Sample Collection and Field Process Control Sampling

OWEA Operator Workshop
September 25, 2007
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Presentation Outline

- Review of Basic Sample Collection Techniques
- 40 CFR 136
- CFR 136.3 Identification of sample containers and holding times / pres. Techniques and approved test methods for analytes
- Process Control Sampling (large vs. small systems)
- pH and alkalinity as a control measure
- Nitrification / Denitrification and effects on plant processes

40 CFR 136

- Code of Federal Regulations pertaining to,
- Proper analytical test methods 136.3
- Proper sampling containers
- Holding times and preservatives
- www.access.gpo.gov/nara/cfr/waisidx_03/40cfr_03.html
- Google Search

Review of Sample Containers, holding times and preservatives

- Typically all samples store @ 4 degrees Celsius in addition to chemical preservatives. Some revision to 40 CFR to temperature requirement holding requirement
- pH and Dissolved Oxygen – plastic or glass, perform immediately
- CBOD5 – plastic or glass, 48 hours from end of composite period
- Solids analysis (TSS, TDS, % solids, % volatiles) – plastic or glass, 7 days
- Ammonia-N – plastic or glass, 24 hours from collection or 28 days if pres'd w/ H_2SO_4

Containers, Preservatives, Holding Times continued

- Fecal coliform – sterile plastic or glass, sodium thiosulfate if chlorinated, 6 hours, again @ 4 degrees Celsius
- Oil and Grease – glass only, pres'd w/ HCl or H_2SO_4
- Metals – vary
  - Cr-6 – no preservative
  - Total Hg HNO3
  - Low Level Hg – HCl, sample collection method 1631
  - CN – Free or Total – NaOH
  - Most other metals – no preservative required
  - Dissolved Metals – filtration through 0.45 micron filter (field or laboratory)

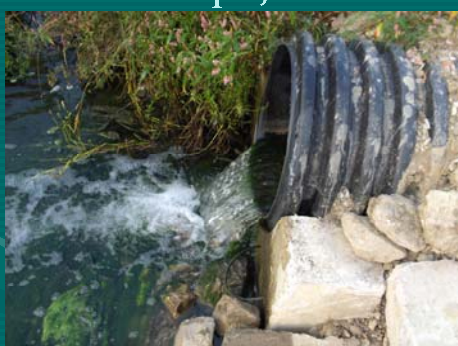
Basic Lab and Process Control

- BOD-5 – Biochemical Oxygen Demand. Represents dissolved oxygen depletion a wastewater will have on a receiving stream as it continues to degrade. (Carbon and Nitrogen Demand)
- CBOD-5 – Same as above, but represents carbonaceous component (organic based) – nitrogen demand (Ammonia). Nitrification Inhibitor added to bottles / dilution water
- BOD-5 = CBOD-5 + Nitrogen (Ammonia)
- Used to determine strength of waste and plant removal efficiencies

Field Sampling

- Grab sample – sample representing a snapshot in time representing flow @ that instant
  - pH, DO, temperature, oil and grease, fecal / E. coli
- Composite Sample – can be
  - Multiple Grab samples of equal volume and time intervals (usually minimum of 3, 30 minutes apart), so composite can be collected over 1 hour period
  - 24 hour Timed Composite – equal volume aliqouts @ equal timed intervals
  - Flow Proportional Composite – Sampler and Flowmeter work together to collect larger aliquots during peak flow periods. Most representative composite sample
  - Neat Composite – each aliquot is collected either in timed or flow proportional intervals, but separated in separate containers. Often used industrially to identify peak loading periods

Grab Sample, Location



Composite Sampling Station

Composite Sampler

Process Control Monitoring, the other half of field sample collection

- All systems require some level of routine process control monitoring
- Larger the facility, higher frequency of sample collection and monitoring
- Smaller the facility, lesser frequency of sample collection….but other test methods may be required to ensure the best treatment

Process Control Monitoring

- 30 minute settling – activated sludge process control monitoring method that helps an operator determine the settling characteristics of the biomass under aeration
- With experience, one can get an idea of sludge age and possible treatment changes

30 minute settling

- During test, operator can observe
- Clarity of water "decant" above the settled sludge during and after 30 minutes.
  - Dark Water – excess BOD5, septicity /underaeration
  - Presence of stragglers (young sludge) or pin floc (old sludge)
  - Physical structure of the floc, i.e. big and fluffy, small and granular, light brown to dark brown
  - These physical observations can help determine effluent variability. Need increased aeration, wasting that may improve effluent. Adequate aeration / overaeration?
  - Observe denitrification – popping sludge

30 minute settling (on the road)

Other Process Control Measures

- Spin test
- Alkalinity
- Ammonia test kits
- These are recommended tests for process control monitoring by OEPA Compliance Assistance Group for package plant operation

Process Control Monitoring

- MLSS – mixed liquor suspended solids – Sample collected from the end of aeration tank prior to clarification. Collect this sample from end of aeration basin just prior to secondary clarifier
  - MLSS along with 30 minute settling can be used to calculate SVI – Sludge Volume Index, SVI indicates how much volume is occupied by 1 gram of settled sludge.
  - Ideally 100 – 1 gram of settled sludge occupies 100 ml, the lower the number the more dense the sludge…granular
- RASSS – return activated sludge suspended solids – sample of concentration settled sludge from clarifier
  - MLSS, RASSS, Flow are used to determine MCRT – Mean Cell Residence Time or Solids Residence Time
  - RASSS along with sludge judging can help determine return flow adjustments

Process Control Monitoring

- MCRT – Mean Cell Residence Time is calculated by dividing solids in the system divided by solids leaving the system (waste and effluent)
- Depending on the type of activated sludge MCRT's range from 5 – 20 days.
- Often smaller plants rely on older sludges, due to low loadings and high ammonia loadings.
- With small systems, it's easy to waste too much

Process Control Monitoring

- pH and dissolved oxygen of aeration tank – critical to know routinely in small or large plants
- pH – critical due to effluent limits
  - Also, can be an indicator of many things, dumps, presence of good nitrification or a quick indicator that pH adjustment is needed
  - Most limiting factor to biological activity, aerobic, anaerobic and attached growth systems are pH dependent (6.5-9.0). Personal Preference 7.5-8.0 SU. 7.0 is on the lower end of the pH scale.

Process Control Monitoring

- pH / alkalinity relationship
- Often the terms are interchanged
- They are related, but not exactly the same
- pH is a measurement of Hydrogen H^+ and Hydroxyl OH^- ion activity in solution. $H^+ = OH^-$ pH = 7.0 SU
- Alkalinity is a water's acid neutralizing capacity (the higher the alkalinity, the more anions it can take before pH drops)
- Since many wastewater processes produce acidic byproducts, these 2 terms are often used interchangably
- This is the importance of understanding alkalinity

Process Monitoring

- Dissolved Oxygen – critical to aeration tanks. Typical consistent values during aerated cycles 2-3 mg/l.
  - Consistently higher than this you may be overaerating. Operator needs to also weigh the value of adequate mixing. Mixing with air is very inefficient, but it's probably the right choice for most treatment systems
  - 30 scfm / 1000 cubic feet of aeration basin
  - May contribute to settling problems and possibly denitrification in the clarifier

Process Control, Large vs. Small

- Large Systems – solids inventory can be managed more efficiently with MCRT
- Small Systems – solids inventory tend to be controlled more by MLSS, RASSS, SVI
  - Tend to be much higher SRT's in small plants vs. large plants
  - Often controlling small plant by MCRT tends to wasting too much
  - Due to low daily loadings w/ often high NH3 leads to the need for longer MCRT

Process Control Large Plant

- May have to take into account
- Primary removal efficiency
- Sand Filter Loading Rates (hydraulic and solids)
- Trickling Filter – Organic loading rate (lbs BOD5 / 1000 cubic foot / day)
- SBR's – basically have 2-3 different plants based on the # of SBR's onsite

Process Control, Small Plant

- On top of the standard 30 min, MLSS, RASSS, Sludge Judging, small plants
- Often need additional monitoring of pH, alkalinity
- Due to the type of system being serviced by extended aeration
- Truck Stops, Rest Areas need extra attention or more than you would expect

Process Control

- Truck stops and rest areas
- Heavy concentrations of NH3
- Fluctuations in traffic and people, seasonal / holiday weekends impact these systems
- Often require supplementation of additional alkalinity
  - Sodium Bicarbonate (baking soda), NaOH, Lime
  - pH of aeration basins can easily drop to 4 SU during high loads of ammonia (urine)

Process Control, Small Plant

- Often package plant expansion… install another package plant
- Flow Equalization / diversion is critical
- Even loadings to each plant to keep them consistent with one another

Alkaline Addition

- Sodium Bicarbonate – Baking Soda $NaHCO_3$ – for small plants, probably the method of choice
  - Just dump it in
  - High alkalinity addition
  - Tends not to overshoot the pH
  - Lime – caking issues
  - NaOH – can easily overadd and have too high of pH, issues with gelling below 55 F

Arm and Hammer Baking Soda

- Great source of alkalinity
- Helps w/ odors (just like the fridge)
- Used in aeration basins and anaerobic digesters
- Kitty litter, toothpaste, frig
- Corrosion control, swimming pools
- Flocculent improvement

Process Monitoring (on the road) Package WWTP Aeration Basin

Process Control, Nitrification Process

- Nitrification requires ample alkalinity and dissolved oxygen
- Nitrification is a nitrogen conversion process vs. a consumption process (BOD5) by nitrifying bacteria (nitrosomonas and nitrobacter)
- Highly aerobic, requires 4 mg/ l d.o. for every mg/l ammonia, so if you have d.o. issues, check raw ammonia’s
- NH3 is converted to nitrite by nitrosomonas. Nitrite is converted to nitrate by nitrobacter.

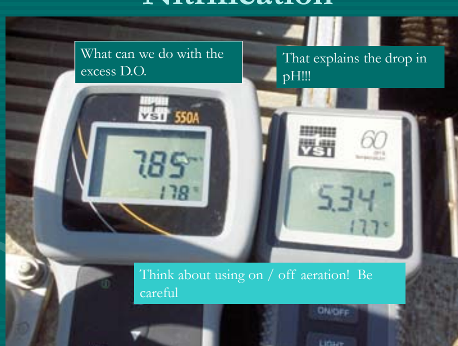
Nitrification

- This process produces acidic ions (anions) (Nitrate) that consume alkalinity, leading to a possible drop in pH
- Nitrification requires 7mg / l alkalinity as CaCO3 for every mg/ l of NH3 present to buffer the formation of nitrates
- Some of these locations, influent ammonia concentrations can be well over 100 mg/l
- Nitrification of these streams leads to alkalinity deficits and low pH in the aeration basin

Nitrification / Alkalinity Relationship

- Influent 100 mg/l NH3 and alkalinity of 400 mg/l
- Need to reach ammonia effluent of 1 mg/ l would require 700 mg/l of alkalinity to buffer nitrate formation and prevent a drop in pH
- Recover approximately 50% of alkalinity consumed through denitrification
- 700 – 400 = -300 mg/l alkalinity deficit + 200 mg/l recovered alkalinity = -100 mg/l, alkalinity deficit
- This type of scenario will lead to low pH in the aeration system and clarifier, which could lead to other treatment issues.
- Goes unattended, nitrification stops and then BOD5 removal can be affected

Nitrification



Nitrification / Denitrification

- In order to denitrify, nitrification must be occurring
- To maintain compliance with ammonia levels, you must stay aerobic, make adjustments slowly in small intervals! Not enough D.O. / mixing ammonia will increase
- If denitrification is occurring in the clarifiers and you observe low pH in the aeration basin, you may be able to use on /off aeration
  - Make sure it's denitrification and not hydraulic washout of the clarifier(s)
- This will help recover alkalinity by driving off nitrates in the aeration basin during off cycles, plus reduce the chance for popping sludge in the clarifiers
- By recovering some alkalinity you reduce the demand for alkaline addition
- At capacity systems, you may have to maintain aeration and add alkaline materials consistently
- Nitrifiers are pretty touchy, they double population 1x / day vs. heterotrophs that reproduce about every 20 minutes

Nitrification / Denitrification Process

- Nitrifiers convert Ammonia ultimately to Nitrate
- Heterotrophic / Facultative bacteria can utilize Nitrate in the presence of BOD5 as a source of oxygen when dissolved oxygen is absent
- Nitrate is used as electron acceptor, Nitrogen gas is driven off
- During off cycles in the aeration basin, this is more likely to occur
- Prefer a setup with returns @ controlled rate running continuously

Sampling

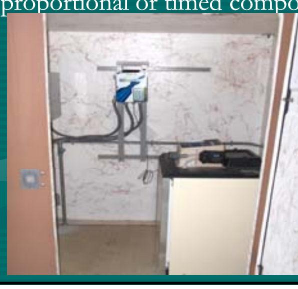
- Composite Sampling
- 3 "grab" composite
- Manual process typically 3 samples ½ hour apart
- Typically, 1 liter each for each sample
- Placed in common composite jug
- So if you're on top of it and nothing comes up, a composite sample could be collected within 1 hour

Sampling

- Composite Sampling – Raw Influent
- Program start and finish by times or number of samples
- Set desired time intervals (as directed in permit or preferred every 30 minutes)
- 48 samples for a 24 hour composite period

Sampling

- Composite sampling – effluent location
- Flow proportional or timed composite



Sample Containers

- New containers should be cleaned
- Even acid rinse to remove any organic residue present on walls of sampling container
- Clean composite jugs between each sampling cycle
- Do not use composite pump to collect fecal samples
- Pump tubes should be cleaned or changed regularly to reduce biofilm growth on the tubes

Sampling

- Effluent Oil and Grease
- Glass Jar Only w/ acid preservative. Oil and grease can stick to plastic
- Try to collect directly into glass container, transfer from dipper to glass could lose oil and grease.

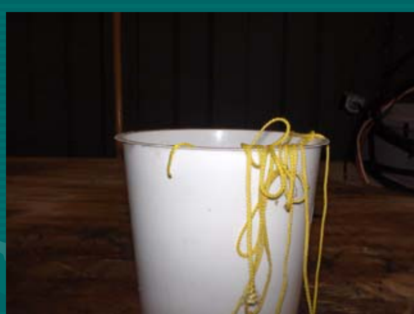
Sampling, cont'd

- Other Sampling Tools
- Core Sampler – "Sludge Judge" – Clarifier Core samples and measurement of sludge blankets. Used for lagoon measurement / sludge sampling
- Polypropylene sample dipper – good for BOD5, TSS, NH3-N grab or manual composite samples
- Telescoping handle dipper – up and downstream, oil and grease, fecals

Sampling Equipment

Sampling Equipment, Vacuum Pump

Sampling Equipment…old reliable



Sampling Review

- Whether the sampling is for compliance or process control, the following applies
- Your process is only as good as the procedures employed to collect sampling
- Be consistent
- Collect representative samples
- Log sample history and results over course of time
- Have quick reference manuals / leaflets for all of staff for sampling procedures (general and specific to your facility)
- Talk to your contract lab for further assistance and questions…they are a resource for you!

Questions and Review

Thank You!

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