

Sampling, Sample Handling and Custody Procedures

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NEORS

Sampling

- Sampling
 - Sampling Methods
 - Bottle Requirements
 - Field Testing
 - Preservation
 - Labeling
 - Chain of Custody
 - Sample Transport
 - Holding Times



Laboratory Receipt

- Chain of Custody
 - Completed Accurately
 - COC vs Samples
 - Holding Times
 - Laboratory Checks
- Anomalies/Documentation
- Sample Login (LIMS)
- Labeling
- Sample Storage



Analysis and Beyond

- Laboratory Analysis
 - Internal Chain of Custody
- Sample Disposal
- Documentation and Record Keeping



Sampling Methods

- Grab Sampling
- Composite Sampling
- Special Sampling

Sampling Method used is usually determined by your Permit or Analysis Method!

Grab Sampling

Grab Sample – an individual sample taken at a single location at a given time.

- It's only representative of the conditions of the sample source at a particular time and location. "Snapshot".
- Some Sample Analysis must be performed on Grab Samples. Examples: Organics (VOC, SVOC), Oil & Grease, Bacteria, Cyanide.

Grab Sampling



Grab Sampling



Grab Samplers (Bailers and Dippers)



Composite Sampling

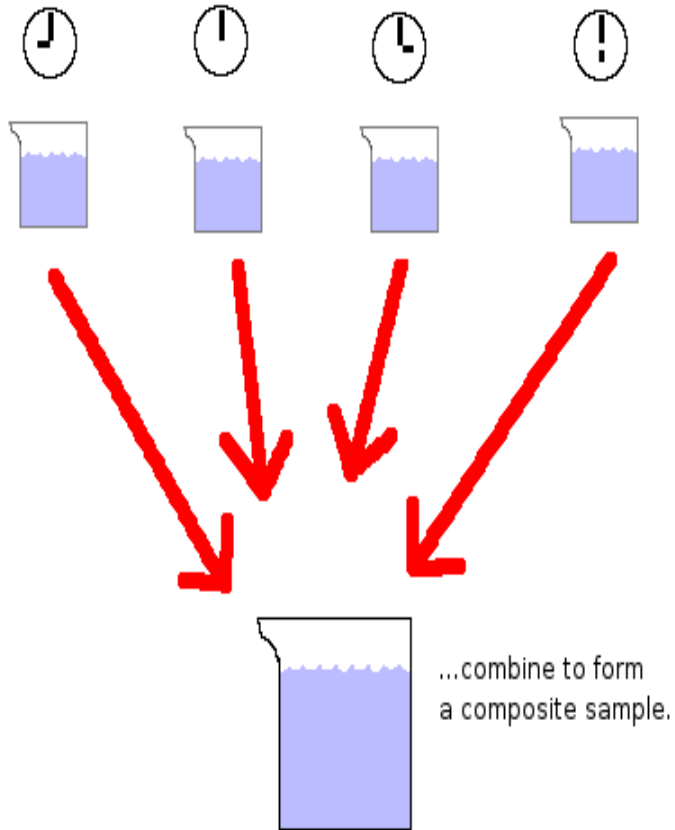
Composite Sample is a collection of numerous discrete samples collected at the same location over a period of time (usually 24 hours).

- Time Composite
- Flow Composite
- Combination of Time and Flow Composite (usually found in most WWTP's)

Examples of Analysis on Composite Samples:
Solids, Nutrients (Nitrogen, Phosphorus), BOD

Composite Sampling

Several grab samples taken at different times
in the same location...



Composite Sampling



Special Sampling - Mercury

Low Level Mercury
Sampling = “Clean Hands/
Dirty Hands” per
Method 1631.

Sampling techniques
outlined specifically
to prevent contamination.



Solid or Sludge Sampling

Sludge or solid sampling using various samplers.

Samples can be analyzed as Grabs or Composites.



Solid, Sludge or Sediment Sampling



Special Sampling

- Bacteria
- Legionella
- Drinking Water:
 - Lead/Copper
 - Nitrate
 - Bacteria

Which Method Should I Use?

The Sampling Method used is based on:

- Regulation (Permits)
- Type of Analysis
- Purpose of Analysis
- Holding Times

Sampling

- The Sample must be Representative.
- Prevent Contamination.
- Sampler safety is always a priority!
- Wear your PPE!

Bottle Requirements

- What kind of bottle should I use?
- What size bottle should I use?
- What analysis is being requested?
- What bottle type/size does the analysis require?
- Is it the sampling method grab or composite?

Bottle Requirements



Bottle Requirements

- Certified Clean Bottles are recommended to prevent contamination.
- Sterile Bottles are mandatory for Bacteria and Legionella.

Bottle Types:

Plastic, Glass (amber or clear),
Vials (amber or clear), Carboys

Bottle Size

40ml, 100ml, 250ml, 500ml, 1000ml, 2-L, 4-L, 9-L, 20-L

***Most Test Methods require specific bottle types and sizes!**

Bottle Types and Volumes

(from Table:1060, Standard Methods Introduction)

<u>Test</u>	<u>Bottle</u>	<u>Volume*</u>	<u>Method</u>
Alkalinity	P, G	200 ml	G, C
BOD	P, G	1000 ml	G, C
Chlorine	P, G	500 ml	G
COD	P, G	100 ml	G, C
CR+6	P, G	300 ml	G
Cyanide	P, G	500 ml	G
Fluoride	P	300 ml	G, C
Metals	P, G	500 ml	G, C
Nutrients	P, G	500 ml	G, C
O&G	G	1000 ml	G
TOC	G	100 ml	G, C
VOC	G	40ml	G

*Some volumes have been reduced significantly with newer technology!

Bottle “Musts”

- Amber Glass (w/TFE Cap)–Organics (SVOC, TPH)
- Glass (w/TFE Cap)– Oil & Grease
- Teflon or Glass – Low Level Mercury 1631
- Sterile Bottles – Bacteria, Legionella
- 40ml Vials – VOC’s (w/TFE cap), TOC
- Plastic – Fluoride, Silica

*Volumes collected are based on the type of analysis and instrumentation being used!

Field Filtering

- Certain analysis requires that the samples be filtered in the field.
- Example: Dissolved Reactive Phosphorus (DRP) and Dissolved Metals require the samples to be field filtered within 15 minutes of sampling through a 0.45 micron filter.
- Samples are preserved (if needed) *after* filtering!



Field Sampling

It's important to know the specific requirements of Permits and Methods when Field Sampling.

Does sampling require? *Field Duplicates*
Field Blanks
Trip Blanks (VOC)

If so, at what frequency? What is the protocol?

Field Testing

- Some analysis should be performed in the Field due to extremely short Holding Times and stability.
- Examples of Field Testing:
 - pH
 - Dissolved Oxygen
 - Conductivity
 - Chlorine
 - Turbidity
- Field Testing can also be used as a screening tool!
- Documentation!!



Field Preservation

- Any analysis requiring preservation should be done in the field.
- First, the sample is collected in the appropriate container, then a specified amount of preservative is added to a specified pH.
- Examples: Nutrients require H_2SO_4 to $\text{pH} < 2$.
Metals require HNO_3 to $\text{pH} < 2$
Cyanide requires NaOH to $\text{pH} > 12$
Most analysis requires immediate refrigeration!



Bottles, Preservation, Volumes and Holding Times

(from Table:1060, Standard Methods Introduction)

<u>Test</u>	<u>Bottle</u>	<u>Volume</u>	<u>Method</u>	<u>Preservation</u>	<u>Holding Time</u>
Alkalinity	P, G	200 ml	G, C	Refrigeration	14 days
BOD	P, G	1000 ml	G, C	Refrigeration	48 hrs
Chlorine	P, G	500 ml	G	Analyze Immediately	0.25 hr
COD	P, G	100 ml	G, C	H ₂ SO ₄ pH<2, Refrig.	28 days
CR+6	P, G	300 ml	G	Refrigeration	24 hrs
Cyanide	P, G	500 ml	G	NaOH pH>12, Refrig.	14 days
Fluoride	P	300 ml	G, C	Refrigeration	28 days
Metals	P, G	500 ml	G, C	HNO ₃ pH<2	6 mos
Nutrients	P, G	500 ml	G, C	H ₂ SO ₄ pH<2, Refrig.	28 days
O&G	G	1000 ml	G	HCL pH<2, Refrig.	28 days
pH	P, G	50 ml	G	Analyze Immediately	2 hr
TOC	G	100 ml	G, C	H ₂ SO ₄ pH<2, Refrig.	28 days

Sample Labeling

All sample bottles collected need to be properly labeled.

Labels Must Include: *Sample ID/Location*

Sample Date and Time

Preservative

Sampler Name/Initials

Labels May Include: *Sampling Method (G, C)*

Test(s)

Labels should be water proof and adhere to the bottles. Ink and writing must be legible.

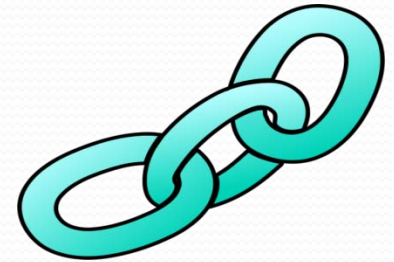
Example: Bottle Label

Sample ID:		
Sample Location:		
Sampled By:		
Sample Date/Time:		
Preservative:		
Analysis:		
Method (G or C):		

Chain of Custody

- A Chain of Custody is a chronological document or “paper trail” that shows the seizure, custody, control, transfer, analysis and disposition of a sample(s).

- Who's the Client (originator)?
- Who Sampled and when?
- Who Transported and when?
- Who Received the samples and when?
- What Samples are received?
- What Analysis are required?
- Are there any Anomalies?



Chain of Custody (COC)

- **A Chain of Custody (COC) must accompany all samples.**
- The COC must be filled out completely and properly:

Client or Sample Originator

Sample ID/Location

Sample Date and Time

Sampling Method (grab or composite)

Sample Matrix

Preservation

Analysis Required


Signed (Date and Time) by the Sampler,
Transporter and Laboratory

Example of a Chain of Custody

Company Name: _____
Address: _____
Email or Fax: _____
Contact: _____ **Phone:** _____

LAB COC ID: _____
Program/Project: _____
Report To: _____
Bill To: _____

Reporting Turn Around Time (Please Check):
 Rush 24hrs
 Rush 48hrs
 Rush 7 Days
 Standard 14 Days

CHAIN OF CUSTODY-ANALYTICAL SERVICES													
Sampler(s) : Comments:			Grab/G / Composite = C	Matrix = Potable Water (PW), Sewage (SE), Stormwater (SW), Recreation Water (RW), Water (W), Solid/Soil (S), Special (SP)	Number of Bottles	Preservative Type 1. HNO ₃ 2. H ₂ SO ₄ 3. H ₃ PO ₄ 4. HCL 5. NaOH 6. Na ₂ SO ₃ 7. Cool 4 C 8. None 9. Other(s)	ANALYSES REQUESTED					Temperature °C	 "Protecting You, Our Water, Tomorrow"
Sample Identification	Date Collected	Time Collected											
1													
2													
3													
4													
5													
6													
7													
8													
9													
10													
Relinquished by: (Signature)	Date	Time	Received by: (Signature)			Date	Time	Sample(s) Rec'd on ICE? Y or N					
Relinquished by: (Signature)	Date	Time	Received by Laboratory: (Signature)			Date	Time	Comments:					

Sample Transport

- Most analysis require the samples be place on ice immediately after sampling.

Exceptions: Metals and Legionella



- Samples must be transported on ice, usually in plastic or Styrofoam[®] coolers.
- Samples should be taken to the Lab as soon as possible via courier or overnight shipping.

Holding Times

- Analysis Holding Times must be considered when sampling and transporting samples.
- Examples of Short Holding Times:
 - Bacteria: 8 hrs
 - Hexavalent Chromium: 24 hrs
 - Ortho-phosphorous: 48 hrs
 - Turbidity: 48 hrs
 - BOD: 48 hrs
 - Sulfite: 48 hrs
 - Nitrate: 48hrs

Samples Arrive at the Lab

A Chain of Custody (COC) must accompany all samples!

- Lab Check:
- Did the samples make it to the lab safely?
 - Is there a COC? Is it filled out properly?
 - Were the samples received on ice?
 - Do sample bottles and labels match the COC?
 - Are the correct tests requested?

Lab Check Continued...

Anomalies: Are there missing or broken samples?



Is there enough sample to perform the test(s) requested?

Were the samples received with enough time to analyze them before the holding time expired?

All anomalies must be documented on the COC!

Lab Check Continued...

- Take and Record all Sample Temperatures.
- Check and record pH for all Field Preserved samples:
 - Are the samples at the proper pH?
 - Is additional preservative needed?
- Are any other checks or preparation needed (filtering, BOD pH adjustment, Chlorine check, etc...)?

Log the Samples into the LIMS System

Create a COC/Sample in the LIMS System

- Create a Unique Sample ID
 - A unique Sample ID helps Track the Sample from Sampling to Login through Analysis and Disposal!

Example: ETE1510220001

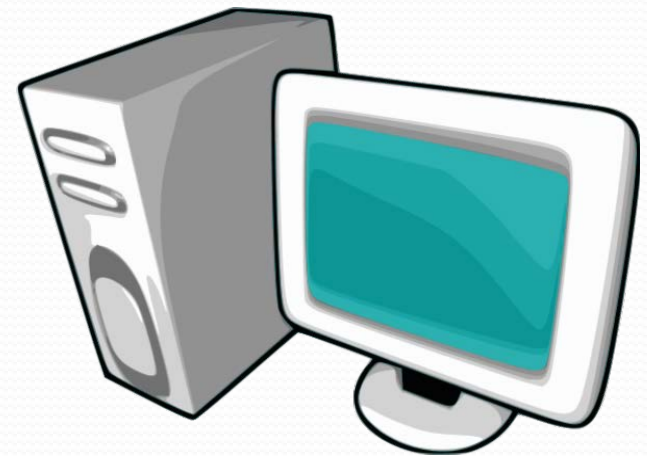
Easterly Treated Effluent = ETE

Sampled or logged in on 10/22/2015 = 151022

First ETE logged that day = 0001

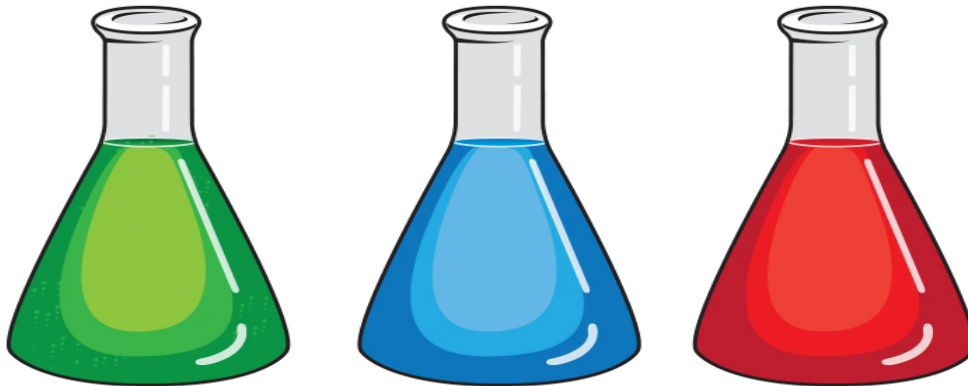
Log the Sample(s) into the LIMS System

- Client/Sample Originator Information
- Sample ID/Location
- Sample Date and Time
- Sampling Method (Grab or Composite)
- Sample Matrix
- Test(s) Requested
- Storage Location
- Anomalies/Comments



Processing a Composite Sample

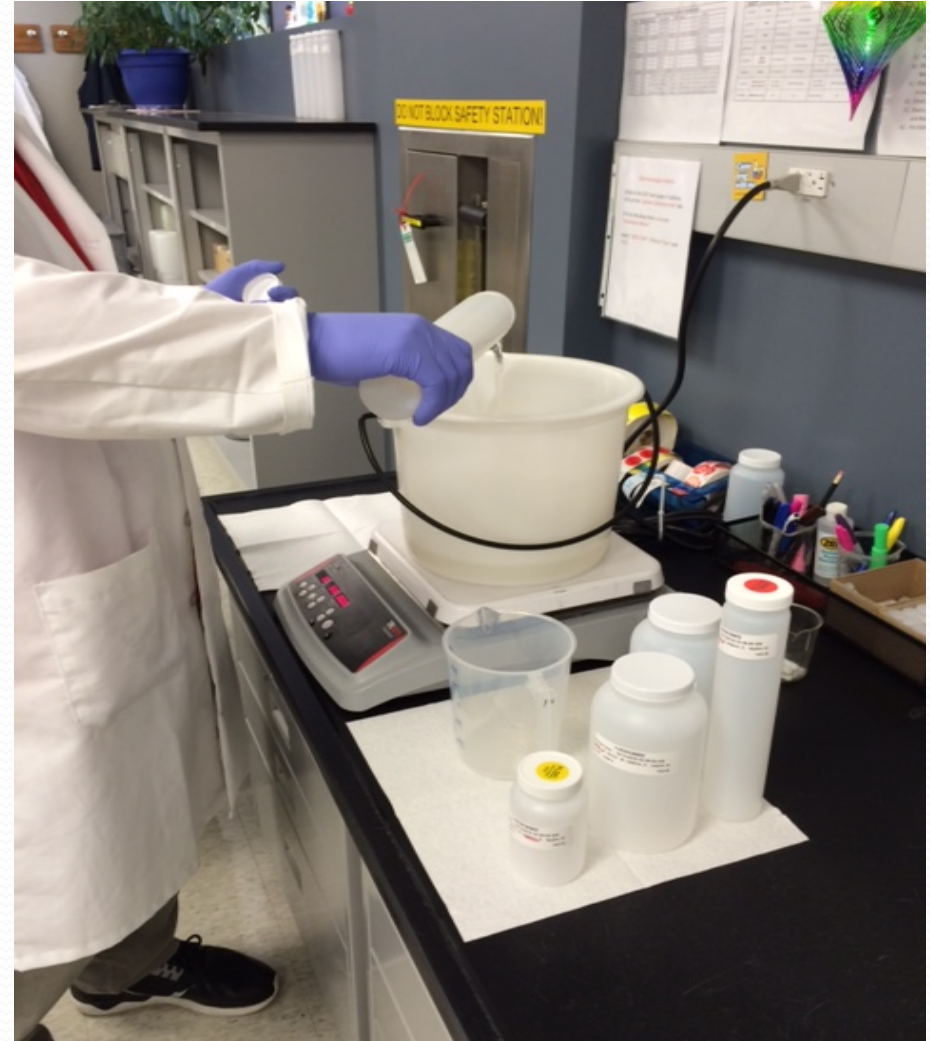
- Homogenize and/or Composite the Samples
- Aliquot (split off) Sample Portions
- Preserve the Samples Portions (if needed)
- Document homogenization and aliquoting
- Document Preservation



Processing a Composite Sample



Processing a Composite Sample



Label the Sample Bottles

Sample Labels should contain:

- Sample ID/Location
- Sample Date/Time
- Preservative
- Storage Location
- Tests (optional)



- Labels should be water proof and adhere to the bottles.*
- Additional preservation labels are recommended!*

Bottle Label

ETE1510220001

Easterly Treated Effluent

10/22/2015 08:00

Preservation: (circle one)

None HNO₃ H₂SO₄

NaOH HCL Na₂SO₈

Storage Location: O6M17

Sample Storage

- Tests requiring the Samples to be stored at 0-6°C must be stored in the appropriate refrigerator or cooler.
- Metals, Mercury and Hardness samples can be stored at room temperature.
- Legionella samples must be kept at room temperature.
- *Sample Storage Must be Secure!*



Cold Storage 0-6°C



Cold Storage 0-6°C

- Volatile Organic (VOC/VOA) samples must be segregated and stored separate from other refrigerated samples.
- Drinking Water, CLP and Evidentiary samples must be stored in a secure/locked refrigerator.
- Standards and reagents *should not* be stored with samples!

Cold Storage 0-6°C



Metals Storage (Room Temperature)



Sample Analysis/ Internal COC

The Samples are now ready for Analysis!

An Internal Chain of Custody (COC) must accompany the samples during and throughout analysis.

- Who Requested the sample and when?
- Who Relinquished the sample and when?
- Who Received the sample and when?
- Who Analyzed the sample and when?
- Who Returned the sample and when?
- Who Received the sample and when?

Sample Request Form (Internal COC)

Northeast Ohio Regional Sewer District

Sample Request Form

Test: _____ Preservative: _____

Analyst: _____ Date/Time Needed: _____

<u>Sample ID</u>	<u>Collect Date/Time</u>	<u>Sample Point</u>	<u>Storage Location</u>	<u>Comment</u>
SP-1510220001	10/21/15 15:00	Sample 1	O5	
SP-1510220002	10/21/15 15:15	Sample 2	O5	
SP-1510220003	10/21/15 15:30	Sample 3	O5	

Total Samples: 3

Relinquished: _____ Date/Time: _____ Received By: _____ Date/Time: _____

Relinquished: _____ Date/Time: _____ Received By: _____ Date/Time: _____

Analysis Complete/Data Reported

- Once the analysis is complete, the Final Report will be issued.
 - WWTP Data Report to Ohio EPA
 - Internal Report to our Industrial and Environmental Surveillance Group
 - External Report for External Clients

Sample Disposal

- Sample Disposal is set up by the Laboratory which is guided by Permits and regulatory bodies (NELAC).
- Our Laboratory generally disposes of samples 30 days after the Final Report has been issued.
- Some samples (or portions) are consumed during the analysis. This should be documented on the Sample Request Form/Internal COC.

Sample Disposal

Samples must be disposed of according to the Laboratory Disposal SOP or Chemical Hygiene Plan (CHP).

- Potentially Hazardous Samples (such as bulk chemicals, Cyanide, Mercury) must be retained and disposed of properly.



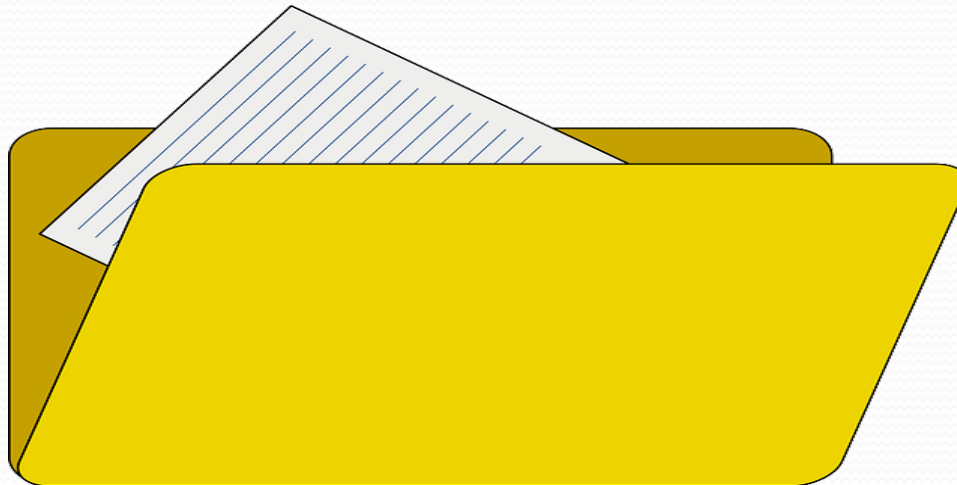
Sample Disposal

- Routine and Non-Hazardous samples can usually be disposed of internally.
- Sample bottles and containers should be rinsed and recycled (if possible).
- **All Sample Disposal must be documented!**



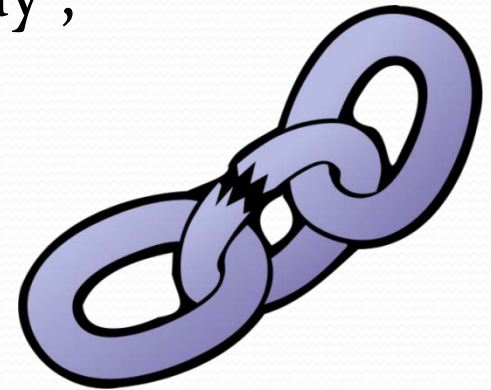
Records and Record Retention

- All Records must be retained according to the organization's record retention policy which is usually guided by their regulatory body.
- Most laboratories keep their records for 10 years.



Legal Aspects!

- A Chain of Custody is a chronological document or “paper trail” that shows the seizure, custody, control, transfer, analysis and disposition of a sample(s).
- If there is an incorrect procedure or “break” at any interval in the “Chain of Custody”, the data can be deemed questionable.
- Document, document, document!!



Thank you!

