

Sample Collection and Analysis

Dan Line

NCSU Water Quality Group

Quality Assurance Project Plan

- **Project Description**
- **Technical Design**
- **Project Organization & Responsibilities**
- **Project Schedule**
- **Sampling Procedures, Chain of Custody**
- **Analytical Procedures**
- **Quality Control Procedures**

Sample Collection

- **Manual**
 - Single Grab(s); composite; etc.
- **Passive**
 - Single grab(s); composite; etc.
- **Powered (automated)**
 - Single grab(s); composite; etc.
- **Keep Lid on Container & hands out**
 - Sample and lab container

Sample Volumes

- **Nitrogen & phosphorus series**
 - TKN, NO₃, NH₃, TP, OP: ~30 ml
- **Suspended sediment**
 - TSS, TDS, TS: 250 ml
- **Aggregate organics**
 - COD 30 ml; BOD 1000 ml
- **Semi volatile organics (PAHs), pesticides**
 - 2000 ml for the group
- **Metals**
 - Total metal content: 20 ml

Sample Preservation

Analyte	Preservation	Holding Time
NH ₃ , TKN	4 deg C; pH<2 H ₂ SO ₄	28 days
OP	4 deg C; filter immed.	48 hrs.
TP	4 deg. C; pH<2 H ₂ SO ₄	28 days
BOD	4 deg. C	48 hrs.
TSS	4 deg. C	7 days
Pesticides	4 deg. C; pH 5-9	7 days

Sample Preservation

- **Cool to 4 deg C**
 - Refrigerated sampler, ice
- **Lower pH to less than 2**
 - Put acid in sample bottles before sample collected
- **Holding Time**
 - Pick up samples quickly

Sample Analysis Method

- **Preservation/preparation Required**
 - OP: filter immed. vs dissolved P lab filters
- **Detection limit**
 - Lower MDL for some analyses
 - Match with water

Sample Analysis Method (costs)

Analyte	MDL	Cost
NH ₃ , NO ₃	0.04 ppm	\$13-25
TKN, TP	0.05-.15 ppm	\$20-40
TSS, TS	1 ppm	\$12-25
FC, FS	10 cfu/100ml	\$15-40
Metals	2-10 ppb	\$15-30
Pesticides	5-100 ppb	\$80-200

Quality Assurance- Field

- **Sample Contamination**
 - Equipment/container blanks, trip blanks
- **Sample Representativeness**
 - Field duplicates, splits
- **Sample Integrity**
 - Standard or Spike Samples

Quality Assurance- Field

- **Equipment/container Blanks**
 - Run distilled water through sampler, analyze
 - Fill sample bottle with distilled water, analyze
 - Do 10%, should be <MDL or 10% of mean
- **Field Duplicates**
 - Collect 2 samples same way; analyze
 - No statistical difference, LC diff < 10% of mean for TP, TKN, NO₃, TSS; FC < 20%
- **Standards or Spikes**
 - Make standard and treat like sample; analyze

Quality Control- Lab

- **State Certified lab**
- **Components**
 - Blanks, spiked blanks, equipment blanks
 - standards
 - Duplicates
 - Within lab
 - Outside lab

Quality Control

- **Long Creek Example**
 - 5 samples sent to 2 labs
 - 25 duplicates within lab; no stat diff.
 - 7 trip blanks; only 1 TKN, NO₃, TP >MDL
 - 2 equipment blanks; minimal level

Quality Control (fixes)

- **Clean Containers**
- **Check Handling- hands**
- **Clean Equipment**
- **Qualify data**
 - Add measure of variability
 - Discard data

Quality Control

- **Data Review**
 - Outliers (>2 stand. dev): check typos, covariates
 - LC at site D, various lab typos
- **Maintain Consistency in Collection**
 - Sampler intake moves, location moves
- **Develop Written Plan**
 - Different people have procedure, don't forget

Summary

- **Develop and Maintain Good Procedures**
- **Preserve Samples**
- **Use Duplicates, Blanks on 10% of Samples**
- **Review Data**
- **Write Procedures to Maintain Consistency**