Wastewater Sampling Procedures, Protocols and Approved Methods for Laboratory Analysis

Speaker:
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“Thousands have lived without love, not one without water.”

-W.H. Auden
Sampling Definition, Objective and Purpose
What is sampling?

the collection of truly representative samples, which when analyzed, yield results reflecting the actual quality of the water samples
What is the objective of sampling?

To collect a portion of material small enough in volume to be transported conveniently and handled in the laboratory while still accurately representing the material being sampled.
What is the purpose of sampling?

• compliance with regulatory requirements
• evaluation of performance efficiency of WTP system
• evaluation of effectiveness of in-house waste minimization measure/s
Sampling Stations
Influent

- raw and untreated wastewater
- collected at points where the flow is highly turbulent and upstream or above the plant return lines
Effluent

- treated or untreated wastewater
- collected at (the) most representative site downstream from all entering wastewater prior to the discharge into the receiving body of water
Influent vs. Effluent

Basic WTP/STP System

(for illustration purposes only)
Outfall

• the discharge point of a waste stream into a body of water
Types of Samples
Grab or Catch

- a sample collected at a particular time and place that can represent only the composition of the source at that time and place
Composite

- a combination of grab samples collected at the same sampling point at different times
Split Samples

- samples taken from the same sample container after thoroughly mixing or compositing the sample
Safety and Health Considerations
Hazards

Review the work area and assess the prevalence and severity of the four types of hazards:
Hazards: Physical

- physical hazards such as slipping, tripping, falling, suffering muscle strain, or encountering power lines
Hazards: Chemical

- chemical hazards such as acute or chronic exposure to toxic compounds
Hazards: Biological

- biological hazards such as anthropods or water-borne diseases or diseases spread by other disease-causing organisms
Hazards: Weather

- weather hazards such as heavy rains, strong winds, lightning or excessive heat
Hazards

Identify potential hazards and seek to diminish their severity whenever possible.
To reduce the risk of exposure to pathogens through ingestion or dermal contact, do not carry food or cigarette while sampling and ensure that any skin lesions are protected from possible contact with wastewater.
Personal Protective Equipment
Hard Hat
Face shield

Goggles
Earmuffs
Gloves
Safety Shoes
Tyvek Suit
Respirator Mask
Face Mask
Sample Handling Components
Container Requirements

Plastic or Glass – one may be preferred over the other depending on the type of parameters

Physico-Chemical Parameters: **TSS, BOD, COD, COLOR**

Plastic Gallon
Container Requirements

Plastic or Glass – one may be preferred over the other depending on the type of parameters

Physico-Chemical Parameters: Oil & Grease
Container Requirements

Plastic or Glass – one may be preferred over the other depending on the type of parameters

Physico-Chemical Parameters: **Heavy Metals**

High-Density Polyethylene
Container Requirements

Plastic or Glass – one may be preferred over the other depending on the type of parameters

Physico-Chemical Parameters: Phenols & Nutrients
**Container Requirements**

Plastic or Glass – one may be preferred over the other depending on the type of parameters.

Physico-Chemical Parameters: **Fecal & Total Coliform**

Coliform Bottles

- w/o chlorination
- with chlorination
Preservation Methods

Refrigeration: Cool at 4°C

Chemical Addition:

- Acidification (pH adjust <2)
- Biocides (HgCl solution)
- Special Cases (NaOH for CN analysis)

Ice Chest
Maximum Holding Time

- Determination
- Container
- Minimum Sample Size (mL)
- Sample Type
- Preservation
- Maximum Storage Recommended/Regulation
Please refer to handouts for the complete list of special sampling and handling requirements per determination.
Sample Contamination Prevention Tips
Field measurements should always be made on-site or on (a) separate sub-sample(s).
Sample container(s), new or used, must be cleaned according to the recommended methods.
Only the recommended type of sample container for each parameter should be used.
Water sample containers should be employed for water samples only.
Recommended preservation methods must be followed.
The inner part of sample containers and caps should not be touched with bare hands, gloves, clothing, fabric, etc.
Sample containers which have been sterilized for microbiological sampling must remain sterile until the sample is collected.
Samples must NEVER be left to stand in the sun.
Samples must be submitted to the laboratory as promptly as possible and must not exceed the prescribed holding time.
Effluent Sampling
Considerations in Collecting Effluent Samples:

1. The sampling station must be representative of the discharge facility.
2. Samples should not be collected from stagnant areas.
3. The sampling area should be in a well-mixed and turbulent area and solid settling is minimal.
4. The volume taken must be sufficient for analysis.
Effluent Sampling may be done in 2 general ways:

- using an intermediate container like buckets, pails, dipper, etc.
Effluent Sampling may be done in 2 general ways:

directly with sampling container
Effluent collected at the sampling station prior to discharge into the water body.
Effluent Sampling

« effluent samples being transferred into a plastic gallon
Observe zero head space for BOD analysis.

Effluent Sampling

Effluent samples being transferred into a plastic gallon
Effluent samples being transferred into a wide mouth bottle
For oil & grease analysis, cover mouth of bottle first with aluminum foil before placing plastic cover.
Fecal coliform sample containers which have been sterilized for microbiological sampling must remain sterile until the sample is collected.

Effluent Sampling

Effluent samples being transferred into a coliform bottle
For split sampling, if necessary:

- Pouring of effluent sample into a plastic gallon
For split sampling, if necessary:

- **Effluent Split Sampling**
  - Pouring of effluent sample directly into sampling containers
For split sampling, if necessary:

...
Use permanent marker to label containers to avoid erasures, smearing or blotting.
signing sealed containers of effluent samples

Secure seal of sample containers and affix initials of PCO.

« preparing effluent samples for transport

Effluent Sampling
Effluent Sampling

« preparing effluent samples for transport

Don’t forget the ice!
Chain-of-Custody
What is Chain-of-Custody (CoC)?

Chain-of-Custody (CoC) is the documentation of the possession and handling of samples.
What is the purpose of CoC?

• Creates legal “paper trail”
• Reduces miscommunication
• Provides accurate tracking for systems audits
CoC Components: Field Logbook

- Hardbound
- Clear and complete
- Legal document
- Firsthand sampling info is recorded
- Documents any unusual circumstances
CoC Components: Sample Labels

- I.D. No. (unique to each sample)
- Location No. (may be the same or different from sample ID#)
- Sampling Date (Months/day/year)
- Sampling Time
- Type of analyses requested
- Others/Remarks
CoC Components: Sample Seals

• Detect unauthorized tampering with samples up to the time of analysis
**CoC Components: Analyses Request Sheet**

- accompanies sample to the laboratory
CoC Components: CoC Record

- includes custody on analyses transfer (e.g., signature and time) shipment (container, packaging, sample tracking)
CoC Components: Continuation of CoC

- CoC procedures in the laboratory (receipt of sample, and so on)
Significant Wastewater Control Parameters
Significant Wastewater Control Parameters

- Color
- Temperature
- pH*
- BOD-COD-DO
- Total Suspended Solids (TSS)

- Oil and Grease
- Heavy Metals
- Nutrients
- Coliforms

*pH stands for puissance de l’Hydrogène, a French phrase which literally means “potential/strength of hydrogen.”
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Basic Analyses
Basic Analyses: Gravimetric Method

• This method of water analysis is used for the quantitative determination of an analyte (the ion being analyzed) based on its mass.
Basic Analyses: Titrimetric Method

- This method of water analysis is a laboratory procedure whereby an unknown quantity of a particular substance is measured by adding it to a standard reagent with which it reacts in a definite and known proportion.

Automatic Titrator
Basic Analyses: Spectrophotometric Method

• This method of water analysis measures how much a chemical substance absorbs light by measuring the intensity of light that passes through a sample solution.

Spectrophotometer
Basic Analyses: Potentiometric Method

• This method of water analysis measures pH, conductivity, DO, Ion and other determination/parameter of water quality using electrodes.
Memorandum Circular No. 012 Series of 2016

Guidelines for the Designation of DENR-Recognized Environmental Laboratories

DENR Administrative Order No. 98-63

https://emb.gov.ph/online-services/

Pursuant to the provisions of Presidential Decree No. 984 otherwise known as the “Pollution Control Decree of 1976”, PD No. 1586 establishing the Environmental Impact Statement (EIS) System in the Philippines, and by virtue of Executive Order No. 192, Series of 1987 and Executive Order No. 292 otherwise known as the “Administrative Code of 1987”, the Department of Environment and Natural Resources (DENR), hereby adopts and promulgates the following guidelines for the designation of DENR recognized laboratories.

01. “DENR recognized environmental laboratories” are laboratories that are authorized to generate environmental data in connection with the Environmental Impact Assessment (EIA), Environmental Monitoring, and research activities, and regulations and implementation of policies, and other Management and Protected Areas.
Guidelines for the Designation of DENR-Recognized Environmental Laboratories

DENR Administrative Order No. 98-63

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Certificate of Recognition

Republic of the Philippines
DEPARTMENT OF ENVIRONMENT AND NATURAL RESOURCES
Vicente Avenue, Diliman, Quezon City

This

CERTIFICATE OF RECOGNITION
C.R. No. 034/2008

is hereby granted to

after having been assessed and found to comply with the documentation, analytical performance and other technical requirements of Administrative Order No. 65, Series of 1998. Guidelines for the Designation of DENR Recognized Environmental Laboratories.

This certificate is valid for three years from date of issue unless otherwise revoked or cancelled.

In testimony whereof, I have hereunto signed this Certificate and issued the same this thirty-first day of April, year two thousand and eight at Quezon City, Philippines.

[Signature]

Secretary

ENVIRONMENTAL LABORATORY RECOGNITION PROCESS
Q & A

Speaker: JENNILYN C. VICENTE
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Thank You!

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