SALMON FOREVER

SUNNY BRAE SEDIMENT LAB

HY 99

LABORATORY PROCEDURE FOR TOTAL SUSPENDED SOLIDS

Redwood Sciences Laboratory

USDA Forest Service

Arcata, CA

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Data Forms

1. Arrange the sample bottles in order by:
   Station ID
   Data Dump #
   Bottle Number
2. Start a new data form for each station.
3. Data forms are filled out as each data dump is processed.
4. Under no circumstances should information for more than one station be recorded on the same data form.
5. You may include more than one data dump on a single form if the dumps are in sequential order.
6. Carefully read the bottle labels.
7. Transfer all data from bottle labels to the lab data forms (double-check the data as it is entered).
8. The bottles are labeled as follows:
   Data Dump Number - Station ID - Bottle Number
   For example, 04DOL11 would indicate data dump 4, station DOL, bottle number 11. In addition, the first bottle in the dump will have the number of bottles in the dump in parentheses.
9. There are three types of samples and lab forms:
   TTS (Turbidity Threshold Samples)
   - Samples are collected in ISCO 1000ml plastic bottles (the nominal sample volume is 350ml).
   - These samples are collected under data logger program control when pre-established turbidity threshold criteria are met.

   AUX (Auxiliary Samples)
   - AUX samples are similar to TTS samples but are manually triggered via the data logger program.

- AUX samples are collected when too few samples have been collected during a storm and when equipment has malfunctioned.
- The label, in addition to normal identification, should also include "AUX".

DIS (Depth Integrated Samples)

- Samples are collected in 500ml (1-pint) glass "milk" bottles.
- These samples represent the cross-sectional average sediment concentration and are used as "truth" to correct the TTS pumped samples (which are not flow-averaged, but are point samples).
- A simultaneous pumped sample is collected, via the data logger program, while the field crew manually collects the DIS bottle.
- The plastic ISCO bottle, in addition to the normal identification, should also include "DI". (The DIS and matching ISCO "DI" bottle should always be analyzed as a pair; e.g. they both would require sand fraction analyses to permit comparison later).

Volume Marks

1. Volume marks are made on all bottles in the field.
2. The volume mark on plastic ISCO bottles is made on a strip of 3M nylon first aid tape on the edge of the bottle.
3. The volume mark on glass DIS bottles is made on the vertically etched strip with a pencil.
4. Check the volume mark for accuracy.
5. Place bottle on a level counter and loosen the cap if it is a plastic bottle.
6. Compare the volume mark on the tape or etched glass to the actual water level (read the level from the bottom of the meniscus).
7. If the difference between the volume mark and the actual sample volume

is more than 1.0cm attempt to determine the cause (cap not tight, cracked bottle, tape did not adhere, mark missing, unknown cause, etc.).

- Assign a SC (Sample Code) of 6 on the lab form.
- After processing the sample, fill the bottle to the field volume mark then weight the bottle and record the weight.
- Make notes in "Comment" column.

Preparing Filters

1. Always handle filters with forceps.
2. A fingerprint weighs approximately 0.0001 gm. This adds a 10% error for a sample weighing 0.0010g.
3. When first taking filters out of the box, inspect them carefully since they have a tendency to stick together; separate them as necessary. Hold each filter up to the light to verify there are no holes. Discard defective filters.
4. Write the filter ID number on "furry" side of filter with light pressure from a dull-pointed "Ultra-Fine Sharpie" marker. Underline the numbers so that they will not be confused when read upside down. Wait 10 minutes to allow the ink to dry before rinsing filter.
5. Record filter ID number on initial tare sheet. If a filter is contaminated or punctured, discard that filter and record it as a "discarded" on tare sheet (the "missing" filter is then accounted for).
6. Seat filters, slightly off-center, with furry side down on the vacuum manifold.
7. Turn on the vacuum and rinse the filter several times with lab grade water to remove any loose fibers and to check for holes. If there is a hole, air will whistle through it and make a jet like sound.
8. Turn off the vacuum and carefully remove filter. Place the filter on a

wire rack and allow 1/2 hour for the filters to air-dry.

9. Place filters on a Teflon or glass pan and heat in oven at 105°C for 1/2 hour.
10. Remove pan from oven and place in desiccator cabinet to cool for at least 1/2 hour before weighing. Do not remove filters from desiccator cabinet until ready to weigh since they will absorb moisture from the air.
11. Take out only a few filters at a time. If the last filter in your batch has been out of the desiccator cabinet for more than 10 minutes before weighing, you have taken out too many filters. Reduce the number of filters next time a new batch is removed. The filters should not be weighed when the relative humidity in the desiccator is more than 28%. Try to keep humidity level in the desiccator between 10-25%. When the humidity approaches 20%, place the desiccant in the oven to bake overnight. Never weigh filters when your hair or clothing is wet.
12. Weigh each filter and record the weight on initial tare sheet. Store prepared filters in clean, dust-free, container or, a pan covered with aluminum foil. Do not stack or overlap the filters.

Suspended Sediment Determination

1. Process filters in numerical order.
2. Place a pre-tarred filter, numbered side down, on vacuum manifold.
3. Record the filter ID number on the appropriate suspended sediment data sheet.
4. Transfer the initial weight value to the data form before weighing. Use a separate sheet for each station.
5. Turn on vacuum and wet the filter with lab grade water to check for tears or holes. Attach the plastic funnel cup.

6. If the display on the top loading balance does not read 0.0 g, press th TARE button before placing the bottle on the balance.
7. Weigh the sample with the cap off.
8. Record the weight (xx.x) under the "Total Bottle Wt." column.
9. Pour the sample from the bottle into funneling cap. For faster filtration, try to pour the clear water through first without disturbing the sediment on the bottom. Rinse the inside of the cap onto filter.
10. Rinse sediment at bottom of the bottle onto filter, using lab grade water. Rinse the sample bottle several more times, making sure to remove all of the sediment. It may take several filters per sample to assure a reasonable filtering speed and drying time; if it takes more than two seconds per drop of filtrate, then another filter should be used.
11. Rinse the plastic funnel cup thoroughly with squirt bottle. Carefully rinse the cup O-ring and threads over filter after cup is removed.
12. Turn off vacuum and carefully remove filter and place on wire rack for one-half hour.
13. Record the number of filters used under the column labeled "Filter Total". If total number filters used is one, the notation in column Filter Total would be "11", or one-of-one. If two filters are used, the notation would be '12' (one-of-two) for the first filter, and "22" (two-of-two) for the second filter, and so on. Since it is difficult to determine how many filters per bottle will be required, process the bottles in numerical order (so enough space is available for multiple filters on the data sheet).
14. Remove any large debris (e.g. leaves, wood, algae, hair, etc.) with tweezers, and describe it in the Comment column. Try to remove the debris while the water is in the funnel to avoid the loss of sediment.

If this proves too difficult, carefully remove the debris while the filter is on the drying rack.

15. Record spills, errors, or notes in the comment column of the data form. It is important to record any observations or suspicions that may explain unusual results.

Drying and Weighing

1. After 1/2 hour on wire rack, place filters in a clean pan and heat at 105C for 1 and 1/2 hours.
2. Remove pan from oven and place in desiccator to cool for at least 1 hour before weighing.
3. Open the right sliding door of the balance (if you are left-handed move the anti-static device to the left side).
4. Check the pan for debris, and if present, gently brush it off with one of the brushes inside the balance. Close the sliding door.
5. Check the display and if it does not read 0,0000g then press the TARE button and wait several seconds before continuing, (this must be done with the sliding doors closed).
6. Open the sliding door and carefully place the filter on the center of the weighing pan then close the door.
7. Wait until the right-most digit stabilized and the "g" is displayed, and then record the weight on the data sheet.
8. Open the door and remove the filter.
9. Close the door.
10. Check the final weight against the initial weight. The final weight should be larger. If the initial weight is larger than the final weight try to determine where the error occurred.

Data Form Completion

1. Make sure all the data is on the data form and check it for errors.
2. See that all pertinent remarks are recorded.
3. Confirm that the station, date, and your initials are filled out on top of data form.
4. The "Sample Code" column indicates the quality of the sample and processing procedure. The codes are:

Sample Code	Condition	Description	Sample
0	No Problems	No Problems	Good
1	Final Weight less than Initial Weight	Error is 0.0005g or less and sediment is not evident	Good/Fair
2	Excess Debris	A significant portion of the sample weight is not sediment	Poor
3	Lost/Spilled Slug Excrement Weighing Error	A significant portion of the sample was lost or spilled; Slug excrement in sample	Worst
4	Low Volume	Processed, less than 150ml	Poor
5	Low Volume	Not Processed, less than 150ml	Discarded
6	Volume Mark Error	Volume Mark and Actual Sample volume differ by more than 1.0cm	Fair/Poor
7	Not Used		
8	Light Debris	Many small pieces or debris, but not a significant portion of the sample	Good/Fair

1. After double checking the data forms, file the data forms.
2. A computer program will compute the concentration in ppm of the total suspended sediment (including sand fraction, if present) and the concentration in ppm of the sand fraction (if it was separated). The computation is as follows:

$$\frac{(NetWt._sand_fraction)+(NetWt._suspeneded_sediment)}{Volume} x1{,}000{,}000 = ppm$$

$$\frac{NetWt._sand_fraction}{Volume} x1{,}000{,}000 = ppm$$

Where *NetWt.* = (Final weight - Initial weight)

Washing Procedure

1. Wash all sample bottles, lids, and glassware, using Alconox powdered soap and hot water. Rinse thoroughly with tap water, and then with lab grade water twice.
2. Set bottles upside down on pegs on the glassware dryers and turn switch on. Put lids on blue racks in the fume hood.
3. Tare the bottles, after making sure they are completely dry and are at room temperature.
4. Record the weight in appropriate column and place the lids back on the bottles when both are completely dry.
5. When the bottles are completely dry, tarred, the lids replaced, and everything has been double-checked, the labels can be removed. First, check with the supervisor before removing any labels. Once approved, this step simply involves removing the labeled tape from the bottles, and returning bottles to the crates, or erasing the pencil mark on the etched portion of the glass DIS bottles.

6. Watch for cracks, holes, or collapsed corners in plastic bottles. If a defects are noticed, discard the bottle after the label is removed.

Notes

- The samples should be processed in approximately the same order in which they arrive at the lab. This limits the amount of evaporation from the bottles, reduces fading of the labels, and generally keeps the processing as parallel to the sampling as possible. Cover the bottles with black plastic to minimize light and fading of the labels. Store bottles in a cool location. Bottles that have been stored over a month or two may have significant evaporation and growth of algae.
- Some bottles arrive empty or nearly empty. Check the field volume mark on the bottle to see if it was low in the field. Test for leaks by placing the bottle on paper towels for several hours. After processing, fill the bottle and place it on its side on paper towels to determine if the sampled leaked through the cap-seal.
- Leave the electronic analytical balance on 24-hours a day, all year.
- The standard weight, next to the balance, should be weighed after every ten filters. If the weighing session is taking longer than usual, weigh the standard weight before the 10th filter. Record the weight in the notebook next to the balance and notify the supervisor any significant changes occur.
- Prepare and weigh about 50-200 new filters periodically, depending on backlog of samples and the size of the crew in the lab.
- Monitor the number of bottles that are ready to be processed and organize then as you proceed.
- Be aware of data dumps that have a higher priority and process them as directed.

- The sample bottles are very unstable and will fall over very easily. Therefore, never remove a bottle cap and set the bottle down to do something else. Keep the cap on until you are ready to filter the sample.
- Plan the low-concentration tasks (e.g. washing or stripping labels) when you feel you will be least alert.
- Turn the oven on first thing in the morning. Turn the oven off at night unless the desiccant is re-charging.
- Dry desiccant at 105 C for 4 hours or overnight. Keep the desiccator door closed as much as possible; transfer desiccant quickly. Periodically grease the door seal with silicon lubricant.
- Samples are normally acidified in the field. If a sample requires acidification in the lab use, three drops of 1:1 HCL per sample. Samples should be acidified as soon as possible to reduce fungal growth and to flocculate the sediment, which speeds up filtering. (laboratory turbidity measurements, if required, will be taken before acidification). Once the samples reach the lab, try not to disturb the samples after acidification, to allow the sediment to settle.
- Use the following procedure to make a new batch of acid. **CAUTION:** Use face shield and gloves whenever handling acid. To make a batch of 1:1 HCL, add 100 ml of acid to 100 ml of water, in that order, **acid to water**. If acid should contact your skin, wash the area thoroughly with copious amounts of water ONLY, and notify your supervisor. A sodium bicarbonate solution should be kept near the acid; this will neutralize the effects of acid spilled on lab surfaces only. Mixing baking soda and water makes a sodium bicarbonate solution. This solution should never be used to neutralize acid spilled on skin, since the heat produced by neutralization will increase cellular damage. Use acid only

under the fume hood in the lab.

Sand Fraction Determination

1. This process may be requested for selected samples. This procedure would be completed before the routine filtering steps.
2. This procedure uses the 0.063mm mesh sieve to separate sands from silts and clays. Determination of particles larger than sand size would require additional sieves, such as 0.5 mm, 1.0mm and 2.0 mm. When determining more than one size fraction, the required sieves are stacked together, with the largest mesh on top and decreasing in size to the smallest mesh. Each sieve is rinsed and transferred to a separate filter.
3. Pour the sample through 0.063mm sieve <u>with the pan in place beneath the sieve</u>. First, wet the sieve with a squirt bottle to reduce the surface tension. Rinse the bottom pan out with lab grade water before starting new bottle. Rinse the sample bottle into the sieve using lab grade water from a squirt bottle. Tilt the sieve in pan (keep the pan flat on the counter) and "chase" the sediment to the lower side of the sieve with a stream of water from the squirt bottle.
4. Rinse sieve thoroughly, up to 10 times, so that only sand particles remain in the sieve, and the suspended sediment fraction goes into the pan underneath. Use water efficiently to reduce the total volume that requires filtering.
5. Chase and rinse the sand particles from the sieve into a container (one with a pour-spout will make transferring the sample into the funnel much easier during the filtering process). Rinse the sieve thoroughly so that all the sand particles are transferred into the container.
6. Process the sand fraction sediment portion using the normal filtering

method yielding the first filter (or more if necessary) for the sand fraction.

7. Pour the suspended sediment fraction (< 0.063mm) from the pan into a pour-spout container. Rinse the pan thoroughly with the squirt bottle to remove all of the sediment. Continue to process the suspended sediment fraction using the normal filtering method.
8. Record which filters are "sand fraction" and which are "suspended sediment fractions" in the comments section of the data.
9. When finished, place the bottom of the sieve upside down to allow the remaining water to drain.

Volume Measurement for a Missed Bottle Weight (SC6)

1. After processing the sample, add filtered water to the bottle until the bottom of the meniscus matches the volume mark on the side of the bottle.
2. Weigh the bottle, with the water, but without the cap, and write the value in total weight column. If they're no other problems with the sample, place a sample code (SC) of 6 on the data sheet. Pour the water from the bottle into a graduated cylinder, note the total volume, measured from the bottom of the meniscus (+/- 1ml), and write this volume in the comment column.

Large Filter Procedure

1. If the sediment sample contains a large amount of sediment and is taking an excess amount of time to process (usually more than 6-8 filters), then switch to a large filters.
2. Place a large filter on the Buchner funnel vacuum apparatus. (Remember to record the filter I.D. number on the lab form.)

3. Weight and record the sample's weight on to the lab form.
4. Slowly pour the water from the bottle onto the filter. Aim for the center of the filter and be very careful not to spill any of the material over the edges of the filter. Squirt lab-grade water into the sample bottle and rinse all the sediment over to one corner of the bottle. Slowly pour the sediment onto the center of the filter. Do this procedure several times without splashing or spilling sediment over the edges of the filter. Since the funnel is larger than the filter sediment can spill over the edges and lodge itself on the underside of the filter. This could cause problems, especially if this sediment falls off unnoticed during the drying or weighing procedure.
5. Do not discard the filtrate from the vacuum flask. Save both the filtrate and the sample bottle portion that hasn't been washed out. Set these aside for the time being.
6. Turn off the vacuum and with the rounded ends of two spatulas; fold the large filter in fours, leaving the filter I.D. showing on top.
7. Put the large filter on the drying rack. Rinse the spatulas through the funnel and then rinse the funnel into the flask.
8. Set up a small filter onto another vacuum flask apparatus. Make sure to record the filter I.D. on the lab form. Pour the filtrate (from the filter flask) onto the small filter. Rinse the flask three times. Repeat the same procedure with the sample bottle. Rinse the small filter funnel as normal and place filter on drying rack.
9. The large filters will be oven-dried for 2 hours (instead of the usual 1 and 1/2 hours). Weigh the filters after cooling in the dessicator for one hour. The large filters absorb moisture rapidly than the small filter. Remove no more than three large filters at a time from the dessicator and weigh them quickly.

10. Oven-dry the large filters again for 1/2 hour.
11. Cool for the filters for 1/2 hour in the dessicator and weigh a second time. Make sure that the weight difference between the first and second weighing is 4% or less. If the weight difference is greater then 4% or greater than 0.5mg then repeat oven-drying for another 1/2 hour, cool in dessicator for another 1/2 hour and weigh. Repeat procedure until the weight difference of the filter stabilizes at 4% or below.
12. Record the second and third weight (only if needed) in the comment section of the lab form.
13. When processing a sand fraction sample, use the large filter for the suspended portion and the small filter for the sand portion.