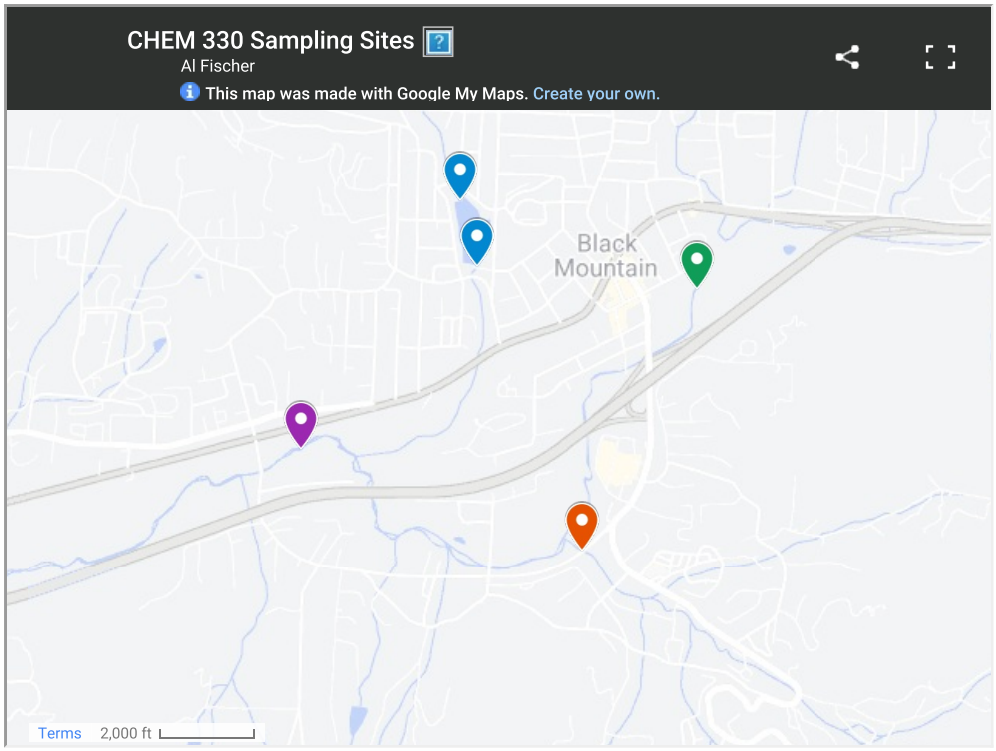
Class Project

The goal of the lab project this semester is to provide the Town of Black Mountain (TOBM) with *accurate* data they can use as a baseline dataset for their stormwater program. The results from your analyses will be shared with Black Mountain. Since this data will be used to inform TOBM about the water quality in the Swannanoa River and its tributaries, it is imperative that it is of the highest quality! As you work through each lab be vigilant about avoiding contamination, and use appropriate quality control measures to check your accuracy and precision as you work. You *must* get an accurate answer before turning in your final report; if your results are not accurate or have unacceptably low precision you must repeat the analysis again to obtain high-quality data.

One intent for this project is to help you become self-sufficient in the lab and help you learn how to know that your data is likely to be accurate. As such, there will be little day-to-day direction of your activities from your instructor, and it will be up to you and your lab partner to plan your time and implement appropriate practices in your lab work.

Sample Collection

Samples will be collected in several locations in Black Mountain, NC (shown below).



1. (This step has been done for you.) Wash 3 1-L plastic sample bottles with hot water and rinse 3X with DI water. Seal the bottle until sample collection.
2. (This step has been done for you.) Fill one of the bottles with DI water as a field blank.
3. Fill out information on attached datasheet.

> ⚠ Note
>
> You will need to make measurements of dissolved oxygen (DO) and temperature with the YSI meter.

4. Collect water samples – be mindful of contamination as you do this!
    1. Use labeling tape and a permanent marker to place a label on the sample bottle listing the sampling site, your name, and the date.
    2. Open the field blank while you are collecting samples.
    3. Enter the water (if it is safe to do so) and wait a minute for any sediment to settle or get carried away.
    4. Open the bottle and rinse it with a small amount of water from the river; discard the rinse water.
    5. Fill the bottle with water from the river, making sure to sample upstream of where you are standing.
    6. Immediately cap the bottle and keep it capped until sample processing or analysis.
    7. Cap the field blank; keep the field blank and sample bottles near each other during transport.
5. Process the samples.
    1. Filter the samples through a 0.45 μm filter using a Millipore apparatus.
    2. Place the filtered sample into a clean plastic bottle (or wash the old one if needed).
    3. Take approximately 250 ml of sample and place it in a clean plastic bottle. Acidify with sulfuric acid to pH < 2. This sample will be used for nitrate analysis. (You can use pH paper to measure the pH.)
    4. Place all samples in a refrigerator at 4°C until analysis.

Analysis

For each analysis you perform, you should create a daily plan and check in with Dr. Fischer prior to beginning the analysis. When you finish the analysis, you should share your results with Dr. Fischer prior to moving on to the next analysis. It is in your best interest to process as much data as possible in real time.

You may need to make solutions and gather equipment as part of your lab work, even if they were provided during previous labs. The main exceptions to this will be any solutions requiring the use of concentrated acids or bases, or exceptionally toxic chemicals (e.g. mercury).

Laboratory Practice

It is imperative that you avoid contamination during your lab work to avoid inaccurate results! Remember to wash all glassware with Alconox and warm water and then rinse it three times with deionized water prior to beginning analysis. (The exception to this is the phosphate analysis – remember that Alconox contains a high percentage of phosphate!) Do not assume the glassware is clean!

It is also important that your results are as accurate and precise as possible. Remember to use the right glassware for each sitution.

- Use volumetric pipettes to deliver a specified volume. Do not combine pipettes to get a desired volume; instead, find the pipette of appropriate volume (or ask for one if you can't find it).
- Use volumetric flasks to contain a specified volume.
- Record all certain digits plus one uncertain digit when writing down data.

It is also important that you have high statistical confidence in the data. Measure all samples in triplicate and report

Required Analyses

You should conduct the following analyses on your samples:

> ⚠ Note
>
> As you work through the following methods, make sure to keep track of any deviations you make from the standard method provided. For example, if the method calls for sodium chloride and you use potassium chloride instead, make that clear.

- Nitrate (Colorimetric/NitraVer 5 Method)
  - Passing \(R^2 > 0.990\)
  - Passing QC is \(\pm 10\%\) of the expected value
  - Analyze samples in triplicate
  - Report as mg nitrate per L with 95% confidence interval

    > ⚠ Note
    >
    > You must use the Agilent 8453 Diode Array UV-visible Spectrometer for this analysis. You will need to get trained by Matt Burleson, CHPH Instrumentation Specialist prior to using the instrument. Please contact him to arrange training.

- Phosphate (Colorimetric Method)
  - Passing \(R^2 > 0.990\)
  - Passing QC is \(\pm 10\%\) of the expected value

- Analyze samples in triplicate
- Report as mg phosphate per L with 95% confidence interval

> ⚠ Note
>
> You must use the Agilent 8453 Diode Array UV-visible Spectrometer for this analysis. You will need to get trained by Matt Burleson, CHPH Instrumentation Specialist prior to using the instrument. Please contact him to arrange training.

- Alkalinity (potentiometric titration - use the USGS Method)
  - Analyze samples in triplicate
  - Make sure to standardize your acid against a primary standard
  - Report as mg/L CaCO\(_3\) using the Gran plot method and give the 95% confidence interval
- Hardness (complexometric titration with EDTA - use EPA Method 130.1)
  - Analyze each sample in triplicate
  - Make sure to standardize your EDTA against a primary standard
  - Report as mg CaCO\(_3\) per L with 95% confidence interval
- Total Dissolved Solids (gravimetric - use EPA Method 160.1)
  - Report your results as mg filterable residue per L
  - *If you have enough sample* perform this analysis in triplicate and provide the 95% confidence interval
- Dissolved Oxygen (collected at site using YSI meter)
- Temperature (collected at site using YSI meter)
- Conductivity (use the electrode provided in class)
- Total Anions (use EPA Method 300.0)
  - Use 5 standards that bracket your sample(s).
  - A passing standard curve will have an \(R^2 > 0.9995\).
  - Use a rinse blank after your high standard
  - A passing QC is \(\pm 10\%\) of the expected value; run the QC after your standards, after every 10 samples, and always finish the run with a QC.
  - Analyze samples in triplicate
  - Analyze blanks once each (field blank, rinse blank, and lab/reagent blank)
  - Report results as mg nitrate per L with 95% confidence interval

> ⚠ Note
>
> You must use the Ion Chromatograph for this analysis. You will need to get trained by Matt Burleson, CHPH Instrumentation Specialist prior to using the instrument. Please contact him to arrange training.

If time / resources allow, we will also complete one or more of the following:

- Analysis of suspended sediments using scanning electron microscopy

> ⚠ Note
>
> You must use the SEM for this analysis. You will need to get trained prior to using it.

- Mercury concentration

> ⚠ Note
>
> You must use the flow-injection mercury system for this analysis. You will need to get trained by Dr. Fischer prior to using it.

Waste Disposal

- Waste for nitrate, phosphate, hardness, and anion analyses can be combined in a the waste bucket provided in lab.
- Neutralized waste from the alkalinity titration can be disposed of down the drain.
- Dissolved solids wastes can be disposed of in the trash and/or down the drain.

Reporting

You should present your results in two ways:

1. A professional-quality lab report written in the style of a USGS water quality report. (This counts toward your lab grade.)
2. A professional-quality pamphlet showing a summary of the results, prepared in the style of a municipal consumer confidence report (CCR). (This will count as part of your final exam grade for lecture.)

There will likely be a lot of overlap between these two documents – if you plan appropriately you may be able to use many of the same figures and data tables. The focus of the lab report is the lab work and the results from it, with some comparisons to other data sources for context. The focus of the CCR-type report should be on summarizing the results and their implications for a general audience.

I encourage you to use a "divide and conquer" approach to writing this report – you each likely have different skills that you can use to benefit the group, so that breaking up the writing and figure preparation will both be more efficient and will produce better results.

Lab Report

This report should be written as if it were a professional document prepared to be shared with aquatic chemists and other water-quality professionals who are familiar with water chemistry and chemical analysis. You can assume this knowledge when writing it (e.g. you do not need to explian what a titration is, but rather can just say you performed a specific type of titration).

- You should follow my style guide for scientific writing when writing your report.
- You should also read my guide to publication-quality figure. All figures in your reports should be "publication quality".
- Some example reports are available via this link (PDF) and this link (PDF).
- An example "A" student report (from another class) can be viewed here.
- A Word template for your report is available here.
- The rubric I will use to grade your report is available as a PDF download here.

We will conduct one or two rounds of peer-review on these reports. For the first round, aim to have the introduction written and whatever portion of the other section you are able to complete based on the analyses you've finished. For the second peer-review, you should fix any errors noted in the first round and have the report almost complete.

Your report should include the following sections:

1. Title
2. Abstract
3. Introduction
    1. Purpose and Scope
    2. Previous / Similar Studies (if applicable)
    3. Description of the Study Area (including map)
4. Methods
    1. Surface Water Sampling
    2. Chemical Analysis (subsection or paragraph for each parameter)
5. Results and Discussion
    1. Results of your analyses, with comparisons to previous / similar studies / generally accepted values
6. Conclusions
7. Works Cited

Consumer Confidence Report

This report should be written as if it were a professional document designed to be shared with the general public. It should be designed to educate people who are unfamiliar with water quality parameters about the importance of each parameter and how it relates to the bigger picture. Write this as if you were explaining your lab work to a family member or friend who is not a scientist.

A good example CCR is the City of Raleigh CCR (PDF). Every public water supply is required to provide a CCR to it's users (including WCU!), so feel free to browse for additional examples.

Your CCR should include a short (~1 paragraph) summary of each parameter measured and why it's important. You should also include the data you measured with "typical" values for context. It is important to explain why each parameter is important and also give context as to whether each value you measured is normal or cause for alarm.

> ⚠ Note
>
> CCRs are provided for drinking water. Since we are measuring surface water as part of a stormwater program, this is not a true CCR! It is just a convenient format for us to use for reporting the results.
