel.

b. the practicality of installing and maintaining automatic equipment, if appropriate.
c. the practicalities of safely interrupting a stream of moving liquid sludge or cake when manually sampling and the nature of the chamber or tank design with respect to stratification of liquid sludges.

6. 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[2]. Therefore, manual composite sampling may be required.

7. ISO 5667-13 describes multiple sample collection devices.

8. For collecting solid sludge samples[3]:
    a. 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 and not just from the surface layer.
    b. For either 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.
    c. 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.
    d. When sampling drying beds, divide each bed into quarters. From the center 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.
    e. 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. For collecting liquid sludge samples:
    a. It should be noted that thin liquid sludges (of lower solids content) require the preparation of relatively large volumes of the sampled material to provide sufficient dry matter to facilitate a truly representative analysis of constituents, e.g. metals. The analyst should always be consulted as to the quantities of sludge required, and the sample reduced accordingly in the field before returning to the laboratory[4].
    b. All of the laboratory sample containers will 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 QA samples. The sampler shall coordinate extra volume requirements with the laboratory.
    c. Collect samples in a manner that represents the entire flow at the sampling point over the entire sampling period[5].
    d. These procedures should be followed when sampling from a tap[6]:
        - 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.
    e. Before drawing a sludge sample, rinse each piece of sampling equipment three (3) times with sample to reduce the chance of contamination from the previous grab.
    f. To prevent solids separation in the sample, use glass, PTFE-coated stirring rods, or stainless-steel spoons to mix the sample before splitting or transferring any portion of it to another container.
    g. 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.
    h. 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 result in solids separation and settling, and affect sample solids content,

[2]USEPA 833-B-89-100: POTW Sludge Sampling and Analysis Guidance Document
[3]ISO 5667- 13: Guidance on Sampling Sludge

[5]USEPA 833-B-89-100: POTW Sludge Sampling and Analysis Guidance Document
[6]USEPA 833-B-89-100: POTW Sludge Sampling and Analysis Guidance Document
[4]ISO 5667- 13: Guidance on Sampling Sludge

depending on the location of the tap (top, side or bottom of the pipe).

i. 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.

j. If the sludge solids tend to separate into different fractions care must be given to mixing the samples adequately to obtain a representative sample. If the fractions cannot be mixed, then separate samples are to be collected. This is because some pollutant parameters are predominantly associated with the solid fraction while others are associated with the liquid phase.

10. Sampling equipment must be made of materials which will not contaminate or react with the sludge. The best material choices are glass and stainless steel because they are relatively inert.[7]
11. Sludge flux (weight/time) and/or flow data volume/time, if available, must be collected and reported with the analytical results.

12. 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.
- relocation of parts of solid sludge piles where needed to access deeper layers.
- provision of safety gear and warnings on any specific hazards present.

[7]USEPA 833-B-89-100: POTW Sludge Sampling and Analysis Guidance Document

4.1.3 Sludge Sample Containers and Preservatives

1. *Figure 6* presents standard sample collection containers and preservatives for sludge samples generated from a wastewater/effluent treatment plant.

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 being used at the laboratory.

3. Caution should be exercised since 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.[8]

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.[9]

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 consists of keeping the temperature between 2°C and 8°C.

[8]ISO 5667- 13: Guidance on Sampling Sludge
[9]USEPA 833-B-89-100: POTW Sludge Sampling and Analysis Guidance Document

Figure 6: Sludge Sample Containers and Preservatives

Sludge Parameter	Sample Container Minimum Size	Standard Preservative (verify with lab method to be used)
Anions		
Cyanide	P, G 1,000-ml wide mouth PTFE lined lid	NaOH > 12 pH, < 13 pH, cool < 4˚C approx 2-ml 10N NaOH
Metals		
Trace Metals	P, G acid washed 1,000-ml wide mouth	HNO_3 < pH 2
Chromium-Hexavalent (VI)	P, G acid washed 300-ml wide mouth	cool < 4˚C
Mercury	P, G acid washed 500-ml wide mouth	HNO_3 < pH 2 cool < 4˚C
ZDHC MRSL V1.1		
Alkylphenol (AP) and Alkylphenol Ethoxylates (APEOs): Including all Isomers	Three G 1,000-ml PTFE lined lid wide mouth	0.008% $Na_2S_2O_3$ V/W cool < 4˚C
Chlorobenzenes and Chlorotoluenes		
Dyes – Azo (Forming Restricted Amines)		
Dyes – Carcinogenic or Equivalent Concern		
Dyes – Disperse (Sensitising)		
Flame Retardants		
Glycols		
Otho-Phthalates – Including all ortho esters of Phthalic Acid		
Polycyclic Aromatic Hydrocarbons (PAHs)		
Perfluorinated and Polyfluorinated Chemicals (PFCs)	G 1,000-ml NO PTFE lining wide mouth	0.008% $Na_2S_2O_3$ V/W cool < 4˚C
Chlorophenols	G 1,000-ml PTFE lining wide mouth	H2SO4, pH <2 0.008% $Na_2S_2O_3$ V/W, cool < 4˚C
Organotin Compounds	G 1,000-ml acid washed PTFE lining wide mouth	keep cool between 2˚C and 8˚C
Halogenated Solvents	Three x 40-ml VOA vial, no headspace	1:1 HCl, HNO_3, or H_2SO_4 pH <2 keep cool between 2˚C and 8˚C
Volatile Organic Compounds (VOC)		
ZDHC MMCF		
Heavy metals	P, G 1000 ml wide mouth	As the dry solid sludge samples will be taken, preferably sludge cakes, there is no need for preservation of the sample. Sludge samples after collection shall be kept cool until sample preparation. Reference samples, if indicated, shall be stored in a climatized storeroom.
Total Organic Carbon, TOC	G, 300 ml wide mouth	
Extractable Organic Halogen Compounds, EOX	G, 300 ml wide mouth	
Adsorbable Organic Halogen Compounds, AOX, if requested	G, 300 ml wide mouth	

P= plastic, G= amber glass

4.1.4 Sludge Sample Custody

Each sample shipment requires a Chain of Custody log that is 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 to protect the integrity of the sample by ensuring only authorised persons have custody of the sample. An example Chain of Custody form is presented in *Appendix A*. The sampling team must maintain physical custody, or use custody seal tape on the cool boxes, until the samples are handed off to the shipping company or laboratory.

4.1.5 Sludge Sample Shipments

1. Use a 24-hour (overnight) delivery service for samples that are shipped to a laboratory.

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

3. *Appendix B* provides sample shipping forms that may help to avoid delays in customs.

4. Samples will 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 duration of transportation. Shipping containers that leak fluid, such as melting ice water, will likely be returned to the shipper.

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

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

7. The sampler shall be responsible for all handling, processing, and custody of the samples, including taking samples to the nearest servicing airport, bus station, or other carrier.

4.2.0 Laboratory Analysis/Testing for Sludge

4.2.1 Sludge 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.
    a. The laboratory shall use the shipping container temperature indicator bottle reading as the sample temperature.
    b. To determine the temperature, invert the bottle several times, remove the cap, and insert a calibrated thermometer.
    c. Allow a minimum of three (3) minutes, but not 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.
    d. If a temperature indicator bottle is not present in the shipping container, an alternative means of determining shipping container temperature can be used.
    e. Under no circumstances shall a thermometer or any other device be inserted into a sample bottle for the purpose of determining shipping container temperature.
    f. Other devices, such as an infrared thermometer, which can measure temperature may be used if they are calibrated ±1°C.
    g. The desired sample temperature when it is received at the laboratory is 2°C to 8°C.
        i. If the sample temperature is greater than 4°C and less than 10°C, the laboratory shall note the issue and provide an exception report with the sample test report.
        ii. 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. 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.

4.2.2 Sludge Sample Holding Time

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

2. *Figure 7* presents both the recommended and maximum holding times for each of the sludge testing parameters.

3. If a sample exceeds the maximum holding time the test results will be reported. However, any test results for samples that exceeded the maximum holding time must be flagged with the following data qualifier: "Maximum holding time exceeded. Red flag in the ZDHC Gateway – Wastewater Module. Probable error in results due to the holding time"

Figure 7 – Sludge Sample Holding Time

Sludge Parameter	Recommended Holding Time	Maximum Holding Time
Anions		
Cyanide	24 hours	14 days
Metals		
Trace Metals	28 days	6 months
Chromium-Hexavalent (VI)	24 hours	28 days
Mercury	-	28 days
ZDHC MRSL V1.1		
Alkylphenol (AP) and Alkylphenol Ethoxylates (APEOs): Including all Isomers	-	**Extraction:** 7-days from collection **Analysis:** 40-days from extraction
Chlorobenzenes and Chlorotoluenes	-	
Chlorophenols	-	
Dyes – Azo (Forming Restricted Amines)	-	
Dyes – Carcinogenic or Equivalent Concern	-	
Dyes – Disperse (Sensitising)	-	
Flame Retardants	-	
Glycols	-	
Halogenated Solvents	7 days	14 days
Organotin Compounds	24 hours	7 days
Perfluorinated and Polyfluorinated Chemicals (PFCs)	-	**Extraction:** 7-days from collection **Analysis:** 40-days from extraction
Otho-Phthalates – Including all ortho esters of Phthalic Acid	-	
Polycyclic Aromatic Hydrocarbons (PAHs)	-	
Volatile Organic Compounds (VOC)	7 days	14 days
ZDHC MMCF		
Heavy metals	28 days	6 months
Total Organic Carbon, TOC	7 days	14 days
Extractable Organic Halogen Compounds, EOX	7 days	14 days
Adsorbable Organic Halogen Compounds, AOX, if requested	7 day	14 days

4.2.3 ZDHC Sludge Parameters

The sludge parameters are listed in the ZDHC Wastewater Guidelines.

4.2.4 Required Reporting Limits and Standard Test Methods for Sludge

1. The ZDHC approved standard analytical/test methods and required reporting limits for each of the sludge parameters are presented in the ZDHC Wastewater Guidelines.

2. All test results will be reported on a dry-weight basis.

3. The required reporting limits were established with consideration to achieving these levels with good laboratory practices.

4. The methods are based on requirements in the European Union, the United States of America, India, and China. Alternate methods, such as those required by the regulatory agency in the region in which the wet manufacturing occurs, may be used with prior ZDHC review.

4.2.5 Quality Systems

1. The minimum quality assurance measures for organic chemical analysis of wastewater and sludge are presented in *Figure 4*.
2. The quality assurance testing in *Figure 8* is required for each of the ZDHC sludge parameters. This data will be reported with the sample results and used by ZDHC to assess data quality and validate the analytical data

Figure 8: 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	< MDL	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 re-calibrate, stability standards	
Internal Standard	For GC methods. Substance with physico-chemical properties similar to the analyte	50 – 150%	Every sample	Correction of injection error. Method specific: surrogate as alternative	Choice of internal standard or surrogate or a combination thereof 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	

B. In case of problematic samples (e.g. internal standard recovery too low, phase separation issues etc.)				
Duplicate	Duplicate undergoing complete process	< 35 % RPD	Problematic samples only	Flag results
Matrix Spike Matrix Spike Duplicate	Spike onto sample matrix (duplicate) undergoing complete process	Method specific	Problematic samples only	Flag results
C. For method validation				
Multi-Point Calibration	Min. 5-point calibration excl. origin	$r^2 \geq 0.995$	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	Reference to ZDHC Wastewater Guidelines; various methodologies can be applied.	≤ WWG RL Ideal: ≤ ½ WWG RL	Validation	Sensitivity, LoD, LoQ

Note:

- General suitability of the analytical approach is demonstrated via method validation.
- Matrix to be encountered for wastewater and sludge analysis is less diverse/concentrated than for process chemicals (ZDHC MRSL).
- Therefore, initial method validation is deemed to sufficiently cover most sample types and a comparatively leaner QC program 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 local authority shall prevail.

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

Figure 9 – Required Quality Assurance Testing for Sludge

Parameter	Field Blank	Method Blank	Calibration Check*	Lab Duplicate	ISTD and Surrogate
Anions					
Cyanide	-	X	X	X	-
Metals					
Trace Metals	-	X	X	X	X
Chromium-Hexavalent (VI)	-	X	X	X	-
Mercury	X	X	X	X	-
ZDHC MRSL V1.1					
Alkylphenol (AP) and Alkylphenol Ethoxylates (APEOs): Including all Isomers	-	X	X	X	X

* calibration check conducted with a second source standard

Chlorobenzenes and Chlorotoluenes	-	X	X	X	X
Chlorophenols	-	X	X	X	X
Dyes – Azo (Forming Restricted Amines)	-	X	X	X	X
Dyes – Carcinogenic or Equivalent Concern	-	X	X	X	X
Dyes – Disperse (Sensitising)	-	X	X	X	X
Flame Retardants	-	X	X	X	X
Glycols	-	X	X	X	X
Halogenated Solvents	X	X	X	X	X
Organotin Compounds	-	X	X	X	X
Perfluorinated and Polyfluorinated Chemicals (PFCs)	X	X	X	X	X
Otho-Phthalates – I ncluding all ortho esters of Phthalic Acid	-	X	X	X	X
Polycyclic Aromatic Hydrocarbons (PAHs)	-	X	X	X	X
Volatile Organic Compounds (VOC)	X	X	X	X	X
ZDHC MMCF					
Heavy metals	Mercury	x	x	x	x
Total Organic Carbon, TOC	_	x	x	x	_
Extractable Organic Halogen Compounds, EOX	_	x	x	x	_
Adsorbable Organic Halogen Compounds, AOX, if requested	_	x	x	x	_

4.2.6 Sludge Sample Storage

1. All samples will be stored under custody at between 2°C to 8°C (unless otherwise specified by the analytical method).

2. The samples may be disposed of 60 days after the final laboratory report is issued.

4.3.0 Reporting and Deliverable Requirements

Reporting[11] here means the submission of test data/results onto the ZDHC Gateway – Wastewater Module by ZDHC Accepted Laboratories on behalf of suppliers. It is vital that the integrity of test data in the Gateway is at its highest possible standard. This will, among other things, support the ZDHC Roadmap to Zero Programme in making scientific and data driven decisions necessary to shape the future of our industry.

1. All test data must be reported using the ZDHC Gateway Electronic Data Reporting (**EDR**) System. The EDR guidelines and reporting template are publicly available on the ZDHC website.

2. The use of reporting formats other than those approved in the EDR will be deemed non-conformance. Such data is unacceptable and resubmission in the specified format will be required.

3. 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, available on the ZDHC website.

[11]For more information please read the latest version of the ZDHC Wastewater Guidelines and the ZDHC Gateway User Terms and Conditions document.

4.4.0 Data Validation

- ZDHC routinely requests laboratory QA/QC information from randomly selected ZDHC Accepted Laboratories. The following information may be requested for data review:

    a. Calibration Curves
    b. Method Blank Instrument Reports
    c. Instrument Calibration Check Sample Reports
    d. Laboratory Control Sample Instrument Reports
    e. Laboratory Duplicate Instrument Reports
    f. Matrix Spike and Matrix Spike Duplicate Instrument Reports
    g. 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.

Appendix A
Example Chain of Custody Form

CHAIN of CUSTODY

Page ___ of ___

Facility Name:	Project: **ZDHC Wastewater Evaluation**	Method of Shipment
Facility Address:		
Fcility Contact:	Sampler's Name: / Sampler's Contact Info:	Special Detection Limit/Reporting

Sample I.D.	Lab Sample No.	No. of Containers	Matrix: Soil	Matrix: Water	Matrix: Air	Matrix: Other	Prsv.: Yes	Prsv.: No	Sampling Date	Sampling Time													Turn Around Time (working days)	REMARKS

Please Refrence the ZDHC Wastewater Guide for required standard test methods and reporting limits.

Sample Received Intact: Yes No		Temperature received: Ice No ice	
Relinq. by sampler (Sign & Print Name)	Date Time	Received by (Sign & Print Name)	Lab Work No.
Relinquished by	Date Time	Received by	
Relinquished by	Date Time	Received by	
Relinquished by	Date Time	Received by laboratory	Date Time

Appendix B
Shipping and Customs Forms

If the following forms are completed and accompany the samples during international shipment, the chance for shipping delays in customs will be minimised:

1. Safety Data Sheet (SDS) for water samples. This illustrates that the wastewater samples are not some type of known hazardous material. Search on Google to find some publicly available examples.

2. Customs declaration forms for various countries, such as the one presented below, can be found at this link: https://www.bing.com/images/search?q=Customs+Declaration+Form+PDF&FORM=IDINTS

CUSTOMS DECLARATION	May be opened officially	CN 22
Designated operator		Important! See instructions on the back
☐ Gift ☐ Documents ☐ Commercial sample ☐ Other		Tick one or more boxes
Quantity and detailed description of contents (1)	Weight (in kg)	Value (3)
For commercial items only If known, HS tariff number (4) and country of origin of goods (5)	Total weight (in kg) (6)	Total value (7)
I, the undersigned, whose name and address are given on the item, certify that the particulars given in this declaration are correct and that this item does not contain any dangerous article or articles prohibited by legislation or by postal or customs regulations. Date and sender's signature (8)		

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.

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