MOHAWK

COLLEGE OF APPLIED ARTS AND TECHNOLOGY

CHEMICAL, ENVIRONMENTAL, AND BIOTECHNOLOGY DEPARTMENT

EDTA Titrations 2: Analysis of Calcium in a Supplement Tablet; Analysis of Magnesium in Epsom Salt; Hardness of Water

by Professor David Cash

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This Experiment is a 3 hour Analytical Chemistry laboratory exercise. It is designed for students in a common second term course of a 2-year diploma program (Biotechnology, Environmental, or Health Technician).

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Experiment 6

Complexometric Titration (2): Use of an EDTA Solution to Analyse for Calcium and Magnesium and to Determine the Total Hardness of Tap Water

OBJECTIVE

Complexometric volumetric titrations with **EDTA** (**e**thylene**d**iamine**t**etraacetic **a**cid) will be performed. The comprehension and skills learned will be transferable to other laboratory and workplace situations.

- A secondary standard EDTA solution will be employed to determine the calcium ion content of a dietary supplement tablet.
- A secondary standard EDTA solution will be employed to determine the percent by mass of magnesium ion in a sample of Epsom Salt (nominally magnesium sulfate heptahydrate).
- A secondary standard EDTA solution will be employed to determine the total hardness of a tap water sample.

REFERENCE

Harris, Chapter 3, pages 265-281.

INTRODUCTION

Reaction of EDTA with Calcium Ions and Magnesium Ions

In contrast with a metal such as zinc, the EDTA complexes of calcium and magnesium are not as stable. For the metal ions in water:

$$Zn^{2+}(aq) + EDTA^{4-}(aq) \rightarrow Zn(EDTA)^{2-}(aq) \quad K_f = 3.2 \times 10^{16}$$

$$Ca^{2+}(aq) + EDTA^{4-}(aq) \rightarrow Ca(EDTA)^{2-}(aq) \quad K_f = 5.0 \times 10^{10}$$

$$Mg^{2+}(aq) + EDTA^{4-}(aq) \rightarrow Mg(EDTA)^{2-}(aq) \quad K_f = 4.9 \times 10^{8}$$

pH Requirement for Titration of Calcium and Magnesium with EDTA

Since EDTA is an acid substance with four weak acid dissociations, the reactions with metal ions are pH dependent. The metals that react strongly with EDTA can be titrated in acidic solution. Zinc is an example. The metals that react more weakly with EDTA must be titrated in alkaline solution. Calcium and magnesium are examples.

To ensure consistent results of titrations, the pH of the solutions must be controlled by using **buffer** solutions. The minimum pH required for successful titration of **calcium** ions with EDTA is about **8**, and that for **magnesium** ions is about **10**. Both of these metals are difficult to titrate with EDTA, especially magnesium.

Epsom Salt
Epsom salt is the common name for magnesium sulfate heptahydrate (**$MgSO_4 \cdot 7H_2O$**). This substance is found as a mineral deposit in several places around the world. It has uses in the dyeing industry, as a plant growth fertilizer, and as a solution in water that can be used to draw excess water out of the feet by soaking the feet in the solution. It is sold in the pharmacy and supermarket for consumer use.

The mineral Kieserite is magnesium sulfate monohydrate (**$MgSO_4 \cdot H_2O$**), a second stable hydrate of magnesium sulfate.

Anhydrous magnesium sulfate (**$MgSO_4$**) absorbs water strongly. It is used in chemical synthesis as a means of removing traces of water from organic substances as part of a purification process.

Measurement of Total Hardness of Water Samples
Total hardness is usually measured in one of two ways. It is measured either by a titration using EDTA, or by a soap titration method. In the EDTA titration, it is assumed that the total hardness is due to the presence mainly of calcium and magnesium ions.

A sample of the water buffered at **pH 10** is titrated with a standard solution of EDTA. The calculation is based on an equation written as if all the hardness were due to **calcium carbonate**. The reaction is 1 mol to 1 mol.

$$\mathbf{CaCO_3 + EDTA^{4-}(aq) \rightarrow Ca(EDTA)^{2-}(aq) + CO_3^{2-}(aq)}$$

Calculation of Total Hardness
The total hardness can be calculated from the titration values. The units of hardness are based on the fictional assumption that the hardness ions are all calcium:

Water Hardness = mg / L or ppm of $CaCO_3$

Definition of the ppm Unit for a Dilute Aqueous Solution
For very dilute solutions in water, since the mass of **1.0 L** of the solution is very close to that of **1.0 L** of pure water, the total mass is very close to **1.0 kg = 1000 g = 1,000,000 mg**.

On this assumption, the solute concentration of a dilute aqueous solution in units of **mg / L** is called **parts per million**, or **ppm**.

Be Very Careful About the ppm Unit.
There is a **different** definition of ppm for **solid mixtures** and yet another for **gaseous mixtures**.

Sample Calculations

Analysis of a Calcium Supplement Tablet

Example 1
A supplement tablet containing (nominally) about **300 mg** of calcium ion was dissolved, filtered, and diluted to **100 mL** volume. Several **2.00 mL** samples of the total solution were titrated with **0.0100 M EDTA** solution by the method of this experiment. The mean corrected titration volume was **13.65 mL** (**0.01365 L**).

Calculate the calcium content of the supplement tablet in **mg** units.
State the value to the **nearest mg**.

Answer
One way to calculate the result is shown:

$$\text{Mass of Calcium (mg)} = \underbrace{\text{Volume EDTA (L)} \times \text{Molarity EDTA (mol / L)}}_{\text{Calculates mol of EDTA}} \times \underbrace{\frac{\text{1 mol Calcium}}{\text{1 mol EDTA}}}_{\text{Converts to mol of Calcium}} \times \underbrace{\frac{\text{40.08 g Calcium}}{\text{1 mol Calcium}}}_{\text{Converts to g of Calcium}} \times \underbrace{\frac{\text{1000 mg}}{\text{1 g}}}_{\text{Converts to mg of Calcium in Sampled Volume}} \times \underbrace{\frac{\text{100 mL Total Volume}}{\text{2 mL Sampled Volume}}}_{\text{Converts to Total mg of Calcium}}$$

$$\text{Mass of Calcium (mg)} = 0.01365\ \text{L} \times 0.01000\ \text{mol / L} \times \frac{\text{1 mol Calcium}}{\text{1 mol EDTA}} \times \frac{\text{40.08 g Calcium}}{\text{1 mol Calcium}} \times \frac{\text{1000 mg}}{\text{1 g}} \times \frac{\text{100 mL Total Volume}}{\text{2 mL Sampled Volume}}$$

Mass of Calcium = <u>274</u> mg

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Sample Calculations (Cont.)

Analysis of an Epsom Salt Sample

Example 2
A sample of Epsom Salt of mass **0.7567 g** was dissolved uniformly in distilled water in a **250 mL** volumetric flask. Portions of the magnesium ion solution of volume **10 mL** were titrated using a **0.01000 M** solution of EDTA by the method of this experiment.
The mean corrected titration volume was **12.25 mL** (**0.01225 L**).

Calculate the percentage by mass (**% w / w**) of the magnesium in the Epsom Salt sample.
State the value to **two** (**2**) places after the decimal point.

Answer
One way to calculate the result is shown:

$$\text{Percentage Magnesium (\% w / w)} = \underbrace{\text{Volume EDTA (L)} \times \text{Molarity EDTA (mol / L)}}_{\text{Calculates mol of EDTA}} \times \underbrace{\frac{\text{1 mol Magnesium}}{\text{1 mol EDTA}}}_{\text{Converts to mol of Magnesium}} \times \underbrace{\frac{\text{24.312 g Magnesium}}{\text{1 mol Magnesium}}}_{\text{Converts to g of Magnesium}}$$

$$\times \underbrace{\frac{\text{250 mL Total Volume}}{\text{10 mL Sampled Volume}}}_{\text{Converts to Total g of Magnesium}} \times \underbrace{\frac{\text{100 \%}}{\text{mass of Sample (g)}}}_{\text{Converts to \% Magnesium}}$$

$$\text{Percentage Magnesium (\% w / w)} = \text{0.01225 L} \times \text{0.01000 mol / L} \times \frac{\text{1 mol Magnesium}}{\text{1 mol EDTA}} \times \frac{\text{24.312 g Magnesium}}{\text{1 mol Magnesium}}$$

$$\times \frac{\text{250 mL Total Volume}}{\text{10 mL Sampled Volume}} \times \frac{\text{100 \%}}{\text{0.7567 g}}$$

% w / w Magnesium = <u>9.84</u> % w / w

This Section Continues on the Next Page →

Sample Calculations (Cont.)

Water Hardness Analysis

Example 3
A **100 mL** (**0.100 L**) sample of tap water was titrated with **0.0100 M** EDTA solution.
The corrected titration volume was **14.80 mL** (**0.01480 L**).

Determine the total hardness in **mg / L** = **ppm** of calcium carbonate. **$CaCO_3$ = 100.1 g / mol**
State the value to the **nearest ppm**.

Answer
The "fictional" **ppm** calcium carbonate in the sample can be determined by finding **mg / L** of **$CaCO_3$**. The balanced equation of the reaction is taken to be:

$$CaCO_3 + EDTA^{4-}(aq) \rightarrow Ca(EDTA)^{2-}(aq) + CO_3^{2-}(aq)$$

One way to calculate the result is shown:

$$\text{Calcium Carbonate (mg / L)} = \underbrace{\text{Volume EDTA (L)} \times \text{Molarity EDTA (mol / L)}}_{\text{Calculates mol of EDTA}} \times \underbrace{\frac{1\text{ mol } CaCO_3}{1\text{ mol EDTA}}}_{\text{Converts to mol of } CaCO_3} \times \underbrace{\frac{100.1\text{ g } CaCO_3}{1\text{ mol } CaCO_3}}_{\text{Converts to g of } CaCO_3} \times \underbrace{\frac{1000\text{ mg}}{1\text{ g}}}_{\text{Converts to mg of } CaCO_3 \text{ in Sampled Volume}} \times \underbrace{\frac{1000\text{ mL Total Volume}}{100\text{ mL Sampled Volume}}}_{\text{Converts to Total mg / L of } CaCO_3}$$

$$\text{Calcium Carbonate (mg / L)} = 0.01480\text{ L} \times 0.01000\text{ mol / L} \times \frac{1\text{ mol } CaCO_3}{1\text{ mol EDTA}} \times \frac{100.1\text{ g } CaCO_3}{1\text{ mol } CaCO_3} \times \frac{1000\text{ mg}}{1\text{ g}} \times \frac{1000\text{ mL Total Volume}}{100\text{ mL Sampled Volume}}$$

Calcium Carbonate (mg / L = ppm) = <u>148</u> ppm

Note: For a **<u>100</u> mL** sample of water titrated with **<u>0.01000</u> M EDTA** solution, the total hardness value is **<u>10</u> times the titration volume in mL**.

Calcium Carbonate Hardness (100 mL Sample) = Titration Volume of 0.01000 M EDTA (mL) × 10

Name		Day		Start Time	

PRE-LABORATORY PREPARATION

To be completed before the laboratory session.
To be submitted before beginning the experiment (15 points).

You are invited to bring your own water sample to the laboratory for hardness analysis.

Questions: Answer in the space provided. **Show work**.
Your Mohawk College ID Number is **nnnnnnXYZ**.

Analysis of a Calcium Supplement Tablet
A supplement tablet containing (nominally) about **300 mg** of calcium ion was dissolved, filtered, and diluted to **100 mL** volume. Several **2.00 mL** samples of the total solution were titrated with **0.0100 M EDTA** solution by the method of this experiment.
The mean corrected titration volume was **14.YX mL** (**0.014YX L**).

Q-1. Calculate the calcium content of the supplement tablet in **mg** units. (6 points)
State the value to the nearest mg. Show work. See **Example 1** on page 3.

0.014YX L = _____ **mL**

Calcium Content (mg) = _____ mg

The PRE-LABORATORY PREPARATION Continues on the Next Page →

PRE-LABORATORY PREPARATION (Cont.)

Analysis of an Epsom Salt Sample
A sample of Epsom Salt of mass **0.74ZY g** was dissolved uniformly in distilled water in a **250 mL** volumetric flask. Portions of the magnesium ion solution of volume **10 mL** were titrated using a **0.01000 M** solution of EDTA by the method of this experiment.
The mean corrected titration volume was **12.X5 mL** (**0.012X5 L**).

Q-2. Calculate the percentage by mass (**% w / w**) of the magnesium in the Epsom Salt sample. (6 points) State the value to two (2) places after the decimal point. Show work.
See **Example 2** on page 4.

0.74ZY g = _____ **g** **0.012X5 L** = _____ **L**

Percentage by Mass of Magnesium (% w / w) = _____ % w / w

Water Hardness Analysis
A **100 mL** sample of water was titrated with **0.0100 M** EDTA solution.
The corrected titration volume was **1Z.Y0 mL** (**0.01ZY0 L**).

Q-3. Determine the total hardness in **ppm** calcium carbonate. (3 points)
State the answer to the nearest ppm. See **Example 3** on page 5.

1Z.Y0 mL = _____ **mL**

Calcium Carbonate Hardness (100 mL Sample) = **Titration Volume of 0.01000 M EDTA (mL) × 10**

Total Hardness (ppm) = _____ ppm

PRE-LABORATORY PREPARATION **Total =** **/ 15**

PROCEDURE

For this Experiment you will Work with a Partner.
Record the name of your partner in the **DATA TABLES AND REPORT** section.

Ensure that the fume hood fans are switched **ON** and are operating.

There are no special disposal instructions for this experiment. All solids and solutions may safely be disposed of by way of the municipal solid waste containers or the sinks. When using a sink for disposal of calcium and magnesium ion solutions or EDTA solution, run the cold municipal tap water at the same time.

A. Preparation of Glassware and Apparatus

The following **clean** glassware and laboratory apparatus is required for the experiment:

For class use:
- □ a **100 mL** grad. cylinder

For each pair of students:
- □ a spatula
- □ a glass stirring rod
- □ a short stem funnel
- □ a plastic buret funnel
- □ a long stem funnel

For each pair of students (cont.):
- □ two small beakers
- □ a rubber pipet squeeze bulb
- □ a **50 mL** buret and its stand
- □ a **250 mL** volumetric flask and its stopper
- □ a **100 mL** volumetric flask and its stopper (extra equipment)

For each pair of students (cont.):
- □ a **10 mL** transfer pipet
- □ a **2 mL** transfer pipet (extra equipment)
- □ a small watch glass to fit a small beaker
- □ a large watch glass to fit a large beaker
- □ all available erlenmeyer flasks
- □ a large (**250 mL** or **400 mL**) beaker

A-1. Clean the glassware and apparatus if necessary with a 1 % solution of detergent in warm water. See **Cleaning and Drying of Glassware** on page **Error! Bookmark not defined.**. Rinse the cleaned glassware and apparatus with tap water and then with distilled water. To avoid breakage, do not leave any glassware standing in an unstable position.

A-2. The instructor will set up one or more hot-plates. Using large beakers, heat **25 mL** of distilled water per student for the filtration step in **Part C**. The filtration of Part C will be slow. Move on to Part C as soon as possible.

You and your partner may divide the work in any way you wish. You may work together, or you may split up and work separately on different parts. You may prepare two burets for titrating simultaneously.

The Experiment Parts may be done in any order. However, the filtration of Part C will be slow and should be done as soon as possible.

B. Buret Preparation

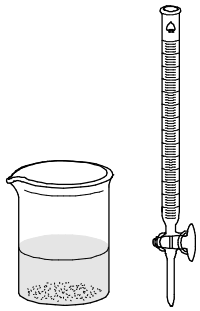
The buret filled with EDTA solution will be used in all other parts of the experiment.

B-1. Take a buret stand and a **50 mL** buret to your bench station. You should have a clean **10 mL** volumetric pipet, a **2 mL** volumetric transfer pipet (extra equipment), two small beakers and at least three erlenmeyer flasks. Dry the outside of the buret, the pipets, the beakers and the flasks.

B-2. Assemble the buret **securely**, and check that the buret tap is working. Drain the buret and pipets upside down in the buret stand. Check that the inner walls of the buret and the transfer pipets are clean and that the capillary tips are not broken or plugged. **It is not possible to do a good analysis with dirty glassware**.

B-3. **Label** a clean small beaker to be used for the supplied EDTA solution. Into this beaker, pour about **20 mL** of the EDTA solution, using the beaker volume markings. The EDTA solution will be assumed to be **<u>0.01000</u> M** unless you are informed otherwise.

B-4. Rinse the inside walls of the beaker with the EDTA solution. Pour the solution into the buret, rinsing the inner walls of the buret with the solution. Drain some of the solution out through the tip of the buret into a waste beaker or flask.



Rinse the small plastic buret funnel also, if it is to be used.

B-5. Repeat the entire rinse process and collect the rinse solution again. The third time, take a larger volume in the beaker and fill the buret to near the **0.00 mL** mark, **clearing the tip of air bubbles**.

Discard all of the rinse portions into the sink with the cold water running.

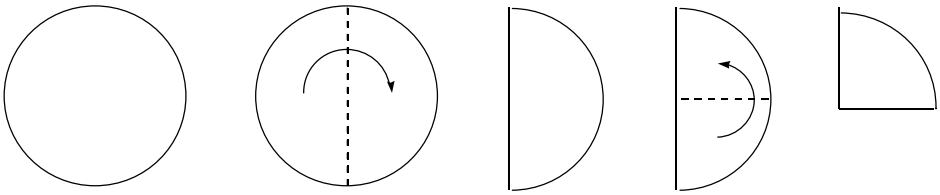
Gravity Filtration with Filter Paper

The instructor will demonstrate the folding, **tearing** and fitting of the filter paper to the funnel.

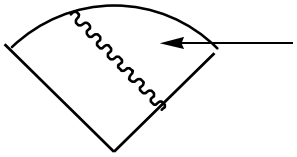
Use any **fast** 11 cm filter paper, Fisher P8 Creped, Whatman #4 or a similar type.

Fold and tear the filter paper circle as shown (see below). Keep the paper clean. Fit your paper into your funnel carefully. The stem of the funnel should reach near the bottom of a receiving vessel if possible.

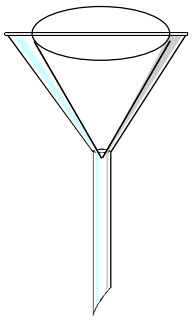
Advanced Folding of a Filter Paper Circle



Fold the filter paper circle twice into a quarter shape as shown above.
Do not crease the paper sharply. This will weaken the fibres and cause tearing to occur.



Tear off one corner of one of the pockets.
Now when you open the other pocket, and place the filter paper cone into your funnel, it will fit better.



Use distilled water from your wash bottle to make the filter paper cone stick to the glass.
This paper will fit better and filter more rapidly.

Quality Control Analysis of Calcium in a Supplement Tablet

The instructions are based on a target of **300 mg** of calcium in the form of calcium citrate per tablet. If the samples differ from this calcium content in form or amount, amend the procedure accordingly.

The amount **300 mg** of calcium is too large for convenient titration with a **0.1000 M EDTA** solution. A more convenient amount is **6.0 mg**. A calcium tablet will be placed in water and the resulting suspension will be filtered through paper. The filtrate will be diluted to **100 mL** volume, and then repeat **2 mL** samples will be titrated. Each **2 mL** sample will contain about **6.0 mg** of calcium.

C. Calcium Sample Preparation

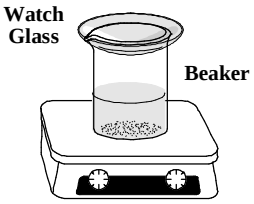
Webber Naturals brand tablets containing **300 mg** of calcium as calcium citrate have been used successfully for this analysis. These tablets disintegrate rapidly into water. Tablets containing calcium in the form of calcium carbonate produce large amounts of foam when dissolved using hydrochloric acid, which is inconvenient.

The filtration of the sample will be slow. Begin this part as soon as possible.

C-1. Record the name and type of sample in **Table D** in the **DATA TABLES AND REPORT** section. Record a description of the tablet. What is the chemical form of the calcium?

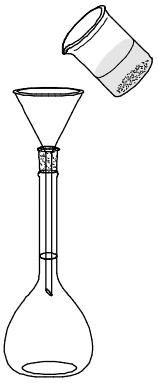
C-2. Label a clean large beaker (**250 mL** or **400 mL**) in such a way that you will be able to identify it later in a crowd of other beakers.

C-3. **Do this in the fume hood**. Place one tablet into the labeled clean beaker. Add about **10 mL** of dilute **6 M HCl** solution. Swirl the beaker to disintegrate the tablet material. Cover the beaker with a large watch glass. Heat the contents gently on a warm (**not hot**) hot plate if necessary for a few minutes. The tablet will not dissolve totally.



Caution: If calcium carbonate is present there will be foaming. The calcium in some supplement tablets is in the form of calcium carbonate ($CaCO_3$) which is insoluble in water, but dissolves in hydrochloric acid. The reaction will produce a large amount of a foam of carbon dioxide gas bubbles.

C-4. When any foaming has ceased, remove the beaker from the hot plate and take it out of the fume hood. **Caution: hot**.



C-5. **Use a fast filter paper** and a clean long stem funnel. Follow the instructions on page 11 to fold the filter paper. Decant as much fluid as possible from the solid in the beaker. Filter and transfer the solution **quantitatively** from the beaker, into a clean **100 mL** volumetric flask (extra equipment).

Note: there may be some soluble starch in the tablet, so the filtrate may appear cloudy. This will not cause a problem when titrating the solution.

C-6. Rinse the beaker, the solid residue, the filter paper, and the funnel with **three 5 mL** portions of **hot** distilled water, adding the wash water to the volumetric flask. Keep the volume of the washes to a minimum.

C-7. Add distilled water to the flask to about one cm below the mark line. Fill the flask to the mark line using a dropper pipet.

C-8. Stopper the flask with a clean stopper. Hold the stopper in place with one hand. Turn the flask over **slowly** at least **17 times** to ensure that the solution is mixed and is completely uniform.

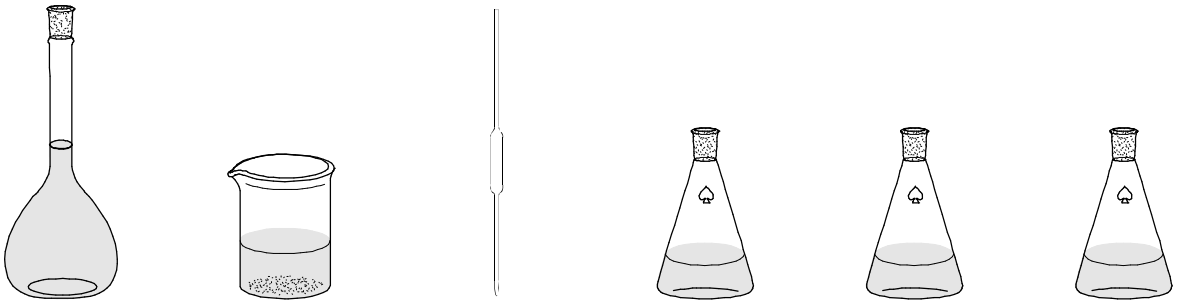
D. Quality Control Analysis of Calcium in a Supplement Tablet

D-1. Label a clean small beaker to be used with your calcium ion solution from the **100 mL** volumetric flask. Rinse this beaker with about a **20 mL** volume of calcium ion solution from your volumetric flask. Use this portion of the solution to rinse out the **2 mL** transfer pipet as well. Collect and discard the rinsing solutions into the sink.

D-2. Repeat the rinsing and discard the solution again. On the third refill, take about **15 mL** to **20 mL** of the calcium ion solution into the beaker.

D-3. The erlenmeyer flasks for the titrations must be clean but the insides need not be dry. **Check that your squeeze bulb is clean and dry inside**.

D-4. Transfer by pipet one **2.00 mL** portion, of the calcium ion solution from its beaker into each of three clean erlenmeyer flasks. Wipe the tip of the pipet dry before each delivery. If you are unsatisfied with your pipetting technique in any transfer, discard the sample in that erlenmeyer flask, rinse the flask well with distilled water, and do it again.



Solution of Calcium Ion (100 mL) | **Intermediate Vessel** | **2 mL Transfer Pipet** | **Three Clean (But not Dry) Erlenmeyer Flasks**

D-5. **Do this in the fume hood**. Add to each erlenmeyer flask:
   a. Distilled water approximately to the **20 mL** mark.
   b. About **10 mL** of **pH 10** buffer solution.
   c. Three or four (**3 or 4**) drops of **eriochrome black T** indicator solution.

D-6. **Mix well**. The indicator colour should be **cherry red** at this point. **Eriochrome black T** is **cherry red** when complexed with calcium at pH 10. It is **sky blue** when it has been displaced from the calcium by EDTA at the end-point of the titration. At the end-point, the colour change will be to a clear, **bright sky blue** colour (not **purple**).

Note: If the colour intensity of the solution is too pale, more indicator solution may be added.

This Section of the PROCEDURE Continues on the Next Page →

D. Quality Control Analysis of Calcium in a Supplement Tablet (Cont.)

D-7. Titrate in turn each **2.00 mL** portion of the **100 mL** Calcium ion solution with EDTA solution from the buret. Record the **initial volume** and the **final volume** to two places after the decimal point to the nearest **0.05 mL**, **in ink**, in **Table D** (**Trial 1**) of the **DATA TABLES AND REPORT** section.

D-8. The end-point colour change is from **cherry red** to **clear, sky blue**. This reaction occurs much more slowly than most titration reactions. Go slowly in both volumes and time at the end-point. The solution will become **purple – grey** as you near the end-point; it may seem almost colourless. Continue until you see a **clear, sky blue** colour. Consult the instructor if you have difficulty with the colours.

D-9. Repeat titrations are expected to have the same titration volume to the end-point. In the repeat trials you can add all but the final **few mL** rapidly, using the first titration volume as a guide. Record all volumes in **Table D**. Refill your buret with EDTA solution as necessary.

D-10. Continue doing trials until you have **three** acceptable trial titration volumes within a range of no more than **0.20 mL**. Consult the instructor if you have difficulty with this standard.

D-11. **The indicator correction for an EDTA titration is usually zero**. Determine an end-point indicator volume correction (**indicator blank**) as follows. Add **25 mL** of distilled water to a clean erlenmeyer flask. Add **4 drops** of indicator solution and **10 mL** of the buffer solution without any calcium ion solution being present. If the solution is **clear sky blue** (end-point colour), the indicator blank is zero, **0.00 mL**.

If the solution is **purple – grey** or **cherry red**, possibly due to impurity metal ions, determine the volume of EDTA solution required to produce the same **clear sky blue** colour you used for your end-point recognition during the procedure. This should be a very small volume; e.g. **0.05 mL** or **0.10 mL**. Record the indicator blank volume in **Table D**.

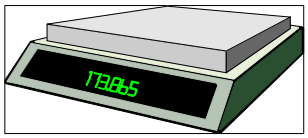
Note: If the indicator correction has been determined for another part of the experiment, use the same value here.

D-12. **When you have completed titrating**, show the instructor your titration values and have your data table initialled by the instructor.

E. Epsom Salt Sample Preparation

E-1. Record the brand name of the Epsom Salt in **Table E** in the **DATA TABLES AND REPORT** section. Record a description of the material. What chemical compound of magnesium is assumed to be present?

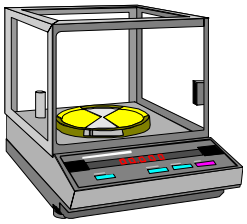
E-2. Place **<u>0.70</u> g – <u>0.80</u> g** of solid Epsom Salt into a clean weighing boat using a **top-loading balance**.



Top-Loading Balance

Do not fill or empty a container over a balance.

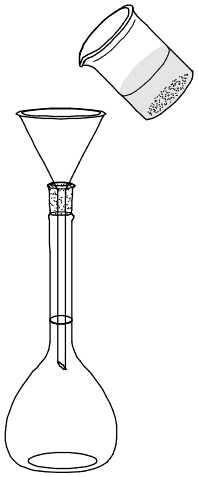
E-3. Weigh the boat plus solid on an **analytical balance**. Record the mass observation **in ink** to four places after the decimal point in **Table E**.



Analytical Balance

E-4. From your weighing boat, transfer the Epsom Salt sample into a small clean beaker. Re-weigh the boat plus any remaining solid on an **analytical balance**. Record the mass observation **in ink** to four places after the decimal point in **Table E**.

E-5. Add about **20 mL** of distilled water to the beaker. Using either a short- or long-stem funnel, transfer the solution **quantitatively** from the beaker, into a clean **250 mL** volumetric flask.



E-6. Continue to add small portions of distilled water to the beaker until the entire sample of Epsom Salt is transferred into the volumetric flask.

E-7. Add distilled water to the flask to about one cm below the mark line. Fill the **250 mL** volumetric flask to the mark line using a dropper pipet.

E-8. Stopper the volumetric flask with its clean stopper. Hold the stopper in place with one hand. Turn the flask over **slowly** at least **17 times** to ensure that the solution is mixed and completely uniform.

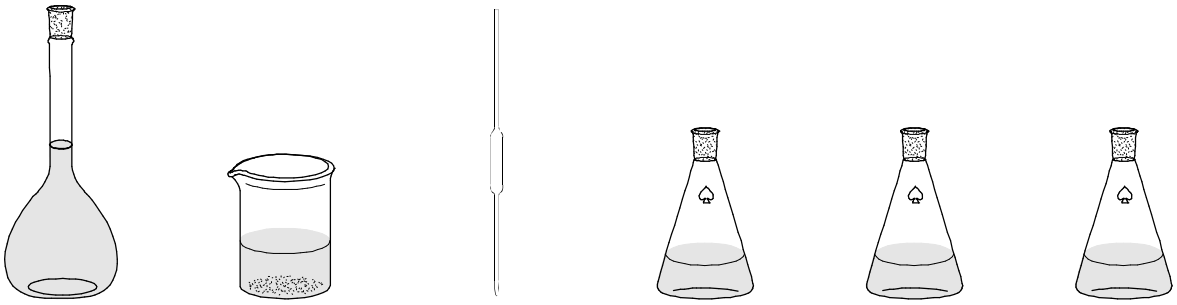
F. Titration of Epsom Salt Samples

F-1. Label a clean small beaker to be used with your Epsom Salt solution from the **250 mL** volumetric flask. Rinse this beaker with about a **20 mL** volume of Epsom Salt solution from your volumetric flask. Use this portion of the solution to rinse out the **10 mL** transfer pipet as well. Collect and discard the rinsing solutions into the sink.

F-2. Repeat the rinsing and discard the solution again. On the third refill, take about **40 mL** to **50 mL** of the Epsom Salt solution into the beaker.

F-3. The erlenmeyer flasks for the titrations must be clean but the insides need not be dry. **Check that your squeeze bulb is clean and dry inside**.

F-4. Transfer by pipet one **10.00 mL** portion of the Epsom Salt solution from its beaker into each of three clean erlenmeyer flasks. Wipe the tip of the pipet dry before each delivery. If you are unsatisfied with your pipetting technique in any transfer, discard the sample in that erlenmeyer flask, rinse the flask well with distilled water, and do it again.



Solution of Epsom Salt (250 mL) | **Intermediate Vessel** | **10 mL Transfer Pipet** | **Three Clean (But not Dry) Erlenmeyer Flasks**

F-5. **Do this in the fume hood**. Add to each erlenmeyer flask:
   a. Distilled water approximately to the **20 mL** mark.
   b. About **10 mL** of **pH 10** buffer solution.
   c. Three or four (**3 or 4**) drops of **eriochrome black T** indicator solution.

F-6. **Mix well**. The indicator colour should be **cherry red** at this point. **Eriochrome black T** is **cherry red** when complexed with magnesium at pH 10. It is **sky blue** when it has been displaced from the magnesium by EDTA at the end-point of the titration. At the end-point, the colour change will be to a clear, **bright sky blue** colour (not **purple**).

Note: If the colour intensity of the solution is too pale, more indicator solution may be added.

This Section of the PROCEDURE Continues on the Next Page →

F. Titration of Epsom Salt Samples (Cont.)

F-7. Titrate in turn each **10.00 mL** portion of the **250 mL** Epsom Salt solution with EDTA solution from the buret. Record the **initial volume** and the **final volume** to two places after the decimal point, **in ink**, in **Table F** (**Trial 1**) of the **DATA TABLES AND REPORT** section.

F-8. The end-point colour change is from **cherry red** to **clear, sky blue**. This reaction occurs much more slowly than most titration reactions. Go slowly in both volumes and time at the end-point. The solution will become **purple – grey** as you near the end-point; it may seem almost colourless. Continue until you see a **clear, sky blue** colour. Consult the instructor if you have difficulty with the colours.

F-9. Repeat titrations are expected to have the same titration volume to the end-point. In the repeat trials you can add all but the final **few mL** rapidly, using the first titration volume as a guide. Record all volumes to the nearest **0.05 mL** in **Table F**.

F-10. Refill your buret with EDTA solution as necessary. Continue doing trials until you have **three** acceptable trial titration volumes within a range of no more than **<u>0.20</u> mL**.
Consult the instructor if you have difficulty with this standard.

F-11. **The indicator correction for an EDTA titration is usually zero**. Determine an end-point indicator volume correction (**indicator blank**) as follows. Add **25 mL** of distilled water to a clean erlenmeyer flask. Add **4 drops** of indicator solution and **10 mL** of the buffer solution without any magnesium ion solution being present. If the solution is **clear sky blue** (end-point colour), the indicator blank is zero, **<u>0.00</u> mL**.

If the solution is **purple – grey** or **cherry red**, possibly due to impurity metal ions, determine the volume of EDTA solution required to produce the same **clear sky blue** colour you used for your end-point recognition during the procedure. This should be a very small volume; e.g. **<u>0.05</u> mL** or **<u>0.10</u> mL**. Record the indicator blank volume in **Table F**.

Note: If the indicator correction has been determined for another part of the experiment, use the same value here.

F-12. **When you have completed titrating**, show the instructor your titration values and have your data table initialled by the instructor.

Water Hardness Ions Analysis of Tap Water

It has been necessary to run the cold tap water in the chemistry laboratories for a long period of time, usually about 45 to 60 minutes, in order to get consistent results for this analysis. Possibly the water standing in the pipes is contaminated with metal ions, perhaps zinc or copper from the pipes. Normal results for Hamilton tap water are in the range of **130 ppm** to **150 ppm** hardness.

G. Water Hardness Ions Analysis of Tap Water

You are invited to bring your own water sample to the laboratory for hardness analysis.

G-1. Record the source of the water sample in **Table G** of the **DATA TABLES AND REPORT** section.

G-2. Add a **100 mL** sample of your water or tap water to a clean erlenmeyer flask, using a **100 mL** graduated cylinder.

G-3. **In the fume hood**. Add about **10 mL** of **pH 10** buffer solution to the erlenmeyer flask.

G-4. Add four (**4**) drops of eriochrome black T indicator solution to the erlenmeyer flask. Mix well. The solution in the flask should be **cherry red** in colour. At the end-point, the colour change will be to a clear, **bright sky blue** colour (not **purple**).

G-5. Refill your buret with EDTA solution as necessary.
Record the **start volume** reading to 2 places after the decimal point to the nearest **0.05 mL** in **Table G**.

G-6. Titrate as in **Part D**, to the cherry red-to-sky blue end-point colour change.
Record the **final volume** reading to 2 places after the decimal point to the nearest **0.05 mL** in **Table G**.

G-7. Repeat until you have satisfactory titration results for **two** (**2**) titrations differing by no more than **0.20 mL**. Have your data table initialled by the instructor.

The REPORT Instructions are on the Next Page →

REPORT

Table D

R-1. Determine and enter the corrected titration volume for each trial.

R-2. Circle the three trials agreeing to within **0.20 mL**.
Calculate and enter the mean corrected titration volume of the three acceptable trials.

R-3. Calculate the experimental calcium content of the supplement tablet in **mg** units.
State the value to the **nearest mg**. Show work. See **Example 1** on page 3.

Table E

R-4. Record the Brand, description and the mass data for the Epsom Salt sample

Table F

R-5. Determine and enter the corrected titration volume for each trial.

R-6. Circle the three trials agreeing to within **0.20 mL**.
Calculate and enter the mean corrected titration volume of the three acceptable trials.

R-7. Calculate the experimental percentage by mass (**% w / w**) of the magnesium in the Epsom Salt sample. State the value to **two** (**2**) places after the decimal point. Show work. See **Example 2** on page 4.

Table G

R-8. Determine and enter the corrected titration volume for each trial.

R-9. Circle the two trials differing by no more than **0.20 mL**.
Calculate and enter the mean corrected titration volume of the two acceptable trials.

R-10. Determine the experimental total hardness in **ppm** calcium carbonate.
State the answer to the **nearest ppm**. See **Example 3** on page 5.

Bonus Questions

See pages 24 and 26 for the **Bonus Questions**.

Name		Day		Start Time	

DATA TABLES AND REPORT

The following **Tables** are to be used for recording observations and measurements. Measured values are to be recorded in the heavily shaded cells **IN INK**. Leave this page open on your bench during the experiment period. Have it initialed by the instructor on completing each section.

The completed and initialed **DATA TABLES AND REPORT** section must be handed in along with any additional pages you may submit as your report.

Name(s) of Partner(s): _____ + _____

Table D: Quality Control on a Calcium Supplement Tablet

Description of the Calcium Supplement Tablet

Brand Name and Description	Chemical Form of the Calcium

EDTA Solution Molarity = 0.01000 M

End-Point Indicator Volume Correction (Indicator Blank)

Final _____ mL - Start _____ mL = _____ mL

Titration Volumes for Calcium Ion Samples (In Ink to 2 places after the decimal point)

	At Least Three Trials are Mandatory Additional Trials Only if Necessary					
	Trial 1-Ca	Trial 2-Ca	Trial 3-Ca	Trial 4-Ca	Trial 5-Ca	Trial 6-Ca
Final Volume (mL)						
Start Volume (mL)						
Titration Volume (mL)						
Indicator Blank Volume (mL)						
Blank Corrected Titration Volume (mL)						

Instructor's Initials on Completion: _____ (5 points)

The DATA TABLES AND REPORT Section Continues on the Next Page →

DATA TABLES AND REPORT (Cont.)

Table D: Quality Control on a Calcium Supplement Tablet (Cont.)

Mean Corrected Titration Volume of **Three** Acceptable Trials (Within a Range of **0.20 mL**)
Circle the Acceptable Trials. State to 2 Places After the Decimal Point.

Mean Corrected Titration Volume = _____ mL Range = _____ mL

Calculate the experimental calcium content of the supplement tablet in **mg** units.
(9 points) State the value to the nearest mg. Show work. See **Example 1** on page 3.

Experimental Calcium Content of the Tablet (mg) = _____ mg

Table E: Epsom Salt Sample Preparation

Epsom Salt Sample Target Mass: 0.70 - 0.80 g

Weighings of Epsom Salt Sample (In Ink to 4 Places after the Decimal Point)

Container + Solid (g)	Container + Residue (g)	Mass of Epsom Salt Sample (g)

Description of the Epsom Salt Unknown

Brand Name and Description	Nominal Chemical Formula

The DATA TABLES AND REPORT Section Continues on the Next Page →

Name		Day		Start Time	

DATA TABLES AND REPORT (Cont.)

Table F: Titration of Epsom Salt Samples

EDTA Solution Molarity = 0.01000 M

End-Point Indicator Volume Correction (Indicator Blank)

Final _____ mL - Start _____ mL = _____ mL

Titration Volumes for Epsom Salt Samples (In Ink to 2 places after the decimal point)

	At Least Three Trials are Mandatory Additional Trials Only if Necessary					
	Trial 1-Mg	**Trial 2-Mg**	**Trial 3-Mg**	**Trial 4-Mg**	**Trial 5-Mg**	**Trial 6-Mg**
Final Volume (mL)						
Start Volume (mL)						
Titration Volume (mL)						
Indicator Blank Volume (mL)						
Blank Corrected Titration Volume (mL)						

Instructor's Initials on Completion: _____ (5 points)

Mean Corrected Titration Volume of **Three** Acceptable Trials (Within a Range of **0.20 mL**) **Circle the Acceptable Trials**. State to 2 Places After the Decimal Point.

Mean Corrected Titration Volume = _____ mL Range = _____ mL

The DATA TABLES AND REPORT Section Continues on the Next Page →

DATA TABLES AND REPORT (Cont.)

Table F: Titration of Epsom Salt Samples (Cont.)

Calculate the experimental percentage by mass (**% w / w**) of the magnesium in the Epsom Salt sample. (8 points) State the value to **two** (**2**) places after the decimal point. Show work.
See **Example 2** on page 4.

Experimental Percentage by Mass of Magnesium (% w / w) = _____ % w / w

Bonus Question 1 (5 points) Answer below or attach another page.

a. Calculate the **theoretical** percentage by mass (% w / w) of magnesium in Epsom Salt. State the value to one place after the decimal point. Show work.

b. Assume that the Epsom Salt sample was 100 % pure. Calculate the percentage error in your experimental result for percentage of magnesium in the Epsom Salt. Show work.

c. Assume instead that your result was completely accurate. What could account for obtaining a result that is lower than the theoretical value? What could account for obtaining a result that is higher than the theoretical value? Explain fully.

The DATA TABLES AND REPORT Section Continues on the Next Page →

Name		Day		Start Time	

DATA TABLES AND REPORT (Cont.)

Table G: Total Hardness of Tap Water

Description of the Sample: _____

EDTA Solution Molarity = 0.01000 M **Sample Volume = 100 mL per Titration**

Total Hardness Titration Volumes (In Ink to 2 places after the decimal point)

	At Least Two Trials are Mandatory Additional Trials Only if Necessary					
	Trial 1-H	**Trial 2-H**	**Trial 3-H**	**Trial 4-H**	**Trial 5-H**	**Trial 6-H**
Final Volume (mL)						
Start Volume (mL)						
Titration Volume (mL)						
Indicator Blank Volume (mL) (Same as in Table D or F)						
Blank Corrected Titration Volume (mL)						

Instructor's Initials on Completion: _____ (5 points)

Mean Corrected Titration Volume of **Two** Acceptable Trials (Within a Range of **0.20 mL**)
Circle the Acceptable Trials. State to 2 Places After the Decimal Point.

Mean Corrected Titration Volume = _____ mL Range = _____ mL

Determine the experimental total hardness in **ppm** calcium carbonate. (3 points)
Use the Mean Corrected Titration Volume. State the answer to the **nearest ppm**.
See **Example 3** on page 5.

Calcium Carbonate Hardness (100 mL Sample) = Titration Volume of 0.01000 M EDTA (mL) × 10

Experimental Calcium Carbonate Hardness (ppm) = _____ ppm

The DATA TABLES AND REPORT Section Continues on the Next Page →

DATA TABLES AND REPORT (Cont.)

Bonus Question 2 (5 points) Answer below or attach another page.

The simplified calculation for hardness of a water sample given on the previous page is correct when the sample size is **100** mL and the EDTA is **0.01000** M. Explain or demonstrate **numerically** why this is true.

Mark Sheet for Experiment 6 **Total = 100 points**

Category	**Points**
Pre-Laboratory Preparation	**/ 15**
Attendance: Punctuality, Diligence, Clean-Up.	**/ 25**
Data Acquisition and Recording In Ink /5 Proper Number of Digits /5 Results Initialed Mg Results /5 Ca Results /5 Water Results /5	**/ 25**
Calculations	**/ 20**
Analytical Result for Percentage Magnesium in Epsom Salt Result Correct Within: ± 0.50 % = 20 ± 0.95 % = 16 ± 1.80 % = 12 ± 3.43 % = 8 ± 6.51 % = 4	**/ 20**
Analytical Result for Hardness of Tap Water Result Correct Within: ± 5 ppm = 20 ± 9.5 ppm = 16 ± 18 ppm = 12 ± 34 ppm = 8 ± 65 ppm = 4	**/ 20**
Bonus Section: Question 1 / 5 Question 2 / 5	**/ 10**
Comments: **Total =**	**/125**