Field Test Kit (II): Measurement of Water Hardness

Experimental Procedure and Post-lab Assignments
Submit your answers to the questions found in the shaded boxes of the procedure by the announced due date. The work can be divided up among the group members, but everyone in the group must hand in a complete set of work under their own name. Please include printout from any calculations done using EXCEL or another program.

Indicator Control Samples
Halfway fill a well with deionized water; halfway fill another well with hard water. Add a few drops of pH buffer to each well. Add enough Erichrome Black T to cover the tip of a micro-spatula and then gently stir each well. At the titration end point the sample should change from the color of the hard water well to the color of the deionized water well.

Field Kit Design
You are to design a test kit that will enable technicians to go out into the field to quickly and efficiently make a series of measurements of water hardness in the range of 0 to 500 ppm. You will need to consider the sample size to be used, the concentration of the titrant and the method of delivering the titrant (refer back to the first week of the laboratory). You should design a test kit that will allow a large number of measurements to be made without the need to carry bulky glassware and large, heavy volumes of titrant. Assume that at most the technician can carry 1 liter of titrant.

Assume a sample volume in liters with a hardness of 500 ppm. Calculate how much titrant solution you need to complex all of the calcium in the sample with EDTA to reach the end point of the titration. You need to find a sample size for each concentration of titrant, EDTA solutions will be available at concentrations of 0.1 M, 0.05 M, and 0.01 M, that can be titrated with a reasonable number of drops of titrant. The technician should not have to count out 400 drops and a titration performed with only a few drops will not be accurate.

Once you have calculated appropriate sample sizes to use with each concentration of titrant you will then titrate five samples with each of the available titrants in order to get three small data set. Make sure to add enough Erichrome Black T to cover the tip of a micro-spatula and a few drops of pH buffer per milliliter of test sample. You can test to make sure the pH is high with the pH indicator paper. using the method of delivering titrant to the sample that you have chosen. You will need to count drops of titrant and convert to volume if the titrant delivery tool you have chosen is not calibrated with units of volume.

Using your titrant volume data and the known concentration of titrant used calculate the water hardness or calcium concentration in ppm (mg/l) for each sample titrated. Since the true value of the water hardness is known, 500 ppm, this will enable us to assess the accuracy of the different methods tested.
The t-table can be used to test for determinate or systematic error in an experiment when
the procedure is run on a sample of known composition as you have just done. In the
inequalities (equations 5 and 6) \( \bar{x} \) is your experimental mean value, \( s \) is the standard
deviation of your data set, \( N \) is the number of trials in your data set, \( \mu \) is the true known
value and \( t \) is the value from the t-table at the confidence level you are using. If there is
no systematic error present then the difference between the true value and your mean
value is due to random error or noise in your data is all

\[
|\bar{x} - \mu| > \frac{ts}{\sqrt{N}} \quad \text{Systematic Error Present} \quad (6)
\]

\[
|\bar{x} - \mu| < \frac{ts}{\sqrt{N}} \quad \text{No Systematic Error} \quad (7)
\]

Table 1 – t Table

The “Degrees of Freedom” is equal to the number of data points in your set minus one (N-1)
for determining a confidence interval for a data set. This is because mathematically only N
different quantities can be calculated from a set of N data points. For our data set we have
calculated a mean value, this leaves N-1 data points left over to calculate the confidence
interval. Likewise, when we compare two data sets our degrees of freedom equals \( N_1 + N_2 - 2 \)
because we have used 2 degrees of freedom in calculating a mean value for each data set.

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**Postlab Questions**

1) What was your sample size for each titrant?

2) How did the volume of titrant actually used compare to the amount calculated? What could account for the difference?

3) What were the mean values of hardness in ppm for each data set?
   What were the standard deviations for your data sets?
   How did your mean values and standard deviation compare to the true value of the stock hard water solution?

4) What were your 95% confidence intervals? Is the true value of the calcium ion concentration in your confidence interval?

5) What is the smallest volume of titrant that you can add to the sample?
   What is the smallest number of moles of titrant that you can add to the sample?
   How will this affect your accuracy in measurement of a 10 ppm sample?
   How will this affect your accuracy in measurement of a 500 ppm sample?

6) Is there systematic error in your method? Use statistical methods to determine this.

7) Describe the method that the technician should use. What sample size, titrant concentration and method of titrant delivery do you recommend?
   Estimate how many titrations of water samples with a hardness of 500 ppm that the technician can perform with 1 liter of titrant given your method?

**Challenges**

Measure the water hardness of the “mystery” sample with your test kit apparatus. Submit your experimental data, calculated mean and standard deviation. We will use the t-table to determine if your results are statistically equal to the true value using the equation:

$$
\pm t = (\bar{x} - \mu) \frac{\sqrt{N}}{s}
$$

Where $\mu$ is the known true value. Extra credit on the group voted scores will be given for having results that are statistically equal to the true value.

How would you modify your test kits to measure samples that have hardness in the 500 to 5000 ppm range? Would you have the same precision and accuracy in 500 to 5000 ppm range as you did in the 0 to 500 ppm range? How would your precision and accuracy change?
**Group Evaluation**

Evaluate the performance of the other members of your group on a 0 to 4 scale. Your rating should be based on your co-worker's preparation, quality and quantity of data collected, laboratory record keeping, assistance to other group members in lab and on post-lab questions, safety practices, experimental technique, and contribution to solving problems.

**You cannot give any two group members the same rating.**

**You must write a 1-line justification for each rating.**

**Your instructor will not reveal your ratings to other students.**

**Your instructor will also rate you, and reserves the right to adjust your ratings if they are felt to be inaccurate.**

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