

# WASTEWATER SAMPLING

## OVERVIEW OF SAMPLE COLLECTION

KELLY WALLACE

PACE ANALYTICAL SERVICES, INC.

919-618-1498

[Kelly.Wallace@pacelabs.com](mailto:Kelly.Wallace@pacelabs.com)



- ❖ WHY DO WE SAMPLE
- ❖ SAMPLE LOCATIONS
- ❖ SAMPLE TYPES
- ❖ LAB WORTHY SAMPLE PROTOCOL
- ❖ QUALITY DATA AND REPORT REVIEW

# WHY DO WE SAMPLE

- 
- **Legal Requirements/Compliance**
    - NPDES Permit/Pretreatment Permit
    - 503 Sludge Regulations-Annual Sludge Report
  - **Process Monitoring and Control**
    - Optimize Treatment Efficiency
  - **Historical Data Collection.....**
    - Trends to Predict Plant Expansions
    - Seasonal Conditions



# SAMPLING POINT

Sampling point chosen should:

- Be representative of the discharge
- Be appropriate for the wastestream conditions
- Have no re-cycle streams
- Allow for unrestricted access at all times
- Be where flow is turbulent and well-mixed
- Include all outfalls that appear in the Permit



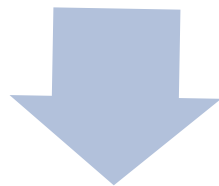
# REPRESENTATIVE SAMPLE



# SAMPLE SHOULD BE:

- Well Mixed
  - Not in an eddy or against the wall or at the bottom of a channel
  - Avoid areas where settling occurs or where large chunks of floating debris exist
- Upstream of Recycle Streams
  - Collect influent samples at a point upstream of any recycle stream discharge
- Uniformity
  - May have to take numerous samples to determine which is most representative spot

IDENTIFY SAMPLE POINT



ALWAYS SAMPLE AT THAT POINT SAME TIME SAME DAY



KEEP SAMPLING DEVICE AT SAMPLE  
LOCATION



# NPDES PERMIT SAMPLING

## B. EFFLUENT LIMITATIONS AND MONITORING REQUIREMENTS - FINAL

During the period beginning January 1, 2015 and lasting until December 31, 2019 the permittee is authorized to discharge from 3001 Firestone Parkway in Wilson, North Carolina, more specifically see Part I, Section A, of permit, into the City Sanitary Sewerage System. Such discharges shall be limited and monitored by the permittee as specified below.

Effluent Characteristics		Discharge Limitations		Monitoring Requirements		Detection Limits mg/L
Parameter	Units	Daily Max.	Monthly Average	Frequency City	Frequency IU	Sample Type
Flow	GPD	350,000	250,000	ES	D	report
BOD <sub>5</sub>	lbs/day	-----	-----	SA*	SA**	composite
COD	lbs/day	-----	-----	SA*	SA**	composite
NH <sub>3</sub>	lbs/day	-----	-----	SA*	SA**	composite
pH min-max	Units	5.0-11.0	-----	SA*	SA**	grab
TSS	lbs/day	-----	-----	SA*	SA**	composite
Chlorides	lbs/day	1043	-----	SA*	SA**	composite
Oil/Grease	mg/L	300	-----	SA*	SA*	grab
Cadmium	lbs/day	-----	-----	SA*	SA*	composite
Chromium	lbs/day	-----	-----	SA*	SA*	composite
Copper	lbs/day	-----	-----	SA*	SA*	composite
Cyanide	mg/L	-----	-----	SA*	SA*	grab
Lead	lbs/day	-----	-----	SA*	SA*	composite
Mercury	lbs/day	-----	-----	SA*	SA*	composite
Nickel	lbs/day	-----	-----	SA*	SA*	composite
Zinc	lbs/day	-----	-----	SA*	M*	composite
Arsenic	lbs/day	-----	-----	SA*	SA*	composite
Molybdenum	lbs/day	-----	-----	SA*	SA*	composite
Selenium	lbs/day	-----	-----	SA*	SA*	composite
Silver	lbs/day	-----	-----	SA*	SA*	composite

There shall be no discharge of floating oil/grease.

All monitoring reports are due no later than the 30<sup>th</sup> day of the following month of the sampling event.

Semi-annual monitoring must occur between January 1<sup>st</sup> and March 31<sup>st</sup> and July 1<sup>st</sup> and September 30<sup>th</sup> of each year.

(A) denotes annually, (SA) denotes semi-annually, (Q) denotes quarterly, (M) denotes monthly, (ES) denotes every sample.

(D) denotes daily: Flow rate recorded daily and sent to the City of Wilson WWMF by the 15<sup>th</sup> day of the following month.

(\*) denotes one (1) working day during a given sampling event.

(\*\*) denotes two (2) consecutive working days during a given sampling event.

Monitoring results shall be reported on the City of Wilson Self-Monitoring Report form and submitted to the Wastewater Management Facility. Permittee shall retain all records and information resulting from monitoring activities required by this permit including all records of analyses performed and calibration and maintenance of instrumentation and recording from continuous monitoring instrumentation for minimum of three (3) years.



# SAMPLE TYPES

## STREAM



## COMPOSITE



## GRAB



# GRAB SAMPLES

- Individual sample, usually taken manually
- Collected over period of time <15 minutes
- Used for parameters that cannot be held, are unstable or difficult to preserve
- Sample volume depends on number of analyses to be performed [Don't forget QA/QC samples...duplicates, "Murphy's Law", etc.]
- Represents conditions that exist at time of sampling [and not necessarily conditions at any other time]

# WHEN DO WE COLLECT GRAB SAMPLES

- Effluent screening to see if a pollutant is present
- Where statistical relationship can be established between grab/composite data
- When NPDES Permit has instantaneous limit, based on grabs
- When checking for extreme conditions
- When checking for “slug loads”



# GRAB PARAMETERS

- pH
- Temperature
- Dissolved Oxygen
- Residual Chlorine
- Micro (fecal)
- Cyanide
- Phenol
- Volatile Organic Analyses [VOAs]
- Low level Mercury

# LOW LEVEL MERCURY

- CLEAN HANDS/DIRTY HANDS SAMPLING  
PROTOCOL-Method 1669
- SAMPLE AND FIELD BLANK NEEDED

# VOLATILE ORGANICS (VOA)

- METHOD 624.1
- TOTAL TOXIC ORGANICS
- 1,4-DIOXANE
- NO HEAD SPACE
- PRESERVED AND UNPRESERVED VIALS DUE TO CHANGE IN METHODOLOGY



# COMPOSITE SAMPLES

- Formed by a number of discrete samples
  - Collected over period of time >15 minutes
  - Collected over a workday
- Can be taken with automatic sampler or manually
- Used to determine average concentration during compositing period
- Used for most compliance samples
  - NPDES Permit limited parameters



# COMPOSITE TYPES

- Time Composite
  - equal volumes taken at equal time intervals
- Flow Proportional Composite Method 1
  - equal time intervals with sample volume varying according to flow
- Flow Proportional Composite Method 2
  - equal volumes taken at varying time intervals in proportion to the flow
- Continuous Composite [i.e. on-line analyzer]
  - taking a small amount of flow continuously

# TYPICAL NPDES PARAMETERS

- GRAB SAMPLE

- pH
- Dissolved Oxygen
- Temperature
- Total Residual Chlorine
- Fecal Coliform
- Cyanide
- Oil and Grease
- Volatile Organic Compounds [VOCs]  
[EPA Method 624]

- COMPOSITE SAMPLE

- BOD/CBOD
- COD
- TSS/TDS
- Nutrients [TN, TP]
- Metals
- Whole Effluent Toxicity  
[Bioassay]
- Acid Extractable and  
Base Neutral Organics  
[EPA Method 625]



# SAMPLE EQUIPMENT



# GENERAL SAMPLING FREQUENCY

- Permit Requirements
  - Required Frequency [daily, weekly, monthly]
  - Required Sampling Points
  - Required Analyses
- Operational Parameters [Process Control]
  - Preference/experience of ORC/staff
  - “Cost” to analyze each sample

# OPERATIONAL CONTROL SAMPLING

- No NPDES permit requirements [no certified lab, no specified frequency]
- Often conducted by plant operators [not for DMRs]
  - Effluent [Residual  $\text{Cl}_2$ , pH, D.O., Temperature]
  - Effluent Grab samples [ $\text{NH}_3$ -N, phosphorus]
  - Aeration Tank D.O.s
  - Mixed Liquor [Settleable Solids, MLSS, SVI]
- Can be conducted several times per day 365 days per year [based on ORC decision]



# LAB WORTHY SAMPLE

LAB WORTHY

EQUALS

COMPLIANT

EQUALS

LEGALLY DEFENSIBLE



# PATH TO SUCCESS

FOLLOW PERMIT  
PROTOCOL-SAMPLE  
SCHEDULE-APPROVED  
SAMPLE EQUIPMENT

COLLECT SAMPLES IN  
PROPER CONTAINERS  
AND COMPLETE ALL  
PAPERWORK (ID, DATE  
AND TIME OF  
COLLECTION)

REVIEW LAB REPORT IN  
A TIMELY MANNER.  
CHECK ANY PERMIT  
LIMIT EXCEEDENCES

# SAMPLE CONTAINERS



# SAMPLE CONTAINERS

- All Samples MUST be Labeled
- Waterproof Labels and Ink Should Be Used
- Label Should Include:
  - Sample ID/Location
  - Date and Time of Sample Collected
  - Initials of Person Collecting Sample
  - Preservation Method
  - Requested Analyses

Pace Analytical  
9800

Town of Holly Springs  
Weekly

Client Sample ID: \_\_\_\_\_

Date Collected: \_\_\_\_\_

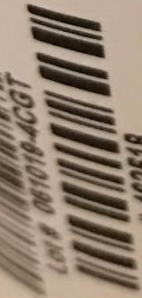
Collected By: \_\_\_\_\_

Ortho Phosphate / Water / 250mL Filter



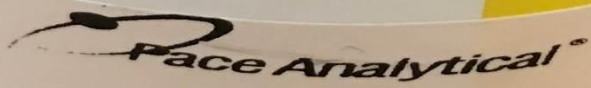
Contains  
Sulfuric Acid

AFFIXED  
C&G



Lot # 061019-4CGT

Container # 462518



**Preparation Date:**  
6/10/2019

**Chemical Expiry:**  
9/14/2021

**125cc Natural  
Leakproof W/M w/  
38PP cap**

**Chemical Lot:**  
3118041  
**Sulfuric Acid  
Solution**

Danger! May be corrosive  
burns and eye damage.  
Do not breathe dusts.  
face, hands and clothing.  
handling. Wear proper  
protection. Use in a  
well-ventilated area.  
Immediately flush with water.  
IF INHALED: Remove person  
to a position of comfort and  
rest. Rinse skin with water.  
Remove clothing and shoes.  
If swallowed: Do not induce  
vomiting. Drink water.  
Small amount.  
Avoid skin contact.  
Wash thoroughly with water.  
Do not eat, drink or smoke.  
Do not get on face or clothing.  
Do not breathe dusts.

# CHAIN OF CUSTODY

- Name of WWTP
- NPDES Permit #
- Sampling Location
- Sample ID
- Initials of Collector
- Composite Start Time
- Composite End Time
- Grab Sample Time
- Container(s) Description
- Preservative(s) Used
- Sample Pretreatment
- What tests to be run
- Relinquished and Received Spaces
- Comments Section



## CHAIN-OF-CUSTODY Analytical Request Document

Chain-of-Custody is a LEGAL DOCUMENT - Complete all relevant fields

Company:		Billing Information:	
Address:			
Report To:		Email To:	
Copy To:		Site Collection Info/Address:	
Customer Project Name/Number:		State:      County/City:      Time Zone Collected: /      [ ] PT [ ] MT [ ] CT [ ] ET	
Phone:	Site/Facility ID #:	Compliance Monitoring?	
Email:		[ ] Yes      [ ] No	
Collected By (print):	Purchase Order #:	DW PWS ID #:	
	Quote #:	DW Location Code:	
Collected By (signature):	Turnaround Date Required:	Immediately Packed on Ice:	
		[ ] Yes      [ ] No	
Sample Disposal:	Rush:	Field Filtered (if applicable):	
[ ] Dispose as appropriate    [ ] Return	[ ] Same Day    [ ] Next Day	[ ] Yes      [ ] No	
[ ] Archive: _____	[ ] 2 Day [ ] 3 Day [ ] 4 Day [ ] 5 Day		
[ ] Hold: _____	(Expedite Charges Apply)	Analysis:	

\* Matrix Codes (Insert in Matrix box below): Drinking Water (DW), Ground Water (GW), Wastewater (WW), Product (P), Soil/Solid (SL), Oil (OL), Wipe (WP), Air (AR), Tissue (TS), Bioassay (B), Vapor (V), Other (OT)

[illegible]

LAB USE ONLY- Affix Workorder/Login Label Here or List Pace Workorder Number or  
MTJL Log-in Number Here

**ALL SHADED AREAS are for LAB USE ONLY**

Container Preservative Type **										Lab Project Manager:									

\*\* Preservative Types: (1) nitric acid, (2) sulfuric acid, (3) hydrochloric acid, (4) sodium hydroxide, (5) zinc acetate, (6) methanol, (7) sodium bisulfate, (8) sodium thiosulfate, (9) hexane, (A) ascorbic acid, (B) ammonium sulfate, (C) ammonium hydroxide, (D) TSP, (U) Unpreserved, (O) Other

\*\* Preservative Types: (1) nitric acid, (2) sulfuric acid, (3) hydrochloric acid, (4) sodium hydroxide, (5) zinc acetate, (6) methanol, (7) sodium bisulfate, (8) sodium thiosulfate, (9) hexane, (A) ascorbic acid, (B) ammonium sulfate, (C) ammonium hydroxide, (D) TSP, (U) Unpreserved, (O) Other

Analyses					Lab Profile/Line:
					Lab Sample Receipt Checklist:
					Custody Seals Present/Intact Y N NA
					Custody Signatures Present Y N NA
					Collector Signature Present Y N NA
					Bottles Intact Y N NA
					Correct Bottles Y N NA
					Sufficient Volume Y N NA
					Samples Received on Ice Y N NA
					VOA - Headspace Acceptable Y N NA
					USDA Regulated Soils Y N NA
					Samples in Holding Time Y N NA
					Residual Chlorine Present Y N NA
					Cl Strips:
					Sample pH Acceptable Y N NA
					pH Strips:
					Sulfide Present Y N NA
					Lead Acetate Strips:

LAB USE ONLY:  
Lab Sample # / Comments:

Customer Remarks / Special Conditions / Possible Hazards:	Type of Ice Used:      Wet      Blue      Dry      None				SHORT HOLDS PRESENT (<72 hours) :    Y    N    N/A			LAB Sample Temperature Info:	
	Packing Material Used:				Lab Tracking #:			Temp Blank Received:    Y    N    NA	
	Radchem sample(s) screened (<500 cpm):    Y    N    NA				Samples received via:			Therm ID#:	
					FEDEX	UPS	Client	Courier	Pace Courier
Relinquished by/Company: (Signature)	Date/Time:	Received by/Company: (Signature)			Date/Time:	MTJL LAB USE ONLY			
Relinquished by/Company: (Signature)	Date/Time:	Received by/Company: (Signature)			Date/Time:	Table #:			
Relinquished by/Company: (Signature)	Date/Time:	Received by/Company: (Signature)			Date/Time:	Acctnum:			
						Template:			
						Prelogin:			
					Trip Blank Received: Y N NA				
					HCL MeOH TSP Other				



# SAMPLE RESULTS DELIVERY

- REPORT SHOULD INCLUDE
  - LABORATORY CERTIFICATION NUMBER
  - DATE AND TIME OF SAMPLE ANALYSIS
  - SAMPLE ID, DATE AND TIME OF COLLECTION
  - LABORATORY TEST RESULTS
  - ANY FOOTNOTES FOR ANALYSIS
  - COPY OF YOUR COC
  - QUALITY CONTROL DATA

# LABORATORY RESULTS

- ORC IS RESPONSIBLE FOR COMPLIANCE OF ALL SAMPLE AND ANALYTICAL DATA
- REVIEW REPORTS AS SOON AS YOUR LAB DELIVERS THE REPORT
- SAMPLE EARLY!! DO NOT WAIT UNTIL LAST DAY OF MONTH, QUARTER, ETC TO SAMPLE
- HAVE YOUR LAB INCLUDE PERMIT LIMITS ON REPORT



# DID YOU CALCULATE DATA CORRECTLY?

- MAKE SURE YOUR CALCULATION FORMULAS ARE UP TO DATE
- Geometric Mean for Fecal Coliform Values
  - Cannot Use “TNTC” Any More
- Arithmetic Mean for all Other Parameters
  - Less Than ( $<$ ) Values - Use Zero [per DWQ]
  - Greater Than ( $>$ ) Values - Use the Value
- Quarterly Average Calculation
  - Remember: Take average of all analyses... NOT average of the 3 monthly averages

# LABORATORY SUCCESS

- NOTIFY YOUR LAB WHEN YOU GET NEW PERMIT OR WITH ANY CHANGES TO YOUR PERMIT-Limits, Sample frequency, etc
- COMMUNICATE ANY CHANGES TO SAMPLE SCHEDULE
- PROVIDE YOUR LAB WITH COPY OF YOUR PERMIT OR SAMPLING SCHEDULING
- HAVE LAB INCLUDE PERMIT LIMITS ON REPORT

# LABORATORY SUCCESS

- DO YOUR LABORATORY HOMEWORK!!
  - CHECK REFERENCES
  - GET CERTIFICATIONS FROM LAB
  - CHECK QUALITY CONTROL DATA ON REPORT
- DEVELOP/MAINTAIN GOOD RELATIONSHIP WITH YOUR LAB
- GET REFERENCES FROM OTHER PLANTS ABOUT LAB
- REVIEW YOUR LAB'S AUDITS, PT STUDIES

# LET ME SAY THIS AGAIN

- BE FAMILIAR WITH SAMPLING POINTS AND EQUIPMENT
- KNOW YOUR SAMPLE SCHEDULE
- KNOW WHAT SAMPLE CONTAINERS ARE REQUIRED, HOLD TIMES, PRESERVATIVES, ETC.
- IF YOU DIDN'T WRITE IT DOWN, YOU DIDN'T DO IT!!
- DOCUMENT, DOCUMENT, DOCUMENT
- BE PROACTIVE-ORDER SAMPLE KITS EARLY, STICK TO SAMPLE SCHEDULE, REVIEW DATA PROMPTLY

**THANK YOU!!**

**[Kelly.Wallace@pacelabs.com](mailto:Kelly.Wallace@pacelabs.com)**

**919-618-1498**

**[www.pacelabs.com](http://www.pacelabs.com)**