**Redwood National Park**

**Sediment Laboratory Procedures**

**For Analyzing Suspended Sediment**



By Vicki Ozaki and Carrie Jones

September 2012

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| **Revision Dates:** | **Changes** | **Initials** |
| 2/24/2015 | 1. Check filters for holes. 2. 50th filter QA for weights. 3. Minimum volume for SSC < 150 mL. 4. Turbidity ≤ 5 NTU not processed. 6. Run sample in HACH 3x 7. Additional comments. 8. Filters OK to be in desicant cabinet > 24 hrs as long as humidity < 20%. | RBdK |
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Sediment Laboratory Procedures

For Analyzing Suspended Sediment

# Introduction

This Standard Operating Procedure provides a step-by-step process for analyzing suspended sediment in the lab.

# Preparing the Sediment Lab

1. **Set the room thermostat** to 68°C (the scale is stable at a room temperature between 66-71°C).
   * + To set the temperature use the ˄˅ buttons on right hand side of the thermostat.
     + Press System and select: cool or heat
     + Select: fan (auto or on)
2. **Turn on the oven** - It must reach a temperature of at least 100°C, preferably 105°C). It takes about a 1/2 hour to reach this temperature.
3. **Turn on the scale.** Press and hold ON/OFF button until numbers appear on the display. If it doesn’t turn on, check to make the surge protector is on.It takes about a half hour for the scale to warm up.



**Precisa Model XB-220A Scale**



**Hach 2100N Turbidimeter**

1. **Scale Calibration Check**

* The scale should be clean and read 0.0000g when nothing is on the scale. If not press the “t” button (tare). The scale’s margin of error is + 0.003.
* **Test the scale’s accuracy** with the weights provided **at least once a month**.
* Record the date, weight and your initials on the Scale Calibration Log in the Sediment Lab Logbook.
* If the weight is off by more than 0.1g re-level the scale and test weights again. If the scale remains more than 0.1g off, notify the laboratory supervisor and make arrangements to have the scale calibrated.

1. **Turn on the Hach 2100N turbidimeter (Large bench top instrument).** ON/OFF switch located on the back of the meter above the power cord. It takes about a 1/2 hour to warm up.
2. **Turbidimeter Calibration Check**

**Check the calibration** with the five formulin solutions standards **at least once a month.**

Prepared Standards

* + - Remove the <0.1 NTU Standard from the plastic case and set it aside – it should never be shaken. Leave the remaining standards in the case and close the case’s lid.
    - If the Standards have been used within the last seven days, invert the standards in the case 10 times.
    - If the standards have been sitting undisturbed for longer than seven days, shake the standards in the case for 2-3 minutes, then let the standards stand undisturbed for five minutes.
    - Thoroughly rinse the outside of each vial with ethanol, holding the vial by the top only or with a glove.
    - After drying and immediately before using each standard apply a thin bead of silicone oil from the top to the bottom of the vial. Use only enough to coat the vial with a very thin layer of oil. After applying the oil carefully wipe the vial down with the black oil cloth using the designated “oily” velveteen side to remove any excess oil. Return the oiling cloth to its plastic bag.

Read Turbidity of Each Standard

* + - **Handling the Standard only by the top**, insert the vial into the Hach matching up the arrow on the standard with the mark on the Hach sample compartment.
    - Record the readings for each standard vial in the appropriate column on the Turbidimeter Calibration Log sheet in the Sediment Lab Logbook.
    - The NTU value should be within **±10%** of the standard vial value. If the NTU value is not within ±10% of the standard vial value the Hach needs to be recalibrated.
      * It is suggested that the Hach be recalibrated every three months regardless of the calibration checks.
      * For calibration procedures refer to the Manual.

After completing the calibration check replace the standards in the plastic case and store it in a safe place

1. **Wash the graduated cylinder and turbidimeter sample bottle** with the distilled water **at least 3 times**. Shake out the excess water. Leave upside down on paper towels to drain.
2. **Rinse the forceps and spatula with distilled water** then refill the squirt bottle with more distilled water.
3. **Prepare the Suspended Sediment Data Analysis Form**

NOTE: Suspended sediment samples at Redwood Creek gaging stations are taken by observers (depth integrated samples) and are in glass bottles. Suspended sediment at Prairie Creek stations are sampled by an ISCO pump sampler and stored in plastic bottles. Generally, sediment samples from only one station are recorded on a lab form (ie. generally don’t mix bottles from different stations on the lab forms). Sediment samples are logged chronologically starting from the beginning of the water year.

There are two different sediment laboratory data sheets—one for Redwood Creek stations and one for Prairie Creek Stations (See example next page).

On each form:

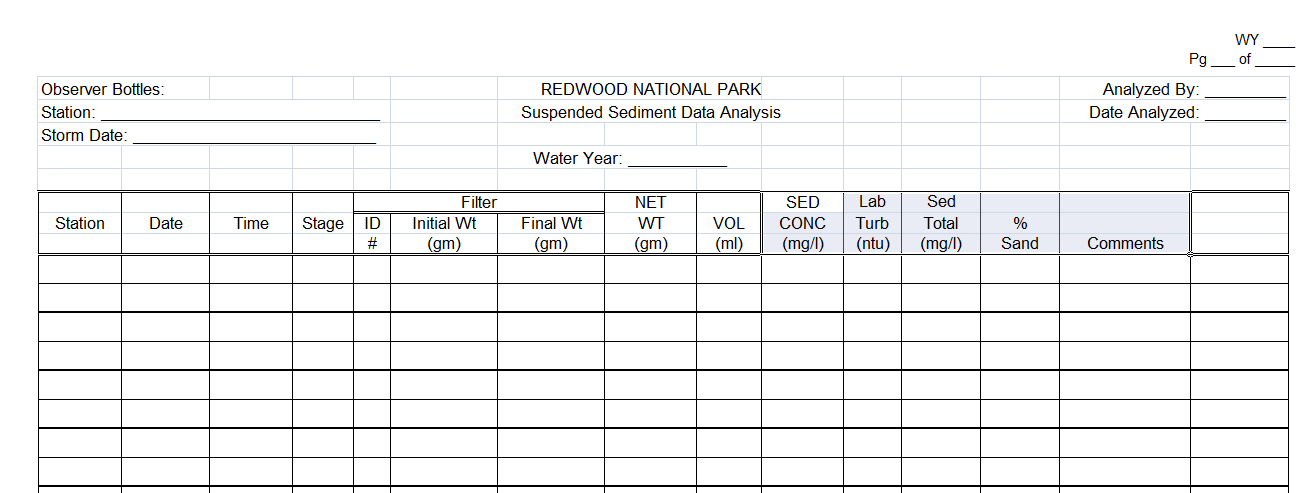
* Fill out the Header Information for the station (top left):

Redwood Creek lab sheets: Station Name and Storm Date.

Prairie Creek lab sheets: Station Name, Storm Date, Pull Date, and Number of bottles filled.

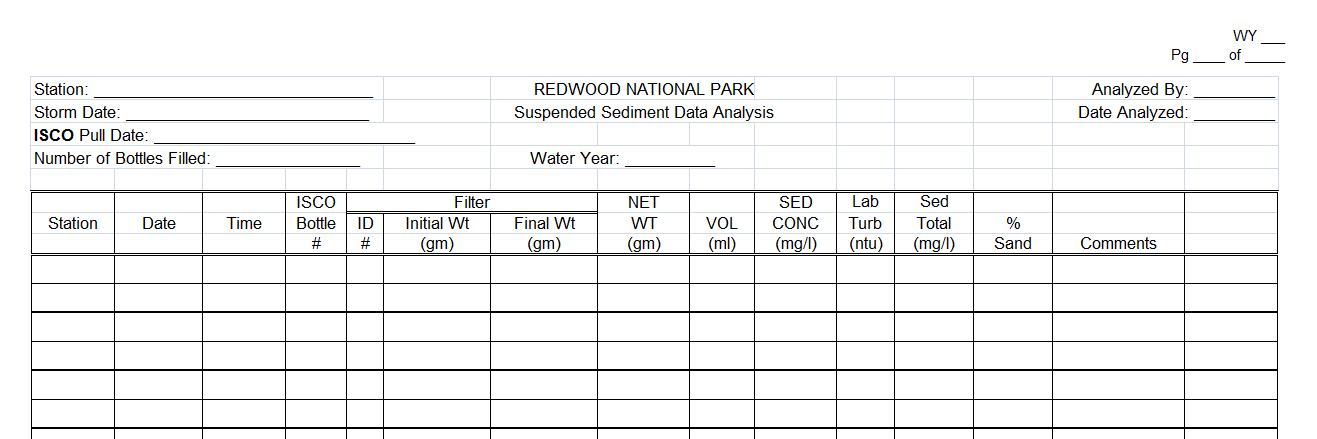
* Fill out the Water Year, Analyzed By and Date Analyzed (top right).

Suspended Sediment Data Analysis Lab Sheets

Observer and ISCO bottles

**ISCO Bottle Lab Sheets** – Prairie Creek Gaging Stations

**Observer Bottle Lab Sheets** –Redwood Creek Gaging Stations



For each bottle:

* Fill out the Station Code (3 letter code), Date, Time, and ISCO Bottle number or Stage. For each bottle, you will fill out two lines on the lab sheet. The first line is for recording data for the Filter 1 (fines) and the second line is for recording data for Filter 2 (sand). See example below.

**Code Location**

**ORK** Redwood Creek at Orick

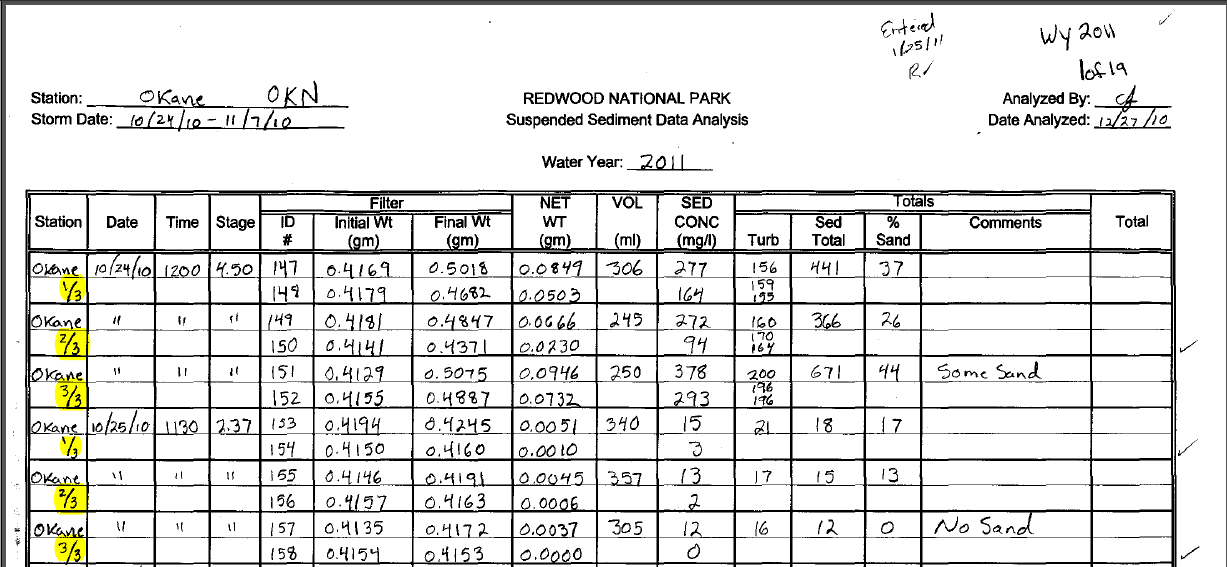
**OKN** Redwood Creek at Blue Lake (aka O’Kane)

**PAB** Prairie Above Boyes Creek

**PRW** Prairie at Wolf Creek Bridge

**LLM** Little Lost Man Creek

**LM** Lost Man Creek



* + For Prairie Creek samples, ISCO bottle numbers should be from 1 - 24.
  + For Redwood Creek, there should be 3 bottles per sample from the O’Kane gaging station and 2 bottles per sample from the Orick gaging station. Multiple bottles are annotated on the lab sheet in the Station Name column as 1/3, 2/3 and 3/3 for O’Kane or 1/2 and 2/2 for Orick.

# Preparing Filters

**Never touch the filter with your bare hands!**

1. **Separate filters:** Grab a stack of filters from the bag with forceps. Hold filters with forceps and gently fan the edge of the filters with a Kimwipe.
2. **Label filters:** Put a filter on a clean tray using forceps. Hold the filter with a Kimwipe. Write a filter ID number (*ID #*) with a very fine-tip Sharpie on the top of the filter. Every WY start at 1 and label in successive order. Every 50th filter write a “Q” after the #. This filter will not be used for SSC but pre-washed and weighed a 2nd time to be used for Quality Assurance of our scale. Use light pressure when writing so the filter won’t tear. This number will be used to keep track of pre-weights. Store labeled filters in box with the numbers visible on top. Note: Pre-label enough filters to last for several days of lab work. This can be done at the end of the day.
3. **Prewashed filters:** Using forceps, place a new labeled filter in the filter apparatus. Turn on the vacuum pump and **rinse the filter 3 times**. Wait until the filter has an even color before turning the vacuum pump off.

Use forceps to gently lift the edge of filter and place prewashed filter in a baking pan. Make sure the filters are not touching.

When the pan is full, **dry the filters in the oven for 1 hour and 30 minutes**. Make sure to record the time filters were placed in the oven or set a timer. After removing filters from the oven, place filters into the desiccator cabinet for at least 20 minutes.

1. **Weigh pre-washed filters:** Check the thermostat to make sure the room temperature is near 68°C. The scale performs best at a temperature between 67-71°C. Make sure air conditioning is turned off since it can affect the scale.

Each filter is weighed twice. Place the filter on scale, write down the weight on scratch paper. Lift the filter off the scale and let it go to 0.000 or Tare. Then reweigh the filter for the second weight. The two filter weights should be within 0.003. Otherwise weigh the filter a third time.

**Record the *Filter ID#*** and **the lowest weight** in the*Initial Wt*. column on the Sediment lab form.



Materials needed to label filters. Left to right on tray: Filter, forceps, small spatula, and Sharpie pen.

# Measuring Turbidity

**Check the turbidity of each sample** if it hasn’t already been done.

1. Shake the sample bottle vigorously to mix up the sediment and set aside while you get organized. When you are ready slowly invert the sample bottle 3 times, quickly pour some of the sample into the turbidity sample vial (fill to the line). Only hold the turbidity sample vial by the rim above the fill-line. Screw the cap on and **clean off the outside of the bottle with a Kimwipe followed by the oiled velvet rag**.

Note: Oil the velvet rag once a day, when working in the lab, by placing 3 drops of oil on a bottle and rubbing the oil into the rag. Store the rag in a plastic bag.

1. Gently invert the sample turbidity vial by turning it upside down a couple of times then insert the vial into the turbidimeter. Align the arrow on the sample vial with the mark on the Hach sample compartment and close the cover. Repeat this process **3** times. **Record the highest number the meter reads (integer value only)** on the lab datasheet. If the reading is **100 NTU’s or more**, pour the sample back into the ISCO bottle and **repeat the procedure two more times.** Record each reading on the lab datasheet. If one of the readings is significantly different do a 4th reading.

**If turbidity is < 5 NTU’s don’t analyze for suspended sediment, dump sample in sink drain. Record comment on Lab sheet “Not processed, NTU < 5” to the right of the sample #.**

Pour the turbidity sample and the remaining ISCO or glass bottle sample into the graduated cylinder. **Measure the volume and record the sample’s volume on the lab datasheet** (VOL column).



Vacuum pump and components of the filtration system



Assembled filtering system

0.063 mm mesh sieve on top of the filter

**If volume is < 150 mL don’t analyze for suspended sediment, unless turbidity is very high > 100 NTU, dump sample in sink drain. Record comment on Lab sheet “Not processed, Vol. < 150 mL” to the right of the sample #.**

# Filtering Suspended Sediment

1. Pick up a 1.0 micron filter with forceps/spatula, check for holes, and put it in the filter holder with the writing side up. If possible, use the numbered filters sequentially. Put the O-ring on top of filter and run forceps along the edge of the flat plastic O-ring to make sure the filter is seated, and screw the top on securely.
2. Place the 0.063 mm mesh sieve on top of the filter. The paper filter will catch fine sediment and the sieve will catch coarse suspended sediment.
3. Top off squirt bottles with distilled water.
4. Hook up the filtration system to the vacuum pump. Turn on the vacuum pump.
5. Slowly pour the sample from graduated cylinder into the funnel cup or filter holder and mesh sieve. Don’t let the water overtop the sieve.

Use the squirt bottle with distilled water to **rinse the cylinder, sample bottle, bottle lid, and turbidity bottle** (anything that has come in contact with the sample) **3 times each.**  Wash all of the sediment into the filter holder and sieve. Rinse the sides and bottom of the bottle to ensure that all the interior surfaces of the bottle have been well rinsed. Last few rinses should be with the bottle upside down.

Take label off of the ISCO bottle and stack in the corn. Bottles will be rinsed out at the end of the day.

**REMEMBER TO: Empty Wastewater Flask Frequently Into The Sink.**

Be careful not to overfill the wastewater flask. Otherwise the wastewater will get sucked into the vacuum pump – VERY BAD!!!

Multiple Filters -Some samples have high concentration of suspended sediment, so you may have to use several filters for one sample Generally this is samples with turbidity values > 150 NTU.

One Filter – If the sediment sample water overtops the sieve while analyzing the sample, you will have to combine the sample onto one filter. In this case, you will rinse the sediment in the sieve onto the filter. The sample will be analyzed as one filter rather than breaking the sample down in to coarse (sand) and fine sediment.

1. **Rinse the sand in the sieve**. Strap the vibrator to the hand holding the sieve. Turn on the vibrator while rinsing the sample with distilled water making sure all the water drains into the filter holder. Once the sand in the sieve is thoroughly rinsed, set the sieve aside and rinse the inside of the filter holder to wash fine sediment onto the paper filter.
2. **Turn off the vacuum pump**. Take off the lid and O-ring. Before removing the filter, make sure most of the water has been extracted out of the filter; otherwise, the filter tends to stick to the drying pan.
3. Use forceps to carefully remove bugs from the filter and discard. Leave other organics such as twigs on the filter.
4. Use forceps or a spatula to lift the filter out of the filter holder and place on a metal or glass drying pan. This filter is referred to as Filter 1-fines. Record the Filter *ID#* on the lab sheet.
5. Empty the flask of water into the sink.
6. **Place a new labeled filter** in the filter holder and screw everything in place. Turn on the vacuum pump and rinse the sand from the sieve onto the filter paper. Turn off the pump and remove the filter and place it on the drying pan. This is referred to as Filter 2-sand. Record the Filter *ID #* on the lab sheet.
7. **Reset work space.** Shake water out of glass containers and refill squirt bottles with distilled water.
8. **Drying Filters**

* Dry all filters in the oven for 1 hour and 30 minutes at a temperature between 100 -105 ⁰C.
* Place dried filters into the desiccator cabinet for cooling (**minimum of 20 minutes**) before weighing.
  + Check the percent humidity in the desiccator cabinet before removing the filters from the oven. The humidity in the cabinet should never exceed 25% (add fresh desiccant if necessary).

1. **Weigh Filters**
   * **Check the scale**; it should be on, clean, and reading 0.0000g. If it doesn’t, press “T” to zero it out.
   * Pull out one filter at a time out of the desiccator cabinet and place it on the scale. Close the scale door. Wait until the scale settles on a number and write down the weight (use scratch paper). Make sure air conditioning is off since it can affect the scale. Lift the filter off the scale and let it go to 0.000 or Tare. Then reweigh the filter for the second weight. The two filter weights should be within 0.003. Otherwise weigh the filter a third time.

Record the Filter *ID#* (if not already done) and the lowest weight (*Final Wt*.) on the Suspended Sediment lab form.

* + Place the weighed filter on the counter until you do the final calculations.

# Final Lab Datasheet Calculations

* + **Net Weight (*NET WT* column; gm) -** The net weight is the difference between Final weight and Initial weight.
  + **Suspended Sediment Concentration (*SED CONC* column**; mg/l**) -** To calculate sediment concentration, multiply the net weight (*NET WT*) by 1,000,000 and divide by the volume (*VOL*). There should be a *SED CONC* calculated for Filter 1 (fines) and Filter 2 (sand).
  + **Total Sediment (*Sed Total* column;** mg/l) – is the sum of the *SED CONC* for Filter 1 and Filter 2.
  + **Percent Sand *(% Sand* column) -** To calculate the percentage of sand of the sample, take the sediment concentration of the filter with sand (*SED CONC* Filter 2) and divide by the total concentration (*Sed Total*); then multiply by 100.
  + **Comments –** Record spills, errors or notes in the comment column. It is important to record any observations or suspicions that may explain unusual results. Also provide a reliability code (developed by C. Jones) which are a visual estimate of sediment on the filter.

***Reliability Codes***(See photos of sediment in the Appendix A):

**Code Description**

0 No Sand

1 Normal-No comments;Sand and/or organics < 40%

2 Some Sand: Sand and/or organics > 40% but less than 50%

3 Lots of Sand: >50%

4 Lots of Sand and/or Organics: Sand and organics > 50%

5 Some Coarse Sand/Coarse Sand: > 50% coarse sand

6 Lots of Coarse Sand: > 50% coarse sand but usually much higher

7 Bad or questionable data

8 No sieve/no filter- Sand not measured for various reasons

9 Misc

999 Broken or dropped bottle or Not Analyzed

# Sediment Lab Clean-Up

Clean up the lab when you are finished for the day.

* + - * + Wash out all of the sample bottles and caps and remove the labels or clean off the writing. Re-label bottles after they dry. Recap bottles and place in crates̶ 24 ISCO bottles per milk crate.
        + Rinse the graduated cylinder and turbidity meter sample bottle with distilled water. Shake out the excess water. Leave upside down on paper towels to drain.
        + Rinse the forceps and spatula with distilled water.
* Return all equipment to original location.
* Refill bottles with distilled water.
* It is also a good idea to prewash filters for the next lab session. If time allows, label filters and take turbidity measurements on new bottles.
* Make a gallon of distilled water. It takes about 4.5 hours. Start one gallon in the morning as soon as you come in and start another one about 5 hours later. Distilled water can be transferred to the empty water gallon containers stored in the sediment lab hallway.
* Desiccant - Keep the desiccant cabinet door closed as much as possible and transfer desiccant quickly. Periodically grease the door seal with silicon lubricant. There are two boxes of desiccant per shelf.

When the humidity in the desiccator starts to approach 25%, Dry out the desiccant in the oven (105⁰C).Take desiccant pack from one shelf and put it in the oven for the day with the sediment samples. Return the dried desiccant to the desiccator cabinet at the end of the day. Repeat with the desiccant on other shelf the next time in the lab. The desiccant should last a long time and need to be dried out about 3-4 times a year.

# Sediment Laboratory

# Tips and Tricks and General Work Flow

Turn on the scale, oven and turbidimeter as soon as you get in the lab so they can warm up.

Prewash filters. I usually use all three pans. Place pans in the oven.

Do turbidity measurements on any new bottles that have come in from the field as soon as possible.

Weigh prewashed filters from the desiccator cabinet. Use the wire racks if the pans are still in the oven.

Analyze suspended sediment samples but **plan to stop about two hours before you go home**.

Weigh and do final calculations for the processed filters in the desiccator cabinet.

Clean up the lab by putting things away and cleaning and crating bottles and caps.

Label and cap clean, dry ISCO bottles.

Label filters.

**General Notes**

**Turbidimeters** – The portable turbidity meters reads higher than the Hach 2100N Bench Top meter. Randy developed a relationship between the portable and the Bench Top.

**Sediment Laboratory Production Rate**: For light to moderate suspended loads, about 15 bottles per day can be analyzed in 6.5 hours which includes washing pre-filters. Note: This is for samples that don’t require additional lab analysis for organics or size breaks other than sand. For Water Year 2012 Carrie ran the lab for 52 days and analyzed about 600 bottles.

# Appendix A: Reliability Codes and Photos with Examples of Filter Descriptions

**Code Description**

0 No Sand

1 Normal-No comments;Sand and/or organics < 40%

2 Some Sand: Sand and/or organics > 40% but less than 50%

3 Lots of Sand: >50%

4 Lots of Sand and/or Organics: Sand and organics > 50%

5 Some Coarse Sand/Coarse Sand: > 50% coarse sand

6 Lots of Coarse Sand: > 50% coarse sand but usually much higher

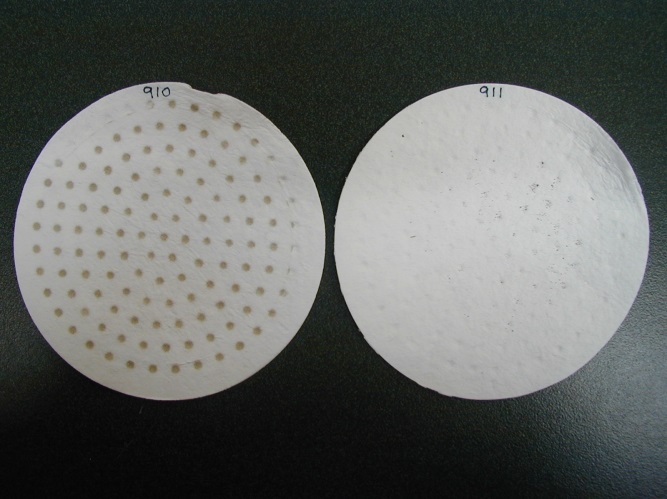
7 Bad or questionable data

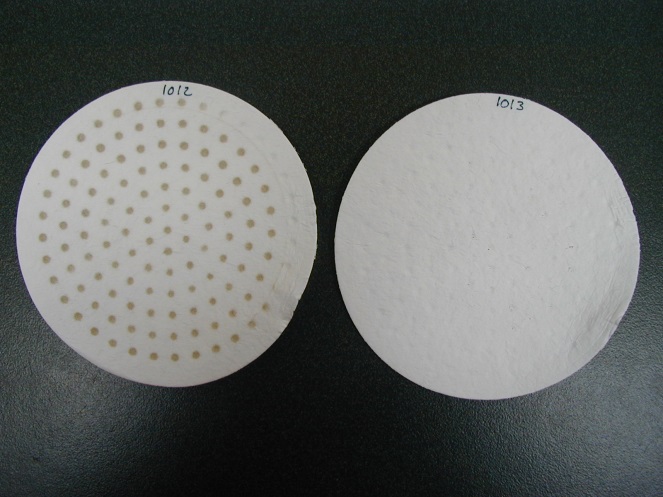
8 No sieve/no filter- Sand not measured for various reasons

9 Misc

999 Broken or dropped bottle or Not Analyzed

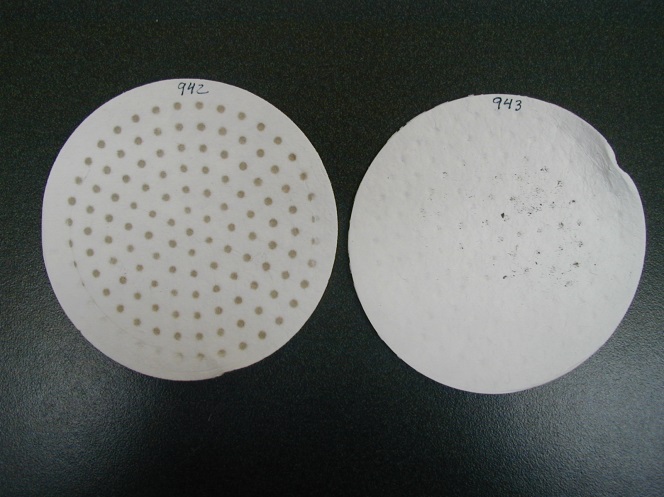
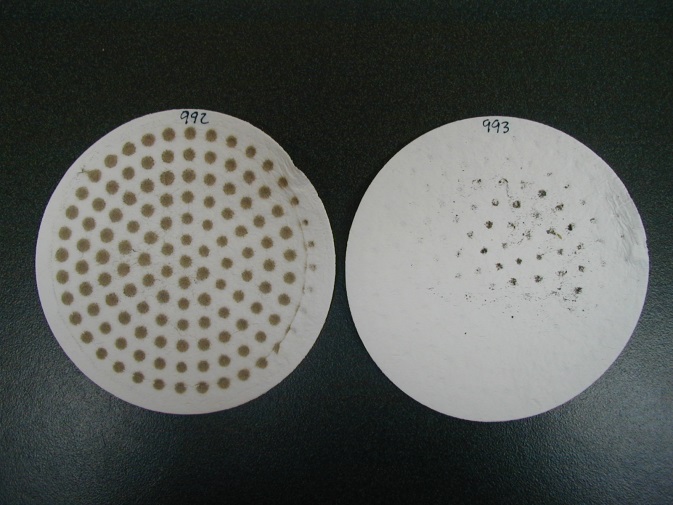
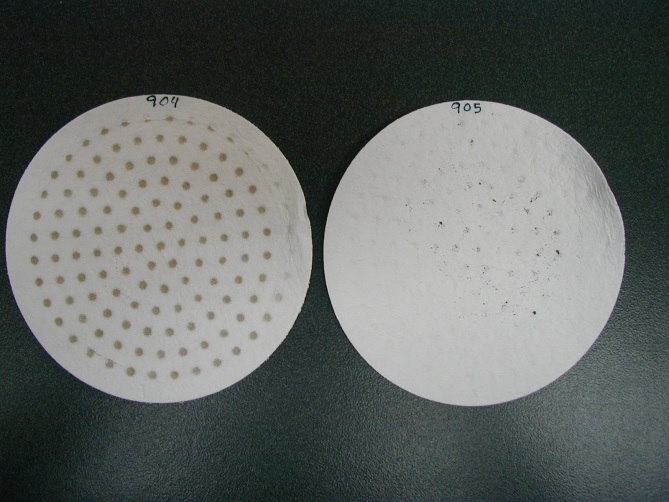
**Reliability Code Description**

 0 No Sand (but may contain some sand but not enough to weigh)



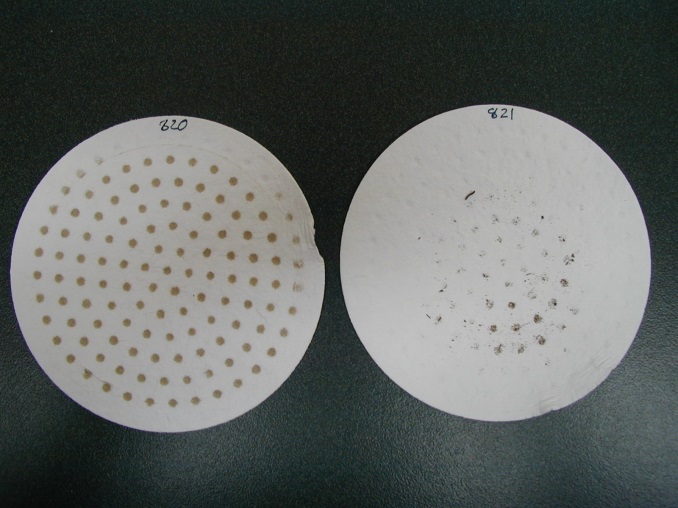
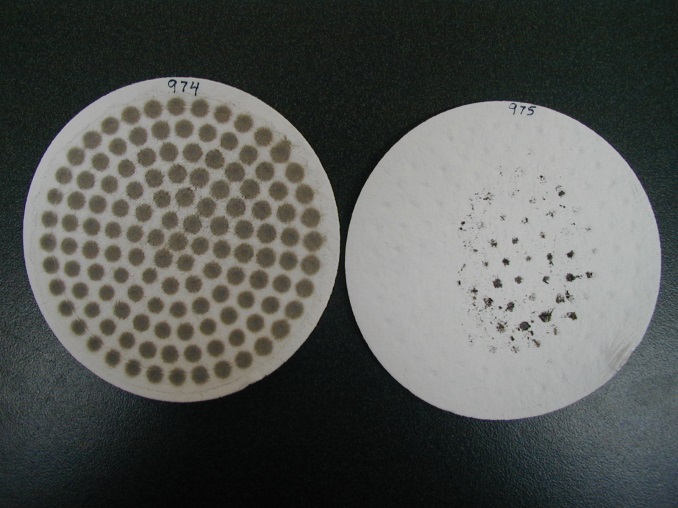
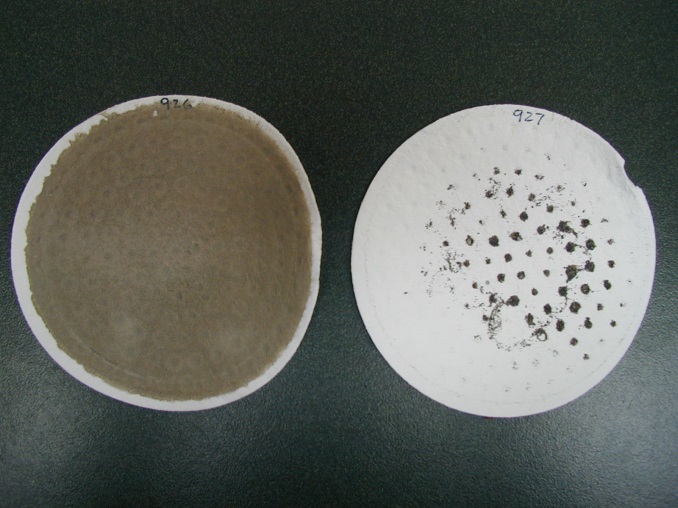
**Reliability Code Description**

1 Normal- No comments; Sand and/or organics < 40%



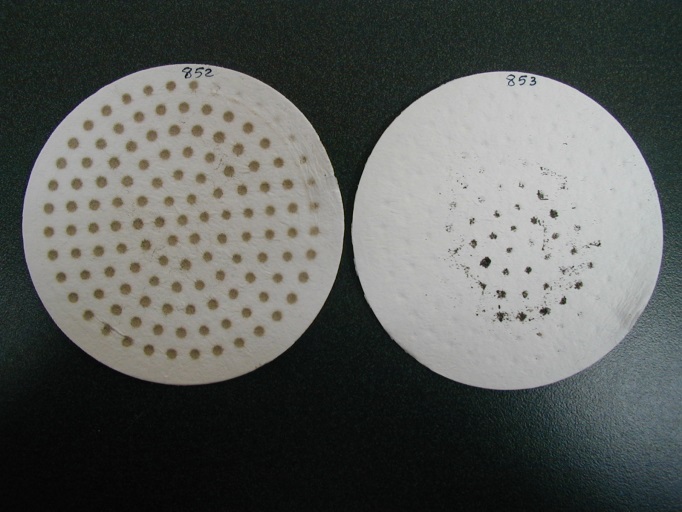
**Reliability Code Description**

2 Some Sand: Sand and/or organics > 40% but less than 50%



**Reliability Code Description**

3 Lots of Sand: >50%



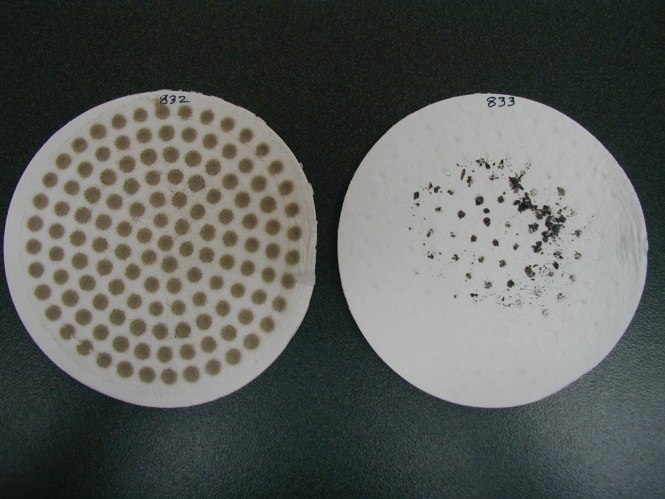
**Reliability Code Description**

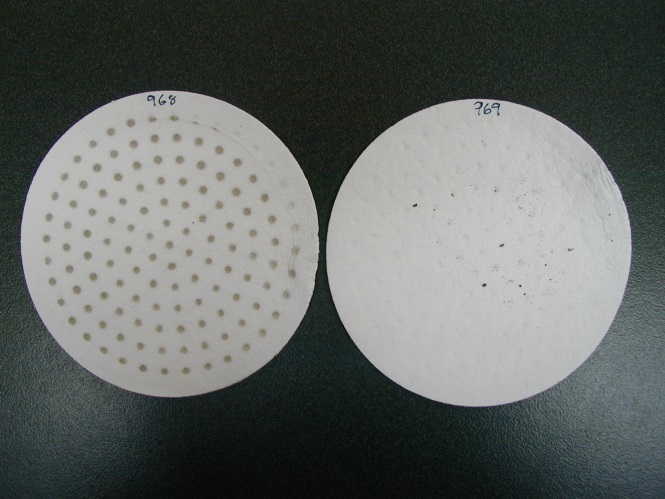
4 Lots of Sand and/or Organics: Sand and organics > 50%



**Reliability Code Description**

5 Some Coarse Sand/Coarse Sand: > 50% coarse sand





**Reliability Code Description**

6 Lots of Coarse Sand: > 50% coarse sand but usually much higher

NO PHOTOS at this time

# Appendix B: Equipment List

Oven (preferably vented)

Scale (Precisa Model XB-220A) and calibration weights

Turbidity meter – Hach 2100N (with formulin calibration standards, empty sample bottles and a soft velvet cloth)

Desiccator cabinet with desiccant

Distilled water maker

Vacuum pump

Filtration system (filter holder, tubing and a wastewater flask)

Vibrator (with a hand strap)

3” Sieve (0.063mm)

Filters (1.0 micron)

Distilled water

Squirt bottle

Glass 500ml graduated cylinder

Kimwipes disposable wipes

Plastic tray (used for holding filters while writing ID numbers)

Glass or metal baking pans

Forceps and spatulas (micro)

Suspended Sediment Data Analysis forms (one for glass bottle samples from observers and one for ISCO bottle samples)

Scratch paper

Pencil

Sharpie ultra fine point permanent marker

Calculator

# Appendix C: Sediment Laboratory Budget

|  |  |  |
| --- | --- | --- |
| General Budget | | |
| Description | Quantity | Cost Estimate |
| Filters (1.0 micron) | 10+ Boxes per year | $500 |
| Kimwipes | Occasionally 30 boxes/case | $100 |
| Scale Calibration | Once per year | $300 |
| Turbidimeter Calibration | Once per year | $125 |
| Temperature Probe Calibration | Once per year | $100 |
| Staffing | 6 PP full time FTE GS-07 |  |
| NOTE: During wet years we need to run the lab continuously during the winter and may need to hire student help. Also cost for lab supplies will increase for Filters. | | |

# Appendix D: Scale and Turbidimeter Calibration Logs

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| **Redwood National and State Parks**  **Scale Calibration Check** | | | | | |
| **Date** | **100 gm** | **Initials** | **Date** | **100 gm** | **Initials** |
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| **Redwood National and State Parks**  **Monthly Turbidimeter Calibration Log** | | | | | | |
| **Date** | **4000 NTU** | **1000 NTU** | **200 NTU** | **20 NTU** | **< 0.1 NTU** | **Initials** |
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| **Precisa Model XB-220A Scale**  **Annual Calibration Log** | | | |
| **Date** | **Certified By** | **Company Name/Contact Info** | **Comments** |
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| **Hach 2100N**  **Annual Turbiditmeter Calibration** | | | |
| **Date** | **Certified By** | **Company Name/Contact Info** | **Comments** |
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