

UNDERSTANDING LAB DATA

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REGULATORY FRAMEWORK

1. What regulations require you to sample?
2. What regulations govern the lab procedures?

WHAT REGULATIONS REQUIRE YOU TO SAMPLE?

1. NPDES Permit
 1. Limits and monitoring requirements page
 2. Chronic Toxicity
 3. PPA
2. 40 CFR Part 403, Pretreatment regulations
 1. Direct monitoring of industrial users
 2. LTMP
3. 40 CFR Part 503

WHAT REGULATIONS GOVERN LAB PROCEDURES?

1. 40 CFR Part 136
 1. The EPA regulations governing approved CWA chemical test methods
2. 15 NCAC 2H .0800s
 1. The NCDEQ regulations governing lab certification

DIFFERENT TYPES OF DATA

1. Regulated/Reportable

1. Influent/Effluent
2. Industrial
3. Biosolids

2. Unregulated

1. Process Control
2. Collections system

DETECTION LIMITS

- MDL, PQL, and RL
 - Method detection limit
 - Practical quantitation limit
 - Reporting limit

MDL

- The EPA says:
 - The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.

PQL

- Lowest level which can be reliably determined within the specified limits of precision and accuracy.
 - MDL's realistic limit
 - Usually 5 to 10 times the MDL

RL

- An instrument-dependent quantity based on the lowest point on the calibration curve.
 - Dependent on the individual laboratory and even the individual run.

DETECTION LIMITS

- Permit
 - DL must show that you met your NPDES permit limits!
- LTMP
 - The state generally wants the lowest detection limits achievable.
 - You must meet the terms of your LTMP
- HWA
 - Your removal rate and your allocation amounts are going to depend on the detection limits.

DETECTION LIMITS

- March 21, 2022 DEQ memo
- “If a higher detection limit than the PQL is used due to matrix interference, you may still use zero (0) as the value— if the lab analysis report identifies the sample, qualifies the result with qualifier code and detailed explanation of interference. Generic matrix interference notations in lab reports or notes are not technically defensible without additional documentation.”

WHAT ABOUT MASS BASED LIMITS

- Pounds formula
- Be aware of average flow and average concentration.
 - Make a table of concentrations from various scenarios:
 - Max permitted, average, average plus 1 SD

DETECTION LIMITS

- DO THE REPORTED RESULTS COMPORT WITH THE LTMP REQUIREMENTS?
 - Send your project manager the PQL page from your LTMP.
 - Ensure that the lab explains itself, in writing.
 - Try another lab?

WAYS TO KEEP YOURSELF AWARE

- Rolling averages
- Averages over time
- Standard deviation
- Trigger values
- Flag QC Failures

ROLLING AVERAGE

- Average of last “N” measurements.
- Gives you an idea of how fast the value is changing in your most recent measurement.
- Useful for process control values like mixed liquor, and bug counts.

AVERAGES OVER TIME

- What is the expected value for a measurement?
- Have a tool that allows you to track the average value for a parameter over a set time period.
- Otherwise, you are really just dealing with numbers out of context.
- Look at a chart or graph of the values over time.

STANDARD DEVIATION

Standard Deviation Formula



Population	Sample
$\sigma = \sqrt{\frac{\sum(X - \mu)^2}{N}}$ X - The Value in the data distribution μ - The population Mean N - Total Number of Observations	$s = \sqrt{\frac{\sum(X - \bar{x})^2}{n - 1}}$ X - The Value in the data distribution $\bar{x}$ - The Sample Mean n - Total Number of Observations

STANDARD DEVIATION

- A measure of how spread out the data is in relation to the average.
 - Easy to do in excel or database.
- Set a SD limit to review.
 - If the value is more than “X” SDs away from the mean, take a closer look.
 - If the data checks out consider investigating further.
 - The POTW or industry might have an issue and not know it.
 - Maybe the process changed and they did not inform you.

EXAMPLE DATA

		TSS		BOD		COD		NH3	TKN	Nox	TN
			mg/L	mg/L			mg/L	mg/L	mg/L	mg/L	mg/L
	StdDev		0	0.23			3.1	0.086	0.320	0.535	0.590
	Max		2.5	3.7			28	0.756	1.940	3.150	4.200
	Min	<	2.5	<	2.0	<	20	< 0.200	0.291	0.230	0.800
	Average		2.5	2.1			23	0.229	1.167	0.892	2.056

TRIGGER VALUES

- KNOW YOUR POTW's LIMITS!
- KNOW YOUR IUs LIMITS!
- However, limits are not enough.
 - Does anyone enjoy writing and/or receiving NOVs?
 - Set a reasonable boundary around the limit and have a conversation or make process changes when you notice something out of the ordinary.
 - Make sure you communicate with your sampling tech and lab techs. Set clear expectations

AVERAGE POLLUTION CONCENTRATIONS FOR DOMESTIC WASTEWATER

SOURCE: CHANGES OF WASTEWATER CHARACTERISTIC DURING TRANSPORT IN SEWERS

Parameter	Concentration, mg/dm ³		
	Strong	Medium	Weak
Total solids	1200	700	350
Dissolved solids (TDS)	850	500	250
Suspended solids	350	200	100
Nitrogen (as N)	85	40	20
Phosphorus (as P)	20	10	6
Chloride	100	50	30
Alkalinity (as CaCO ₃)	200	100	50
Grease	150	100	50
BOD ₅	300	200	100

PROCESS CONTOL

SOURCE: A DATA MINING APPROACH TO THE PREDICTION OF FOOD-TO-MASS RATIO AND MIXED LIQUOR SUSPENDED SOLIDS

Variable	Minimum	Average	Maximum
Q (m ³ /d)	32,564	40,698	86,592
T _{sl} (°C)	10.0	15.9	23.0
pH	7.00	7.6	8.1
MLSS (kg/m ³)	1.97	4.26	6.59
RAS (%)	44.6	90.70	167.6
WAS (kg/d)	3,489	11,123	19,194
F/M (gBOD ₅ /gMLSS·d)	0.03	0.07	0.13
BOD ₅ (mg/dm ³)	127	309	557
COD (mg/dm ³)	384	791	1250
TSS (mg/dm ³)	126	329	572
TN (mg/dm ³)	39.9	77.7	124.1
NH ₄ ⁺ (mg/dm ³)	24.4	49.31	65.9

PROCESS CONTROL

- What are the most useful sampling points?
- What are the most useful parameters to measure?
 - Same as NPDES permit?
 - Proxies: COD / Bug Count / Alkalinity
 - Process ratios: F/M, SVI, MLVSS
- What does the value physically indicate?
- How does it compare to normal operation?

PROCESS CONTROL

- Know the removal rates across your system
 - Just because the Eff is clear does not mean that your process is working well
- Check for a lab error before making process changes
- If a parameter changes is the cause transient?
 - I and I
 - Industrial loading

WHEN LAB RESULTS SHOULD TRIGGER ACTION

- **Document but Continue:**
 - Single outlier within 2x normal range
 - Weekend/holiday flow variations
 - Minor seasonal temperature effects
 - Variations tied to known process changes

WHEN LAB RESULTS SHOULD TRIGGER ACTION

- **Monitor Closely (Trending Concern):**
 - MLSS changing > 10% per day
 - SVI increasing above 200 mL/g
 - Ammonia >5 mg/L in effluent
 - Rapid changes in microbiological activity

WHEN LAB RESULTS SHOULD TRIGGER ACTION

- **Immediate Action Required:**
 - Effluent BOD > 1/2 weekly permit limit
 - Effluent (or secondary) TSS >30 mg/L
 - pH outside 6.0 - 9.0 range
 - DO in aeration <1.0 mg/L or >4.0 mg/L
 - Removal rates slipping below 85%

VERIFY LAB DATA BEFORE MAKING PROCESS CHANGES

- Was the Sample Collected Properly?
 - Timing: Was sample taken during normal operations or during upset?
- Does the data match process observations?
- QC failures noted:
 - Have standards, titrants, probes, etc. been verified?

QUESTIONS TO ASK OF EVERY DATA POINT

- Is it reportable?
 - If so, were all the required regulations followed?
 - Was it a violation?
 - Is it otherwise concerning (HWA, SIU, overloading plant)?
- Does it make physical sense?
 - $TKN > NH_3$
 - $COD > BOD$
 - Negative removal rate
 - Outliers

FLAG QC FAILURES

- Types:
 - Standard failure:
 - Is the result biased high or low?
 - If this is your lab, rerun sample if it is within hold time.
 - Relatively rare in contract labs. If you see it, give your contact a call!
 - BODs
 - Blank above range
 - Is your value close to the DL?
 - If it is close to the detection limit, does it matter?
 - Low level Hg

FLAG QC FAILURES

- BOD
 - How do you treat $>$, $<$, or samples which do not hit at all?
- Low level Hg
 - “If Hg or any potentially interfering substance is found in the field blank at a concentration equal to or greater than the ML (Table I), or greater than one-fifth the level in the associated sample, whichever is greater, results for associated samples may be the result of contamination and may not be reported or otherwise used for regulatory compliance purposes.”

FLAG QC FAILURES

- Matrix spike failures
 - Assuming proper lab procedure, usually indicates interference.
 - If the method is amenable, try:
 - Standard additions
 - Diluting to a different range
 - Try a different method / different lab

WHEN IN DOUBT - DON'T REACT YET

- Resample if possible (within permit monitoring requirements)
- Talk to the lab to discuss unusual results
- Document your concerns in the plant log
- Continue current operations until data is confirmed

BOTTOM LINE

- How confident are you in the data?
- How important is the data?
 - Process control vs NPDES vs compliance enforcement
- **WOULD YOUR DATA HOLD UP IN COURT?**