EXPERIMENT 5: WATER ANALYSIS: SOLIDS

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Abstract

Total solids is the measurement of the suspended and dissolved solids in a sample of water. Suspended solids. Suspended solids are the small solid particles that remains on a water filter and are suspended in water. These also act as visual indicators of the quality of a water sample. Dissolved solids include some organic materials and other inorganic nutrients that pass through a water filter and are dissolved in water. Total Suspended Solids (TDS) is the number of solid particles that are trapped by the filter, while Total Dissolve Solids (TDS) refers to the amount of some inorganic or organic matter that passed the filter. In this experiment, the goal of the researchers is to determine the quantity of the total, dissolved, and suspended solids in a soiled water sample and identify the ions present in the solid particles on the water sample. The group conducted a number of tests to identify the given variables and analyzed the data gathered thoroughly to achieve accurate and precise results.

Introduction

A substance that composed of the chemical element of hydrogen and oxygen is called water. It also exists in solid, liquid and gaseous state. Water has the ability to dissolve many other substances and its versatility as a solvent is essential to living organisms. For some extensive districts, surface water is utilized as the primary drinking water source. Before placing the water to the distribution lines, the water is first channeled into a water-treatment facility where impurities are removed, and bacteria are killed. To properly removed the impurities a water has the contents of a surface water must be correctly known and predictable and tests are utilized to determine the contents of the surface water.

Methods

Materials and Procedures

Materials used in the experiment:

1. Graduated Cylinder
2. 400 mL beaker
3. 150 mL beaker
4. Graduated Cylinder
5. Spatula
6. Matchstick/box
7. Funnel
8. Stirring Rod
9. Iron Stand
10. Bunsen Burner

11. Wire Gauze
12. Evaporating Dish
13. Petri Dish
14. Funnel Support

Total Dissolved Solids (TDS)

Obtain 100 mL of a water sample from your instructor. The water sample may be from the ocean, a lake, a stream, or from an underground aquifer. Preferably the water sample is high in turbidity. Assume the density of your water sample to be 1.01 g/mL

1. Filter the water sample. Gravity filter about 50 mL of a thoroughly stirred or shaken water sample into a clean, dry 100-mL beaker. While waiting for the filtration to be completed, proceed to Part B.

2. Evaporate the filtrate to dryness

a. Clean, dry, and measure the mass (±0.001 g) of an evaporating dish or 250-mL beaker.

b. Pipet a 25-mL aliquot or portion of the filtrate into the evaporating dish or 250-mL beaker. Determine the combined mass of the sample and evaporating dish (250-mL beaker).

c. Use a hot plate or direct flame to slowly heat—do not boil—the mixture to dryness.

d. As the mixture nears dryness, cover the evaporating dish or beaker with a watch glass and reduce the intensity of the heat. If spattering occurs, allow the dish to cool to room temperature, rinse the adhered solids from the watch glass, and return the rinse to the dish.

3. A final heating to dryness. Again, heat slowly, being careful to avoid further spattering. After all the water has evaporated, reduce the heat of the hot plate or maintain a "cool" flame beneath the dish for ~3 minutes. Allow the dish to cool to room temperature and determine its final mass. Cool the evaporating dish and sample in a desiccator, if available.

Total Solids (TS) and Total Suspended Solids (TSS)

1. Evaporate an original water sample to dryness
   a. Clean, dry, and measure the mass (±0.001 g) of a second evaporating dish (or 250-mL beaker).
   b. Thoroughly stir or agitate 100 mL of the original water sample; pipet2 a 25-mL aliquot of this sample into the evaporating dish (250-mL beaker). Record the combined mass of the water sample and evaporating dish (beaker).
   c. Evaporate slowly the sample to dryness as described in Part A.2. Record the mass of the solids remaining in the evaporating dish.
2. Total suspended solids. Collect the appropriate data to determine the total suspended solids in the water sample.

Analysis of Data

1. Compare your TDS, TS, and TSS data with three other chemists in your laboratory who have analyzed the same water sample. Calculate the average value for the TSS in the water sample. Pay head to significant figures.

Chemical Tests

1. Test for carbonates and bicarbonates. With your spatula, loosen a small portion of the dried samples from Part A and Part B and transfer each to separately marked 75-mm test

tubes or watch glasses. Add 1 drop of 6M HNO3 (Caution: HNO3 is corrosive and a severe skin irritant) and quickly and carefully observe.

2. Test for chlorides (halides). To each of the samples from Part D.1, add 1 drop of water, agitate the solution, and add 1–2 drops of 0.01 M AgNO3 (Caution: AgNO3 is a skin irritant) and observe.
3. Test for calcium ion. With your spatula, loosen a second portion of the dried samples from Parts A and B and transfer each to separately marked 75-mm test tubes or watch glasses. Add about 1 drop of water, agitate or stir the solution, and add 1 drop of 1 M K2C2O4 and observe.

Calculations

Prelaboratory

1. List several anions, by formula, that contribute to the salinity of a water sample.

Answer: CO_3^{2-}, HCO_3^-, Cl^-, Br^-, I^-, PO_4^{3-} and SO_4^{2-}

2. Distinguish between and characterize total dissolved solids (TDS) and total suspended solids (TSS) in a water sample.

Answer: Total suspended solids (TSS) refers to the solids that are not dissolved in water, these solid remains can be caught using a filter. TSS can include a wide variety of material, such as silt, decaying plant, industrial wastes and sewage. Total dissolved solids (TDS) comprise inorganic salts and some small amounts of organic matter that can be dissolved in water.

3. Experimental Procedure, Part A.2c, d. Explain why a “cool flame” is important in heating a solution to dryness

Answer: A cool flame is important in heating a solution to dryness because if a hot flame is used and it is heated too quickly it can cause bubbles known as bumping in the solution which means that a hot flame is not ideal in heating a solution to dryness.

4. a. What is an aliquot of a sample?

Answer: An aliquot of a sample is part or portion of the total amount of the solution that is usually used to perform tests on a sample.

b. What is the filtrate in a gravity filtration procedure?

Answer: Gravity solution is a process where unwanted solids are removed from a solution. In this process the solution that passing through the filter is the filtrate in a gravity filtration procedure.

d. How full (the maximum level) should a funnel be filled with solution in a filtration procedure?

Answer: A funnel should be filled with solution in a filtration procedure in a maximum level at two thirds of the funnel.

5. Experimental Procedure, Part D. What observation is "expected" when:

a. An acid (nitric acid, HNO3) is added to a solution containing carbonate or bicarbonate ions?

Answer: The observation expected when HNO3 is added to the solution is that the formation of CO_2, H_2O will occur, and it will leave behind salt.

b. Silver ion is added to a solution containing chloride (or bromide or iodide) ions?

Answer: The observation expected when silver ion is added to a solution containing chloride ions is that silver and chloride ions would make an ionic compound, thus a precipitate would be formed at the bottom of the solution.

6. A 25.0 mL aliquot of a well-shaken and filtered sample of river water is pipetted into an evaporating dish. The sample was heated to dryness. Assume the density of the river water was 1.01 g/mL.

Total Dissolved Solids (TDS)

Answer:

1. Mass of evaporating dish (g) <u>26.217</u>

2. Mass of water sample
   plus evaporating dish (g) <u>51.467</u>

3. Mass of water sample (g) 25.25

4. Mass of dried sample
   plus evaporating dish (g) <u>35.291</u>

5. Mass of dissolved solids in 25-mL
   aliquot of filtered sample (g) 9.074

6. Mass of dissolved solids per total
   mass of sample (g solids/g sample)
   Show calculation 0.3594

7.Total solids (g solids/kg sample, ppt) 359.4

7. The following data were collected for determining the concentration of suspended solids in a water sample (density =1.01 g/mL).

	Trial 1	Trial 2	Trial 3	Trial 4	Trial 5	Trial 6
Volume of sample(mL)	25.0	20.0	50.0	25.0	20.0	25.0
Mass of sample (g)	25.3	20.2	50.5	25.3	20.2	25.3
Mass of dry solid (g)	10.767	8.436	21.770	10.826	8.671	10.942
Mass of solid/mass of sample(g/g)	0.426	0.418	0.431	0.428	0.429	0.432

a. What is the average TSS in the water sample? Express this measurement in ppt (parts per thousand, g/kg).

Average TSS = $\frac{0.426+0.418+0.431+0.428+0.429+0.432}{6}$ = 0.427g/g ($\frac{1000\,g}{1\,kg}$) = 427ppt

b. Calculate the standard deviation and the relative standard deviation (% RSD) for the analyses.

X	(X-MEAN)^2
0.426	0.000001
0.418	0.000081
0.431	0.000016
0.428	0.000001
0.429	0.000004
0.432	0.000025
Total = 0.427	Total = 0.000128

$\sigma = ¿ \quad \sqrt{1/6}\left(0.000128\right)$ = 0.00462

RSD = (100) (0.00462)/0.427 = 1.08%

Water Analysis: Solids Report Sheet

A. Total Dissolved Solids (TDS)

	Trial 1
Mass of evaporating dish (beaker) (g)	48.74 g
Mass of water sample plus evaporating dish (beaker) (g)	73.64 g
Mass of water sample (g)	24.90 g
Mass of dried sample plus evaporating dish (g)	48.94 g
Mass of dissolved solids in 25-mL aliquot of filtered sample (g)	0.20 g
Mass of dissolved solids per total mass of sample (g solids/g sample)	0.0080 g/g
Total dissolved solids (TDS) or salinity (g solids/kg sample, ppt)	8g/kg
Average TDS of the sample	8 ppt

Solutions

$mass_{\text{water sample}}$ = 73.64 g – 48.74 g = 24.9 g

$mass_{\text{dissolved solids}}$ = 48.94 g – 48.74 g=0.20 g

$$m=\frac{g\,solid}{g\,sample}=\frac{0.20\,g}{24.9\,g}=0.0080\,g\,solids/g\,sample$$

$$m=\frac{g\,solid}{kg\,sample}=\frac{0.20\,g}{24.9\,g\,x\,1\,kg/1000\,g}=8\,g\,solids/g\,sample$$

B. Total Solids (TS) and Total Suspended Solids (TSS)

	Trial 1
Mass of evaporating dish (beaker) (g)	41.87 g
Mass of water sample plus evaporating dish (beaker) (g)	66.96 g
Mass of water sample (g)	25.09 g
Mass of dried sample (g)	0.71 g
Mass of total solids in 25-mL aliquot of filtered sample (g)	0.42 g
Mass of total solids per total mass of sample (g solids/g sample)	0.0167 g/g
Total solids (TS, g solids/kg sample, ppt)	16.7 g/kg
Average TS of the sample	16.7 ppt
Total suspended solids (TSS, g solids/kg sample, ppt)	8.7 ppt
Average TSS of the sample	8.7 ppt

Solutions

$$m=\frac{g\,solid}{g\,sample}=\frac{0.42}{25.09\,g}=0.0167\,g\,solids/g\,sample$$

$$m = \frac{g\,solid}{kg\,sample} = \frac{0.42}{25.09\,g\,x\,1\,kg/1000\,g} = 16.7\,g\,solids/g\,sample$$

$TSS = TS - TDS$

TSS = 16.7 *g solids /kg sample* – 8 *g solids/ kg sample*

TSS = 8.7 ***g solids/ kg sampl***e

C. Chemical Tests

Test	Observation	Conclusion
1. $-¿(TDS)$ $2-¿, HCO_3^{¿}$ $CO_3^{¿}$	No Bubbles	No carbonates or bicarbonates
$-¿(TS)$ $2-¿, HCO_3^{¿}$ $CO_3^{¿}$	No Bubbles	No carbonates or bicarbonates
2. $-¿(TDS)$ $-¿, I^{¿}$ $-¿, Br^{¿}$ $Cl^{¿}$	With Bubbles	No chlorides
$-¿(TS)$ $-¿, I^{¿}$ $-¿, Br^{¿}$ $Cl^{¿}$	With Bubbles	Chlorides are present
3. $2+¿(TDS)$ $Cl^{¿}$	Without Bubbles	No calcium ion
$2+¿(TS)$ $Cl^{¿}$	With Bubbles	No calcium ion

Write a summary of your assessment of the quality of your water sample.

The result in the first test which is determining whether carbonates and bicarbonates exists is negative from both Total Solids (TS) and Total Dissolved Solids (TDS). Also, there were no bubbles formed from the observation. The second test also brings out the same result in the Total Dissolved Solids which means that no chlorides are present, but bubbles are observed. The Total Solids also had a reaction (with bubbles) but resulted an opposite of the reaction occurred in TDS which is chlorides are present. The last test had no reaction both in TDS and TS and there were no bubbles observed in TDS, but bubbles existed in TS.

Laboratory Questions

1. The collected water sample is not filtered. Will this oversight result in the TDS value being reported too high or too low? Explain.

Answer: Since the collected water sample is not filtered the Total Dissolved Solids (TDS) value reported will be too high because the mass of dried solids in the sample would weigh more.

2. The evaporating dish was not properly cleaned of a volatile material before its mass was determined. When the sample is heated to dryness the volatile material is removed. As a result of this technique error, will the reported TDS be too high, too low, or unaffected? Explain.

Answer: Because an error occurred in the experiment the Total Dissolved Solids (TDS) will be too high because the reported mass of the evaporating dish was higher, but the actual mass of the dish was lower, which will increase reported mass of the Total Dissolved Solids (TDS).

3. Some spattering of the sample onto the watchglass does occur near dryness. In a hurry to complete the analysis, the chemist chooses not to return the spattered solids to the

original sample and skips the first part of Part A.3. Will the reported TDS for the water sample be too high or too low? Explain.

Answer: The result will be inaccurate because the chemist did not follow the instructions clearly. Therefore, the result will be too high.

4. The sample in the evaporating dish is not heated to total dryness. How will this error in technique affect the reported value for TDS—too high, too low, or unaffected? Explain. TSS—too high, too low, or unaffected? Explain.

Answer: The reported value for the TDS will be too high because the recorded value for TSS will also be too high.

5. As the sample cools, moisture from the atmosphere condenses on the outside of the evaporating dish (beaker) before the mass is measured. Will the presence of the condensed moisture increase or decrease the reported TDS in the water sample? Explain.

Answer: The presence of the condensed moisture will increase the reported TDS in the water sample because of the excess mass of the condensed water.

6. The sample in the evaporating dish (beaker) is not heated to total dryness. As a result of this technique error, will the reported value for total solids (TS) be too high, too low, or unaffected? Explain.

Answer: Due to the excess water in beakers, both the TDS and TSS will evaluate, thus the reported value for TS will be too high since it is equal to the sum of TDS and TSS.

7. Suppose the water sample has a relatively high percent of volatile solid material. How would this have affected the reported mass of:

Answer: If the water sample possesses relatively high percent of volatile solid material, the amount of recorded dissolved solids will be too low. This is caused by the high amount of volatile solid, which evaporates quickly, being released into the air during the heating process. Supposedly, there are more dissolved solids, but the mass of the evaporated volatile solids would no longer be recorded along with the remaining solids. Consequently, the recorded number of total solids would also be too low. The initial mass of the water sample still has both the volatile and the non-volatile solids, thus a high mass of water will be recorded. By using it as a divisor with the low amount of remaining solids after the heat-drying, the resulting value of total solids will be too low. On the other hand, the recorded amount of suspended solids still remain after the sample water is heat dried, hence the unaffected recorded amount.

8. When several drops of 0.010M Ag NO_3 are added to a test sample, a white precipitate forms. What can you conclude from this observation? Explain.

Answer: After the amount of total dissolved, and suspended solids are determined, these are then to be subjected to chemical tests. If several drops of Silver Nitrate are added to the solids and formed a white precipitate, it basically means that chlorides are present in the water sample. This reaction is caused by single replacement between the ions. The anion Chloride will replace the Nitrate to yield silver chloride and a by-product nitrate. The white precipitate formed is the silver chloride being visible in the solution.

Result and Discussion

The results for the Total Dissolved Solids were measured and recorded. The measured mass for; evaporating dish/beaker is 48.74g, water sample plus evaporating dish is 73.64g, water sample is 24.90, dried sample plus evaporating dish is 48.94g, dissolved solids in 25-mL aliquot of filtered sample is 0.20g, and dissolved solids per total mass of sample is 0.0080g/g. The total dissolved solids (TDS) or salinity is 8g/kg and the average TDS of the sample is 8ppt.

The result for the Total Solids and Total Suspended Solids were also measured and recorded. The measured mass for; mass of evaporating dish is 41.87 g, mass of water sample plus evaporating dish is 66.96 g, mass of water sample is 25.09 g, mass of dried sample is 0.71 g, Mass of total solids in 25-mL aliquot of filtered sample is 0.42g, and mass of total solids per total mass of sample is 0.0167 g/g. The total solids are 16.7 g/kg, and the average TS of the sample is 16.7 ppt. While the total suspended solids are 8.7 ppt and average TSS of the sample is 8.7 ppt.

For the Chemical Test, it was observed and recorded. The observation and conclusion for;

$-¿(TDS)$ $2-¿, HCO_3^{¿}$ $CO_3^{¿}$ are no bubbles and no carbonates or bicarbonates and same data for the total solids of $-¿$ $2-¿, HCO_3^{¿}$ $CO_3^{¿}$. For $-¿(TDS)$ $-¿, I^{¿}$ $-¿, Br^{¿}$ $Cl^{¿}$ are with bubbles and no chlorides while for the total solids of it, the chlorides are present. For $2+¿(TDS)$ $Cl^{¿}$ are without bubble and no calcium ion however the reaction is the only different for the total solids since it bubbled.

Conclusion

The total dissolved solids (TDS) or salinity is 8g/kg and the average TDS of the sample is 8ppt. The total solids are 16.7 g/kg, and the average TS of the sample is 16.7 ppt, while the total suspended solids are 8.7 ppt and average TSS of the sample is 8.7 ppt. For the chemical test, the

$2+¿(TDS)$
$Cl^{¿}$ are without bubble and no calcium ion however the reaction is the only different for the total solids since it bubbled.