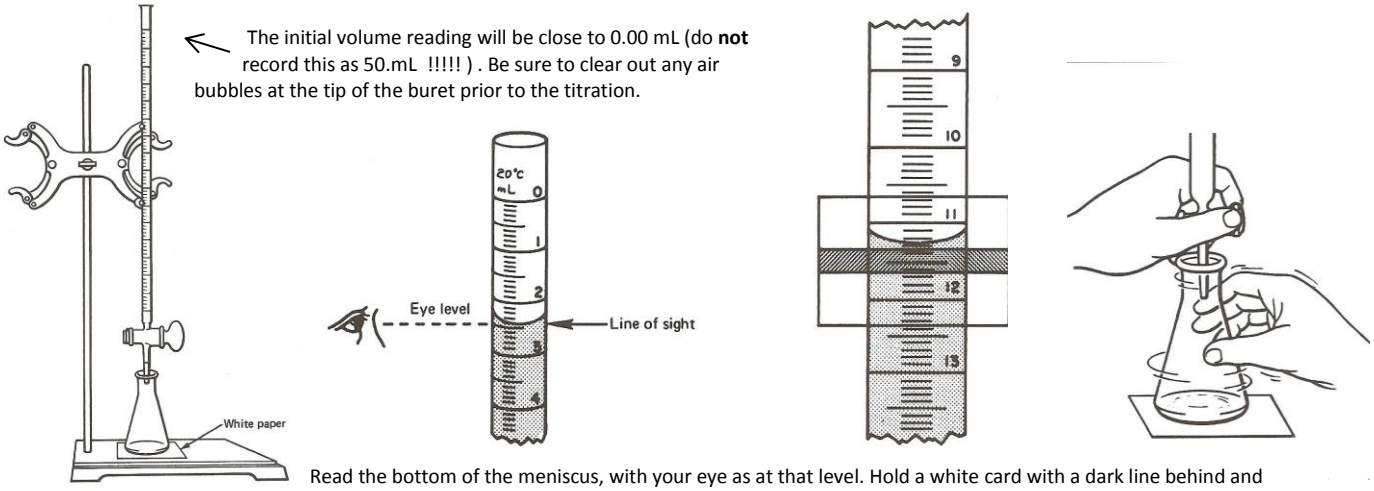
EXPERIMENT C. DETERMINATION OF CALCIUM IN WATER.

Introduction: Water "hardness" is a function of the amount of Ca^{2+} present in the tap water we drink. The water has been in contact with limestone ($CaCO_3$) and some of that dissolves in the water. Many caves are formed by erosion of the limestone by water. Well water contains a fair amount of Ca^{2+} which makes using soap difficult since the Ca^{2+} precipitates the soap as "soap scum". Using detergents avoids this problem. In fact, an old method of determining water hardness was to add soap solution, a drop at a time to a sample of tap water, shake it, and when soap bubbles start to form – the Ca^{2+} has all been reacted with the soap. We use water "softeners" to take out the Ca^{2+} or replace it with Na^+ which never precipitates with anything.

A more precise determination of the Ca^{2+} employs a reaction with a large spiderlike molecule, ethylenediaminetetraacteate (EDTA) which wraps around the Ca^{2+} forming a soluble complex ion. We add some ammonia to remove two H^+ ions from the EDTA, giving the Ca^{2+} a chance to bond with the EDTA in a 1:1 ratio .

Since both Ca^{2+} and EDTA (with or without the Ca^{2+}) are colorless in aqueous solution, we need an indicator, eriochrome in this case, which turns a wine red in the presence of Ca^{2+} ions, and dark blue without the Ca^{2+} . So we add the indicator to the water prior to adding EDTA, then as all the Ca^{2+} is taken up by the EDTA, we will see a color change – an "endpoint".



Read the bottom of the meniscus, with your eye as at that level. Hold a white card with a dark line behind and below the meniscus. A finger also works. Read the volume going down. This reading shows 11.28 mL.. For the purpose of this experiment, the volume can be read to the nearest 0.1 mL – so round to 11.3 mL. if you wish – or If you can read to the nearest 0.05 mL that would be fine too. Future labs may require precision of +/- 0.01 mL.

Once the endpoint has been reached, the volume of EDTA can be found by subtracting the initial volume reading from the final reading. (call this x mL) The EDTA has a standard concentration of 0.0100 M. The Ca^{2+} reacts in a 1:1 mole ratio with the EDTA and the $CaCO_3$ (the source of the Ca^{2+}) is determined in mg $CaCO_3$/L water . Since the molar mass of $CaCO_3$ is 100 g/mole, the number of milligrams of $CaCO_3$in the sample of water (say y mL) is found by:

vol EDTA	concentration	mole ratio	molar mass	metric conversion

$$x\ \text{mL EDTA} \times \frac{.0100\ \text{mole EDTA}}{1000\ \text{mL}} \times \frac{1\ \text{mole } CaCO_3}{1\ \text{mole EDTA}} \times \frac{100\ \text{g } CaCO_3}{1\ \text{mole } CaCO_3} \times \frac{1000\ \text{mg}}{1\ \text{g}} = x\ \text{mg } CaCO_3 \quad !!!!$$

Now divide the mg $CaCO_3$ by the liters of water used in the titration. (convert your buret volume to L)

$$\frac{x\ \text{mg } CaCO_3}{y\ \text{mL water}} \times \frac{1000\ \text{mL}}{1\ \text{L}} = \text{your mg } CaCO_3\text{/ L}$$

This is also called ppm (parts per million) which is how your water company reports the "hardness"

Procedure: Work individually.

Materials	Chemicals
Burette & stand	50 mL 0.01 M EDTA
3 125 mL Erlenmeyers	tap or drinking H_2O
3 beakers	Eriochrome
Funnel, watch glasses	NH_3 buffer
1 50 mL grad cylinder	oxalic acid (optional)

- Obtain a buret and clamp it as shown in the diagram.
- Obtain about 50 mL EDTA solution in your 150 mL beaker
- Rinse your buret with about 5-10 mL EDTA and make sure the valve turns smoothly and is neither too loose or too tight. Ask the instructor for help in adjusting the valve and clearing out any bubbles at the tip of the buret, then place a funnel and fill the buret to near the 0 mark with EDTA. Record the initial volume.
- Obtain some tap water or your favorite mineral water (i.e. Arrowhead or other spring water). Do Not use purified water – as many brands have virtually no calcium. Pour this into a 400 mL beaker as your supply. (you will not need 400 mL – more like 75 or 100 mL)
- Obtain about 50 mL ammonia buffer solution (pH 10) in your 150 mL beaker. Cover it with a watch glass to reduce the odor of ammonia Label your 3 beakers so you don't mix them up.
- Use your grad cylinder; measure out 25.0 mL spring water and pour it into an Erlenmeyer. Add 1 drop of Eriochrome indicator and 10 mL of ammonia buffer. Titrate the mixture to a royal blue end point. If you use less than 10 mL of EDTA to titrate the water, or more than 25 mL , do a quick calculation and use an appropriate amount of water for the next run. An initial run like this is often called a Q&D (quick and dirty) run, used to approximate what the next sample sizes should be. Don't use this value in calculation of the ppm. Tap water contains a lot of Ca^{2+} so use only 10 mL tap water. The spring waters have less calcium, so start by using 25 mL.
- Do three more trials, with appropriate volumes of water such that the volume of titrant (EDTA) to be used will be between 15 and 25 mL.

If time allows, do a run on some other water – see how much calcium is there. Record any extra runs on your notebook and submit the signed copy sheet with the report or turn it in at the end of the period – as your instructor prefers

EXPERIMENT C. CALCIUM IN WATER REPORT. **Name** _____
Section _____

Data: Description of water used: _____

	Q&D run	Trial 1	Trial 2	Trial 3
Volume of water used …….				
Initial buret volume reading ……				
Final endpoint reading ……				
Volume of EDTA used ……..				

Calculations:

	Q&D run	Trial 1	Trial 2	Trial 3
Milligrams of $CaCO_3$ …….				
mg/L (ppm) ……				

show setup of one calculation here.

Average value of ppm in the 3 trials: _____

Questions:

1. Some bottled water brands contain little or no calcium (ex "zero water") also distilled or deionized water is sometimes sold so people can drink "pure" water. Do you think it is healthy to drink these types of waters ? Why or why not ?

2. EDTA is a "chelating agent" used in the treatment of lead poisoning. Pb^{2+} behaves in a similar way as Ca^{2+} . Why would the doctors advise you not to take calcium supplements if you are being treated for lead poisoning ?

3. Look up the structure of EDTA, draw it on the back of this page. What are some remarkable features of this molecule ?