Introduction:

Alkalinity is an important water quality parameter. It refers to the capacity of water to neutralize acids. Naturally-occurring bases (i.e. alkaline compounds) in water such as bicarbonate, carbonate, and hydroxide ions react with $\text{H}^+$, lowering the acidity of water. Without this acid-neutralizing capacity, the pH of natural waters can drastically change in the presence of acid pollutants, such as coming from acid-mine drainage. Thus, measuring alkalinity is important in determining a stream's ability to neutralize acidic pollution from rainfall or wastewater.

From an operational viewpoint, total alkalinity is the number of moles of $\text{H}^+$ required to neutralize 1 L of water sample to the endpoint. In practice, sulfuric or hydrochloric acid is added to the water sample in measured amounts until the three main forms of alkalinity (bicarbonate, carbonate, and hydroxide) are converted to carbonic acid. At pH 10, hydroxide (if present) reacts to form water. At pH 8.3, carbonate is converted to bicarbonate. At pH 4.5, it is certain that all carbonate and bicarbonate are converted to carbonic acid. At this pH all the alkaline compounds in the sample are "used up." Because alkalinity assumes that all the alkaline species is due to calcium carbonate, the result is reported as milligrams per liter of calcium carbonate ($\text{mg/L CaCO}_3$) or ppm $\text{CaCO}_3$.

Objective:

To determine alkalinity in terms of $\text{mg CaCO}_3$ per liter of water, an unaltered sample is titrated to an end point of pH 4.5 measured with a pH meter. The sample must not be filtered, diluted, concentrated, or altered in any way. The sample should be refrigerated at 4°C and run as soon as practical.

Materials:

- pH meter
- 50-mL buret
- 100-mL graduated cylinder
- 0.0100 M HCl
- Methyl orange indicator

Procedure:

Part I. Prepare HCl Solution

1) *Per bench*: Prepare 1.00 L of 0.0100 M HCl using a 0.1000 M standard HCl solution a 1-L volumetric flask. Use DI water to dilute to the mark. You will share this 1-L acid with other students in your bench for Part III.

Part II. Calibrate pH meter (Each pH meter will be shared by two students)

1) Calibrate your pH meter according to the manufacturer’s direction using calibration buffers. Make sure to rinse the electrode thoroughly with DI water in between each reading.
Part III. Titration (Individual, so you must take turns with the pH meter)

1) Fill a 50-mL buret with your prepared 0.0100 M HCl solution, making sure to remove any bubbles from the buret tip. Remember to record the initial volume reading.

2) Measure 100-mL of your water sample using a graduated cylinder. Carefully transfer in an Erlenmeyer flask by pouring down the walls of the flask. Add 2-3 drops of bromocresol green.

3) Measure and record the initial pH of your water sample.

4) Titrate with HCl, being careful to stir thoroughly but gently.

5) Titrate to pH 4.5. Around the end point of the titration, bromocresol green converts from the blue base form to a yellow acid form; thus a green color indicates when pH is around 4.5. As soon as you see a localized change in color of indicator that disappears upon mixing, slow down the addition of titrant and keep track of the pH. Record the volume of titrant used to reach a pH of 4.5.

6) Repeat the titration once or two more times until you get reproducible volume readings (within 5% will be good).

Part IV. Estimating alkalinity with a test strip

1) Following the instructions from the box, use a test strip to estimate the alkalinity of your water sample. You will compare your result here the result from titration.

Calculations: Potentiometric titration to pH 4.5.

NOTE: Alkalinity assumes that all the alkaline species is due to calcium carbonate. Thus, your result must be reported as milligrams per liter of calcium carbonate (mg/L CaCO$_3$) or ppm CaCO$_3$.

1. Complete and balance the following titration equation:

\[
\text{“Alkalinity”} + \text{H}^+(\text{aq}) \rightarrow \text{CaCO}_3(\text{aq})
\]

2. Using the mole ratio between CaCO$_3$ and H$^+$ in the balanced equation and the volume and molarity of titrant (H$^+$) used to reach the equivalence point pH of 4.5, determine the number of moles of CaCO$_3$ neutralized by H$^+$ at the equivalence point.

3. Using the result in the previous step and the volume of water titrated, calculate the alkalinity as mg CaCO$_3$/L water. The molar mass of CaCO$_3$ is 100.09. You must report the average alkalinity of 2-3 titrations.

4. In your discussion, compare the results obtained between titration and test strip methods. Comment on the “accuracy” of the test strip relative to titration when determining alkalinity.
Discussion Guide

1. Explain the relationship and principles shown by the results
   This probably won’t apply much in the Alkalinity lab since the only result you have is the triplicate titration results.

2. Point out exceptions or lack of correlation, and attempt to explain any discrepancies you find.
   In this case, compare the two or 3 repetitions (titrations) that you did. Do they agree with each other? Explain reason for agreement (e.g. good technique) or lack of (explain any likely experimental errors)

3. Compare your results to accepted (or expected) values
   We don’t have accepted value, but you might want to discuss what sort of alkalinity is expected for freshwater in Massachusetts. A quick Google search might help, but don’t forget to cite your reference(s). Compare also the result of your test strip with The references section of this handout gives you an idea of style when citing sources. that of titration.

4. State your conclusions (This answers the problem)
   We don’t have a research problem, but we do have a goal for doing the experiment. What was the goal? Was it achieved in this experiment? State the method used to achieve this goal.

5. Comment on the significance of the work
   Again, a Google search will help on this section. What is the significance of knowing the alkalinity of surface/fresh water? Discuss it here.

References: