Working with a Certified Water Laboratory

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Overview

• The ‘All Important’ Permit
• Sampling Hints
• Paperwork Matters!
• Reading a Laboratory Report
Labs and Treatment Plants
We’re a Partnership
Know Your Permit

• Know **how** to sample
• Know **where** to sample
• Know **when** to sample
Know **How** to Sample
## Grab or Composite?

<table>
<thead>
<tr>
<th>Outfall</th>
<th>Test</th>
<th>Frequency</th>
<th>Composite or Grab</th>
<th>Sample Type</th>
</tr>
</thead>
<tbody>
<tr>
<td>#001</td>
<td>BOD5</td>
<td>2/week</td>
<td>24 hour composite</td>
<td>Raw Waste</td>
</tr>
<tr>
<td>#001</td>
<td>Flow</td>
<td>7/week</td>
<td>24 hour composite</td>
<td>Raw Waste</td>
</tr>
<tr>
<td>#001</td>
<td>Nitrogen, Total</td>
<td>1/week</td>
<td>24 hour composite</td>
<td>Raw Waste</td>
</tr>
<tr>
<td>#001</td>
<td>Nitrogen, Total Kjeldahl</td>
<td>1/month</td>
<td>24 hour composite</td>
<td>Raw Waste</td>
</tr>
<tr>
<td>#001</td>
<td>pH</td>
<td>2/week</td>
<td>Grab</td>
<td>Raw Waste</td>
</tr>
<tr>
<td>#001</td>
<td>Phosphorus, Total</td>
<td>1/week</td>
<td>24 hour composite</td>
<td>Raw Waste</td>
</tr>
</tbody>
</table>
Composite Sampler
Grab Sampler
Know Where to Sample
Monitoring Locations: Wastewater

Influent
- After preliminary screening
- Aerated grit chamber prior to grit removal
- Force main after grit removal
- Sampling manhole ahead of plant

Effluent
- Acid mix basin
- Post aeration basin
Monitoring Locations: Wastewater

More Effluent locations

• Sampling manhole at the end of the plant
• Prior to UV contact tank
• Prior to disinfection
• After dechlorination process
• Prior to discharge aeration steps
Monitoring Locations: Wastewater continued...

Sludge

- Liquid sludge, sludge storage tank, end of secondary digester
- Cake sludge dewatering cell
- Lagoon sludge
Getting a Representative Sludge Sample

Section 4.43

• All sludge samples shall be collected at a point and in a manner which will yield sample results which are representative of the sludge being tested
Why is this important to you?

• Sludge testing is mainly for metals
• An annual sample should look like an ordinary day in your operation
• What ifs: you sample only from a section that contains stormwater runoff with heavy metals from cars
• What ifs: you sample only from a section where a local dentist had a one-time problem with his or her mercury collection system
Sample little bits over a period of time and mix together

One possible way to get a representative filter cake sample:

• Using a 500 ml glass beaker and a stainless steel trowel, collect approximately 200 ml of sample from the belt filter press as the sludge falls into the roll-off container.
• Collect 7 other samples in 30 minute intervals
• Mix in a stainless steel bucket
• Place sample for lab in sample container
• Seal bottle, label with sample number, date, time and refrigerate until delivery to lab
How to Sample Lagoons

- Use a “sludge judge” to collect samples from a randomized gridlike pattern, then composite the collected samples.
Samples from a Liquid Storage Tank

• Sludge tends to be a suspension so samples need to be well mixed in a tank with agitation
• Individual layers must be sampled if the tank has no agitation
• In both cases, grab several samples at different layers or time intervals, mix and place in sample container for lab
Monitoring Locations: Drinking Water

- Distribution System
- Each Active Well
- Raw Water
Know When to Sample

• WPDES permits vary, new requirements may change frequency of testing
• Drinking water regs vary with years—VOC, HAA5, Fluoride, Radioactivity
• Drinking water months for nitrate
Communicating Permit Updates

• Help the lab by sharing all permit updates
  – New test?
  – Drop a test?
  – Different Operator?

• The lab needs to know these important changes!
Helpful Hints for Sampling

“Your results are only as good as your sample collection”
Don’t Reuse a Sterile Bottle

• If something happens, don’t just dump out the sample and start over
• Feel free to ask for extra bottles from the lab
There’s no 5 second rule

- If you dropped a sterile bottle or the lid on the floor, ask the lab for a new bottle
Sampling in the Home
Distribution System Monitoring: Coliform

• Don’t set your filled sample bottles on the dashboard and drive around all day
Balance politeness and sample quality

• If you see the cat licking the faucet
• If you see the faucet is leaking
• If you see the sink is full of dirty dishes
Do Not Use

• A mop sink, outside faucet or a tap that is not generally used for human consumption
• A site that is vacant
• A site that has undergone recent plumbing improvements
• A tap that has any type of treatment
Do Not Use

• A site where the owner or resident is uncooperative
Best Sampling Procedure for Coliform

• Don’t flame the faucet
• Many correct ways to take a sample for bacteria—wipe the tap with alcohol, spray with a dilute chlorine solution
• Don’t overflow the bottle, you’ll flush out the anti-chlorine treatment in the bottle
Quiz Question

• What is the best way to sample for lead and copper in the distribution system?
Lead and Copper Sampling

• Have homeowner run water out of the kitchen tap for a few minutes
• No one should use the faucet for 6-8 hours
• Draw the sample first thing in the morning
Remember...

- Everything about the sample should be correct before it goes to the lab
Cooler Quiz
Cooler Quiz

• True or False:
  It’s OK to take the lab cooler home on the weekend for adult beverages?
There is a method to the madness

- Sample bottles from the lab are chosen for materials, volume and preservatives
Please do not send the lab samples in an old mayonnaise bottle
We can’t always control drop in samples
## Typical tests, bottles used and shipping requirements

<table>
<thead>
<tr>
<th>Test</th>
<th>Bottle</th>
<th>Temperature</th>
<th>Hold Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>BOD</td>
<td>P or G</td>
<td>Cool 4 degrees C</td>
<td>48 hours</td>
</tr>
<tr>
<td><strong>Total Coliform</strong></td>
<td>Sterile plastic</td>
<td>Cool</td>
<td>6 hours</td>
</tr>
<tr>
<td>Ammonia</td>
<td>P or G</td>
<td>Cool 4 degrees C, pH 2</td>
<td>28 days</td>
</tr>
<tr>
<td><strong>Total Kjeldahl N</strong></td>
<td>P or G</td>
<td>Cool 4 degrees C, pH 2</td>
<td>28 days</td>
</tr>
<tr>
<td>Nitrate</td>
<td>P or G</td>
<td>Cool 4 degrees C</td>
<td>48 hours</td>
</tr>
<tr>
<td><strong>Total Phosphorus</strong></td>
<td>P or G</td>
<td>Cool 4 degrees C, pH 2</td>
<td>28 days</td>
</tr>
<tr>
<td>TSS</td>
<td>P or G</td>
<td>Cool 4 degrees C</td>
<td>7 days</td>
</tr>
</tbody>
</table>
Usually effluent bottles are bigger than influent bottles
Oil and Grease

- Oil and grease must be in a glass bottle because oil can stick to plastic.
I know they’re nice, but

- Labs would really appreciate it if you use our sample bottles for their intended purpose.

I don’t know where those sample bottles went to?
This Really Happened!

A homeowner brought in a bottle of water. She said she had two samples in there. She had carefully layered one sample on bottom and one on the top.

We could save a lot on bottles!
Paperwork Matters

- Chain of custody forms help you defend your results in any legal battle.
Here’s a Scenario

• Your industrial pretreatment client denies discharging BOD. You know you took the sample properly. There is no doubt to the accuracy of the results. If you don’t have the proper chain of custody forms, your results are worthless for legal purposes.
Elements of a COC

- Sample number
- Location where sample was taken
- Date and time of collection
- Name of sample collector
- Signatures when sample is handed off to driver and lab
- You should keep a copy
In case you were wondering.....
Turnaround Time

• Labs will help when you are under a deadline, but some samples take more time than others
  • BOD 5 days
  • Lead and Copper 2 weeks for drinking water
  • 6 days is typical for ammonia, TKN, nitrate, phosphorus, metals
• Rush samples 2-3 day turnaround
Sufficiently sensitive methods for wastewater testing

- August 2014 ruling from EPA
- There are often multiple analytical methods available for testing certain pollutants. This ruling specifies that labs must use the best test method that matches the concentration of the pollutant. All labs are aware of this ruling and conform.
sneak a peek
BEHIND THE SCENES
Typical route for van driver
Driver checking materials for route
Checking Temperatures on incoming samples
Logging in date and time on incoming samples
Samples going into system
IT'S THE LAB'S FAULT
Quality Control/Quality Assurance

• Quality Control is the techniques that you use to make sure that your data is valid and traceable.

• Quality Assurance is the whole system that checks, makes adjustments, and takes corrective action to make sure the lab produces quality data.
Laboratory Checks

• For bacteria testing, check each lot of reagents with positive and negative controls to ensure reagents are working correctly

• Check each lot of sterile bottles for sterility even if they have a certificate of sterility
Inorganic Test Accuracy Checks

- Nitrate-calibrate ion chromatograph once a month
- Perform method blanks daily
- Analyze a QC sample every 10 samples
- Analyze known standards on every run: if results don’t pass, your samples are rerun
- Analyze proficiency samples quarterly plus State supplied proficiency samples 1/year
Labs aren’t perfect either
And last but not least....
Reading your report

• Please read it right away!
• The laboratory cannot redo BODs because of 48 hour sample hold times
• Wastewater metal samples are only kept for 10 days
<table>
<thead>
<tr>
<th>Sample ID Number</th>
<th>Sample Description</th>
<th>Test Name</th>
<th>Methodology</th>
<th>Results</th>
<th>Dilution Factors</th>
<th>LOD/LOQ</th>
<th>Preparation Date</th>
<th>Date Tested</th>
</tr>
</thead>
<tbody>
<tr>
<td>ww93222</td>
<td>SW East 001^</td>
<td>Ammonia as N</td>
<td>SM4500-NH₃ B&amp;D</td>
<td>0.3 mg/L</td>
<td>2</td>
<td>0.20/0.40 mg/L</td>
<td>7/1/2016</td>
<td>7/1/2016</td>
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<tr>
<td></td>
<td></td>
<td>Biochemical Oxygen Demand (5 Day)</td>
<td>SM5210 B</td>
<td>17 mg/L</td>
<td>NA</td>
<td>NA</td>
<td>NA</td>
<td>7/1/2016</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Nitrate + Nitrite as N</td>
<td>SM4500 NO₃ E</td>
<td>0.1 mg/L</td>
<td>1</td>
<td>0.02/0.05 mg/L</td>
<td>NA</td>
<td>7/11/2016</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Lab pH</td>
<td>SM4500-HB</td>
<td>6.1 mg/L</td>
<td>1</td>
<td>0.02/0.10 mg/L</td>
<td>NA</td>
<td>7/1/2016</td>
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<tr>
<td></td>
<td></td>
<td>Total Phosphorus</td>
<td>HACH 8190</td>
<td>0.2 mg/L</td>
<td>1</td>
<td>0.02/0.10 mg/L</td>
<td>7/1/2016</td>
<td>7/1/2016</td>
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<td>ww93223</td>
<td>SW West 002^</td>
<td>Ammonia as N</td>
<td>SM4500-NH₃ B&amp;D</td>
<td>0.22* mg/L</td>
<td>2</td>
<td>0.20/0.40 mg/L</td>
<td>7/1/2016</td>
<td>7/1/2016</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Biochemical Oxygen Demand (5 Day)</td>
<td>SM5210 B</td>
<td>56 mg/L</td>
<td>NA</td>
<td>NA</td>
<td>NA</td>
<td>6/30/2016</td>
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<tr>
<td></td>
<td></td>
<td>Nitrate + Nitrite as N</td>
<td>SM4500 NO₃ E</td>
<td>&lt;0.02 mg/L</td>
<td>1</td>
<td>0.02/0.05 mg/L</td>
<td>NA</td>
<td>7/11/2016</td>
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<td></td>
<td></td>
<td>Lab pH</td>
<td>SM4500-HB</td>
<td>6.6 mg/L</td>
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<td>0.02/0.10 mg/L</td>
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<td></td>
<td></td>
<td>Total Phosphorus</td>
<td>HACH 8190</td>
<td>0.08* mg/L</td>
<td>1</td>
<td>0.02/0.10 mg/L</td>
<td>7/1/2016</td>
<td>7/1/2016</td>
</tr>
</tbody>
</table>
What are LOD and LOQ?

• Limit of Detection, LOD
  – LOD is the smallest amount of something that the method can see, barely above zero

• Limit of Quantitation, LOQ
  – LOQ is the amount that the method can reliably and accurately detect
### LOD/LOQ and Preparation Date

<table>
<thead>
<tr>
<th>Sample ID Number</th>
<th>Sample Description</th>
<th>Test Name</th>
<th>Methodology</th>
<th>Results</th>
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<th>LOD/LOQ</th>
<th>Preparation Date</th>
<th>Date Tested</th>
</tr>
</thead>
<tbody>
<tr>
<td>ww81600</td>
<td>Eff</td>
<td>Ammonia as N</td>
<td>SM4500 NH3 B&amp;D</td>
<td>0.18 mg/L</td>
<td>1</td>
<td>0.07/0.24 mg/L</td>
<td>8/7/2015</td>
<td>8/7/2015</td>
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<tr>
<td></td>
<td></td>
<td>Biochemical Oxygen Demand 5 day</td>
<td>SM5210 B</td>
<td>9150 mg/L</td>
<td>NA</td>
<td>NA</td>
<td>NA</td>
<td>8/7/2015</td>
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<tr>
<td></td>
<td></td>
<td>Chloride</td>
<td>EPA 300.0</td>
<td>1332 mg/L</td>
<td>100</td>
<td>20/49 mg/L</td>
<td>NA</td>
<td>8/7/2015</td>
</tr>
<tr>
<td></td>
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<td>Nitrate + Nitrite as N</td>
<td>EPA 300.0</td>
<td>&lt;0.1 mg/L</td>
<td>1</td>
<td>0.1/0.22 mg/L</td>
<td>NA</td>
<td>8/7/2015</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Lab pH</td>
<td>SM4500 HB</td>
<td>5.0 s.u.</td>
<td>NA</td>
<td>NA</td>
<td>NA</td>
<td>8/7/2015</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Total Phosphorus</td>
<td>HACH 8190</td>
<td>17 mg/L</td>
<td>100</td>
<td>2.0/7.0 mg/L</td>
<td>8/7/2015</td>
<td>8/7/2015</td>
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<td>Total Kjeldahl Nitrogen</td>
<td>SM4500-NH3B &amp; NH3C</td>
<td>11 mg/L</td>
<td>1</td>
<td>0.56/1.9 mg/L</td>
<td>8/7/2015</td>
<td>8/7/2015</td>
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<td></td>
<td></td>
<td>Total Suspended Solids</td>
<td>SM2540 D</td>
<td>7150 mg/L</td>
<td>NA</td>
<td>NA</td>
<td>NA</td>
<td>8/7/2015</td>
</tr>
</tbody>
</table>

All LOD/LOQs adjusted to reflect dilution. LOD = Limit of Detection, LOQ = Limit of Quantitation, NA = Not Applicable.

Qualifier: *Results are below the LOQ.

WDNR Laboratory Certification Number: 737109450

WDATCP Laboratory Certification Number: 55-424

The results issued on this report only reflect the analysis of the samples(s) submitted at our lab and may not be construed as an endorsement of the sampling method employed. This report shall not be reproduced except in full, without written approval of the laboratory. The accuracy of these results are limited by the integrity of the sample and the accuracy of the test method. Reports are kept on file for three years.

Report Authorized By: Lindsay Weisbrod

Date ____________________
Accuracy and Precision

• Accuracy is getting the right answer
Precision

- Precision is getting the same answer again and again
- It might not be the right answer, but the test is consistent
Precision vs. Accuracy
Bias

• Associated with accuracy
• When you consistently analyze a sample and find that you always get high or low results from expected
• Like a thermometer that always reads high
What do you think of these ammonia results?

- **Influent Ammonia 34 mg/L**
  - LOD 0.7 mg/L
  - LOQ 2.4 mg/L

- **Effluent Ammonia <0.07 mg/L**
  - LOD 0.07 mg/L
  - LOQ 0.24 mg/L
Questions?
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715-240-0444