Laboratory Week 3   Environmental Science 101

Laboratory Analysis of Stream Samples

Purpose:

1. Determine conductivity, total suspended solids (TSS) and soluble reactive phosphorus (SRP) on your stream samples from last week, using standard methods and quality control. Prepare samples for ICP analysis of potassium.
2. Observe the methods for fecal coliforms, *E. coli* and biochemical oxygen demand (BOD),

1. Laboratory conductivity (Aiken 202)

Using more reliable and precise meters and probes, you will remeasure the conductivity of the 10 stream samples that you collected last week. Rinse a test tube with a small portion of your stream sample to be tested and then fill the tube about 2/3 full. Rinse the probe thoroughly with distilled water and lower it fully into the test tube, moving it up and down a few times to remove air bubbles. Allow the reading to stabilize and record it. Do this for all samples, running at least one in duplicate. Also determine the conductivity of a standard provided by your lab instructor.

2. Total suspended solids (TSS) (Aiken 202)

Total suspended solids are solids in water that can be trapped by a filter. TSS can include a wide variety of material, such as silt, decaying organic matter, and industrial wastes. Read the procedure from Standard Methods for the Examination of Water and Wastewater (handed out in lab) and then follow the simplified procedure in Appendix I. Use the following samples: 1) your group's "disturbed" sample, 2) your group's second sample, and 3) the TSS quality control sample provided. If you have time, run your disturbed sample in duplicate.

3. Soluble Reactive Phosphorus (SRP) (Hills 20)

Soluble reactive phosphorus is primarily a measure of orthophosphate (PO_4), the inorganic form of phosphorus that is readily available to algae. For this lab, you will analyze 3 samples for SRP, following methods provided in Appendix II. Use the following samples: 1) your group's "disturbed" sample, 2) your group's second sample, and 3) the SRP quality control sample provided. Run one of your two unknowns in duplicate.

4. Potassium results from the tracer study (Hills 20)

Potassium will be determined using an ICP-AES (inductively coupled plasma atomic emission spectrometer). The results should be available next week at the lab website. The calibration standards and quality control results will be included in the file. You will run all 10 stream samples, including 1 duplicate and 1 blank (distilled water). To prepare all of your samples for analysis, clearly label 12 ICP tubes with your group letter and sample number (i.e. A-1, A-2,…A-blank, A-dup). Rinse the tube with a small amount of your sample and then fill it about ¾ full. **Do not include your disturbed, TSS sample**.

5. Biological Analyses (BOD and E.coli) (Hills 135)

Biochemical Oxygen Demand (BOD) is a measure of the oxygen used by microorganisms to decompose organic materials found within the water. Fecal coliforms and *E. coli* are indicators of human or animal waste contamination. Both these measurements are critical in determining the state of impacted waters. Eamon Twohig, a technician and graduate student in Plant & Soil Science, will demonstrate the procedures for each. Handouts will be given during the lab. These methods will be used by many of the project groups.

Details of Lab Report

One report will cover two labs (the tracer study and lab analysis). Use units of meters and seconds. The following need to be included:

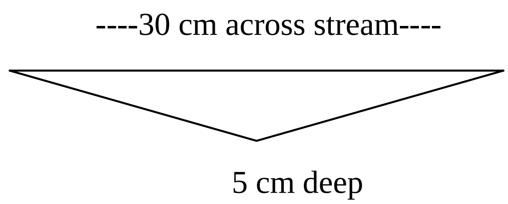
Methods

Provide a brief description of field and lab methods, concentrating on anything that deviated from the lab handouts. Do not reiterate material on the handouts, but refer to it.

Data

Provide the following data in clearly labeled tables or graphs.

1. Field data:
    a. Time and conductivity of your samples
    b. Flow meter readings and cross-sectional area measurements. Because of low flow, we only have one reading at one point. The velocity measured was 0.17 m s^{-1}.



2. Lab data:
    a. Conductivity and K concentration of your samples and related QC samples.
    b. Plot K concentration vs. time and vs. conductivity.
    c. TSS and SRP results from two of your samples and the laboratory QC sample.
3. Flow data:
    a. Calculate velocity to each station based on the time that it took to reach the maximum concentration. In cases that no maximum is clear, abandon all hope (or estimate).

Group	Distance (m)	Group	Distance (m)
A	3.4	H	4
B	8.5	I	8
C	12.5	J	12
D	22	K	16
E	36	L	20
F	50		
G	60.6		

    b. Calculate velocity and discharge based on the flow data above (1b.). Also calculate discharge based on the attached flow data from a stream in the Lye Brook Wilderness Area (Appendix III).
    c. Flux: Calculate the daily export of Na and Cl using data from samples taken on 9/4/01: Stream V= 0.13 m s^{-1}; Cross sectional area = 975 cm^2; Na = 340 mg L^{-1}; Cl = 566 mg L^{-1}.

Discussion

1. How would you rate the quality control data for your laboratory measurements (including the potassium results)? Were the QC results within 5% of their intended value? Were duplicates with 5% of each other? Was the ICP blank for K low enough?
2. Was there a good relationship between K concentration and conductivity? Why or why not?
3. What is the shape of your plot of K concentration vs. time and why?
4. Estimate the longitudinal dispersion coefficient (D_L) from the data using equation 2-12 (handed out in lecture). Drop the e^{-kt} from the equation because no degradation should occur. Remember that M is M per cross-sectional unit area. Assume this to be 1000 cm^2. We added 2 L of 2 molar KCl. Does your result seem reasonable based on values given in the text?

Appendix I

TSS:

Filters have been selected and prepped so ignore A and B. Follow instructions in section C, using 3 samples (your TSS sample, your second sample, and the TSS standard).

1. Record weight of filter
    a. Weigh filter and put it in **labeled** tin using forceps!!! No fingers – oils from hand can artificially increase weight. Label tin with ball point pen – make indent in aluminum – markers burn off. Label tin with Group # and Sample #
2. Set up filter apparatus – funnel, flask, tubing, vacuum
3. Rinse filter with distilled water (approximately 5mL)
4. Put stirring rod in sample and mix on magnetic stirrer
5. While stirring sample, pipet sample into filter and **record volume** used (25mL)
6. Allow all liquid to pass through filter
7. Rinse with distilled water (approximately 30mL) THROUGH PIPET!!!
8. Allow to suction for 3 min. after rinsing.
9. Remove filter, place in same aluminum tray
10. Dry for 12 hours at 103-105 C
11. Record weight

Calculation: (end weight – tare weight) / volume filtered = TSS
Report results in mg/L

2540 D. Total Suspended Solids Dried at 103–105°C

1. General Discussion

a. Principle: A well-mixed sample is filtered through a weighed standard glass-fiber filter and the residue retained on the filter is dried to a constant weight at 103 to 105°C. The increase in weight of the filter represents the total suspended solids. If the suspended material clogs the filter and prolongs filtration, the difference between the total solids and the total dissolved solids may provide an estimate of the total suspended solids.

b. Interferences: See 2540A.2 and 2540B.1. Exclude large floating particles or submerged agglomerates of nonhomogeneous materials from the sample if it is determined that their inclusion is not desired in the final result. Because excessive residue on the filter may form a water-entrapping crust, limit the sample size to that yielding no more than 200 mg residue. For samples high in dissolved solids thoroughly wash the filter to ensure removal of dissolved material. Prolonged filtration times resulting from filter clogging may produce high results owing to increased colloidal materials captured on the clogged filter.

2. Apparatus

Apparatus listed in Sections 2540B.2 and 2540C.2 is required, except for evaporating dishes, steam bath, and 180°C drying oven. In addition:

Aluminum weighing dishes.

3. Procedure

a. Preparation of glass-fiber filter disk: Insert disk with wrinkled side up in filtration apparatus. Apply vacuum and wash disk with three successive 20-mL portions of reagent-grade water. Continue suction to remove all traces of water, and discard washings. Remove filter from filtration apparatus and transfer to an inert aluminum weighing dish. Take care to prevent the dried filter from adhering to the weighing dish. Alternatively weigh dried filter and weighing dish both before and after filtration. Filter material that sticks to the dish must be added to the filter to avoid error. If a Gooch crucible is used, remove crucible and filter combination. Dry in an oven at 103 to 105°C for 1 h. If volatile solids are to be measured, ignite at 550°C for 15 min in a muffle furnace. Cool in desiccator to balance temperature and weigh. Repeat cycle of drying or igniting, cooling, desiccating, and weighing until a constant weight is obtained or until weight change is less than 4% of the previous weighing or 0.5 mg, whichever is less. Store in desiccator until needed.

b. Selection of filter and sample sizes: See Section 2540C.3*c*. For nonhomogeneous samples such as raw wastewater, use a large filter to permit filtering a representative sample.

c. Sample analysis: Assemble filtering apparatus and filter and begin suction. Wet filter with a small volume of reagent-grade water to seat it. Stir sample with a magnetic stirrer, and while stirring, pipet a measured volume onto the seated glass-fiber filter. Wash with three successive 10-mL volumes of reagent-grade water, allowing complete drainage between washings, and continue suction for about 3 min after filtration is complete. Samples with high dissolved solids may require additional washings. Carefully remove filter from filtration apparatus and transfer to an aluminum weighing dish as a support. Alternatively, remove the crucible and filter combination from the crucible adapter if a Gooch crucible is used. Dry for at least 1 h at 103 to 105°C in an oven, cool in a desiccator to balance temperature, and weigh. Repeat the cycle of drying, cooling, desiccating, and weighing until a constant weight is obtained or until the weight change is less than 4% of the previous weight or 0.5 mg, whichever is less. Duplicate determinations should agree within 5% of their average. If volatile solids are to be determined, treat the residue according to 2540E.

4. Calculation

$$\text{mg total suspended solids/L} = \frac{(A - B) \times 1000}{\text{sample volume, mL}}$$

where:

A = weight of filter + dried residue, mg, and
B = weight of filter, mg.

5. Precision

The standard deviation was 5.2 mg/L (coefficient of variation 33%) at 15 mg/L, 24 mg/L (10%) at 242 mg/L, and 13 mg/L (0.76%) at 1707 mg/L in studies by two analysts of four sets of 10 determinations each.

Single-laboratory duplicate analyses of 50 samples of water and wastewater were made with a standard deviation of differences of 2.8 mg/L.

6. Bibliography

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Appendix II

Soluble and Available Phosphorus

Soluble reactive phosphorus (SRP) in water is defined as the quantity of P that reacts with the colorimetric test used (e.g. Murphy-Riley ascorbic acid or stannous chloride blue). The majority of the P being measured is orthophosphate (PO_4^{3-}) found under "normal" pH conditions as either $H_2PO_4^-$ or HPO_4^{2-}.

Because the test acidifies the sample, some non-orthophosphate-P may be converted to orthophosphate and contribute to the test—thus, the name "reactive".

Water samples need to be filtered through 0.45 µm pore-size filters before analysis—thus, the name "soluble". This cutoff between soluble and particulate is somewhat arbitrary and "operationally defined." However if particles are left in the solution, the acidification could easily solubilize sorbed or precipitated P.

SRP has a short hold time because of potential changes in the sample. Most sources recommendation running it within 24 hours of sampling.

We will use the stannous chloride version of the test. The mechanism is that the orthophosphate forms a soluble complex with the added molybdate. This complex is colorless but becomes blue after reduction by adding $SnCl_2$. A number of things can affect the results including i) the freshness of the reagents, ii) the pH of the sample (should be near-neutral), and iii) the presence of interfering ions such as arsenic (positive interference).

SRP determination.

1. In a clean test tube, add 6.0 mL of your filtered water or weak salt extract.
2. Add 2 mL of ammonium molybdate solution and mix.
3. Add 1 mL of stannous chloride solution and mix thoroughly.
4. Allow 5 minutes for color development and measure the absorbance of the sample on a spectrophotometer set at a wavelength of 660 nm.
5. The lab instructor will prepare a series of known standards and provide the absorbance results. Plot concentration vs. absorbance for use as a standard curve.

Appendix III.

This is a stream we have been monitoring in southwestern Vermont. The spreadsheet was developed by the USGS to do discharge calculations from velocity meter measurements. You can ignore everything in blue. Use the data in light green to calculate discharge. Convert to metric units.

HMST v2.9 One-Point Velocity Discharge Measurement Checker With EDIs

By Brian Loving, Hydrologist, US Geological Survey

Station Name:	isco Kelly Stand					Print Header
Station Number:		**WIDTH**		**# of Sections**	0	
Date:	10/26/06	**AREA**		**Sections > 5%**	0	Print Full Sheet
Measurement #:		**VELOCITY**		**Sections > 10%**	0	
Meter Type:	Pygmy			***Standard Error**		Clear Sheet
Rating # (1 or 2)	2	**TOTAL DISCHARGE**				
# of EDI Verticals	5	***Measurement sheet Q is OK if between 0.00 and 0.00***				Select a Bed Typ

EDI Sample #	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**
Location to Sample									

	Distance (ft)	Width (ft)	Depth (ft)	Revolutions	Seconds	Velocity At Point ft/s	Area	Discharge
	1.9	0.15	0.49	49	60	0.816		
	2.2	0.30	0.31	82	60	1.344		
	2.5	0.30	0.31	42	60	0.703		
	2.8	0.30	0.36	52	60	0.864		
	3.1	0.30	0.46	90	60	1.472		
	3.4	0.30	0.51	57	60	0.944		
	3.7	0.30	0.6	36	60	0.607		
	4	0.35	0.59	24	60	0.415		
	4.4	0.35	0.52	38	60	0.639		
	4.7	0.30	0.48	32	60	0.543		
	5	0.30	0.46	11	60	0.207		
	5.3	0.15	0.41	15	60	0.271		

Assume each segment is rectangular. Ignore the Revolutions and Seconds, these were used to calculate Velocity.
Final result should be in cubic meters per second.