Suspended and Dissolved Particulate Matter in Seawater

Introduction

In this experiment you will determine the following parameters for your sample of sea water:

- the density
- the concentration of dissolved solids
- the concentration of suspended particulate matter

Procedure

Obtain a 1-gallon sample of sea water from the stockroom. Write your name and the date on the container. This sample has been especially collected for you and must last you the entire semester. Take good care of it. In part, "good care" means keeping the sample capped when not in use.

Question - Why should the sample container be capped when not in use?

In the instructions that follow, whenever you see "weigh the sample," you should also record the result.

**Part 1. Density Measurement and Gravimetric determination of the concentration of dissolved substances in filtered sea water**

Everyone knows that sea water is salty. In this part of the experiment you will determine how salty your sample is.

Clean a weighing bottle and a lid and rinse carefully with de-ionized water. Shake it to remove excess water. Obtain a 100-mL beaker and label it with your sample number and your initials. Place the weighing bottle and the lid in the beaker and place the beaker in a drying oven for at least an hour. Sec 1 students use oven #1, section 2 students use oven #2, etc… The weighing bottle and its lid will be used later for the measurement of the density. Proceed with parts 2 and 3 of this laboratory until ready to continue. Go to Part 2, entitled “Preparation of seawater by filtration for future analyses”
Density of sea water

*Note: This section is to be performed after Part 2 or Part 3

In this portion of the experiment, you will check the calibration of an automatic pipette and then determine the density of seawater. To determine the density, you will measure the mass and volume of a sample of filtered sea water (from part 2 or part 3 of this laboratory). You will use an analytical balance to determine the mass and an automatic pipette to determine the volume. Record the brand, the model number and the number.

To use the balance, follow these instructions:

1. Use the same analytical balance for all your weighings
2. Brush any left-over powder after use and place the brush on top of the balance
3. Close the balance door after use
4. Clean the balance bench top

- Calibration of the automatic pipette (Finnpipet)

Have ready a clean weighing bottle and cap (not the same as the one you put in the oven). The weighing bottle need not be scrupulously dry. You are going to weigh by difference. Obtain an automatic pipette and a pipette tip from your laboratory instructor.

To use the automatic pipet, follow the directions:

1. Tightly place a disposable tip on the tip cone of the pipet
2. Wet the newly attached tip with the solution being pipetted before any actual pipetting takes place. This is done by filling and emptying the tip 2-3 times.
3. Hold the pipet straight that means vertically. Do not hold the autopipet horizontally or tip up since the liquid will leak into the barrel and damage the pipet. The barrel has to be dry at all times.
4. While holding the pipet vertically in air, depress the operating button to the first stop
5. Dip the tip under the surface of the liquid in the reservoir about 1 cm deep and slowly release the operating button
6. Lift the pipet from the solution, make sure the tip is full
7. Holding the pipet vertically, deliver the solution to your desired container, by depressing the operating button to the first stop. After a delay of about one second, continue to depress the operating button all the way down to the second stop (last stop). This action will empty the tip
8. Release the operating button to the ready position

Note: A typical mistake for students is to confuse the first stop and the second stop during the transfer of the solution.

Set the pipette to deliver 5 mL. Ask your instructor to write the temperature on the blackboard. Record the temperature in the laboratory. Weigh the weighing bottle with the lid on. Rinse a small beaker with deionized water. Pour some deionized water in the
beaker. Using the automatic pipette, transfer 5 mL of de-ionized water from the beaker to the weighing bottle and weigh it again.

**Caution:** Do not put water in the weighing bottle while it is on the balance pan. We have CaCl2 inside the balance chamber to avoid moisture.

Empty the weighing bottle and repeat this procedure twice more. Note: We are weighing by difference.

Your notebook entries will look as follows:

(1) Mass of weighing bottle plus lid and 5 mL deionized water in g  
(2) Mass of weighing bottle plus lid in g 

Mass of deionized water in g 

\[
\frac{(1) - (2)}{5} 
\]

- Density measurements

Remove the first weighing bottle from the oven. Place the weighing bottle and lid in a desiccator. Allow it to cool for a few minutes. Weigh it with the lid on. After making sure that the pipet tip has no droplets in it, use the pipette to transfer 5 mL of Millipore filtered seawater to the weighing bottle and weigh it (lid on).

- Dissolved particulates

Place the weighing bottle (**lid off**) with the weighed portion of filtered seawater in the labeled beaker into the oven. If necessary, ensure that the weighing bottle remains in an upright position. Allow the seawater to dry until the next laboratory period.

*Be sure you keep track of where you put the cap because you will need to put it back on the weighing bottle when you weigh your sample later! It might be a good idea to put the cap in the beaker with the weighing bottle, but not on the weighing bottle itself.*

At the beginning of the next laboratory period remove the sample from the oven, place it in your desiccator, and allow it to cool for 1 hour or so. Weigh it (cap on), and record the mass. Remove the cap, allow the sample to remain on the laboratory bench for 10 minutes, replace the cap, and weigh the sample again. Record the result. (Is there a difference in weights?)

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**Part 2. Preparation of seawater by filtration for future analyses**
A general word about liquid handling: For scrupulously accurate work it is necessary to avoid contaminating your sample with other liquids. A good complete way to assure cleanliness is to:

1. Clean all receiving vessels in the usual way with detergent;
2. Rinse thoroughly with tap water;
3. Rinse at least 3 times with de-ionized water;
4. Rinse again with a small (measured) portion of the sample that will be stored in the container;
5. Shake dry.

For this laboratory wash the following:

- the filter assembly by the above steps 1 to 5
- a 50- or 100-mL beaker by the above steps 1 to 3 and then follow the instructions below
- the filter flask by the above steps 1 to 3 and then follow the instructions below
- two one-liter plastic bottles by the above step 3 and then follow the instructions below
- graduated cylinders by following steps 1 to 3, as explained below

Specific instructions follow.

Assembling the filtration apparatus

Obtain a filtration unit from the stockroom. The equipment is usually handed out to students at the beginning of the laboratory. Assemble the apparatus and connect it to the house vacuum. Use a ring stand and clamp down the unit.

Shake the receiving flask (which has already been rinsed with de-ionized water) to remove excess water - it is not necessary for this container to be bone dry. Shake dry the two one-liter plastic bottles that have already been rinsed with de-ionized water. (You will need the second bottle for part 3 of this experiment.) Using forceps, obtain a Millipore APFF04700 prefilter from your laboratory instructor and put it in the filter assembly. This filter disk is coarse and is specially designed for the purpose at hand. Note that each filter costs about $1.00. Have the set-up checked by your laboratory instructor.

Cleaning in preparation for filtration

Have ready a clean 50- or 100-mL beaker that has been rinsed with de-ionized water. Swirl your container (with the lid on!) of seawater vigorously and transfer a few mL to the beaker. Swirl the beaker to rinse it and discard the water. Your beaker is now ready for use and you are ready to begin filtering your sample.
Swirl your container of seawater vigorously and transfer approximately 50 mL to the beaker. Transfer 5-10 mL of seawater to the filter funnel. Turn on the aspirator, and filter. Carefully remove the filter funnel and lay it on its side on a paper towel. Remove the filter support and lay it on its side on a paper towel. Disconnect the receiving flask, swirl it around to rinse. The filter flask is now clean enough to receive the sample.

Filtration

Reassemble the filtration unit and resume transferring the seawater from the beaker to the funnel, swirling (gently!) the beaker each time to make sure particulates do not sink to the bottom. Using aspiration, filter a total of no more than 750 mL of seawater through the Millipore filter. Note that the filter flask will not hold more than about 450 mL at one time. Since you are unable to filter a full 750 mL at one time, keep an eye on your filtration. You will have to stop near the middle to empty the filter flask before continuing.

Watch the filtration process carefully. Depending on the particle concentration, the filters may clog while water remains in the funnel - a situation that you want to avoid. Perform the filtration with small portions. If it appears to you that the rate of filtration has slowed appreciably before you have transferred 750 mL, then check with your instructor, finish filtering whatever sea water remains in the filter funnel, and proceed to the next section, Removing and storing the filter. In any case, after the filter flask is half to three-quarters full, break the suction as instructed and proceed to the next section.

Storage of the filtered water

Transfer 5-10 mL of the filtered water (measured accurately) to the small graduated cylinder. Use this water as a rinse for the plastic bottle and discard the rinse water. Obtain a large graduated cylinder. Rinse it with a small volume of filtered seawater and discard the rinse.

Transfer the filtered water sample remaining in the filter flask to the rinsed plastic bottle. Label the bottle carefully with your sample number, your name, the date, your laboratory section, and the comment "filtered through Millipore APFF04700." You will need this sample for later work. To preserve its integrity, be sure to cap the bottle containing the filtered water when not in use to avoid evaporation of water, which would cause changes in concentration.

Discard the filter paper.

Part 3. Determination of the concentration of suspended particulates

Note: Please note the concentration of suspended particulates and dissolved solids are two different parameters. DO NOT CONFUSE THEM~
Low concentrations of very small particles are found in seawater. In this part of the experiment you will attempt to separate the particles and then to determine their mass.

You will need a 1-L plastic bottle that has been rinsed and shaken dry. Using forceps obtain a 0.45 micron Osmonics E04WP04700 filter from your laboratory instructor. This filter is especially designed to have low mass and to resist weight changes.

Question - Why are these characteristics of the filter important for this application?

Weigh the Osmonics filter precisely. To weigh the filter, tare a weighing paper and place the filter paper on the tared weighing paper to keep it clean. Place the filter in the filter assembly. Shake out the filter flask from part 2 to ensure that there is no water left in the flask. By following the same procedures that you used in part 2, quantitatively filter a total of approximately 750 mL of unfiltered seawater through the Osmonics E04WP04700 filter (do not use the blue separator of the Osmonics filters):

After the filter flask is half to three quarters full, break the suction and do the following:

a. Transfer 5-10 mL of the Osmonics filtered water to the small graduated cylinder, measure the volume, and record it as \( V_1 \).

b. Use this water as a rinse for the plastic bottle and discard the rinse water.

c. Obtain a large graduated cylinder.

d. Rinse it with a small measured volume of filtered seawater. Record it as \( V_2 \).

e. Transfer the filtered water sample remaining in the filter flask to the large graduated cylinder and record the volume as \( V_3, V_4, V_5, \text{ etc...} \) If you are using a 100 mL graduated cylinder, you will be refilling it several times

f. Next, transfer the filtered seawater from the large graduated cylinder to the rinsed plastic bottle. Note: The total volume of seawater is \( V = V_1 + V_2 + V_3 + V_4 + V_5 + \text{ etc...} \)

When done, remove the filter carefully, place it on a paper towel (particulate side up!) and allow to dry in air for five minutes. Weigh precisely. Place the filter on a watch glass using crucible tongs. Put the filter paper in an oven at 105°C for five minutes. Longer times in the oven cause the disintegration of the filters. Remove, allow to cool, and weigh again to constant weight.

Store the filtered seawater in the second one-liter plastic bottle, which has been rinsed in step b, labeled with your sample number, your name, the date, your laboratory section, and the comment "filtered through Osmonics 04WP04700."

Reminder: Don’t forget to return to part 1.
Part 4. Clean-up and Shut-down

Rinse the filter apparatus thoroughly with de-ionized water and return it to your instructor. Clean the glassware that you used and rinse with de-ionized water. Return the automatic pipette to the stockroom.

Hand in the copy of your data sheets to your instructor before you leave the lab.

Guidelines for Writing a Lab Report

Unless otherwise specified in the laboratory write-up, lab reports should contain the following sections:

1. Abstract. Maximum 100 words. A summary of the important findings. Report the average, do not report individual findings.
3. Experimental methods. Maximum, 1/2 page. A catalog of the types of measurements made and the instruments used (if any).
4. Results and Discussion. The most important section of your report. Length will vary, but will usually be 1 to 4 pages. It need not be a book. Numerical results should be presented as tables or graphs.

A few general guidelines for all reports follow.

- Consolidate all your raw data in one table for efficiency.
- Label tables and graphs carefully and be sure to include units. (Potential for serious points off here).
- Give each table and graph a number and a title. The text should introduce the reader to each table and graph by number with a statement such as, "The measurements needed for the calculation of the density are shown in Table 2."
- Compare results with literature where possible.
- Identify and comment on likely sources of error.
- Strive for brevity and clarity. Avoid like the plague the use of the word "this" without a noun to follow, e.g., "This explains why I got a value of 2.0." Even if you think it is repetitious, be specific, i.e., in this example write instead, "This clever use of the dilution factor explains why I got a value of 2.0."
Report for the Suspended and Dissolved Particulate Matter in Sea Water

Some specific suggestions for the write-up of the first laboratory (Suspended and Dissolved Particulate Matter in Sea Water) follow.

Part 1 Density of seawater and concentration of dissolved solids (1 to 2 pages).

- In the Handbook of Chemistry and Physics, look up the density of pure water at the laboratory temperature you recorded. Your instructor will leave the Handbook in the lab. Alternatively, you can obtain the density from the NIST website or equivalent. Record the reference. Recalling that density, \( d \), is given by:

\[
d = \frac{\text{mass}}{\text{volume}},
\]

use the looked-up density and your recorded masses of pure water to calculate the volume delivered by the automatic pipette. Is the calculated volume the same as the volume "dialed" on the automatic pipette? If not, then what volume do you think the automatic pipette actually delivered. Justify your choice, which will be quite important in all your subsequent calculations. If you are in doubt, discuss this question with your classmates or your laboratory instructor.

- From the measured mass of filtered seawater and the calibrated volume (2 decimal places) delivered by the automatic pipette, calculate the density, \( d \) (in g/mL), of your filtered seawater. How does this density compare with the density of pure water? What is the uncertainty of your measurement?

You can calculate the uncertainty of the density, \( \delta d \), from the following formula:

\[
\delta d = d \times \sqrt{\left(\frac{\delta m}{m}\right)^2 + \left(\frac{\delta V}{V}\right)^2}
\]

Here \( \delta m \) is the uncertainty in the mass and \( \delta V \) is the uncertainty in the volume. You will have to use your judgment in deciding how big each of these quantities is.

- Compile and consider a few published values for the density of seawater. One possible source of data is the LEO-15 web site. Compare the published results with the values you obtained. Of course, the actual value of the density for your sample may be different from published values, but if the discrepancies are large, then it is probably a good idea to re-check your calculations.
• Report the concentration of dissolved solids in your sample of filtered seawater in units of mass/volume. Explain why it is not possible to calculate the molarity of the dissolved solids at this stage.
• Assuming that all the dissolved solids in the sample are NaCl, how much seawater would it take to yield 1 pound of table salt? Use the value for the concentration of dissolved solids; DO NOT USE THE DENSITY OR ANOTHER PARAMETER

Part 3. Concentration of suspended particulates (1/2 to 1 page)

Report the concentration of suspended particulates in mg/(L sea water) and in mg/(kg sea water). Comment on any problems encountered.
Grading of the Lab Report

Suspended and Dissolved Particulate Matter in Sea Water

Please type your lab report and submit a stapled hardcopy to your instructor at the beginning of the next lab period. Write your name on each page and make sure your report is paginated.

Abstract (should include density in g/mL, the Dissolved solids in g/mL and the suspended solids in mg/L and in mg/kg)

Introduction
Experimental Methods
Inclusion of Raw Data
Pipette Calibration-Volume Delivered
Density of Filtered Sea Water
Calculation of the Uncertainty in the Density
Concentration of suspended particulates (mg/L and mg/kg)
Volume of Water to deliver 1 lb of NaCl
Answer to Questions

Please make sure all the above information is included in your lab report. Make sure your answers have the correct number of significant figures and the correct required units. Presentation, your understanding, discussion, explanation and accuracy are taken into consideration when the report is being graded. You may receive bonus points or lose points when appropriate.

Grading Grid

Prepare a grading grid on the cover sheet of your report similar to the following table. Fill in the values that you obtained.

<table>
<thead>
<tr>
<th>Item</th>
<th>Points</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 Abstract</td>
<td></td>
</tr>
<tr>
<td>2 Introduction</td>
<td></td>
</tr>
<tr>
<td>3 Experimental Methods</td>
<td></td>
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<tr>
<td>4 Volume delivered by calibrated autopipet</td>
<td>Your Value</td>
</tr>
<tr>
<td>5 Density of Filtered Seawater in g/mL</td>
<td>Your Value</td>
</tr>
<tr>
<td>6 Uncertainty in Density in g/mL</td>
<td>Your Value</td>
</tr>
<tr>
<td>7 Concentration of Dissolved Solids in g/mL</td>
<td>Your Value</td>
</tr>
<tr>
<td>8 Volume of Water in mL to deliver 1 lb of Table salt</td>
<td>Your Value</td>
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<tr>
<td>9 Concentration of Suspended particulates in mg/L</td>
<td>Your Value</td>
</tr>
<tr>
<td>10 Concentration of Suspended particulates in mg/kg</td>
<td>Your Value</td>
</tr>
<tr>
<td>11 Questions, Presentation, Other</td>
<td></td>
</tr>
</tbody>
</table>

TOTAL